micromeritics®

AutoChem 2920

Automated Catalyst Characterization System

Operator's Manual

V4.00

292-42803-01
July 2009

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Form No. 008-42104-00
Rev. 12/95

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1. GENERAL DESCRIPTION

Organization of the Manual

This manual describes how to install, operate, and maintain the AutoChem II 2920 Analyzer. The manual is divided into the following chapters.

Chapter 1	**GENERAL DESCRIPTION**. Provides a general description and specifications of the analyzer.
Chapter 2	**INSTALLATION**. Provides unpacking and inspection information, and installation instructions.
Chapter 3	**USER INTERFACE**. Describes basic skills for using the software.
Chapter 4	**GENERAL OPERATING PROCEDURES**. Describes operating procedures for the AutoChem II analysis program.
Chapter 5	**FILE MENU**. Provides descriptions for each item on the File menu.
Chapter 6	**UNIT MENU**. Provides descriptions for each item on the Unit menu. (A Unit menu is created for each analyzer connected to your system.)
Chapter 7	**REPORTS MENU**. Provides descriptions for each item on the Reports menu, as well as instructions for using the Peak Editor. Also includes examples of some of the reports available for the analysis program.
Chapter 8	**OPTIONS MENU**. Provides descriptions for each item on the Options menu.
Chapter 9	**TROUBLESHOOTING AND MAINTENANCE**. Describes troubleshooting and maintenance procedures.
Chapter 10	**. ORDERING INFORMATION**. Provides information on ordering parts and accessories for the analyzer.

Several appendices and an index are also included.

Conventions

This manual uses the symbols shown below to identify notes of importance, cautions, and warnings.

Notes contain a tip or important information pertinent to the subject matter.

Cautions contain information to help you prevent actions which could damage the instrument.

Warnings contain information to help you prevent actions which could cause personal injury.

Equipment Description

The AutoChem II 2920 is a highly automated catalyst characterization system. It can perform the following experiments individually or in various sequences:

- Temperature-Programmed Desorption (TPD)
- Temperature-Programmed Reduction (TPR)
- Temperature-Programmed Oxidation (TPO)
- Temperature-Programmed Reaction
- Pulse Chemisorption
- BET Surface Area

The AutoChem II software represents a major advancement in the convenience and control of temperature-programmed analyses because of the high level of automation offered. Very few steps must be physically performed by an operator; the vast majority are controlled with precision by the software.

This automated process is determined by the sample file you create. In addition to information about the sample itself, the sample file contains a list of specific instructions, which the analyzer follows to perform any series of experiments you desire. The instrument monitors the progress of each step, then initiates the next one while continuously controlling temperatures and gas flows. Delays are eliminated, because most experiment steps do not require an operator to initiate them. The potential for human error is reduced, and less labor is required. You can create standard sets of analysis conditions, making it possible for a laboratory manager to standardize procedures for laboratory technicians to use during repeat sample analyses.

Convenient Windows® Interface

The Windows interface makes it easier than ever to create sample files, control analyses, and generate the reports you need. You can:

- Monitor detector signal, time, and temperature data.
- Create and store standard sets of analysis conditions for frequently performed analyses.
- Integrate detector signal peaks both automatically and manually.
- Display and print peak graphs and reports.
- Establish calibration curves for calculation of unknown sample concentrations.
- Reprocess stored analysis data using different parameters.
- Export data in ASCII text format for use in other applications.

Flexible Peak Editing

The AutoChem's powerful Peak Editor enables you to evaluate results quickly and easily, edit peaks, and produce reports that reflect your specific needs. Adjusting peak boundaries is a matter of simply pointing and clicking them to eliminate baseline noise or other undesirable effects. The Peak Editor also enables you to separate composite peaks.

Major Components and Features

Figure 1-1. AutoChem 2920 Analyzer

The AutoChem II 2920 analyzer is designed to offer maximum control, flexibility, and automation. Some features include:

On-screen graphical status display	Enables you to observe the status of automatically controlled operations. In manual mode, the graphical status display allows you to control valves, heat zones, and other components manually.
Point-and-click intuitive user interface	Allows you to create sample files using pre-programmed analysis conditions.
Temperature control	Four separate heat zones let you heat internal gas lines, the thermal conductivity detector, and valves individually, from ambient to 150 ºC.
Furnace control	Lets you set furnace temperatures, their duration, and ramp rates. Sample target temperature can range from ambient to 1100 ºC (or -70 to 1100 °C with the optional CryoCooler).
CryoCooler (optional)	Enables you to start analyses at subambient temperatures. The CryoCooler can also be used to speed throughput by ramping the furnace temperature rapidly down to ambient after an analysis.
KwikCool	Enables you to ramp the furnace temperature rapidly down to near ambient, reducing analysis time and increasing throughput.
Clamshell-type furnace	Heats samples efficiently and offers easy access to analysis ports.
Mass Flow Controllers (MFCs)	Provide precise control of gas flows, using the rates you specify in the sample's analysis file.
Vapor Generator (optional)	Allows analysis using vapors from liquids carried by an inert gas.
Exhaust gas analysis port	Allows sampling of analysis gas after it has passed the thermal conductivity detector for analysis by a mass spectrometer, infrared spectroscope, thermal analyzer, or gas chromatograph.

Diversion ports (with cold trap attached)	Allow trapping of condensibles in the gas flowing from the sample before they reach the thermal conductivity detector. A delay loop, dryer, or mass spectrometer may also be attached.
Thermal Conductivity Detector (TCD)	Highly accurate, gold-plated filaments detect minute changes in the thermal conductivity of gases. Highly linear sensitivity enhances the accuracy of results.
Exhaust ports	Allow attachment of appropriate exhaust systems to meet local safety standards for chemisorption exhaust.
Internal Blending	Allows limited blending of gas mixtures in the analyzer. Useful for multipoint BET surface area analysis, for example, or to mix gases instead of purchasing premixed gas bottles. This feature is enabled using an option on the Gas Flows dialog.

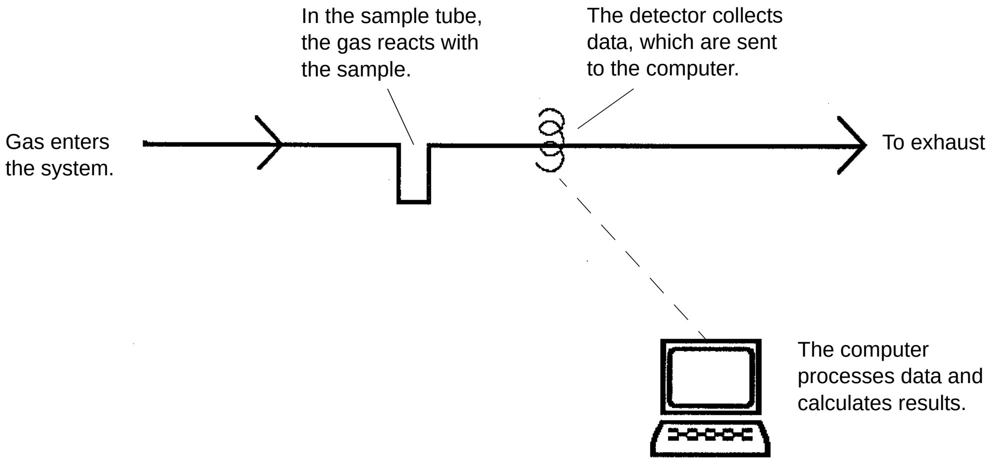
Temperature-Programmed Analyses

This section provides a basic, simplified description of the technique for performing temperature-programmed analyses.

The AutoChem II is a sophisticated instrument that enables you to perform a variety of complex experiments. Most temperature-programmed experiments, however, are based on the following *highly simplified* steps:

1. Gas flows into the analyzer.
2. The gas interacts with the sample as the temperature changes.
3. Gas flows past the detector.
4. The detector collects data.

5. The software plots and calculates results.



How does the detector work?

The detector contains heated filaments; it measures the difference in gas thermal conductivity sensed between the gases flowing over the sample and reference filaments.

The gases flowing past the detector cool the filament by extracting heat. How quickly any type of gas removes heat from the detector is determined by its *thermal conductivity*[1]. A gas with a high thermal conductivity cools the filament rapidly, and more power is required to maintain its temperature. A gas with a lower thermal conductivity removes heat from the filament more slowly.

When the sample reacts with the gas, it causes changes in the composition of the gas and, consequently, changes the thermal conductivity of the gas. These changes are sensed by the detector as an increase or decrease in the amount of power required to maintain the filament at a constant temperature.

What data are collected?

The detector reports the amount of electricity (in volts) required to keep its temperature constant during the analysis.

What role does sample temperature play?

Because the sample's temperature determines how rapidly it interacts with the analysis gas (or if it reacts at all), data are collected over the range of temperatures you specify.

In some experiments, you may wish to start collecting data at a very low temperature to establish a baseline where the gas is completely unaffected by the sample. In other cases, you may wish to collect data after a reaction has begun. In still other experiments, your primary interest may be determining the temperature at which the maximum reaction occurs.

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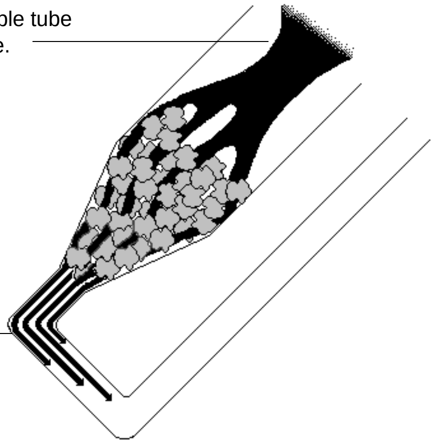
[1] The thermal conductivity of a gas is its ability to conduct heat. Each gas has a distinct thermal conductivity.

<u>Here's one example:</u>

Consider the example of a Temperature-programmed reduction (TPR). During the TPR, a metal oxide is reacted with hydrogen to form a pure metal. This reaction is referred to as "reducing" the metal; for example, TPR of a catalyst containing Platinum. Argon, which has a very low thermal conductivity, is used as a carrier gas. It is blended in a fixed proportion with hydrogen, an analysis gas with a much higher thermal conductivity. Then the gas mixture flows through the analyzer, through the sample, and past the detector.

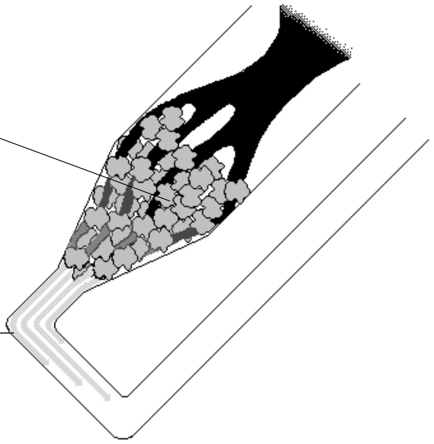
When the gas blend begins flowing over the sample, a baseline reading is established by the detector. This baseline is established at a low enough temperature that no reduction of the sample is occurring, so the baseline level recorded by the detector is that of the thermal conductivity of the two gases in their fixed proportion. In other words, the proportion of gases flowing over the detector is the same as the proportion of gases entering the analyzer, because at the low temperature, there is no interaction with the sample.



Gas flows into the sample tube and through the sample.

Before the reaction begins, the concentration of gases exiting the sample tube is unchanged after flowing through the sample.

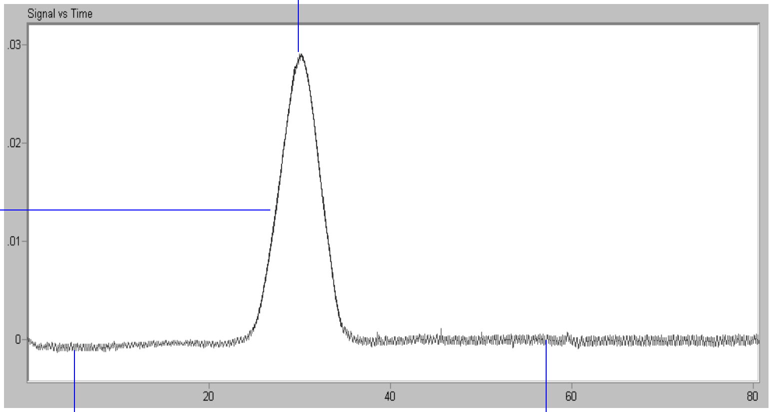
The temperature is then changed, and when a critical temperature is reached, hydrogen atoms in the gas flow react with the sample, forming H_2O molecules. The H_2O molecules are removed from the gas stream using a cold trap. As a result, the amount of hydrogen in the argon/hydrogen gas blend inside the analyzer *decreases*, and the proportion between the two gases shifts in the direction of argon, as does the mixture's thermal conductivity.



As the temperature changes, the sample begins to react with the gas(es) flowing through it.

The concentration of gases exiting the sample tube is altered after passing through the sample. This change in the gas concentration is recorded by the Thermal conductivity Detector downstream.

Since argon has a lower thermal conductivity than hydrogen, the mixture's thermal conductivity consequently decreases. The flowing gas removes heat from the filament more slowly, requiring less electricity to maintain a constant filament temperature. The instrument records the electrical demand as it changes (this is called the detector signal). The detector signal is recorded continuously over a range of temperatures. When these readings are graphed, the data form one or more peaks. Peaks can be positive or negative; negative peaks are show in this example.



2. As the temperature changes, the sample begins to react with one of the gases. Therefore the gas mix is then made up of a larger proportion of the other gas. This causes a shift in the mixture's thermal conductivity. The detector measures this change by recording the change in the amount of electricity required to maintain constant filament temperature.

3. As temperature continues to increase, the interaction reaches a maximum, then begins to diminish.

1. Baseline readings. The gas(es) is (are) not reacting with the sample, so there is no change in the signal from reading to reading.

4. As fewer and fewer sample atoms are available to bond with the analysis gas, there is less and less change in the mix of gases flowing into the analyzer and past the detector, so the thermal conductivity shifts back toward the baseline value.

This example illustrates the fundamental concept upon which the analyzer operates. Of course, the various types of analyses the AutoChem can perform result in different types of traces. For example, a pulse chemisorption analysis results in a series of peaks that gradually increases in size as the sample is dosed with separate increments of gas. Initially, the gas uptake by the sample results in smaller peaks. But when all the active sites are saturated, no more gas can be taken up and the peaks become equal.

Peak Area

The area beneath each peak is calculated to provide information about the volume of gas reacted during the analysis. Calculations are described in Appendix B.

Automatic Operation

The AutoChem II software provides a simple format for you to specify all the analysis conditions for the experiment; you create a sample file which contains sample information and a list of specific steps the analyzer will follow to perform the experiment(s). Then, the instrument automatically performs the analysis, from controlling the gas mixture and flow rate to monitoring the temperature and pressure. Data points are recorded automatically. BET analyses require some operator interface. After analysis, you can use the Peak Editor to adjust the peaks to create reports that contain the data you need, without baseline noise or other undesirable effects.

Because you can specify up to 99 experiments and each experiment can contain up to 99 steps, the AutoChem II can perform a wide variety of preparation and analysis functions automatically.

Cold Trap

In some cases, it is desirable to trap substances resulting from the reaction. In the previous example, H_2O is produced during the analysis. If the gas flow is passed through the cold trap at an appropriate temperature, the water can be removed before the gas flows past the detector. The cold trap can also be used as a delay path for BET surface area experiments.

Injection Loop

Injection loops are provided for injecting carefully measured doses of gases for analyses such as Pulse Chemisorption. The AutoChem II is shipped with a 1.0-cm^3 loop installed; 0.5- and 5.0-cm^3 loops are also included. If you set up your sample file so that a loop is used for introducing gas into the analyzer, the instrument will automatically dose the sample as specified in the sample file.

Sample Preparation and Calibration

Depending on the type of experiment(s) you wish to perform, sample preparation and/or calibration may be required. More specific instructions are contained in the appropriate sections of this manual.

A sample is prepared for analysis by removing unwanted adsorbates from the surface of the sample. This is usually accomplished by flowing gas over the sample and may include heating the sample. The flowing gas may be inert or chemically active gases may be used to activate the surface.

Calibration routines provide the AutoChem II analyzer and software with the appropriate information to convert electrical signals to physically meaningful data such as volume adsorbed, loop volume, and gas concentration.

Analysis Types

Regardless of which type of experiment you perform, the basic concept is the same: the filament detects changes in the gas mixture flowing past it. The sample, gas selection, and analysis conditions determine what changes occur. Some of the analyses you can perform with the 2920 are described in the following sections.

A tutorial for some of the different type of analyses is provided in Appendix J.

TPD Analysis

Temperature-Programmed Desorption (TPD) analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows through the sample and adsorbs on the active sites. Programmed desorption begins by raising the temperature linearly with time while a steady stream of inert carrier gas flows through the sample.

At a certain temperature, the heat overcomes the activation energy; therefore, the bond between the adsorbate and adsorbent will break and the adsorbed species desorb. If different active metals are present, they usually desorb the adsorbed species at different temperatures. These desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which measures the gas concentrations. The volume of desorbed species, combined with the stoichiometry factor and the temperature at which pre-adsorbed species desorb, yields the number and strength of active sites.

TPR Analysis

Temperature-Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperature at which the reduction occurs. An important aspect of TPR analyses is that the sample need not have any special characteristics other than containing reducible metals.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) through the sample, usually at ambient temperature. While the gas is flowing, the temperature of the sample is increased linearly with time and the consumption of hydrogen by adsorption/reaction is monitored. Changes in the concentration of the gas mixture are determined. This information yields the hydrogen uptake volume.

TPO Analysis

Temperature-Programmed Oxidation (TPO) examines the extent to which a catalyst can be oxidized or was previously reduced.

Usually the sample is pretreated and the metal oxides are reduced to the base metal, typically with a gas mixture of hydrogen with either nitrogen or argon. Then the reactant gas, typically 2-5% oxygen with helium, is applied to the sample in pulses or, alternatively, as a steady stream.

The furnace heats the sample tube and sample according to the user-selected temperature program. The oxidation reaction occurs at a specific temperature. The AutoChem II 2920 measures the uptake of oxygen.

When using any mixture of gases for TPR or TPO analyses, make sure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity (see appendix C).

Pulse Chemisorption Analysis

A Pulse Chemisorption analysis determines active surface area, percent metal dispersion, and the average active particle size by applying measured doses of reactant gas to the sample.

The gas reacts with each active site until all sites have reacted. Once the active sites have completely reacted, the discretely injected gas volumes elute from the sample tube unchanged. The amount chemisorbed is the difference between the total amount of reactant gas injected and the amount that eluted from the sample. The quantity of each pulse of reactant gas is determined by the loop volume on an automatically controlled valve. Three loops of different volumes are provided with the analyzer.

BET Surface Area Analysis

The BET Surface Area analysis lets you evaluate total surface area of the catalyst before and after chemisorption. Pore-plugging phenomena which might occur due to the irreversible adsorbed species during chemical reactions, as well as the occurrence of sintering, can be studied.

After outgassing the sample, a mixture of nitrogen and helium (typically 5 to 30% nitrogen) flows over the sample which is immersed in a liquid nitrogen (LN_2) bath. Both the adsorption and desorption of the N_2 are recorded. The amount of nitrogen desorbed at LN_2 temperatures and the sample weight are used to calculate total specific surface area.

The entire BET analysis — or even repeat analyses — is performed *in situ*.

Langmuir Surface Area Analysis

The Langmuir surface area analysis allows you to evaluate the total surface area of the catalyst and is especially useful for adsorbate/adsorbent systems that adsorb only a monolayer. These materials typically exhibit a Type 1 isotherm and are often microporous. The Langmuir surface area analysis may be best applied to zeolites and microporous carbons – typically the Langmuir surface area will exceed the BET surface area for these materials and provide a more accurate estimation of the total surface area.

The Langmuir surface area report may be applied to a wide range of gas concentrations and is not limited to the typical BET range (5-30% N_2).

Total Pore Volume Analysis

The Total Pore Volume is a single-point estimate of the pore capacity of a material. The total pore volume analysis is usually conducted near the saturation pressure of the adsorbate (0.995 P/Po). The total pore volume of a material can be determined on both fresh and used materials, the difference in pore volume may indicate pore plugging and directly relate to changes in the performance of catalysts and adsorbents.

For a high surface area sample (> 100 m^2/g), a quantity less than 50 mg is recommended. The high sensitivity combined with the large quantity of adsorbed gas allows the use of smaller sample quantities while maintaining high precision. The reduced sample quantity will also reduce the likelihood of saturating the high sensitivity detector.

Additional Uses of the AutoChem II 2920

The AutoChem II 2920 may also be used for Temperature-Programmed Reactions, Catalyst Pretreatment, and Isothermal Reactions. These techniques are similar to many of the procedures that have been previously described. The tremendous flexibility of the AutoChem II 2920 allows the use of custom applications.

Catalyst Pretreatment

Catalyst Pretreatment usually consists of activating a catalyst prior to its use in a chemical reaction. For example, when a temperature-programmed reaction is to be performed, the catalyst must be reduced under a flow of H_2 at a specific temperature. The reduction is necessary to create active sites, or reduced atoms of active metals, which are responsible for the activity of a catalyst.

Temperature-Programmed Reaction

A Temperature-Programmed Reaction lets you monitor the products from the reaction between gases and a catalyst at a specified temperature. The AutoChem II 2920 analyzer can be programmed to raise the temperature of a catalyst bed at a constant ramping rate as the gases flow through the catalyst. At the optimal temperature, the gases react in the presence of the catalyst, creating products. The products of the reaction and the excess reactants can be diverted to a gas chromatograph or to a mass spectrometer to be analyzed.

Isothermal Reaction

An Isothermal Reaction is similar to a temperature-programmed reaction except that the catalyst is kept at a constant temperature (isothermal) to perform the catalytic reaction. Both the product of the reaction and the excess reactants can be diverted to a gas chromatograph or to a mass spectrometer to be analyzed.

Applications

Catalytic processes that benefit from TPD/TPR analyses include:

Polymerization
Hydrogenation
Catalyst Cracking
Hydrocracking
Isomerization
Oxidation
Dehydrogenation
Hydrotreating
Alkylation Reforming

System Requirements

Location

When selecting the location of the analyzer, keep the following in mind:

- The analyzer performs best in a relatively constant temperature environment.
- It should be installed on a workbench approximately 0.9 m (36 in.) high, in a location free of drafts from sources such as a forced-air heating or cooling system.
- The analyzer should not be placed near a window; exposure to direct sunlight may cause the temperature to vary.
- Consider your needs for venting the analyzer exhaust. If you are using toxic or hazardous samples, gases, or vapors, you may wish to locate the analyzer near a vent hood. Considerations for venting the analyzer exhausts are discussed in **Venting the Gas Exhausts** on page **2-6**.
- There must be adequate space for several gas supply bottles nearby, as well as for any options, such as the CryoCooler.

Gases

Compressed gases are required for sample analysis with the AutoChem II 2920 analyzer. Gas bottles and a rack for storage of these bottles should be located near the analyzer. As an alternative, outlets from a central source may be provided nearby. Gas sources must be able to supply flow rates of up to 200 cm^3 per minute at 7 psig (50 kPag) for helium and hydrogen, or at 11 psig (75 kPag) for other gases.

Gas purity should be 99.999% and gases should be free of H_2O.

Gas Regulators

High-purity, dual-stage gas regulators should be used with the AutoChem II 2920. (Appropriate gas regulators are available from Micromeritics; refer to **Ordering Information** on page **10-1**.) Dual-stage regulators minimize the effect of pressure drop in the gas bottle, ensuring that a constant supply of carrier gas is delivered to the instrument. This steady pressure of gas contributes to stable baselines and more reproducible analyses. Refer to **Gas Connections** on page **2-3** for instructions on connecting gas bottles and regulators.

Coolant

An inlet port is provided on the front of the furnace for attaching a coolant. A complete discussion of cooling options is contained in Chapter 4. One cooling device, the KwikCool, is included as a standard accessory. The optional CryoCooler may be purchased from Micromeritics. Refer to **Ordering Information** on page **10-1**.

- The KwikCool enables you to cool the furnace rapidly to ambient temperature. This speeds the analysis and allows for more rapid throughput.
- The CryoCooler makes it convenient to perform subambient analyses. In some cases, such as the reduction of Platinum, the reaction begins at approximately -28 °C, so analyses begun at ambient temperature are undesirable. The CryoCooler, in conjunction with the furnace, lets you continuously ramp temperatures from -120 °C to 1100 °C. The CryoCooler can also be used to speed cooling to ambient following an analysis.

Vapor Generator

An optional Vapor Generator can be used with water or other liquids. The Vapor Generator attaches easily to the analyzer, and its operation is controlled by the software.

The Vapor Generator allows an inert carrier gas, such as helium, to be bubbled through the liquid at up to 100 °C. The vapor then passes through a cooler condensation zone, which is maintained at an accurate temperature to ensure a stable vapor pressure. The vapor then passes to the loop valve zone (Fill/Inject valve) for precise injections onto the sample.

Specifications

The following table contains the specifications of the AutoChem II 2920.

Table 1-1. AutoChem II 2920 Specifications

Characteristic	Specification
TEMPERATURE SYSTEM	
Range:	-100 to 1100 °C (with optional CryoCooler) Ambient to 1100 °C (without CryoCooler)
Selection:	Digitally set, 1 °C increments
Ramp rates:	Up to 50 °C per minute within -70 to 500 °C range; Up to 30 °C per minute within 500 °C to 750 °C range; Up to 10 °C per minute within 750 °C to 1100 °C Range
GASES	
Loop (Analysis):	H_2, CO, O_2, N_2O, NH_3 (maximum 15% NH_3 in helium), liquid vapors and others
Carrier	He, Ar, and other inert gases or other mixtures
Preparation:	H_2, O_2, He, Ar, and others
The Corrosion Resistant version of the AutoChem 2920 allows the use of gases such as H_2S and HCl. Review the list of Wetted Materials and also consult Micromeritics for more guidance.	
GAS FLOW RATE	
All Mass Flow Controllers (MFC)	
Manual control:	0 - 100 cm^3/min. (Rate for Hydrogen; other gases have a different range)
GAS DELIVERY	
Inlet ports:	4 each for preparation gas, carrier gas, and loop (analysis) gas
Temperature control:	Internal gas lines and valves can be heated up to 150 °C
SAMPLE TUBE	
Type:	FlowThru^{TM} sample tubes, for use up to 1100 °C, accept powders and pellets up to 9 mm diameter
Material:	Fused quartz

Characteristic	Specification
ELECTRICAL	
Voltage:	85 to 265 VAC
Frequency:	50/60 Hz
Power:	1100 VA
ENVIRONMENT	
Temperature:	15 to 35 ºC operating; 0 to 50 ºC non-operating
Humidity	20 to 80% relative, non-condensing
PHYSICAL	
Height	24.5 in. (62.2 cm)
Width:	26.0 in. (66 cm)
Depth:	22.75 in. (58 cm)
Weight:	130 lbs (59 kg)
COMPUTER	
Minimum requirements:	Computer capable of running Windows® 2000, Windows® XP Professional, or Windows Vista® Business or Ultimate operating system CD-ROM drive 128 megabytes of main memory 1-gigabyte hard drive SVGA monitor (800 x 600 minimum resolution)

WETTED MATERIALS

Standard System

Stainless steel, Gold plated nickel/iron (filaments), Kalrez® or Chemraz® (most O-ring seals), Quartz (sample tubes, thermocouple sheath), Quartz wool (supports sample in tube), Inconel®- sheathed sample thermocouple (can be placed outside tube or sheathed in quartz), PEEK (Poly Ether Ether Ketone - some ferrules), Teflon® tubing (most exhaust lines; TCD exhaust is stainless steel for connection of external devices), Copper (gas supply lines and BET delay path), Nickel plated brass and steel (gas inlet valves), Viton® (seals inside mass flow controllers), Polyaryletherketone/PTFE composite (rotary valves seals), Silicone/Teflon® sandwich (septum).

Characteristic	Specification

WETTED MATERIALS

System with Vapor Generator Option

As above, plus borosilicate glass Vapor Generator flask, Kalrez® or Chemraz® flask gasket.

System with Corrosion Resistant Option

As above, but stainless steel manifolds are protected from corrosion with vapor deposited Silcolloy by Silcotek®, Stainless steel tubing, fittings, ferrules and rotary valves are replaced by Hastelloy-C® versions, Kalrez® is used for all O-ring seals and the seals inside the mass flow controllers, stainless steel grade 316 gas supply lines, stainless steel gas inlet valves with Kalrez® or Chemraz® seals.

2. INSTALLATION

This chapter describes how to unpack, inspect, and install the AutoChem II 2920 analyzer.

Unpacking and Inspection

When you receive the shipping cartons, carefully compare the Packing List with the equipment actually received and check the equipment for any damage that may have occurred during shipment. Be sure to sift through all packing material before declaring equipment missing.

Save the shipping cartons if equipment or parts have been damaged or lost. The inspector or claim investigator must examine the cartons prior to completion of the inspection report.

Equipment Damage or Loss During Shipment

If equipment is damaged or lost in transit, you are required to make note of the damage or loss on the freight bill. The freight carrier, not Micromeritics, is responsible for all damage or loss occurring during shipment. If you discover that equipment was damaged or lost during shipment, report the condition to the carrier immediately.

Equipment Return

Micromeritics strives to ensure that all items arrive safely and in working order. Occasionally, due to circumstances beyond our control, equipment may be received which is not in working order. When equipment has been damaged (either during shipment or in use) and you wish to return the equipment to Micromeritics for repair or replacement, please follow these steps:

1. Pack the instrument in its original shipping carton if possible. If the original carton is unavailable, for a nominal fee Micromeritics can provide another carton for your use.

Failure to package your instrument properly may result in shipping damage.

2. Tag or otherwise identify the defective equipment, noting the defect and, if possible, the circumstances under which the defect occurs.

3. Make reference to the sales order or purchase order for the equipment, and provide the date the equipment was received.

4. Notify a Micromeritics Service Representative of the defect and request shipping instructions. The Service Department will assign a Return Material Authorization (RMA) number to your return and provide shipping information.

Installing the Hardware

The AutoChem 2920 analyzer initially is installed by a Micromeritics representative. The following sections are provided to explain common tasks you may need to perform during use of the analyzer.

Power Supply

Connect the female end of the power cord supplied with your instrument to the power connector located on the side panel. Connect the male end to an appropriate power source.

The power and RS-232 cables supplied with your analyzer are shielded. If you replace or alter any of these cables, they may no longer conform with the European Council Directives.

Follow the manufacturers' instructions for connecting your computer and printer to an appropriate power supply.

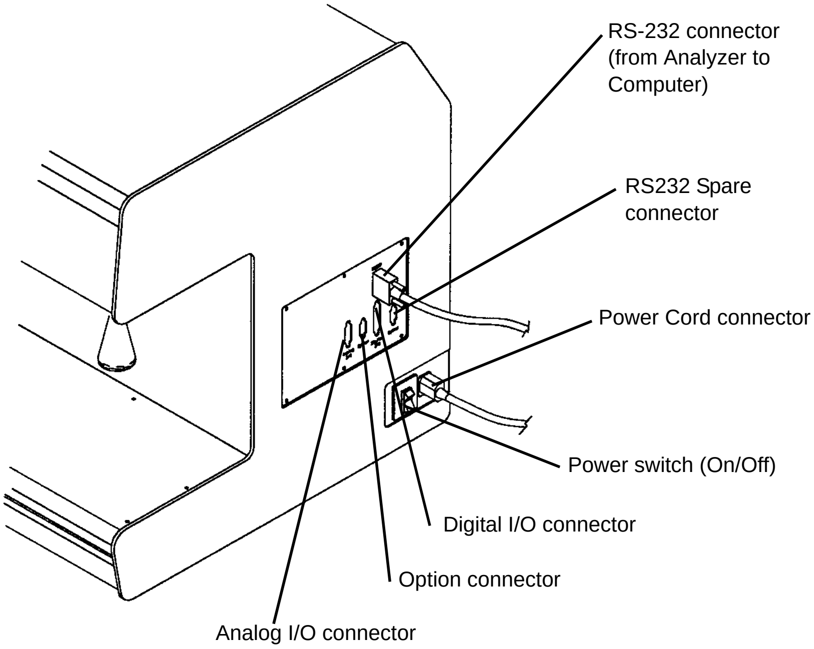


Figure 2-1. Power Switch and Connector Location

Cable Connections

Use the RS-232 cable supplied with your instrument to connect the analyzer to the computer; this cable is connected to the connector labeled **RS232** (refer to Figure 2.1 for location).

When connecting a cable to the connector labeled **Analog I/O** or **Digital I/O**, be sure it is no longer than 2.9 m (9.5 ft).

Do not connect cables to the connectors labeled **Option** and **RS232 Spare**; these connectors are for factory use only.

Auxiliary Inputs and Outputs

Ports are provided for connecting auxiliary inputs and outputs. For example, you may wish to connect a chart recorder to the analyzer for use in gas chromatography.

Refer to Appendix E, page **E-1** for a complete description of the auxiliary input/output function.

Gas Connections

Gas regulators used with the AutoChem II must be of comparable quality to those used for semiconductor manufacturing and gas chromatography. Regulators must avoid or minimize the amount of lubricants and elastomers to which the gas is exposed. Avoid low-quality regulators because the lubrication and elastomers may contaminate the gas stream and cause unstable baselines, catalyst poisoning, and other problems.

Always use two-stage regulators for the carrier gas (to ensure stable flow rates) and for the preparation and loop gas (to minimize regulator lock-up).

High-quality regulators are usually available to suit local gas connections and safety regulations from chromatography gas suppliers and semiconductor gas suppliers, as well as from Micromeritics (refer to **Ordering Information** on page **10-1**).

The AutoChem II 2920 provides inlet connections for up to four preparation gases, four carrier gases, and four loop (analysis) gases. Before connecting gas bottles:

- Make sure that all gas bottles are shut off before connecting them to the analyzer.
- Make sure that no analysis is in progress (the software is idle).

1. Attach an appropriate regulator to the first supply bottle you want to connect. (Copper tubing and brass reducer fittings are supplied with the analyzer. If you are using a gas that is incompatible with brass and copper, substitute compatible tubing and fittings; stainless steel gas lines are available from Micromeritics.) Leave the gas bottle shut-off valve closed until instructed otherwise.

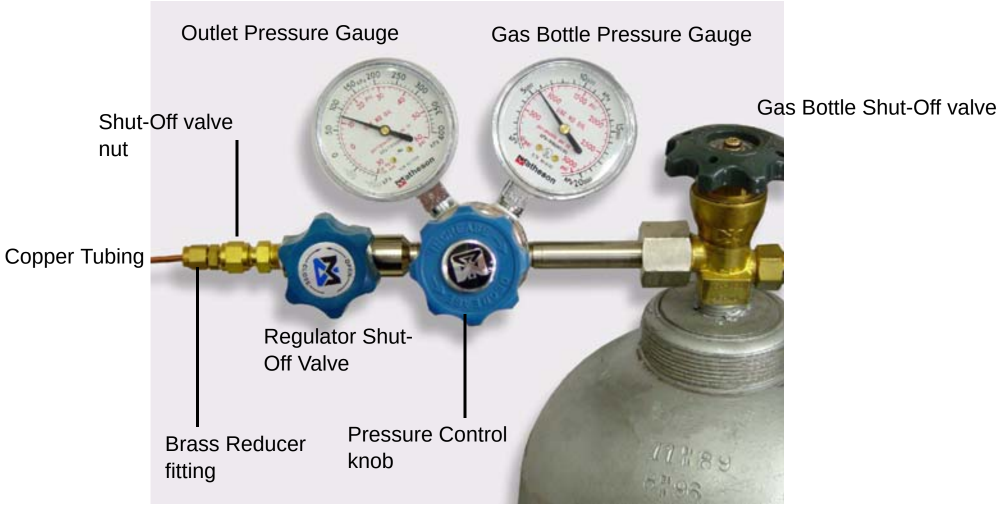


Figure 2-2. Connecting a Gas Supply Bottle to the Regulator

2. If the regulator has a 1/8-in. outlet, proceed to step 3. If the regulator has a 1/4-in. outlet, attach the supplied brass reducer fitting to the outlet of the regulator shut-off valve.

3. Tighten the regulator shut-off valve nut.

Do not over-tighten the fittings. Doing so can collapse the brass fitting and cause a leak.

4. Attach the copper tubing to the regulator or to the brass reducer fitting.

5. Purge the regulator as follows:

    a. Close the regulator shut-off valve by turning it fully clockwise.

    b. Turn the pressure regulator control knob fully counterclockwise.

    c. Open the gas bottle valve by turning it counterclockwise, then close the gas bottle valve.

    d. Observe the gas bottle pressure gauge. If the pressure decreases, tighten the nut connecting the regulator to the gas bottle. If the pressure is stable, proceed to step e.

For hazardous gases, make sure the gas supply equipment is adequately vented.

e. Turn each pressure regulator control knob clockwise until the outlet pressure gauge indicates 5 psig (35 kPag). Then open each regulator shut-off valve by turning it counterclockwise. This action will purge the regulator and copper tubing.

f. Make sure the gas bottle valve is completely closed.

6. Attach the other end of the copper tubing to the appropriate fitting on the side of the analyzer.

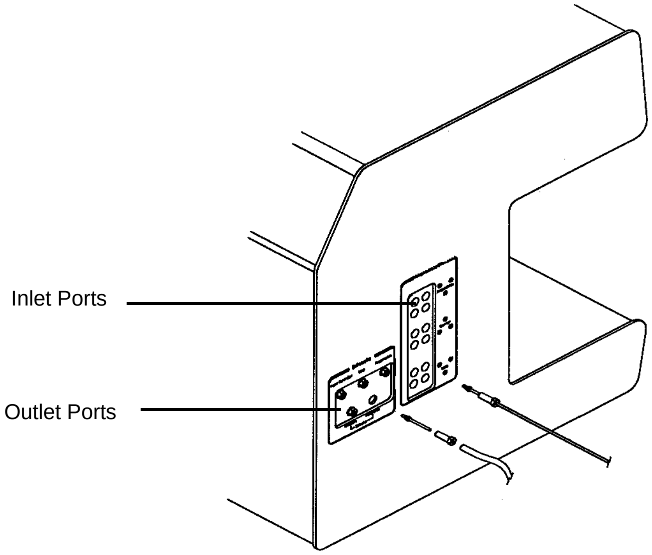


Figure 2-3. Side Panel Connections - Inlets and Exhausts

7. Set the regulator pressure using the following guidelines:

   - A pressure of 18 to 22 psig is appropriate for most applications.
   - When high flow rates are required, you may have to raise the pressure to 30 psig.
   - If you are using a gas supply tubing that is longer than the tubing supplied with your analyzer, you may have to raise the pressure to achieve the desired flow rate.

A form is provided in Appendix A for you to record the name of the gas connected to each inlet port. This form may be helpful in completing the Gas Selections dialog, as described later in this chapter.

8. Repeat steps 1 through 7 for each additional preparation, carrier, or loop (analysis) gas supply bottle you wish to attach.

Venting the Gas Exhausts

Each of the exhaust ports should be safely vented. The appropriate method for venting each port depends upon the gases you are using and the experiments you are performing.

Use extreme caution when establishing your exhaust method, in order to prevent dangerous accumulations or combinations of gases, vapors, or condensibles, particularly when using the optional Vapor Generator. (Instructions for attaching the Vapor Generator are located on page 2-16.)

Vapor Generator Exhaust

The type of venting described for the vapor generator may also apply to other exhausts if the possibility of condensation of liquids is present.

If the Vapor Generator contains toxic or otherwise hazardous liquids, these are vaporized, travel through the system, and are vented through the Vapor Generator exhaust port. After the vapor leaves the heated analyzer, it may cool and return to the liquid state. Accumulation and/or mixing of such condensibles can create a variety of dangerous conditions in the laboratory. It is the operator’s responsibility to ensure safe venting of all exhausts.

Recommended Method

The ideal treatment for vapor exhaust is to run the exhaust through heated extension lines to a fume hood, separate from any other exhausts. Additionally, a downward slope of the extension lines should be carefully maintained to prevent accumulation of any liquids that should condense in the line.

Alternative Method

As an alternative to the method recommended above, you may wish to vent the exhaust through a collection container to collect any condensibles before gases are vented. An example of one such method is shown in the following illustration.

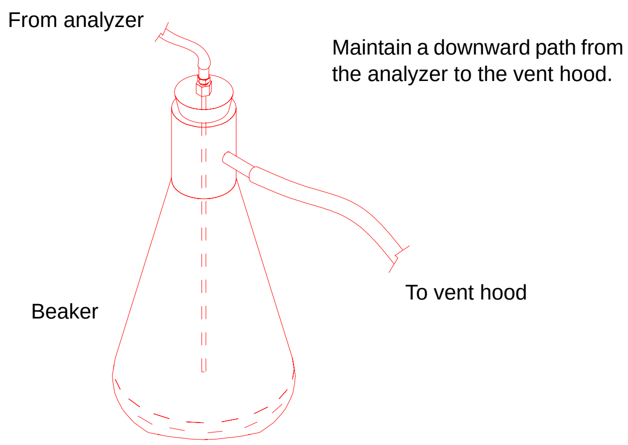


Figure 2-4. Example of a Collection Container for Venting Exhaust

Particular care should be taken if you plan to vent the exhaust from more than one port into a single collection container before diverting the gases. Make sure that you consider any hazards of combining the various exhausts in a single venting container, basing your decision on the materials being used in the experiment.

Results of Improper Venting

If no venting method is provided, the vapor is discharged into the laboratory, potentially causing a hazard for laboratory personnel.

Another possible effect of improper venting is condensation of liquid drops in the exhaust line, which could cause intermittent blockage of the line. As a result, detector signals may become erratic. This is visible in the data as smaller-than-expected peaks, missing peaks, or even larger peaks, depending upon the timing of the drop blocking the exhaust.

Installing the Furnace

Initially, the furnace should be installed on the analyzer. However, it should be removed when performing B.E.T. analyses. Removal instructions follow these instructions.

1. Hold the open furnace next to the support post. Raise the furnace, moving it above the support post until the retaining knob is inserted in the hole in the top of the furnace.

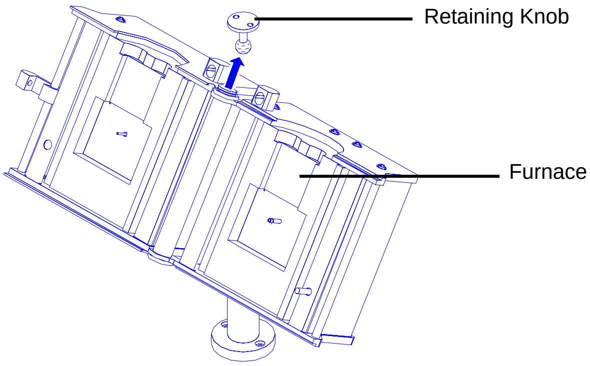


Figure 2-5. Raising the Furnace onto the Retaining Knob

2. When the retaining socket on the bottom of the furnace is located above the support post, gently lower the furnace until it rests securely on the post.

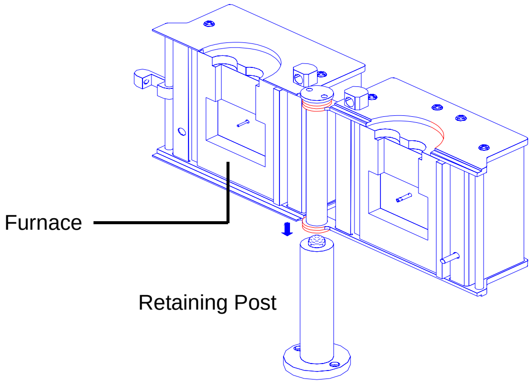


Figure 2-6. Lowering the Furnace onto the Retaining Post

3. When you are ready to begin the experiment, close the furnace around the sample tube and secure the latch.

4. Plug the thermocouple cable and the furnace power cable into the appropriate connectors on the analyzer. These connectors are located on the recessed front panel, and are labeled.

Removing the Furnace

When performing B.E.T. analyses, the furnace must be removed to allow placement of a cold bath dewar around the sample.

1. Unplug both the furnace power plug and the thermocouple cable from the analyzer.

2. Open the latch, then open the furnace.

If used recently, parts of the furnace may be hot. Use the cotton gloves provided with the analyzer to protect your hands.

3. Gently lift the right side of the furnace until it is freed from the support post.

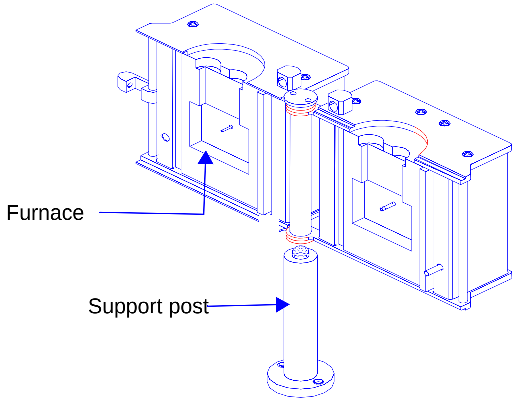


Figure 2-7. Removing the Furnace

4. Lower the furnace, moving it away from the support post until it is free of the retaining knob.

5. Close the furnace and secure the furnace latch. Place the furnace in a location where it will be protected from damage.

Attaching the KwikCool or Optional CryoCooler

KwikCool

A KwikCool unit is shipped with every AutoChem II 2920. This unit allows you to cool the furnace quickly between analyses.

Installation

1. Turn off the analyzer.
2. Attach the KwikCool cable to the connector labeled **Sub Ambient Cooler** on the front panel of the analyzer.
3. Attach the tubing provided to the inlet on the front of the furnace.
4. Attach a supply of dry house air (or nitrogen) to the gas inlet on the front of the KwikCool; the pressure is indicated next to the inlet.
5. Fill a dewar with an ice water bath.
6. Place the KwikCool on top of the dewar.

Figure 2-8. Installing a KwikCool

7. Turn on the analyzer and start the analysis program.

Operation

When you insert an experiment step, a space is provided (on the Temperature Ramp dialog) for you to indicate that you are using the KwikCool. Refer to **"Enable KwikCool" on page 5-38.**

CryoCooler

The CryoCooler is an optional device that enables you to start an analysis at sub ambient temperatures. The CryoCooler cools the sample quickly and holds the temperature at a user-specified value between +20 and -100 ºC.

The CryoCooler unit consists of a cryogenic pump mechanism, a storage dewar for liquid nitrogen (LN_2), and a delivery hose that attaches to the AutoChem furnace.

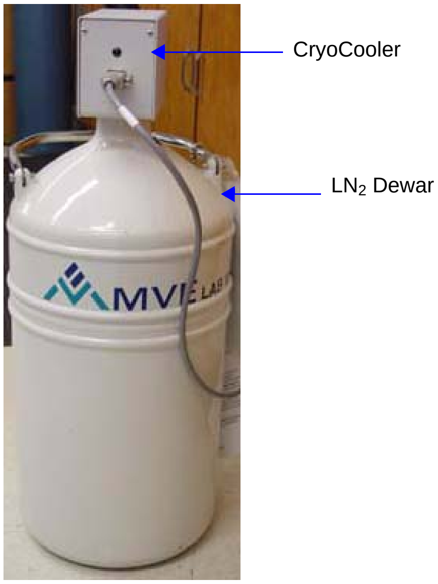


Figure 2-9. CryoCooler Unit

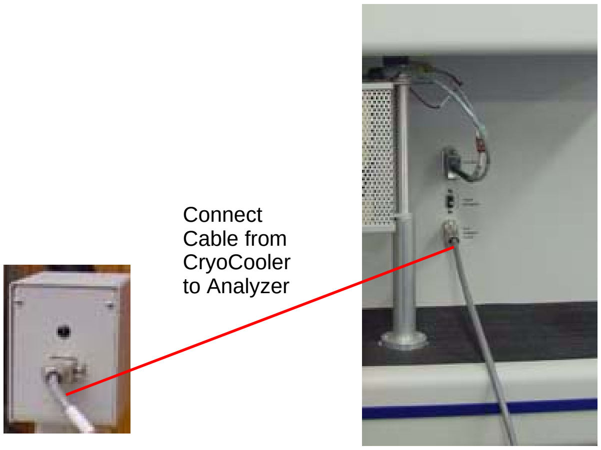
Stable subambient temperatures are achieved by small, frequently repeated injections of LN_2 into the analyzer’s furnace cavity, which contains the sample. (Actually, the stream that reaches the sample compartment is not liquid but cold vapor.)

Always wear a safety face shield or goggles and gloves when handling liquid nitrogen to prevent the possibility of splashing liquid nitrogen into your eyes or onto your skin.

The CryoCooler must be free of internal moisture before insertion into LN_2. If present, moisture will freeze and prevent operation. The system was free of moisture when it left the factory; however, dry air, nitrogen, or other inert gas should be passed through the delivery hose to be sure it has remained dry.

Installation

1. Attach one end of the cable (provided with the CryoCooler) to the connector labeled **Sub Ambient Cooler** on the front panel of the analyzer. Attach the other end of the cable to the connector on the CryoCooler.



CryoCooler **Analyzer Front Panel**

2. To verify the CryoCooler is operating properly before inserting it into LN_2, enable manual control, right-click on the CryoCooler icon, and set the speed to 100%. The green light on the housing should come on and the rotor on the pump should be visibly turning. Turn the CryoCooler off using manual control, but leave the cable connected.

3. Fill the dewar approximately two-thirds full with LN_2.

4. Insert the CryoCooler into the dewar. Boiling will result as the pump mechanism contacts the LN_2 so insert the CryoCooler slowly over a period of several minutes. This permits most of the resulting vapor to escape before the CryoCooler is finally centered on the dewar opening.

Insert the CryoCooler into the LN_2 slowly. Inserting it too quickly will cause the LN_2 to boil-off and possibly splash onto your hands.

5. Place the dewar on the floor near the front of the analyzer or beside the analyzer on its workbench.

6. Attach the delivery hose from the CryoCooler to the furnace inlet tube.

Delivery hose connection
(tighten thumbwheel to secure)

7. Secure the delivery hose by tightening the thumbwheel on the furnace connection.
8. Allow five minutes for thermal expansion and contraction effects to dissipate, then start the AutoChem analysis.

If the CryoCooler fails to respond immediately, it may be due to components having not yet thermally adjusted to their new temperature surroundings or to ice formation from previously condensed moisture. Wait a few minutes and try again. Failure this time is almost certainly due to ice formation.

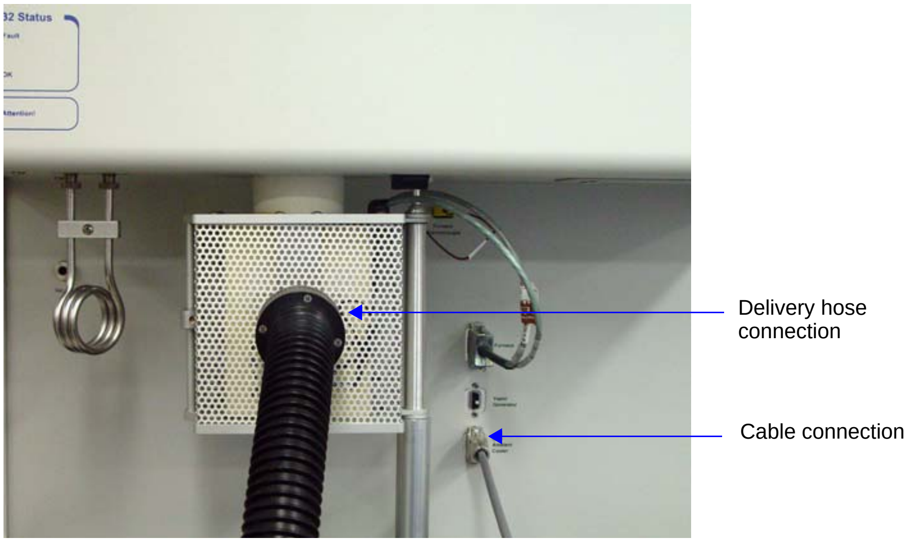
Routine Maintenance

The buildup of ice and frost particles is likely in any device which uses liquid nitrogen in a laboratory environment. Repeated filling and evaporation alone can lead to an accumulation of ice crystals within the dewar. The CryoCooler has been carefully designed to minimize problems from ice. Ice problems can be avoided almost entirely if, on a weekly basis, you empty the dewar and thoroughly dry its interior.

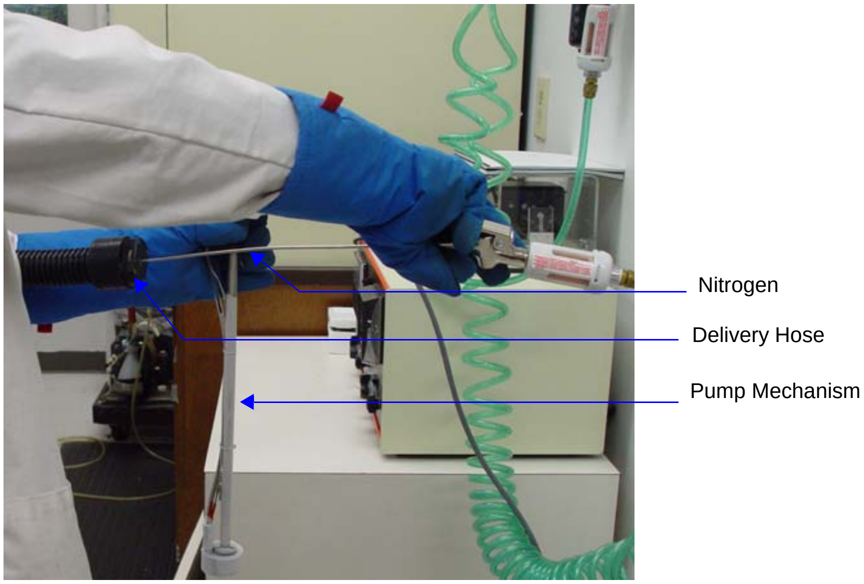
Recovering from an Iced Condition

If ice buildup should occur in the CryoCooler, a thermal overload switch activates and cuts power to the pump motor. The green light will also be off in this condition. Perform the following steps to remove ice buildup:

1. Remove the CryoCooler from the dewar.
2. Unplug the cable from the **Sub Ambient Cooler** connector on the front of the instrument to allow a few minutes for the thermal protector to reset itself.
3. Turn the thumbwheel on the delivery hose connection clockwise to loosen, then disconnect the hose from the analyzer.



4. Blow dry air or nitrogen into the hose and out of the pump mechanism, holding the CryoCooler nearly vertical.



5. Also blow into the pump inlet and into one of the holes of the member connecting the housing and the pump just above its connection point to the pump.

6. It is permissible to heat the pump mechanism with a laboratory heat gun until it is too warm to hold. When doing so, be careful not to heat the plastic delivery hose directly; it will melt if overheated.

Do not apply heat directly to the delivery hose. Doing so may melt the plastic.

7. Slowly insert the CryoCooler back into the dewar and proceed with the sample analysis.

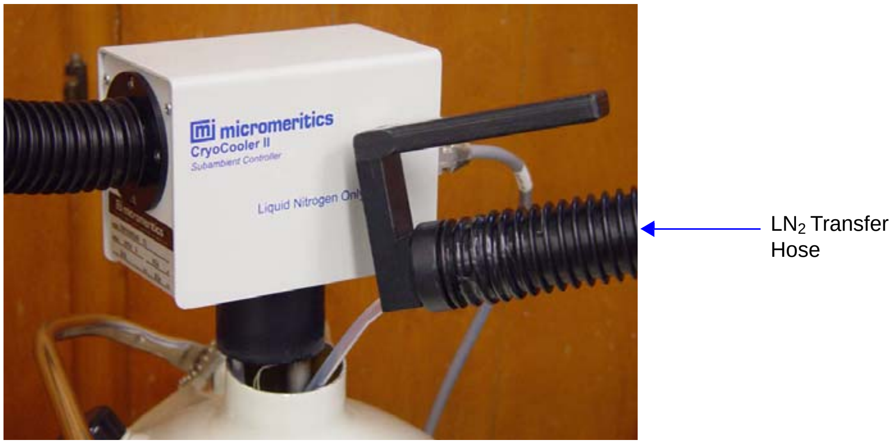
The CryoCooler reservoir retains LN_2 for several days when not used. If its use is temporarily suspended, leave it plugged into the instrument but disconnected from the furnace inlet. An internal heater produces a slight outflow of gaseous nitrogen which prevents ice buildup in the delivery hose.

Replenishing the Dewar

You can replenish the dewar without completely removing the CryoCooler in either of two ways:

- **By adding liquid nitrogen to the dewar.**

  Lift the CryoCooler a few inches above the neck of the dewar, allowing it to rest on the rim of the neck. Then insert the nozzle of an LN_2 transfer system and refill the dewar. The Model 021 LN_2 transfer system is available from Micromeritics (refer to Chapter 10 for ordering information).



Insert the CryoCooler into the LN_2 slowly. Inserting it too quickly will cause the LN_2 to boil-off and possibly splash onto your hands.

- **By attaching the CryoCooler to another dewar.**

  Remove the CryoCooler from the nearly empty dewar and immediately insert it into another full dewar.

Attaching the Optional Vapor Generator

The Vapor Generator enables you to perform analyses using vapor.

Use appropriate safety measures to prevent injury from contact with hazardous liquids used in the Vapor Generator. Information about safely venting the exhausts is found earlier in this chapter.

The majority of the Vapor Generator’s components are internal to the analyzer, and are installed and calibrated by a Micromeritics Service Representative.

To attach the Vapor Generator’s external parts:

1. Fill the Erlenmeyer flask to a depth of at least 50 mm (2 in.) with the desired liquid.
2. Unzip the heating mantle, place it around the flask; do not zip it shut. Screw the flask into the port on the underside of the front panel. The metal cylinder that extends down from the Vapor Generator port is the aerator. When the flask is properly installed, the aerator should extend down into the flask and its contents. Zip the mantle shut.
3. Plug the heating mantle connector into the appropriate connector on the recessed front panel (identified on the panel). Tighten the connector screws.

4. The valve that controls the flow of gas from the Vapor Generator has two states: Bypass and Vapor. When you create the sample information file, select Vapor for the state of the Vapor Generator valve during the portion(s) of analyses in which you wish to flow vapor through the system. The Loop gas is the gas which flows through the Vapor Generator; make sure the gas you are using for vapor generation is connected to one of the Loop gas inlet valves.

Be sure to clean the vapor generator before changing to a different liquid (refer to Chapter 9 for cleaning instructions).

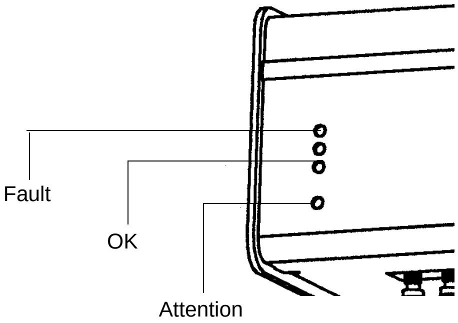
Turning On the Analyzer

If you are connecting the KwikCool, or CryoCooler, do so before turning the analyzer on.

Place the power switch (main breaker switch) on the side panel of the analyzer in the ON (|) position. The power switch is located on the right side of the analyzer next to the power cord connector (refer to Figure 2-1, page 2-2).

When the system is turned on for the first time or after a period of non-use, it should be allowed to warm up for at least three hours before analyses are performed. During this time, a purge carrier gas (such as helium or nitrogen) should be flowed through the system at about 50 cm^3/min.; the heat zones should be set at 110 °C. Refer to **Enable Manual Control** on page **6-12**.

When the analyzer is turned on (but the software is still not running), the uppermost and lowermost indicator lights (the red Fault light and the green Attention! light) on the upper front panel are illuminated. When the software is subsequently started and communication is established with the analyzer, these lights are turned off and the second light from the bottom (the green OK light) is illuminated.



Instructions for starting the software are found later in this chapter. Refer to **Front Panel Indicators** on page **3-1** for a description of the indicators.

Installing the Software

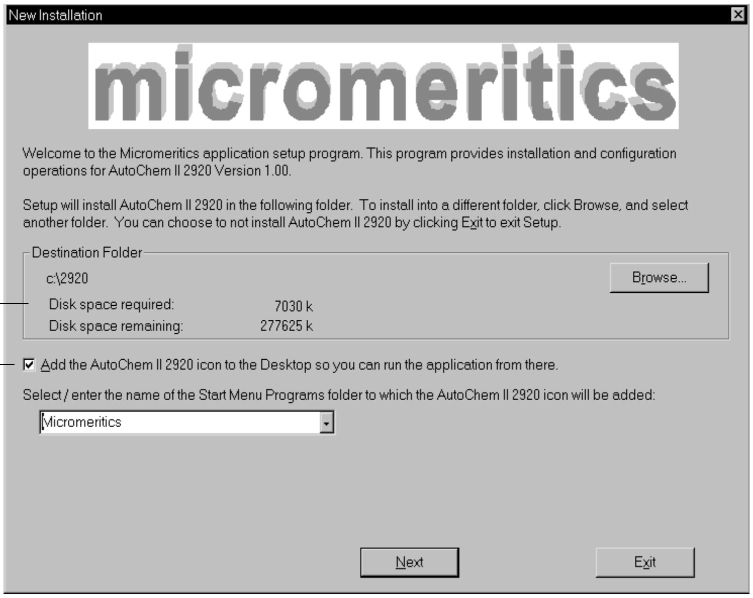
The AutoChem II 2920 program is also available as a standalone option so that you can install it on a computer other than the one controlling the analyzer. This allows you to create or edit sample and parameter files, as well as generate reports on completed sample files. Review the Micromeritics PROGRAM License Agreement for restrictions on using another copy.

Install the AutoChem II application software as follows:

1. Insert the AutoChem program CD into the CD-ROM drive.

Proceed to Step 4 if AutoPlay is enabled on your computer; Setup will start automatically and display the New Installation dialog.

2. Select **Start**, **Run** from the **Status** bar.
3. Enter the drive designator for the CD-ROM, followed by **SETUP** (for example: **e:setup.)**, then click **OK**.
4. The New Installation dialog is displayed.



Welcome to the Micromeritics application setup program. This program provides installation and configuration operations for AutoChem II 2920 Version 1.00.

Setup will install AutoChem II 2920 in the following folder. To install into a different folder, click Browse, and select another folder. You can choose to not install AutoChem II 2920 by clicking Exit to exit Setup.

Destination Folder

c:\2920

Disk space required: 7030 k

Disk space remaining: 277625 k

☑ Add the AutoChem II 2920 icon to the Desktop so you can run the application from there.

Select / enter the name of the Start Menu Programs folder to which the AutoChem II 2920 icon will be added: Micromeritics

Displays the amount of current disk space and the amount required for installation of the application. Also displays the directory into which the application will be installed.

Select this option to add the AutoChem II 2920 icon to your desktop.

You may terminate the installation at any time by clicking Exit. If you do so, you must start the installation program from the beginning.

The **Destination Folder** group box displays the amount of current disk space, the amount of disk space required for the analysis program, and the directory into which the application will be installed. If you prefer a different directory for installation, click **Browse** to select the desired directory.

5. If you want to run the application from the desktop, select the checkbox just below the Destination Folder group box to add an icon.

6. The AutoChem II icon will be added to the Micromeritics folder by default. If you prefer a different folder, enter or select one from the drop-down list.

7. Click **Next**; the Analyzer configuration dialog is displayed.

8. In the Step 1 group box, click the radio button for the number of analyzers to be attached to this computer. If you are attaching two analyzers, be sure your computer has two serial communication ports.

Choose 0 (zero) if you are installing this program for offline data reduction on a computer other than the one controlling the analyzer, or if this is the destination computer in a Move operation.

9. For each analyzer, enter the analyzer’s serial number and communications port number.

10. Click **Next**; the Calibration File Installation dialog is displayed. Read the information displayed and proceed accordingly.

11. After the calibration files have been installed, the Installation Complete dialog containing the Readme file is displayed.

12. Use the scroll bar if you want to read the contents of the file, then click **Finish** to close the dialog.

13. Remove the program CD and store in a safe place. The original program CD contains the calibration files specific to your instrument. Upgrade CDs do not contain calibration files. Therefore, it is important that you store the original program CD in a secure location in the event calibration files need to be reinstalled.

Using the Setup Program for Other Functions

After initial installation of the AutoChem analysis program, the application setup program can be used to:

- Upgrade software
- Add an analyzer
- Move an analyzer from one computer to another computer
- Remove an analyzer from the computer
- Change the analyzer setup
- Reinstall calibration files
- Uninstall the analysis program

To start the application setup program:

1. Ensure that the analysis program is not operating.

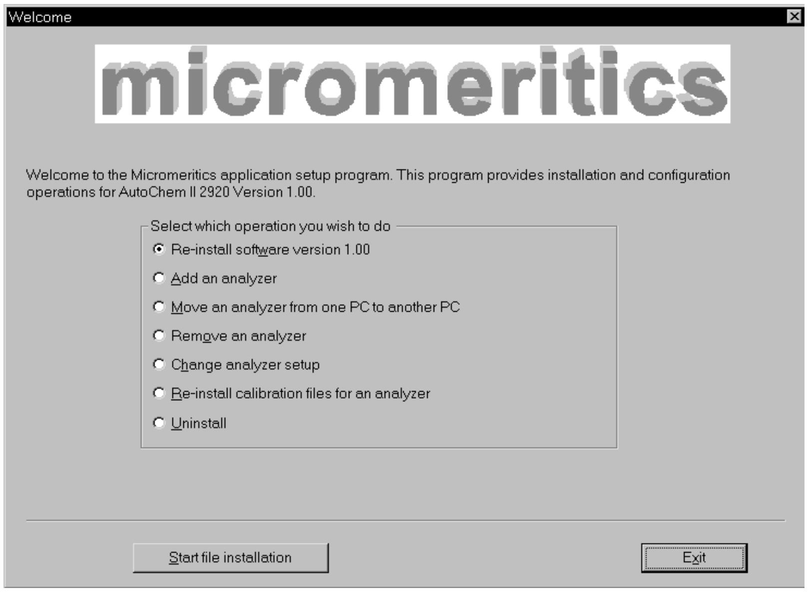
2. Insert the AutoChem HP 2950 Setup CD into the CD-ROM drive.

Proceed to Step 5 if AutoPlay is enabled on your computer; Setup will start automatically and display the New Installation dialog.

3. Select **Start**, **Run** from the **Status** bar.

4. Enter the drive designator for the CD-ROM, followed by **SETUP** (for example: **e:setup.)**, then click **OK**.

5. The setup Welcome screen showing the options available is displayed.

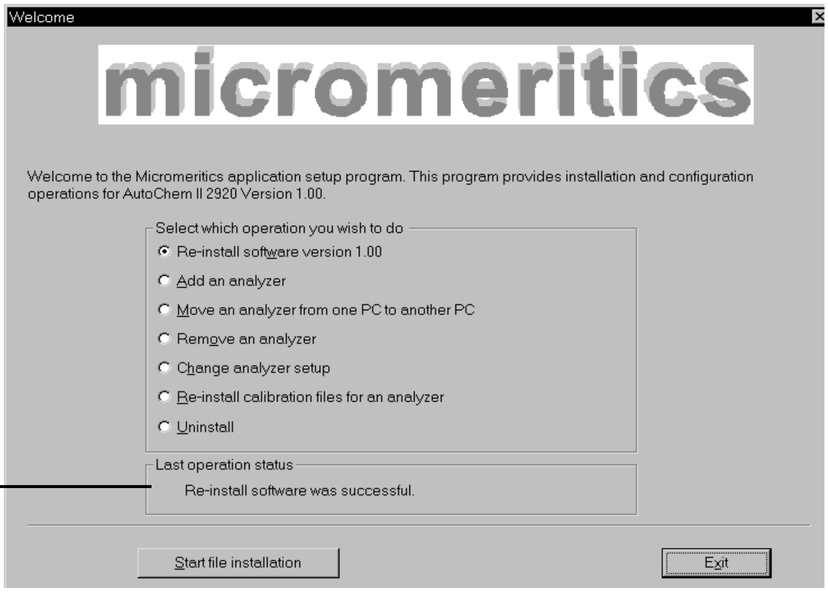


Welcome to the Micromeritics application setup program. This program provides installation and configuration operations for AutoChem II 2920 Version 1.00.

Select which operation you wish to do
- ● Re-install software version 1.00
- ○ Add an analyzer
- ○ Move an analyzer from one PC to another PC
- ○ Remove an analyzer
- ○ Change analyzer setup
- ○ Re-install calibration files for an analyzer
- ○ Uninstall

6. Select the operation you wish to perform. Procedures for performing each operation are in subsequent sections.

   After the requested operation is completed, the setup Welcome screen is again displayed. A confirmation message indicating completion of the operation is shown in the lower section of the dialog.



Welcome to the Micromeritics application setup program. This program provides installation and configuration operations for AutoChem II 2920 Version 1.00.

Select which operation you wish to do
- ● Re-install software version 1.00
- ○ Add an analyzer
- ○ Move an analyzer from one PC to another PC
- ○ Remove an analyzer
- ○ Change analyzer setup
- ○ Re-install calibration files for an analyzer
- ○ Uninstall

Last operation status: Re-install software was successful.

7. After you have completed all desired operations, click **Exit** to close the dialog.

Installing Subsequent Software Versions

When you install a software upgrade, the system installs all of the application files and any status files that do not already exist on the computer. Existing analyzer status files are not affected and default and data files are not overwritten. There are three types of subsequent installation; the software version controlled by the setup program is:

- a later version than the version installed on the computer
- the same version as the version installed on the computer
- an earlier version than the version installed on the computer

The setup program automatically detects which type of installation applies and customizes the selection in the Setup dialog accordingly.

1. Start the Setup program. Choose the software option; remember, only the applicable option will display; it will be one of the following:

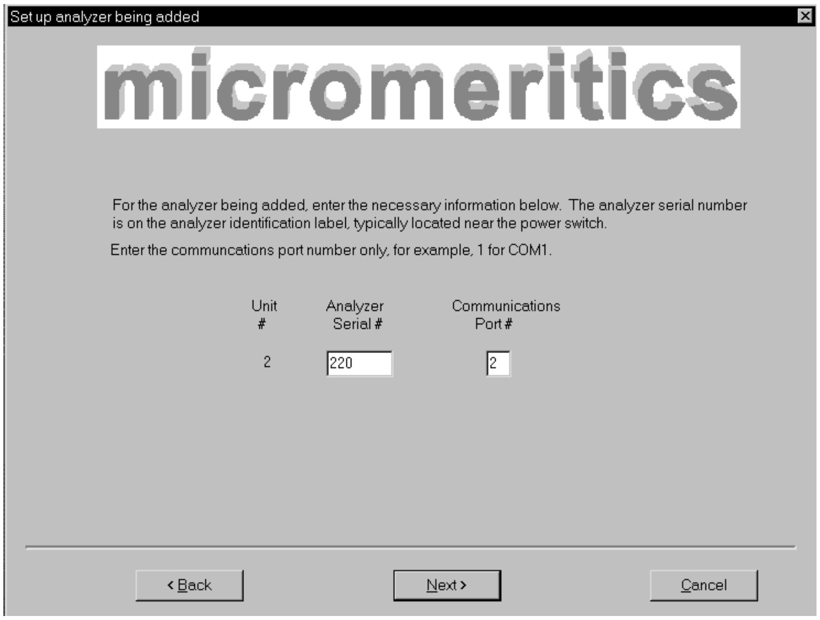
   - Upgrade software to version (number) from version (number)
   - Reinstall software version (number)
   - Downgrade software to version (number) from version (number)

2. Click **Start File Installation**; the application installs the software and redisplays the setup Welcome dialog. If no other operations are desired using this dialog, click **Exit** to close the dialog.

Adding an Analyzer

Add an analyzer to the existing application as follows:

1. Start the Setup program. Select **Add an analyzer**, then click **Next**; the Setup analyzer dialog is displayed:



For the analyzer being added, enter the necessary information below. The analyzer serial number is on the analyzer identification label, typically located near the power switch.

Enter the communcations port number only, for example, 1 for COM1.

Unit #	Analyzer Serial #	Communications Port #
2	220	2

2. Enter the serial number of the analyzer being added, then the communications port to which it is to be connected.

3. Click **Next**; the Calibration Installation dialog is displayed.

4. Select the location of the calibration source files. If the calibration files are located in a directory other than the one displayed, click **Browse** to select the directory. Click **Finish**; a media change dialog is displayed.

5. Click **OK** to install the calibration files. A dialog containing the Readme file is displayed after the calibration files are installed.

6. Click **Exit** to close the dialog.

Moving an Analyzer from One Computer to Another Computer

You can move an analyzer, along with its status and calibration files, from one computer (Source PC) to another computer (Destination PC).

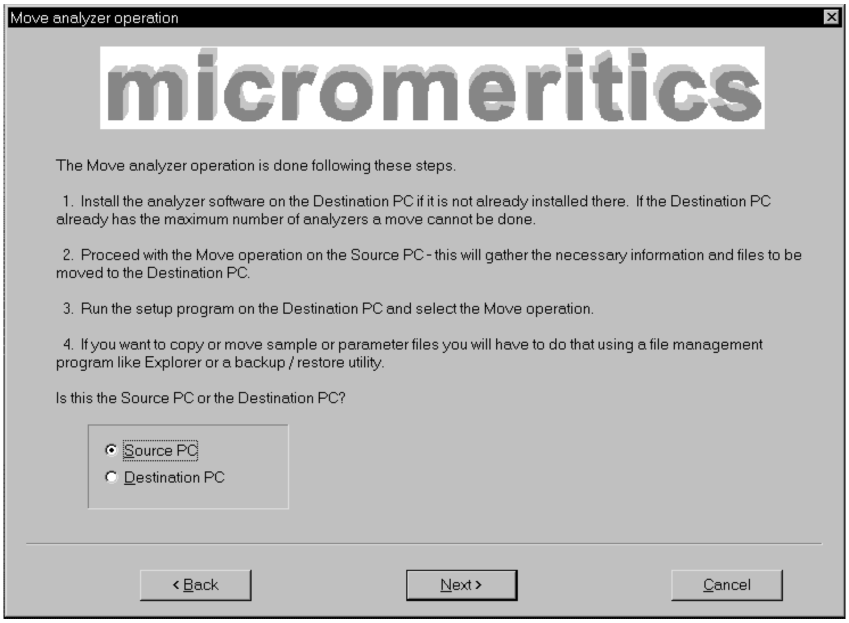
This operation does not move sample or parameter files. To move these files, use a file management program such as Explorer or a backup/restore utility.

1. Install the analysis program on the destination computer. Refer to **Installing the Software** on page **2-18**. Be sure to select 0 as the number of instruments; all related instrument information will be transferred in the **Move** operation.

   If the analysis program is already installed on the destination computer, proceed to Step 2.

2. Start the application setup program on the source computer. Refer to **Using the Setup Program for Other Functions** on page **2-20**.

3. In the Setup dialog, select **Move an analyzer from one PC to another PC**, then click **Next**; the Move analyzer operation dialog is displayed.



The Move analyzer operation is done following these steps.

1. Install the analyzer software on the Destination PC if it is not already installed there. If the Destination PC already has the maximum number of analyzers a move cannot be done.

2. Proceed with the Move operation on the Source PC - this will gather the necessary information and files to be moved to the Destination PC.

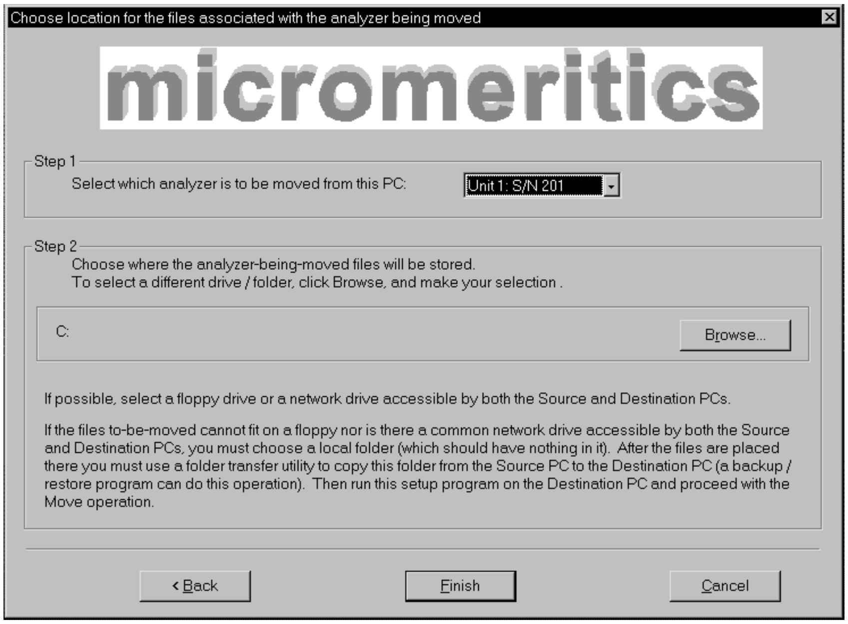
3. Run the setup program on the Destination PC and select the Move operation.

4. If you want to copy or move sample or parameter files you will have to do that using a file management program like Explorer or a backup / restore utility.

Is this the Source PC or the Destination PC?

● Source PC
○ Destination PC

4. Select **Source PC**, then click **Next**; the following dialog is displayed.



Choose location for the files associated with the analyzer being moved

Step 1

Select which analyzer is to be moved from this PC: Unit 1: S/N 201

Step 2

Choose where the analyzer-being-moved files will be stored.
To select a different drive / folder, click Browse, and make your selection .

C:

If possible, select a floppy drive or a network drive accessible by both the Source and Destination PCs.

If the files to-be-moved cannot fit on a floppy nor is there a common network drive accessible by both the Source and Destination PCs, you must choose a local folder (which should have nothing in it). After the files are placed there you must use a folder transfer utility to copy this folder from the Source PC to the Destination PC (a backup / restore program can do this operation). Then run this setup program on the Destination PC and proceed with the Move operation.

5. In the Step 1 group box, select the analyzer that is to be moved.

6. In the Step 2 group box, choose a location in which the moved files will be stored. If possible, choose a floppy drive or a shared network drive. If this is not possible, select a local folder. After the files are placed there, use a folder transfer utility to copy this folder from the Source PC to the Destination PC.

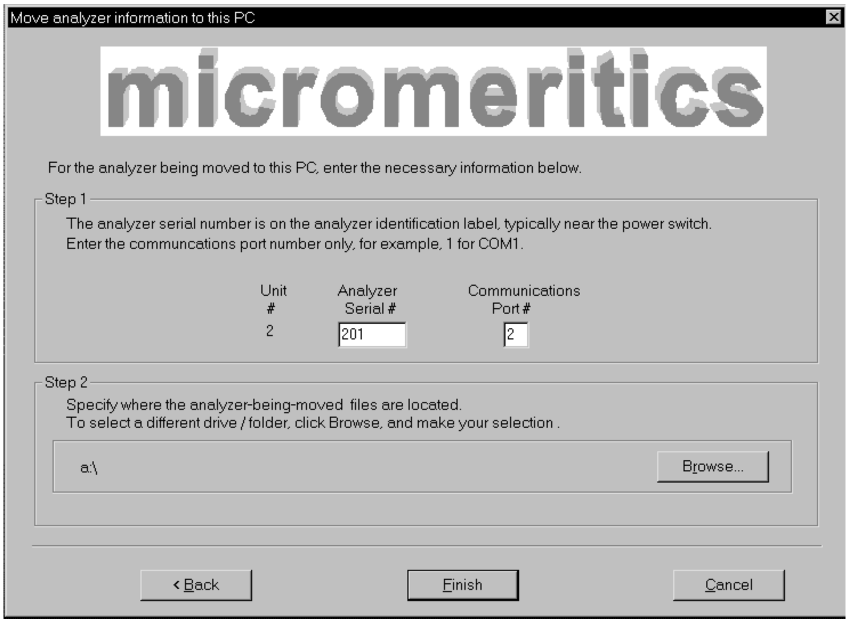
   Calibration files are not included in the moved files. These files must be copied and moved separately to the destination computer. The calibration files are located in a subdirectory (folder) of the Hardware directory. The subdirectory is named 292-(serial number).

7. Click **Finish**; the files are moved and the setup Welcome screen is displayed.

8. Start the application setup program on the destination computer.

9. In the Setup dialog, select **Move an analyzer from one PC to another PC**; the Move analyzer operation dialog is displayed (shown on previous page).

10. Select **Destination PC**, then click **Next**; the following dialog is displayed.



For the analyzer being moved to this PC, enter the necessary information below.

Step 1

The analyzer serial number is on the analyzer identification label, typically near the power switch.
Enter the communcations port number only, for example, 1 for COM1.

Unit #	Analyzer Serial #	Communications Port #
2	201	2

Step 2

Specify where the analyzer-being-moved files are located.
To select a different drive / folder, click Browse, and make your selection .

a:\

11. In the Step 1 group box, enter the serial number of the analyzer being moved and the communications port to which it will be attached.

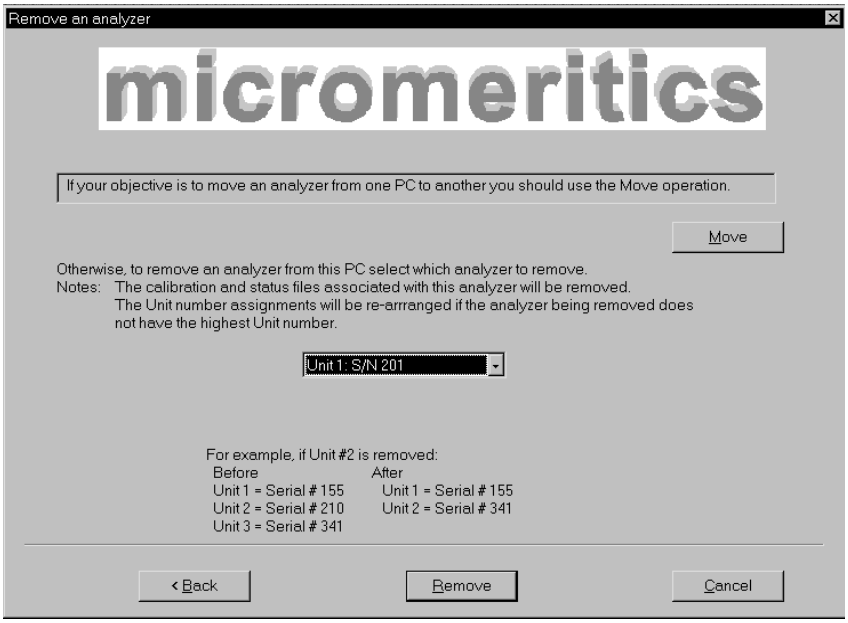
12. In the Step 2 group box, click **Browse** and choose the location of the moved files.

13. Click **Finish**; the files are moved and the setup Welcome dialog is displayed.

Removing an Analyzer

You can remove an analyzer from the computer as follows. When you remove an analyzer, the status files are removed as well.

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions** on page **2-20**.
2. From the Setup dialog, select **Remove an analyzer**, then click **Next**; the Remove an analyzer dialog is displayed.



If your objective is to move an analyzer from one PC to another you should use the Move operation.

Otherwise, to remove an analyzer from this PC select which analyzer to remove.
Notes: The calibration and status files associated with this analyzer will be removed.
The Unit number assignments will be re-arrranged if the analyzer being removed does not have the highest Unit number.

Unit 1: S/N 201

For example, if Unit #2 is removed:

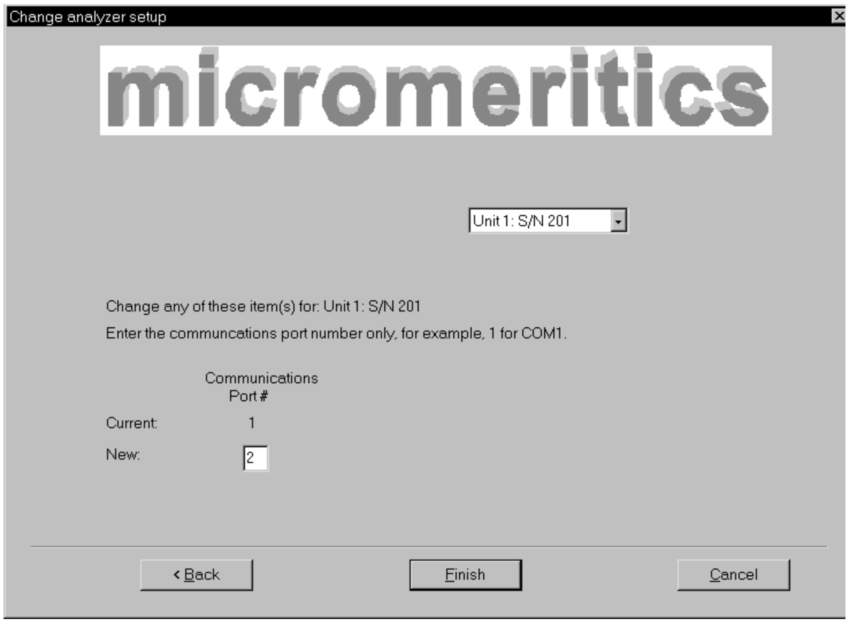
Before	After
Unit 1 = Serial # 155	Unit 1 = Serial # 155
Unit 2 = Serial # 210	Unit 2 = Serial # 341
Unit 3 = Serial # 341	

3. From the drop-down list, choose the serial number of the analyzer you want to remove.
4. Click **Browse**; the analyzer is removed and the Welcome screen is again displayed.
5. Click **Exit** to close the dialog.

Changing an Analyzer Setup

Change the analyzer setup as follows:

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions** on page **2-20**.
2. From the Setup dialog, select **Change analyzer setup**, then click **Next**; the Change analyzer setup dialog is displayed.



Change analyzer setup

Unit 1: S/N 201

Change any of these item(s) for: Unit 1: S/N 201

Enter the communcations port number only, for example, 1 for COM1.

	Communications Port #
Current:	1
New:	2

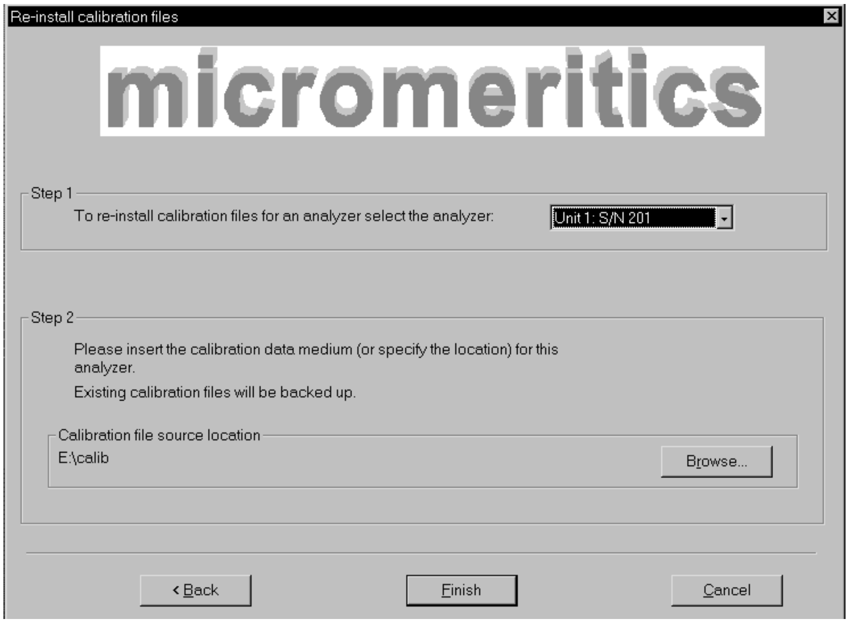
3. From the drop-down list, choose the analyzer you want to change.
4. Enter the new port number in the space provided.
5. Click **Finish**; the change is completed and the Welcome dialog is again displayed.
6. Click **Exit** to close the dialog.

Reinstalling the Calibration Files

You can reinstall the files containing an analyzer's factory calibration data as follows:

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions** on page **2-20**.

2. From the Setup dialog, select **Re-install calibration files** for an analyzer, then click **Next**; the Re-install calibration files dialog is displayed.



Re-install calibration files

Step 1

To re-install calibration files for an analyzer select the analyzer: Unit 1: S/N 201

Step 2

Please insert the calibration data medium (or specify the location) for this analyzer.

Existing calibration files will be backed up.

Calibration file source location

E:\calib

3. Select the analyzer whose calibration files you want to reinstall from the drop-down list. If you have only one analyzer installed, a drop-down list is not included.

4. Insert the CD containing the calibration files; ensure that the CD drive is displayed as the calibration file source location. If not, click **Browse** and choose the CD drive.

5. Click **Finish**; the calibration files are reinstalled and the Welcome dialog is again displayed.

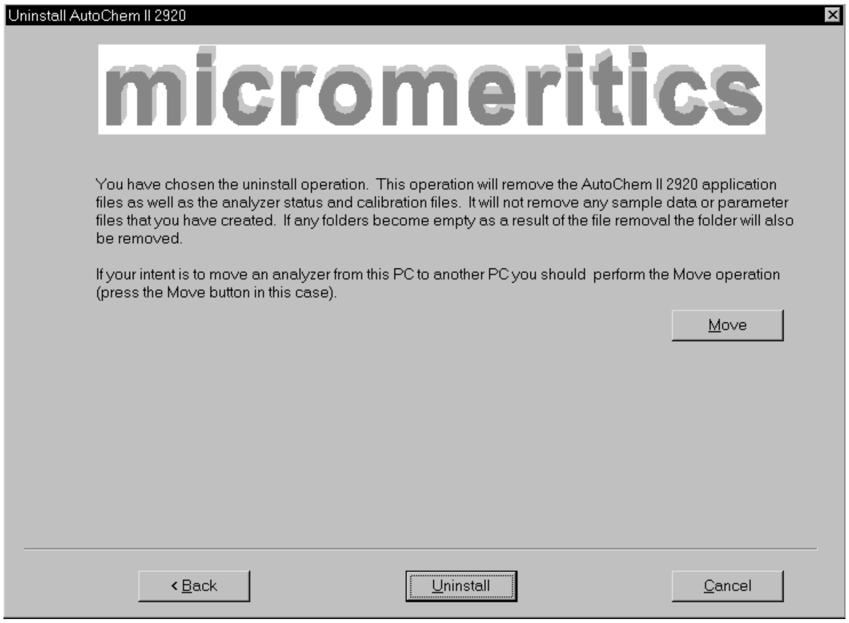
6. Click **Exit** to close the dialog.

Uninstalling the Analysis Program

You can remove the AutoChem II analysis program as follows. When you perform this operation, the application removes the analysis program, status files, analyzer setup files, and resulting empty directories. It does not remove data files.

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions** on page **2-20**.

2. From the Setup dialog, select Uninstall, then click **Next**; the Uninstall dialog is displayed.



You have chosen the uninstall operation. This operation will remove the AutoChem II 2920 application files as well as the analyzer status and calibration files. It will not remove any sample data or parameter files that you have created. If any folders become empty as a result of the file removal the folder will also be removed.

If your intent is to move an analyzer from this PC to another PC you should perform the Move operation (press the Move button in this case).

3. Click **Uninstall**; the Select Uninstall Method dialog is displayed.

Welcome to the Micromeritics Analyzer Setup uninstall program.

You can choose to automatically uninstall this software or to choose exactly which changes are made to your system.

Select the Custom button to select which modifications are to be made during the uninstall. Select the Automatic button for the default uninstall options. Press the Next button to continue.

● Automatic
○ Custom

4. Choose one of the following:

    - Automatic: click **Next**; the system uninstalls the AutoChem II software automatically and the setup Welcome dialog redisplays.
    - Custom: click **Next**; a series of dialogs is displayed, allowing you to choose the files you wish to uninstall. After all files are selected and uninstalled, the setup Welcome dialog redisplays.

5. Click **Exit** to close the Welcome dialog.

Starting the AutoChem Software

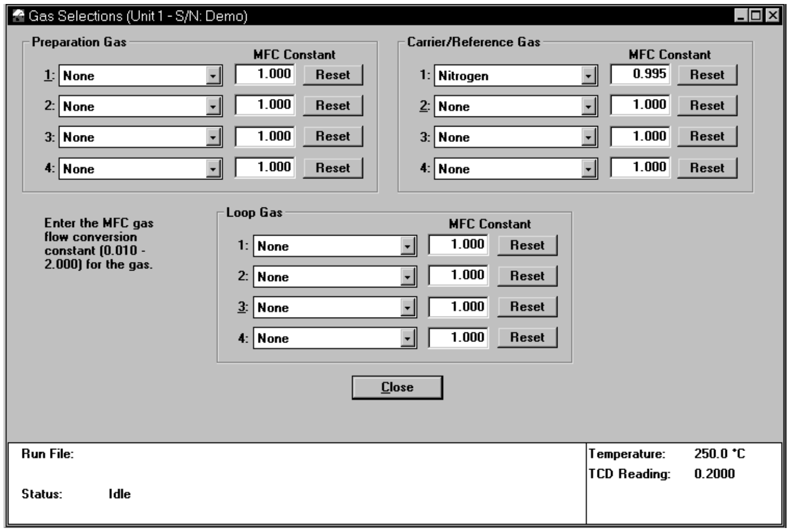
1. Follow standard laboratory procedures to turn on your computer and any attached devices.
2. Select **Start >Programs** from the Windows Task bar.

If you had an icon added to your desktop, simply double-click on the icon.

3. Select the AutoChem 2920 program from the Micromeritics group.

Specifying Gases

After gases are connected to the instrument, select **Unit > Gas Selections** to specify the gases connected to each port.


Gas Selections (Unit 1 - S/N: Demo)

Preparation Gas

	Gas	MFC Constant
1:	None	1.000
2:	None	1.000
3:	None	1.000
4:	None	1.000

Carrier/Reference Gas

	Gas	MFC Constant
1:	Nitrogen	0.995
2:	None	1.000
3:	None	1.000
4:	None	1.000

Enter the MFC gas flow conversion constant (0.010 - 2.000) for the gas.

Loop Gas

	Gas	MFC Constant
1:	None	1.000
2:	None	1.000
3:	None	1.000
4:	None	1.000

Run File:

Status: Idle

Temperature: 250.0 °C

TCD Reading: 0.2000

Select the appropriate gas from the drop-down lists of each port for the Preparation, Carrier/ Reference, and Loop gases.

If a gas or gas mixture you wish to use does not appear in the drop-down list, you can add it to the list using the Gas Defaults option on the Options menu.

1. Close the Gas Selections dialog.
2. Select **Gas Defaults** from the Options menu.
3. Add the gas or mixture to the Gas Defaults list. (Refer to **Gas Defaults** on page **8-13** for additional information.)
4. Reopen the Gas Selections dialog and select the new gas from the list.

If you change a gas connection, it is essential that you update the Gas Selections window to reflect the current gas connections, in order to avoid creating dangerous combinations of gases in the analyzer.

Purging the System

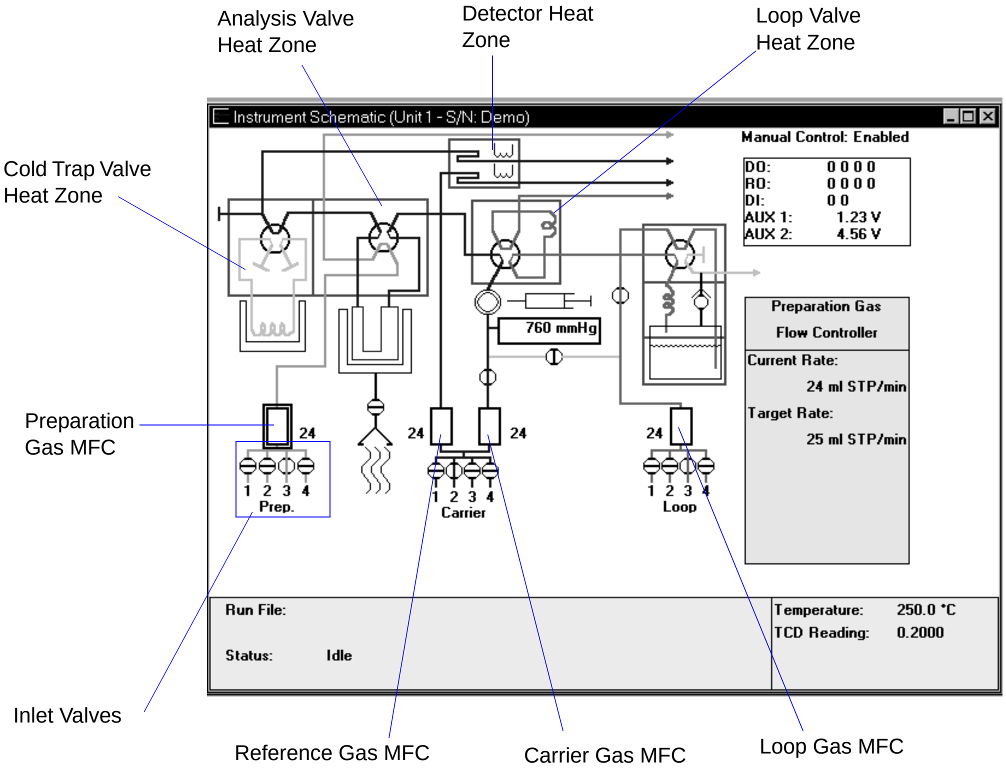
Purging Air

The instrument must be purged of air after installation or after you change the instrument hardware. For example, you must purge the instrument and gas supply lines after changing a gas bottle (that line only must be purged) or changing the loop.

Purge one line at a time. It is the operator’s responsibility to make sure that dangerous combinations of gases are not created while using Manual Control. It may be necessary to flow an inert gas through the system between flows of incompatible gases.

Purge the analyzer’s internal gas lines of air as follows:

1. Select **Unit > Show Instrument Schematic**, then **Enable Manual Control**. (Refer to **Enable Manual Control** on page **6-12** for detailed information on manually controlling the system. The following instructions are sufficient for you to purge the instrument.)



Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Preparation Gas Flow Controller
Current Rate: 24 ml STP/min
Target Rate: 25 ml STP/min

Run File:
Status: Idle

Temperature: 250.0 °C
TCD Reading: 0.2000

Figure 2-8. The Instrument Schematic

2. Set the Cold Trap valve, Analysis valve, Loop valve, and Detector heat zone temperatures to 110 °C.
    a. Position the cursor over the desired valve’s heat zone (the cursor will change to a thermometer) and right-click.
    b. Select **Set temperatures**, then enter 110.
    c. Click **OK**.
3. Wait until the temperature reaches the 110 °C target. (Click on the heat zone to display both the target and current temperatures in the box on the right side of the schematic.)

When you have clicked on the heat zone, the cursor appears as a thermometer. If you have accidentally clicked on the valve, move the cursor slightly and click again.

4. First, purge all air from the preparation gas line.
    a. Position the cursor over the preparation gas Mass Flow Controller (MFC) and right-click.
    b. Select Set flow rate and enter 50.
    c. Click **OK**.
    d. Position the cursor over the four preparation gas valves and right-click.
    e. Open the valve that corresponds to the gas you wish to flow by clicking the appropriate Flow Gas [n] menu choice.
    f. Allow the gas to flow for approximately 20 minutes or until you are certain the lines have been purged of air.
5. Repeat step 4 for each valve to which a gas bottle is connected.
6. Repeat the process (steps 4 and 5) for the carrier and loop gas flows.

Purging Gases

When you change the gas that is flowing through the instrument, or when you change the type of gas connected to a port, you should purge the instrument of the previous gas by flowing the new gas.

Use the instructions for **Purging Air from the System** to purge the previous gas. Be sure to change the Gas Selections dialog to reflect the change in gases. (See **Specifying Gases** on page **2-31** for more information.)

Changing the Gas Flow During an Analysis

When you change gases during an analysis, you may wish to allow the new gas to flow for a period of time before creating conditions that cause the experiment to begin (such as elevating the temperature). In some cases, you may wish to purge one gas (by flowing an inert gas for a period of time) before starting to flow another gas. This may be useful for avoiding undesirable combinations of gases within the analyzer.

To flow an inert gas between incompatible gases, you can insert a Change Gas Flows step (in which an inert gas is flowed for a period of time) and a Wait step (to wait for the inert gas to purge the analyzer) between other steps that involve incompatible gases.

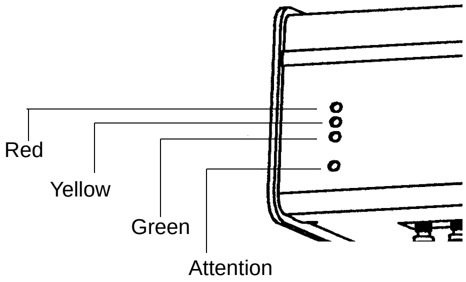
When gas flows are changed while the analyzer is recording data, the gas flow is briefly disturbed. This may result in a brief period of noise or other visible disturbances on the peak data. Either disregard the disturbance or insert a *Wait for stable baseline* step immediately after changing gas flows.

3. USER INTERFACE

This chapter provides a description and the functions of the front panel LEDs (lights), and general instructions for using the software.

Front Panel Indicators

There are four indicator lights (Light-Emitting Diodes—LEDs) on the upper left of the analyzer's front panel.



The top three LEDs show the state of the instrument's communication with the computer.

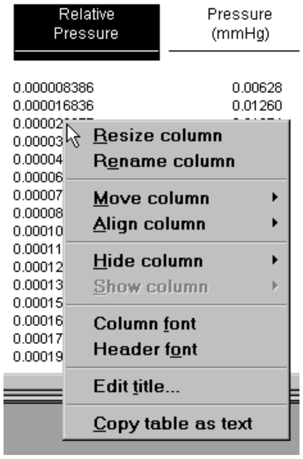
Red (Fault)	There is no communication with the computer (For example, if the 2920 software was exited). When in this state, the Attention! LED (which is green) is also illuminated.
Yellow	Five minutes have elapsed with no communications.
Green (OK)	Normal communications.
Attention!	Blinks to alert the operator that attention is required. For example, if a Wait step requiring action by the operator was included in the list of experiment steps, the LED blinks and the appropriate message appears on the computer screen. After the operator performs the requested action and clicks **OK**, the LED ceases to blink.

Using the Software

The AutoChem II 2920 analyzer operates in the Windows environment and requires familiarity with standard Windows operations such as using the mouse, menus, and dialog boxes. This manual provides brief instructions for standard operations; however, you may need to refer to your Windows documentation or the Windows tutorial to clarify functions which are specific to Windows.

Shortcut Menus

Shortcut menus (sometimes referred to as *context-sensitive* menus or *pop-up* menus) are available for certain components on the instrument schematic when in manual mode, and for onscreen graphs and tabular reports. These menus are accessed by selecting the item for which you wish to display its menu and clicking the right mouse button. For example, right-click in a column of an onscreen report and the following menu is displayed.



Shortcut Keys

Shortcut keys can be used to activate some menu commands. Shortcut keys or key combinations (if assigned) are listed to the right of the menu item. Instead of opening the menu and choosing the command, simply press the key combination. For example, to open a sample information file, press **F2**; the Open Sample Information dialog is displayed.

You can also use shortcut keys to access a menu or any function that contains an underlined letter by pressing **Alt** plus the underlined letter in the command. For example, to access the File menu, press **Alt**, then **F**.

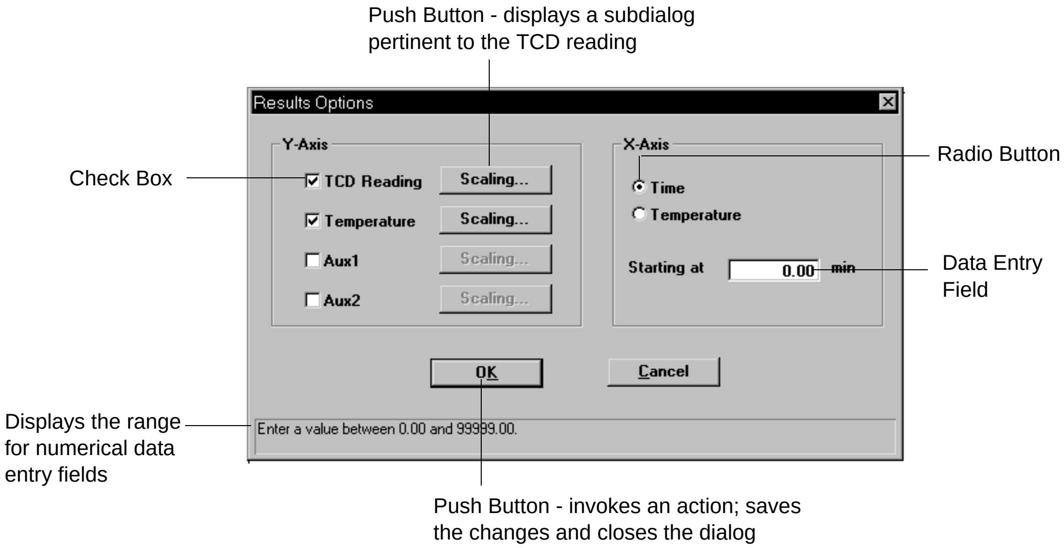
Table 3-1 provides a list of the keys available in the AutoChem 2920 analysis program.

Table 3-1. Shortcut Keys

Key(s)	Function
F2	Open a sample information file Clear the selected field (when using the Select Dates dialog)
F3	Open an analysis conditions file Insert the current date (when using the Select Dates dialog)
F4	Open a report options file Display a calendar (when using the Select Dates dialog)
F6	Tile windows
F7	Cascade windows
F8	Start report
F9	Close all open report windows
Alt + F4	Exit the analysis program
Shift + F2	List sample information files
Shift + F3	List analysis conditions files
Shift + F4	List report options files
Shift + F9	Display action(s) available for selected component on the instrument schematic when manual control is enabled

Dialog Boxes and Subdialog Boxes

Dialog Boxes and Subdialog Boxes are provided for you to enter information to be used by the software. They are sometimes called “windows” and sometimes called “screens.” Dialog boxes are displayed when an item followed by an ellipsis (...) is selected. Subdialog boxes are displayed when a push button on a dialog box is selected. Both types of boxes may contain one or more of the following:



Enter a value between 0.00 and 99999.00.

Push Button - displays a subdialog pertinent to the TCD reading

Check Box

Radio Button

Data Entry Field

Displays the range for numerical data entry fields

Push Button - invokes an action; saves the changes and closes the dialog

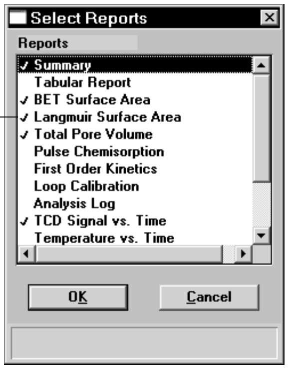
Data Entry Field	The entry in these fields may be numeric (numbers only) or alphanumeric (numbers, letters, or printable characters). If an invalid entry is made, an error message is displayed. When a numerical data entry field is selected, its valid range is displayed in an information bar across the bottom of the dialog.
Radio Button	Small, circular radio buttons are sometimes provided when you must choose only one item from a group of options.
Check Box	Check boxes allow you to choose multiple options from a group of options.
Push Button	Several types of push buttons are found on the 2920 dialogs. When you click a push button, a subdialog appears enabling you to perform a function or enter data pertinent to the subject matter. Other push buttons, such as **OK** and **Cancel**, execute a single command.

Push Button *(continued)*

A push button that you will find frequently on dialogs is **Replace.** This push button allows you to replace the contents of the current file with those of another file. For example, if you are creating an analysis conditions file, you can save time by clicking **Replace**. A dialog opens allowing you to choose the file containing the values you wish to use. After you close the dialog, your new file contains the same values as the file from which they were copied. The old file remains unchanged, even if you edit the values in your new file.

List

A list contains selections from which you may choose one or more items. To choose an item from the list, simply double-click on the desired selection, or highlight the item and press **Spacebar**. A check mark precedes the item when it is selected. Items are deselected in the same manner.

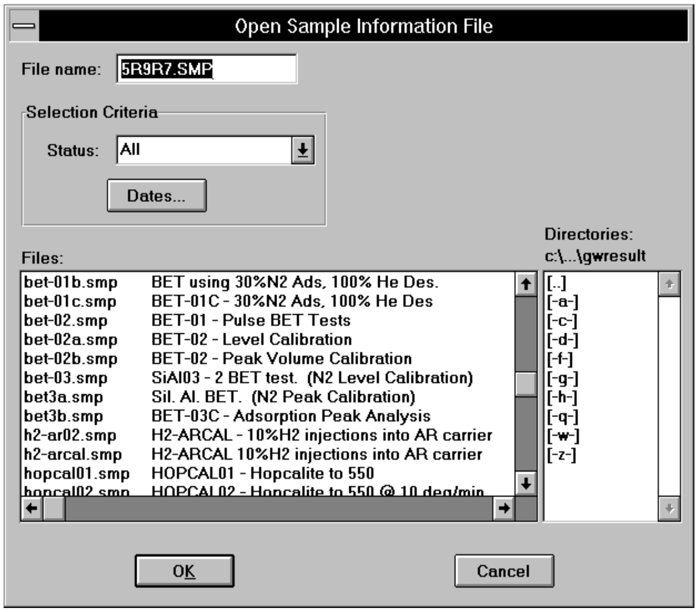


✓ Summary
Tabular Report
✓ BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption
First Order Kinetics
Loop Calibration
Analysis Log
✓ TCD Signal vs. Time
Temperature vs. Time

Indicates report is selected

Selecting Files

Sample information is stored in files and saved under file names. Certain dialogs contain a Files list window that displays a list of files available for the current operation. For example, the Open Sample Information dialog:



File name: 5R9R7.SMP

Status: All

Files:

bet-01b.smp	BET using 30%N2 Ads, 100% He Des.
bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier
hopcal01.smp	HOPCAL01 - Hopcalite to 550
hopcal02.smp	HOPCAL02 - Hopcalite to 550 @ 10 deg/min

Directories:
c:\...\gwresult
[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

From the list of files in the Files list window, move the mouse pointer to the file you wish to open and double-click. If you do not see the desired file, it may be in another directory. Go to the Directories list box and choose the correct drive and/or directory.

You can limit the list of files displayed in the Files list box by choosing one or more of the following:

- Select a file status from the Status drop-down list. Table 3-2 describes each file status.

Table 3-2. File Status and Description

Status	Description
All	All sample information files in the specified directory and within the specified range of dates.
Analyzing	Sample information files that are currently being used for analysis.
Complete	Sample information files that were used in an analysis that has been completed.
No Analysis	Sample information files that have not been used in an analysis.

- Enter a range of dates. Click **Dates**; the Select Dates dialog box is displayed.

  Select **Show Date Range** to enable the **From** and **To** fields so that you can enter beginning and ending dates. You can also double-click in each field to display a calendar to set a date. The range of dates remains the default until the range is changed or **Show All Dates** is selected.

  For convenience, the following shortcut keys are available when the Select Dates dialog is displayed:

F2	Clears the field
F3	Inserts the current date
F4	Displays a calendar from which you may select a date

> **You can change the date format by using the Date and Time function on the Windows Control Panel.**

- Use wildcard characters in the path name you enter in the File name field. Wildcard characters such as * and ? can be used to filter file names. For example, you can limit the list of files displayed to those containing TPD as the first three characters of the file name by entering TPD*.smp.
- Navigate to a different directory. The current directory is displayed just above the **Directories** list box. You can change directories by double-clicking a directory in the **Directories** list box, double-clicking **[..]** to move up one level, or by entering the desired directory in the **File Name** field. For example, enter C:\2920files\sample*.smp to display sample files in the **2920files\sample** directory on your local drive.

File Name Conventions

For sample information files, a default file name (the next available sequence number) and a default extension are displayed when the Files list box is opened. For analysis conditions and report options, only a default extension is displayed with an * (*.ANC, for example). The following table shows the file name extensions for the AutoChem II 2920 analysis program.

Table 3-3. Default File Name Extensions

File Type	Extension
Sample Information	**SMP**
Analysis Conditions	**ANC**
Report Options	**RPO**
Export to disk (ASCII)	**EXP**
Export to disk (Grams 32)	**CGM**
Report to disk	**RPT**
List to disk	**LST**
Calibration	**CAL**
Vapor Generator (option) calibration	**VCL**
The following types are available for reports saved from the Report window:	
Spreadsheet	**XLS**
ASCII	**TXT**
Report	**REP**

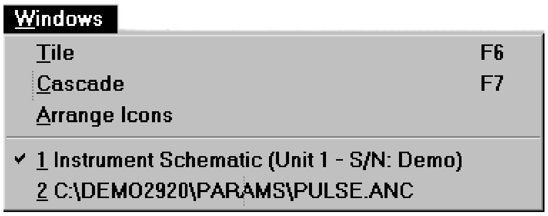
Menu Structure

Main Menu Bar

All functions for the AutoChem II 2920 are accessed from the Main menu bar. Brief descriptions of each menu are provided below; refer to the chapter given in parentheses for a detailed description of the options contained on that menu.

File	Allows you to manage sample and parameter files. (Chapter 5, page **5-1**)
Unit <n>	Enables you to perform analyses. A unit menu is displayed for each attached unit. (Chapter 6, page **6-1**)
Reports	Enables you to generate and close reports. (Chapter 7, page **7-1**)
Options	Allows you to edit sample defaults, specify your system configuration, and select data presentation formats. (Chapter 8, page **8-1**)
Windows	Enables you to arrange the windows and icons on your screen. It also displays the names of all open windows. (this chapter, page **3-10**)
Help	Displays help information. (this chapter, page **3-10**)

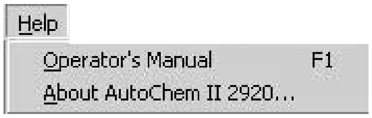
Windows Menu



Tile	Resizes all open windows and arranges them side by side so that the contents of all open windows are visible.
Cascade	Resizes all open windows and arranges them in a stacked manner. The active window is positioned on top of the stack. Each window's title remains visible, making it easy to select other windows.
Arrange icons	Arranges all minimized icons in an orderly manner.

The Windows menu also displays all open files and windows. The current file is preceded with a check mark.

Help Menu



Operator's Manual	Displays the AutoChem II 2920 Operator's Manual in PDF format.
About Win2920	Displays information about the AutoChem II 2920 analyzer.

4. GENERAL OPERATING PROCEDURES

This chapter describes many of the procedures for using the analyzer and the software for analyses. Maintenance procedures are described in Chapter 9. Procedures which generally occur when the analyzer is first installed are included in Chapter 2.

Specifying Sample Defaults

Sample information files include the information required by the AutoChem II 2920 to perform analyses and collect data. The analysis program automatically generates sample information file names and assigns default values. Sample defaults allow you to customize your sample file as well as the file name sequence. It is best to use defaults for materials that you most commonly analyze. You can always edit the values when the sample file is created.

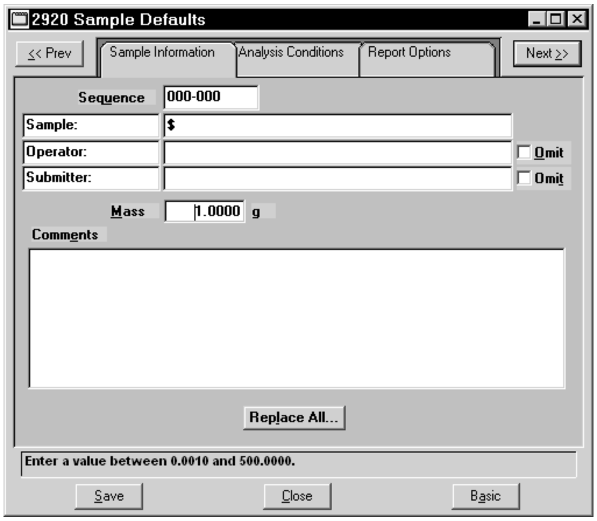
The sample information presentation can be presented in a basic, advanced, or restricted format. Sample defaults can be specified in the basic and advanced formats only; you cannot specify sample defaults using the restricted format.

If desired, you can define parameter files before entering sample defaults (refer to **Defining Parameter Files** on page **4-4**).

Advanced Format

Select Option > Option Presentation and ensure that Advanced is selected as the format.

1. Select **Options** > **Sample defaults**; the 2920 Sample Defaults dialog is displayed



Sequence 000-000
Sample: $
Operator: ☐ Omit
Submitter: ☐ Omit
Mass 1.0000 g

Enter a value between 0.0010 and 500.0000.

2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File** > **Open** > **Sample information**.

3. In the field on the right of the **Sample** line, enter a format for the identification. Be sure to include the **$** symbol if you want the sample file number included as part of the identification.

You can also edit the word Sample. For example, you may prefer to use *Test* or *Material*.

4. Edit the **Operator** and **Submitter** lines as desired. Or, you may have either (or both) of these limes omitted entirely by selecting **Omit**.

5. In the **Mass** field, enter an approximate value for the mass of sample you typically use in your analysis. A more accurate value can be entered in the sample file itself.

6. Click the **Analysis Conditions** tab and enter appropriate information for your most commonly analyzed sample material. Remember, you can always edit this information in the sample file when it is created.

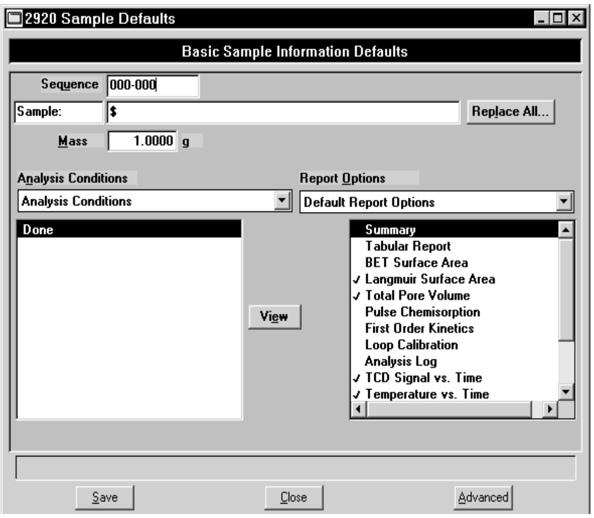
7. Click the **Report Options** tab and choose the desired reports. Again, this selection can be edited in the sample file when it is created.

8. Click **Save**, then **Close**.

Basic Format

Select Option > Option Presentation and ensure that Basic is selected as the format.

1. Select **Options** > **Sample defaults**; the Basic Sample Information Defaults dialog is displayed.



Sequence 000-000

Sample: $

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

Summary
Tabular Report
BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption
First Order Kinetics
Loop Calibration
Analysis Log
✓ TCD Signal vs. Time
✓ Temperature vs. Time

2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File** > **Open** > **Sample information**.

3. In the field on the right of the **Sample** line, enter a format for the identification. Be sure to include the $ symbol if you want the sample file name (sequence number) included as part of the identification.

You can also edit the word Sample. For example, you may prefer to use *Test* or *Material*.

4. In the **Mass** field, enter an approximate value for the mass of sample you typically use in your analysis. A more accurate value can be entered in the sample file itself.

5. Select the down arrow to the right of the **Analysis Conditions** field to choose a default parameter file. The steps included in this file will display in the window below the field. You may view the details of these steps by selecting **View**.

6. Select the down arrow to the right of the **Report Options** field to choose a default parameter file. The reports chosen (preceded with a check mark) for the selected file will display in the list of reports below the field.

7. Click **Save**, then **Close**.

Defining Parameter Files

Analysis Conditions and Report Options files can exist as part of the sample information file or as an individual parameter file. Having these files exist independently allows you to use them over and over again. For example; unless you expect each analysis to use unique sets of analysis conditions and report options, you can create a group of standard parameter files containing the values you expect to use most frequently. The contents of these files can be copied and imported into multiple sample information files, providing speed and consistency. After these values have been copied and imported into the sample information file, they can be edited in any way that you wish. The original parameter file remains intact and ready for its next use.

This makes it possible for one person (such as a laboratory manager or senior chemist) to create standard parameter sets that can be used easily by others (such as laboratory technicians).

Several predefined parameter files (located in the PARAMS directory) are included with the AutoChem II analysis program. These files are examples only and the gases specified in these files may not match the gases connected to your analyzer. If you wish to use one of these files as a starting point, simply create a new file and select **Replace**. A dialog is displayed so that you can select the existing parameter file. After the values from this file are copied into the current file, you can edit them in any way desired.

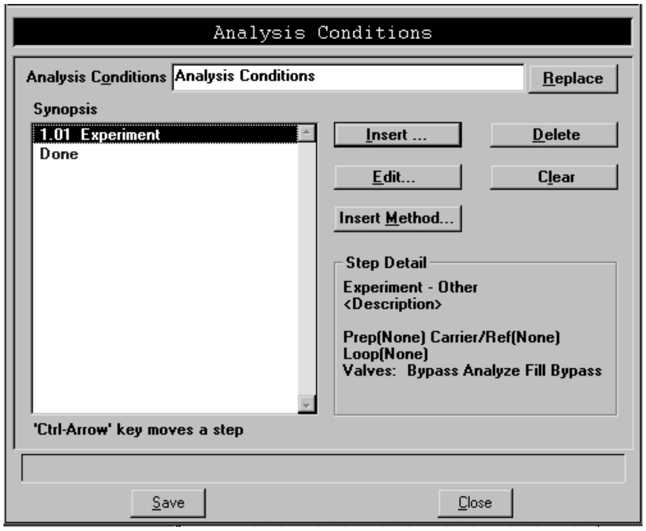
Make sure you save these files to the directory specified as the Parameter File directory (refer to Chapter 8 for additional information). If you do not save these files to the parameter file directory, they will not display in the drop-down lists on the Basic sample file editor.

Parameter files can also be created from the Advanced Sample information window using the **Save as** function on the File menu. Simply select **Save As > Report Options** or **Analysis Conditions** — whichever portion of the sample information file you wish to save.

Analysis Conditions

Analysis conditions specify the data used to guide an analysis.

1. Select **File** > **Open** > **Analysis conditions**; the Open Analysis Conditions File dialog is displayed.
2. Enter a name for the file (up to eight characters) in the File name field.
3. Click **OK**, then **Yes** to create the file; the Analysis Conditions dialog is displayed.



Analysis Conditions: Analysis Conditions

1.01 Experiment
Done

Experiment - Other
<Description>

Prep(None) Carrier/Ref(None)
Loop(None)
Valves: Bypass Analyze Fill Bypass

'Ctrl-Arrow' key moves a step

Refer to Analysis Conditions on page 5-10 for detailed information on the fields contained on this dialog.

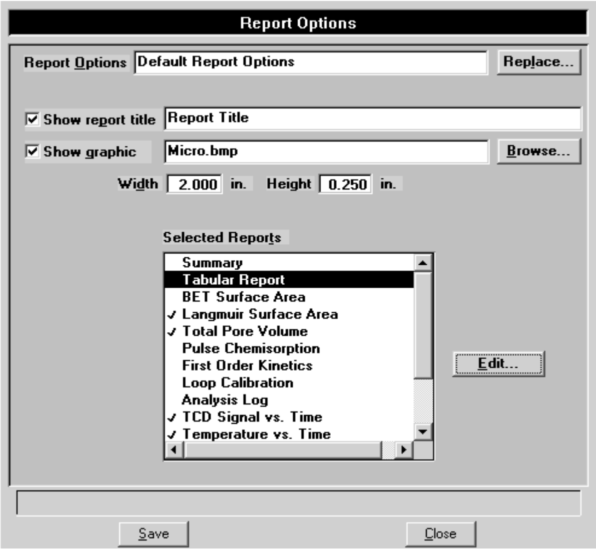
4. Enter a description in the **Analysis Conditions** field. Use a description that will help you identify the conditions specified. You can use up to 42 characters.
5. Click **Insert** to insert steps for the analysis. Each step you insert has an associated dialog, which contains the parameters for that step. You can insert up to 1000 steps in the Synopsis.
6. Choose **Experiment**, or a calibration option, then click **OK**; an associated dialog is displayed.
7. Define the experiment (or calibration) as desired, then click **OK**; the experiment (or calibration) will display in the synopsis window.
8. Click **Save**, then **Close**.

Report Options

1. Select **File** > **Open** > **Report options** ; the Open Report Options File dialog is displayed.

2. Enter a name for the file (up to eight characters) in the **File name** field.

3. Click **OK**, then **Yes** to create the file; the Report Options dialog is displayed.



Report Options: Default Report Options

☑ Show report title: Report Title

☑ Show graphic: Micro.bmp

Width 2.000 in. Height 0.250 in.

Selected Reports

Summary
Tabular Report
BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption
First Order Kinetics
Loop Calibration
Analysis Log
✓ TCD Signal vs. Time
✓ Temperature vs. Time

Refer to Report Options on page 5-39 for detailed information on the fields contained on this dialog.

4. Enter a description (up to 42 characters) in the **Report Options** field. Use a description that will help you identify the types of reports selected.

5. Select **Show report title** and enter the title you wish to appear at the top of the report. Or deselect this option if you do not want a title on your report.

If your company logo exists as a bitmap (bmp) or enhanced metafile (emf), you can have it display in the report header, select Show graphic. Click Browse to select the file; use the Width and Height fields to specify a size.

6. Select (or deselect) the desired report by highlighting the report and double-clicking. Alternatively, you can press the **Spacebar** after the report is highlighted. A report is selected when it is preceded with a check mark.

7. Edit reports as desired.

8. Click **Save**, then **Close**.

Creating Sample Information Files

Every analysis requires a sample information file. The file consists of information groups which, collectively, identify the sample, guide the analysis and specify the data reduction.

A sample file may be created in any of the three different formats:

- **Advanced**
  Provides complete access to all parts of the sample file, allowing you to quickly edit parameters as required.
- **Basic**
  Presents a single window in which you select predefined parameter files for your analysis. You can also switch to the Advanced format to edit parameters if desired.
- **Restricted**
  Presents a single window in which you select predefined parameter files (identical to the Basic format). You cannot, however, switch to the Advanced format for editing purposes. This format is password-protected.

Select **Options > Option presentation** to choose the format you wish to use.

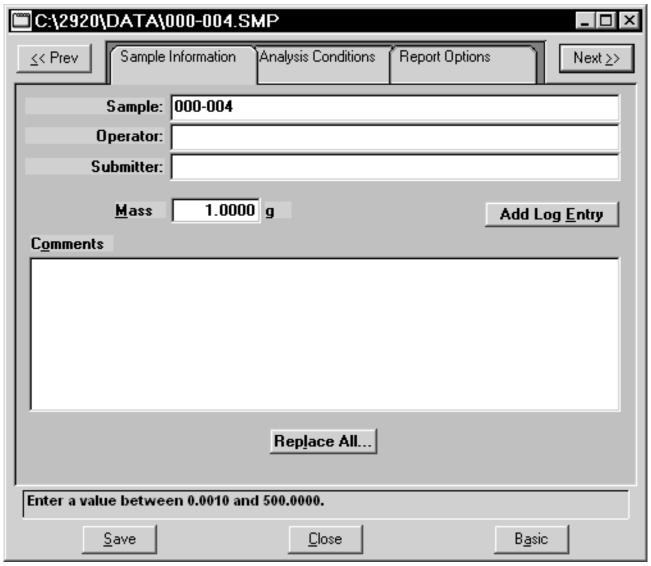
Sample information files can be created in advance of the analysis or they can be created at the time of analysis, whichever is convenient for you.

Advanced Format

The Advanced format presents the sample information editor in a manner similar to that of an index card file. Simply click on the appropriate tab and enter the required information.

1. Select **File > Open > Sample information**; the Open Sample Information dialog is displayed.
2. Accept the next sequenced file number or enter a new name in the **File name** field.

3. Click **OK**, then **Yes** to create the file; the Advanced sample dialog is displayed.



C:\2920\DATA\000-004.SMP

Sample: 000-004

Operator:

Submitter:

Mass 1.0000 g

Comments

Enter a value between 0.0010 and 500.0000.

The values contained on the dialog are those specified in Sample Defaults. You can accept these defaults to create the file or continue with the subsequent steps to edit them.

4. In the **Sample** field, enter a description or add to the existing one.
5. Edit the **Operator** and **Submitter** fields if desired.
6. Use the **Comments** field if desired for any comments about the sample or analysis conditions. Anything you enter in this field will display in the header of some reports.
7. Click the **Analysis Options** tab and define analysis conditions. Refer to **Defining Parameter Files** for instructions on completing the dialog.
8. Click the **Report Options** tab and specify desired report options. Refer to **Defining Parameter Files** for instructions on completing the dialog.
9. Click **Save**, then **Close**.

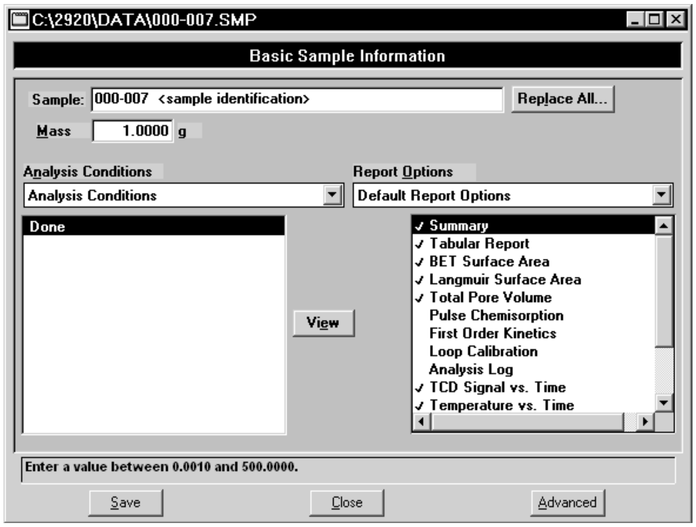
Basic Format

The Basic format allows you to create a sample file by choosing from predefined parameter files. The values in these files can be edited when needed by switching to the Advanced format.

1. Select **File** > **Open** > **Sample information**; the Open Sample Information dialog is displayed.

2. Accept the next sequenced file number or enter a new name in the File name field.

3. Click **OK**, then **Yes** to create the file; the Basic Sample Information dialog is displayed.



C:\2920\DATA\000-007.SMP

Basic Sample Information

Sample: 000-007 <sample identification>

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

✓ Summary
✓ Tabular Report
✓ BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption
First Order Kinetics
Loop Calibration
Analysis Log
✓ TCD Signal vs. Time
✓ Temperature vs. Time

Enter a value between 0.0010 and 500.0000.

The values contained on the dialog are those specified in Sample Defaults. You can accept these defaults to create the file or continue with the subsequent steps to edit them.

4. In the **Sample** field, enter a description or add to the existing one. You can use up to 42 characters.

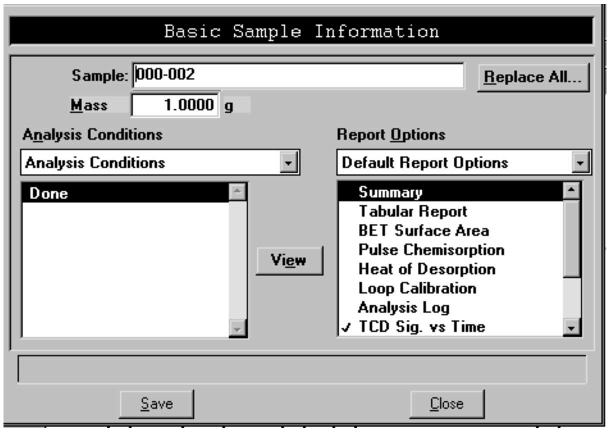
5. Click the down arrow to the right of the **Analysis Conditions** field and choose the file containing the appropriate analysis conditions. After you choose a file, its steps are displayed in the window below the **Analysis Conditions** field. You can view the details of the steps by highlighting the step and clicking **View**.

6. Click the down arrow to the right of the Report Options field and choose an appropriate file. You can select additional (or deselect current) reports by double-clicking on the report(s).

7. Click **Save**, then **Close**.

Restricted Format

Sample information files are created using the Restricted format in the same manner as with the Basic format. The name displayed in the title bar even displays as Basic Sample Information. However, the dialog for this format does not allow you to switch to the Advanced format to edit parameters. Certain menu functions are also disabled with this format.


Basic Sample Information

Sample: 000-002

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

Summary
Tabular Report
BET Surface Area
Pulse Chemisorption
Heat of Desorption
Loop Calibration
Analysis Log
✓ TCD Sig. vs Time

Preparing for an Analysis

Depending upon the standard procedures at your laboratory, some preparation activities may be performed at the beginning of the day or at the beginning of a shift. Regardless of when you choose to perform them, the following activities must be performed before beginning an analysis:

- If the analyzer has been turned off, allow it to warm up for at least 3 hours after turning on. (Refer to **Turning on the Analyzer** on page **2-17**.)
- Check all gas regulator pressure settings. (Refer to **Gas Connections** on page **2-3**.)
- Ensure that there is an adequate amount of cryogen in the dewar. The cryogen level should be approximately 25 mm (1 in.) from the top. (This chapter, page **4-26**.)
- Make sure the sample thermocouple is properly positioned. (This chapter, page **4-11**)
- Update the current ambient temperature and pressure. (Refer to **Environmental Defaults** on page **8-16**.)

Selecting the Thermocouple Position

Before beginning an analysis, the thermocouple must be attached to the analyzer. There are three possible configurations for the thermocouple:

- inside the sample tube
- inside the sample tube surrounded by a quartz sheath
- outside the sample tube

Best accuracy is obtained when the bare thermocouple is placed inside the sample tube. In some cases, however, one or more of the gases used during the experiment may react with the Inconel™ material of which the thermocouple is made. A quartz sheath is provided to protect the thermocouple when the gases or sample used in the experiment may damage it. In this case, the sheath is placed around the thermocouple and the thermocouple is positioned inside the sample tube. When placed inside the tube, you can adjust the thermocouple so that the end is located in the sample or just above it; always try to position the end of the thermocouple as close to the furnace thermocouple as possible.

Positioning the thermocouple along the exterior of the sample tube is less accurate than the other configurations. If the thermocouple is placed outside the sample tube, make sure it is clamped against the sample tube, and that its end does not extend below the point where the sample tube begins to taper.

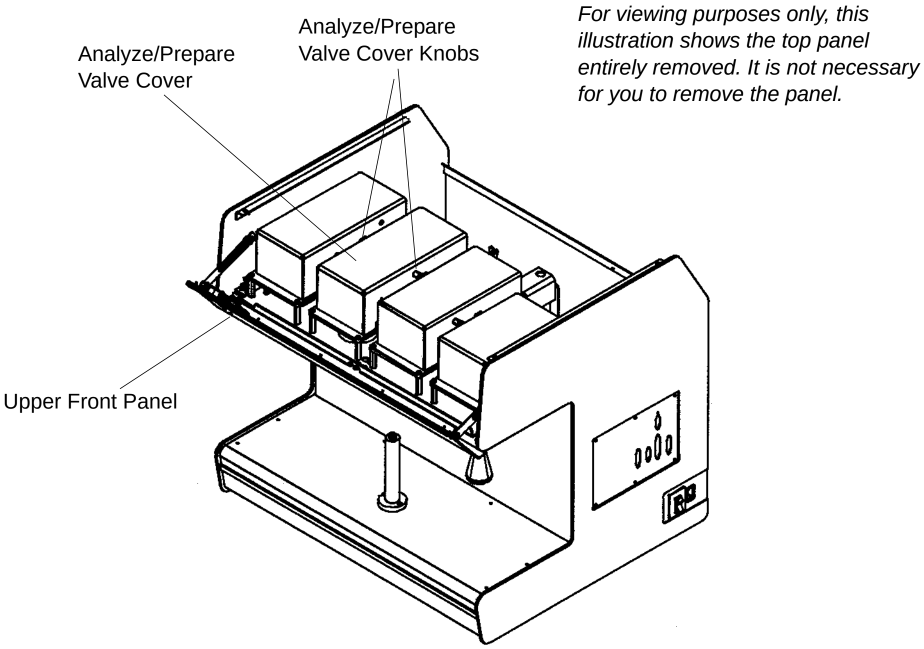
Changing the Position of the Thermocouple

1. Turn the analyzer’s power switch off, then disconnect the analyzer from its power source.

2. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle. Then, using the handle, open the top panel. The top panel is hinged to allow easy access to its components; it is not necessary to remove the top panel.

   Pull down the upper front panel.

3. The Analyze/Prepare valve cover is held in place by two knobs or plungers. Remove the valve cover by pulling the plungers outward until the valve cover is released. Pull the cover up and out.



For viewing purposes only, this illustration shows the top panel entirely removed. It is not necessary for you to remove the panel.

4. Remove the insulation that surrounds the Analyze/Prepare valve.

The sample thermocouple and nearby components may be hot. Make sure you allow the sample thermocouple to cool before removing it. Use the cotton gloves provided with the analyzer to protect your hands. If the zone is not near room temperature, loosening or tightening the fittings may cause damage to components of the instrument.

5. Use an open-ended wrench to loosen the sample thermocouple fitting.
6. Loosen the fitting for the stainless steel plug in the alternate thermocouple opening and remove the plug.
7. Remove the fitting that retains the sample thermocouple.
8. Move the sample thermocouple from its current position to the new position.

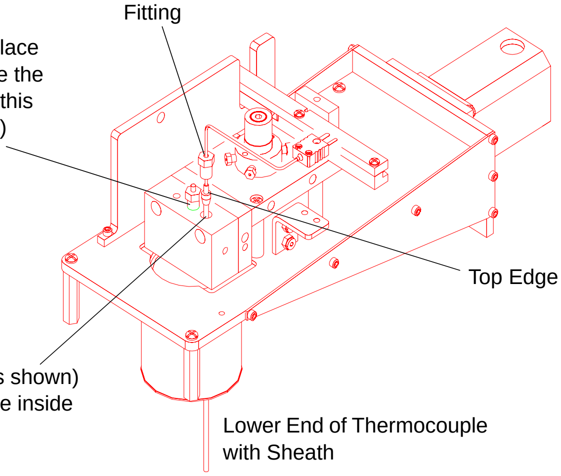
   If you are using the quartz sheath, perform the following before proceeding to the next step. Remember, the sheath can only be used when the thermocouple is being placed inside the sample tube.

   a. Remove the ferrule from the thermocouple, straighten the thermocouple as much as possible.

   b. Insert the thermocouple into the sheath.

   c. Place a larger ferrule around the sheath.

The sheath is very fragile and can be easily broken. Because the sheath is made of quartz, you must use the cotton gloves when handling it. (Oils from your fingers may lower the quartz' melting point.)



Use the left opening to place the thermocouple outside the tube. (In this illustration, this opening contains a plug.)

Use the right opening (as shown) to place the thermocouple inside the tube.

Figure 4-1. Thermocouple Placed Inside the Tube (Using a Sheath)

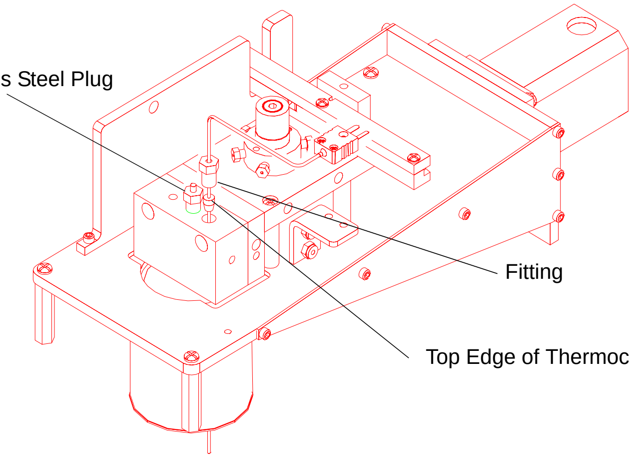


Figure 4-2. Thermocouple Placed Inside the Sample Tube (Without Sheath)

9. Place the stainless steel plug in the unused thermocouple opening and tighten the fitting. Reinstall the sample thermocouple retaining screw.

10. Adjust the exposed portion of the sample thermocouple to the appropriate length. When adjusting the length of the exposed portion, consider the following:

    - The thermocouple's end should be aligned as closely as possible with the furnace thermocouple. (The furnace thermocouple's location is shown in the following sections.)
    - The thermocouple's end should never be placed outside the painted (black), recessed area inside the furnace.
    - If the thermocouple is positioned inside the tube, its end should generally be located approximately 2-3 mm (1/8 in.) above the sample surface. In some cases, you may allow the thermocouple to extend into the sample.
    - If the thermocouple is positioned outside the tube, its end should not extend below the point where the sample tube begins to taper.

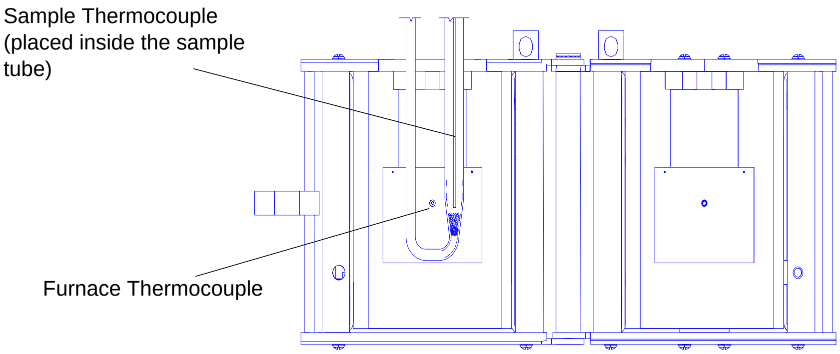


Figure 4-3. The Furnace Thermocouple

11. Tighten the thermocouple fitting.
12. Place the insulation in its original position. Reinstall the Analyze/Prepare valve cover.
13. Return the upper front panel and the top panel to their original closed positions.
14. Turn the instrument's power switch on.

Using the Thermocouple Clamp

If the thermocouple is placed on the outside of the sample tube, you must clamp it to the tube as shown in the following illustrations.

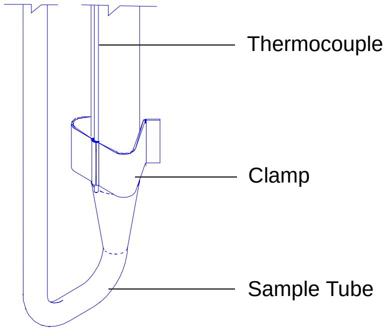


Figure 4-4. The Thermocouple Clamp

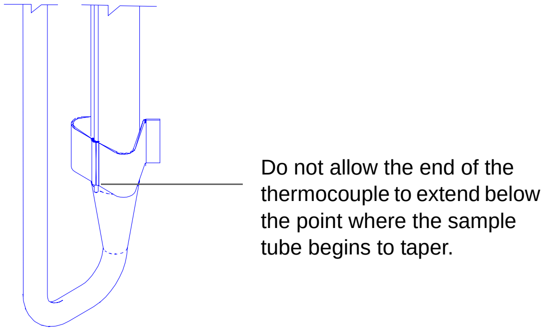


Figure 4-5. Vertical Alignment of the Thermocouple End

Cleaning Sample Tubes

The sample tube should be cleaned (using an ultrasonic cleaning unit) and adequately dried before use to obtain accurate analysis results. You will need the following materials for cleaning sample tubes:

- Ultrasonic cleaning unit
- Alconox® or other suitable detergent
- Rubber gloves or lint-free cloth
- Isopropyl alcohol (IPA) is preferred for cleaning; acetone or deionized water may be used if IPA is unavailable
- Nitrogen or helium
- Fume hood
- Sample tube brush

Clean the tubes in an ultrasonic cleaning unit as follows:

1. Ensure that the ultrasonic cleaning unit is clean.
2. Using a ratio of 5 grams of Alconox (or equivalent detergent) per 500 mL of warm water, fill the bowl of the ultrasonic unit with enough water to cover the entire sample tube.
3. Ensure that the detergent is dissolved before placing the sample tube into the water. If too much detergent is used, it may be difficult to rinse from the sample tube.

Always wear gloves when handling quartz sample tubes. The salts from bare hands lower the normal melting point of quartz (approximately 1300 ºC) to a much lower temperature. Oils from your skin may also destroy the quartz when the tube is heated.

4. Fill the sample tube with warm water and place it in the bowl of the ultrasonic cleaning unit. Turn on the ultrasonic cleaning unit for approximately 15 minutes.
5. Using rubber gloves or a lint-free cloth, remove the sample tube from the bowl.
6. Clean the interior of the sample tube with the brush supplied in the accessories kit.
7. Rinse the sample tube thoroughly with hot water. Then rinse it again with isopropyl alcohol, using a waste container to collect used isopropyl alcohol.

Figure 4-6. Rinsing the Sample Tube

If isopropyl alcohol is unavailable, acetone or deionized water may be used to rinse the sample tube.

8. Using dry, compressed nitrogen or air, dry the interior of the sample tube under a vent hood. Use a tubing extension long enough and small enough in diameter to fit inside the sample tube.

9. Wipe a stopper with a lint-free cloth; make sure the stopper is dry. Label the sample tube and stopper for identification and place the stopper on the sample tube.

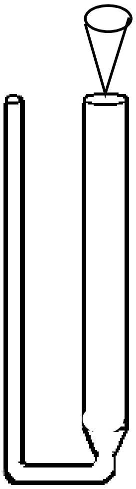


Figure 4-7. Placing the Stopper in the Sample Tube

Preparing the Sample

In order to obtain the most repeatable, accurate results, you should:

- Use approximately the same amount of sample for each analysis (especially if you compare data from sample to sample).
- Make sure the sample is well dispersed in the tube, not against the side or walls of the tube.
- Make sure the material's particle size distribution remains fairly constant from sample to sample. (Large particles of a substance reduce at a different rate than small particles of the same substance.) For samples with a wide particle size distribution, care should be taken that each sample is representative of the entire lot's distribution.

Micromeritics recommends analyzing samples weighing between 20 mg and 2 g. When determining a sample amount, consider that the sample must be contained within the bottom 20 mm (3/4 in.) of the sample tube.

When determining sample size, you may also wish to consider the percentage of active metals in your sample. Materials with lower percentages of active metals may require larger sample amounts for analysis, while materials with high percentages of active metals can be analyzed using smaller samples.

Larger amounts of very fine powders may become packed in the sample tube and block gas flow. The resulting increase in gas pressure may force some of the sample out of the sample tube, causing contamination of, or damage to, the analyzer. A possible solution is to expand the sample bed with quartz wool.

Determining the Mass of the Sample

Always wear gloves when handling quartz sample tubes. The salts from bare hands lower the normal melting point of quartz (approximately 1300 ºC) to a much lower temperature. Further, oils from your skin may destroy the quartz when the tube is heated.

1. Remove the stopper from the wide opening of the sample tube, and place a small amount of quartz wool into the clean, empty sample tube. (Try using a “ball” of quartz wool approximately 8-mm in diameter — loosely packed; some trial-and-error may be needed to determine the correct amount of quartz wool.)

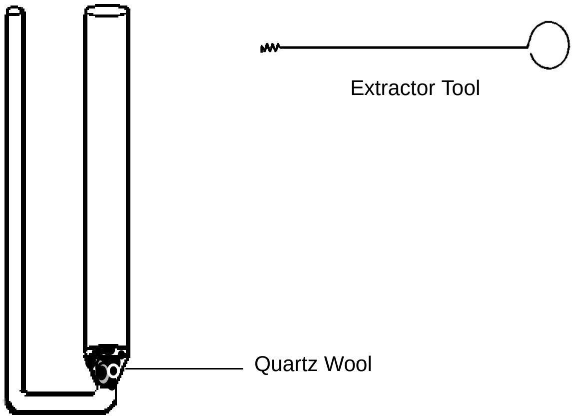


Figure 4-8. Placing Quartz Wool in the Sample Tube

2. Press the quartz wool firmly into place using the extractor tool. Replace the stopper.

3. Place the weighing support onto the balance. Zero the balance and allow it to stabilize.

A form to help you determine the sample’s mass is provided in Appendix A. Make copies of this form and use it for your convenience.

4. Place the sample tube (with stopper) onto the sample weighing support. If you are using the Sample Mass form from Appendix A, record the value as the empty sample tube mass.

Figure 4-9. Weighing the Sample Tube

5. Remove the sample weighing support and sample tube set (tube and stopper) from the balance.

6. Place a sample container onto the balance. Zero the balance and allow it to stabilize.

7. Slowly add the sample to the container. This step enables you to control the size of the sample before it is poured into the prepared sample tube. Do not record the sample mass at this point.

8. Remove the stopper from the tube. Using the funnel supplied with the analyzer, pour the sample from the container into the sample tube. Use a pipe cleaner to clean the walls of the sample tube before weighing the sample.

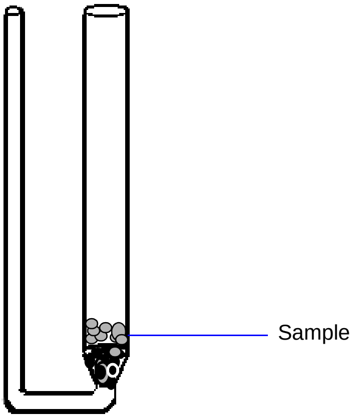


Figure 4-10. Placing Sample in the Sample Tube

Make sure that all of the sample is located in the lowest 20 mm of the sample tube. Any sample located higher in the tube will not be ideally located within the heated zone during analysis.

If you fail to clean any sample residue from the sample tube walls, results will be less accurate. The sample along the walls will be included in the sample weight, but will not be heated to the same temperature (and will therefore not react) as the sample that is properly located in the bottom of the tube.

9. Replace the stopper on the sample tube.

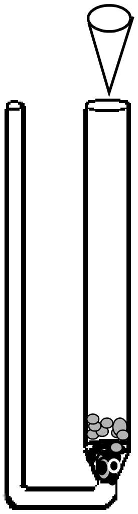


Figure 4-11. Sealing the Sample Tube

10. Determine the mass of the sample tube (with stopper) and sample. If you are using the Sample Mass form from Appendix A, record the value as the **Sample plus sample tube mass**.
11. Subtract the sample tube mass from the sample plus sample tube mass. The result is the sample mass.

The value recorded at this step is prior to sample preparation and analysis on the analyzer. Sample preparation and analysis will usually cause a change in this value. The difference between the *before preparation and analysis* and *after preparation and analysis* values may be useful when estimating how much contaminant is contained in fresh sample.

For some types of analyses, the sample mass measured after analysis is preferred, because many samples contain elevated levels of contaminants and/or moisture which are included in the sample's mass before degassing. This additional mass may affect the analysis results.

For TPR analyses (or other analyses involving small sample quantities), it may be helpful to weigh the sample in a tared container, transfer it to the sample tube, then reweigh the empty sample container after you transfer the sample to the tube. If there is a detectable amount of sample remaining in the container, subtract its mass from that of the sample in the tube.

Installing the Sample Tube on the Analysis Port

Always wear gloves when handling quartz sample tubes. The salts from bare hands lower the normal melting point of quartz (approximately 1300 ºC) to a much lower temperature. Further, oils from your skin may destroy the quartz when the tube is heated.

1. Carefully slide the sample tube retaining nuts and ferrules over the open ends of the sample tube. The larger diameter nuts and ferrules should be placed on the larger side of the tube, and the smaller diameter nuts and ferrules should be placed (narrow end down) on the smaller side of the sample tube.

Be careful not to allow the nuts to slide rapidly down the tube and smash into the curved portion of the tube, or the tube may be damaged.

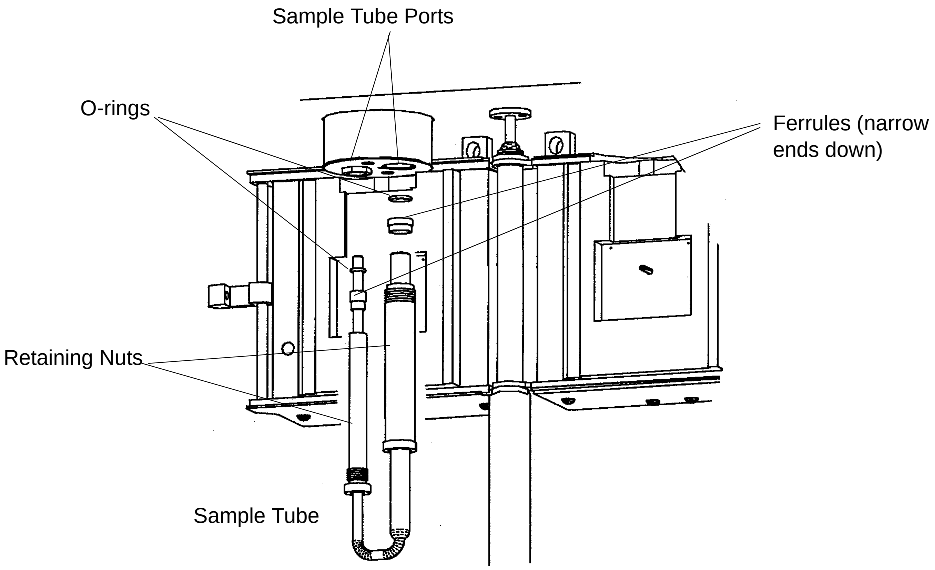


Figure 4-12. Sample Tube Components

2. Place an O-ring around the sample tube near each opening.

3. Remove any sample tube stoppers from the tube immediately before connecting the sample tube to the analyzer. Insert the open ends of the sample tube into the openings under the upper panel, aligning the thermocouple in the proper position (either inside or outside the sample tube; refer to Changing the Thermocouple Position in this chapter).

4. Press the sample tube up into the analyzer until it comes to a stop. Tighten the fittings by screwing the sample retaining nuts until they are finger-tight.

Do not overtighten the analysis port fittings. Doing so may break the sample tube. The O-rings are sealed when the fittings are finger-tight.

Removing the Sample Tube

The furnace and sample tube may remain very hot for some time after analysis has ended. The sample tube retaining nuts are extremely hot, approximately 110 ºC. Use Extreme caution!

It is recommended that you allow the furnace and sample tube to return to ambient temperature before touching them. If the sample tube has cooled, use rubber gloves when removing it. If it is still hot, be sure to use the cotton gloves provided.

1. If the furnace is closed around the sample tube, unlatch and open it.

Support the sample retaining nuts so that they do not fall rapidly out of the analyzer. This may cause severe damage to the sample tube.

2. Loosen the sample retaining nuts, being very careful to support them. As you loosen the nuts, also support the sample tube and pull it gently down out of the analyzer. When the nuts are sufficiently loose, the sample tube, retaining nuts, ferrules, and O-rings should slide out of the analyzer. If necessary, use the extractor tool to pull the O-rings out of the port(s).

3. Inspect the O-rings for cracks or other damage that might cause leaks. If the O-rings are cracked or damaged, replace them before reinstalling the sample tube. (Extra O-rings are shipped with the analyzer.) Otherwise, clean the O-rings you removed, dry them, and reinstall them.

Use only Kalrez$^{\text{TM}}$ O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.

Using the Cold Trap or Delay Path

A **cold trap** is used to condense liquids out of the gases flowing through the analyzer before they reach the detector. Specific instructions for using the cold trap are included in the instructions for performing each type of analysis in the tutorial (Appendix J).

A **delay path** is used to reduce the perturbation (disturbance) of the gas flow caused by injections of gas through the septum.

The cold trap or delay path is installed in the set of ports located under the top panel, to the left of the sample tube ports.

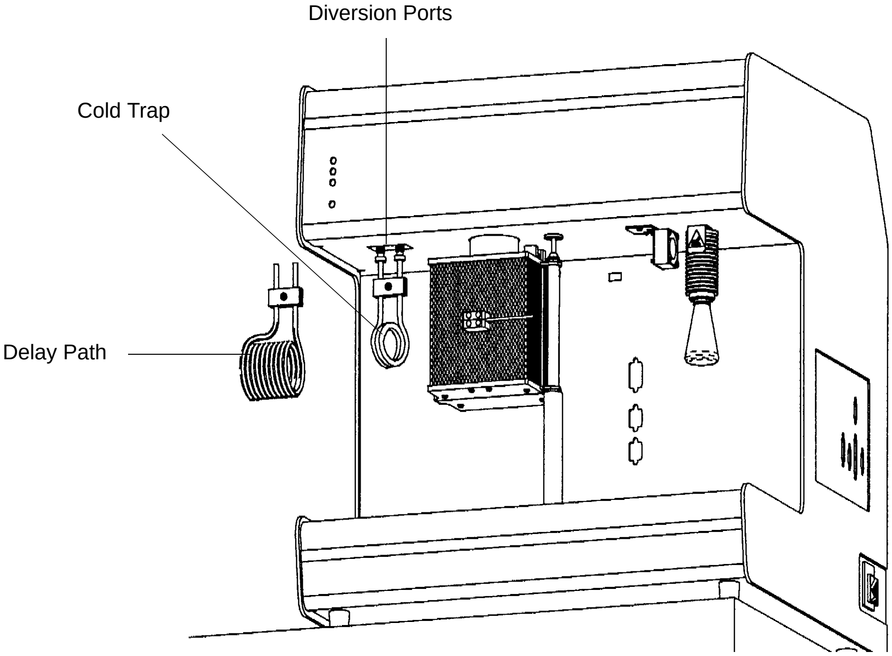
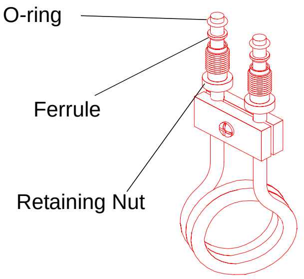


Figure 4-13. Location of the Cold Trap or Delay Path

1. Insert the open ends of the cold trap (or delay path) into the openings under the upper panel. If they are difficult to insert, loosen the attachment nuts by unscrewing them slightly.



The same components are used when installing the delay path.

Figure 4-14. Cold Trap Components

If the nuts are unscrewed until they become free of the analyzer, they will fall out of the analyzer.

2. Press the cold trap (or delay path) up into the analyzer until it comes to a stop; securely tighten the fittings.

Do not overtighten the fittings. A sufficient seal is achieved when the fittings are finger-tight.

3. If you wish to use a dewar to reduce the temperature of the cold trap, fill the dewar with the appropriate coolant. Place it underneath the cold trap so that the cold trap is immersed in the coolant.

Cleaning the cold trap is a regular maintenance procedure. A schedule and instructions are contained on page 9-4.

Using a Dewar

Precautions

Always handle dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. Always observe the precautions listed below.

We recommend the following be observed when handling dewars containing liquefied gases:

- Protect yourself by wearing 1) goggles (or a face shield), 2) an insulated or rubber apron, and 3) insulated gloves.
- When pouring cryogenic liquids from one container to another: 1) cool the receiving container gradually to minimize thermal shock, 2) pour the liquefied gas slowly to prevent splashing, and 3) vent the receiving container to the atmosphere.
- Use a plastic stirring rod when stirring substances in a dewar containing liquefied gases (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it is coated with some type of protective coating.
- Do not remove the mesh covering from the dewar flask. This covering is in place to minimize the risk of flying particles if the dewar is accidentally knocked over or dropped and broken.

- Do not handle heavy objects above the dewar. If unavoidable, place a protective cover over the dewar’s opening. If an object of sufficient weight is accidentally dropped into the dewar, shattering may occur.

Placing the Dewar Under the Cold Trap

To surround the cold trap, the dewar must rest on a small stand. Hold the dewar beneath the cold trap, then raise it until you can slide the stand underneath the dewar. Then lower the dewar until it rests on the stand. Make sure the cold trap is immersed.

Mixing an IPA/LN2 Slurry

An isopropyl alcohol (IPA)/liquid nitrogen (LN_2) slurry is used to maintain cold trap temperatures of approximately -80 °C. Use the following instructions to mix such a slurry if this is the temperature you wish to maintain.

Improperly mixing an IPA/LN_2 slurry could cause injury. If you do not stir the mixture continuously, gas may build up under the surface, causing the liquids to splash out of the dewar.

1. Chill a 600-mL dewar by rinsing it with LN_2. Allow a small amount of LN_2 to remain in the bottom of the dewar (approximately 1 cm deep).
2. Stirring constantly, slowly add approximately 500 mL of IPA. For greatest safety, use a laboratory squirt bottle. Squirt the stream of IPA along the inside edge of the dewar, very near the top, allowing the IPA to flow down the dewar’s inside wall. Do not stop stirring the mixture in the bottom of the dewar, even if stirring becomes difficult. As you continue stirring and adding IPA, the mixture will loosen and become easier to stir.
3. When all the IPA has been placed in the dewar, slowly begin pouring LN_2 into the dewar. Approximately 1 liter is needed. You must continuously stir the mixture as you add the LN_2. As ice chunks form, break them up and stir them down into the mixture. Avoid splashing. Gently knock ice chunks away from the sides of the dewar and continue stirring. Add liquid nitrogen until the slurry is within 25 mm (1 in.) of the top of the dewar.
4. Lift the stirrer out of the slurry and observe as the slurry drips into the dewar; the stirrer should be thickly coated with slurry.

   Your goal is to achieve a slurry that has a thick, syrupy consistency without large chunks. When the slurry is cold enough, small pieces of ice may be present (10 - 20% of the mixture). A little ice accumulation along the sides of the dewar is acceptable.

Checking the Cryogen Level

If you are performing an analysis that requires use of the cold trap and cryogen, check the cryogen level in the dewar. It should be about 25 mm (1 in.) from the top.

The cryogen must not be cold enough to trap the carrier gas or analysis gas. Do not use liquid nitrogen with argon carrier gas. For example, use an alcohol and liquid nitrogen slurry (-80 ºC).

Use appropriate safety procedures when handling all cryogens. Be sure to wear safety glasses and gloves, and observe the precautions listed earlier in *Precautions*.

Cleaning the Dewar

The dewar must be cleaned between uses. Empty the dewar, then rinse it with water. Make sure that it is dry before using it again.

Using the Septum

The septum is used for injecting quantities of gas into the analyzer, such as during a loop calibration. The septum is located in the upper left corner of the recessed portion of the front panel.

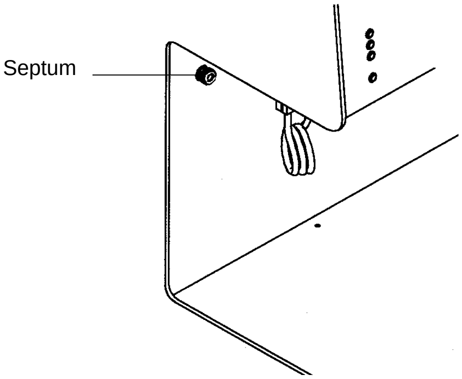


Figure 4-15. Location of the Septum

Accuracy of data is diminished when poor techniques are used for injecting gas through the septum. The following guidelines should help you maximize the accuracy of your data.

Injecting the gas through the septum causes a peak to appear, but it also causes a perturbation in the flow of gas through the analyzer. This perturbation is visible in the peak data. You can minimize this perturbation by injecting the gas more slowly into the septum. However, prolonging the injection causes the peak to spread.

You must develop your injecting method to balance your desire to minimize the perturbation with your need for sharper peaks. The Peak Editor (described on page **7-14**) enables you to adjust your peak data to reduce the effects of perturbation.

- Always hold the syringe by its metal parts away from the needle. Holding the syringe by the glass allows your body heat to affect the volume of gas in the syringe.

- After filling the syringe, allow the syringe to lie on a room-temperature surface, such as that of the workbench, for about a minute. This helps ensure that the syringe and its contents are at room temperature.

- If the gas you are using is lighter than air, do not allow the filled syringe to remain in a vertical position (needle up); the gas will diffuse out and the total volume will be reduced.

- To fill the syringe: first empty it completely, then insert it into a septum accessory installed on the gas regulator. Draw the syringe plunger back until the syringe is completely filled with gas. Remove the syringe from the septum, and allow it to return to room temperature as described above. Then, press the plunger into the syringe until the correct amount of gas is contained in the syringe.

- An alternative way to fill the syringe with nitrogen (evaporating from liquid nitrogen), is to empty the syringe, then hold the tip of the needle just above the surface of a quantity of liquid nitrogen. Draw the syringe plunger back to fill the syringe with nitrogen gas. Allow the syringe to stabilize at room temperature, then push the plunger into the syringe until the correct amount of gas is contained in the syringe.

- Make sure the needle is inserted fully into the septum on the analyzer.

- Make sure that you inject the entire quantity of gas that you have measured into the syringe. (Press the plunger into the syringe completely.)

Cooling Options

The furnace must cool during temperature-programmed analyses if (1) a low temperature is required as part of the analysis, or (2) you simply wish to speed cooling of the furnace in order to increase sample throughput, or reduce analysis time.

Cooling options are listed below. If you wish to speed the cooling of the furnace to increase throughput, the choice may be one of convenience. If cooling is required for your analysis, the CryoCooler may be required.

- **Allow the temperature to return to ambient over time**
  If time is not a consideration, you may wish to allow the furnace to cool over time. It may take two hours or more for the furnace to cool from 1100 °C to near ambient. This may be acceptable when time is not limited.

- **Promote cooling by manually opening the furnace**
  The furnace cools more rapidly when it is open. This option requires the availability of an operator to open and close the furnace at the appropriate times during analysis.

When it has been used recently, the furnace and/or the sample tube may be hot. Use the cotton gloves provided with the analyzer to protect your hands.

- **Connect a cooling gas or cryogen flow to the inlet on the front of the furnace**
  You can connect a cooling flow of an appropriate gas or cryogen to the inlet on the furnace. Read the caution above. This option can provide rapid cooling of the sample, but offers no control of the target temperature or the speed at which it is achieved. The gas or cryogen must be nonreactive, such as nitrogen, air, helium, argon, liquid nitrogen, or liquid argon.

- **Connect the KwikCool to the inlet in the front of the furnace**
  The KwikCool allows you to ramp the furnace temperature rapidly down near ambient, which reduces the time required to complete analyses and increases throughput. Instructions for attaching the KwikCool to the furnace are provided in Chapter 2. A KwikCool is included as a standard accessory with every AutoChem II 2920.

- **Connect the optional CryoCooler to the inlet in the front of the furnace**
  The CryoCooler provides control of the sample temperature from temperatures as low as -70°C. The CryoCooler is required for accurate analyses that include signal recording below ambient. Instructions for attaching the CryoCooler to the furnace are provided in Chapter 2 (refer to Chapter 10 for ordering information).

Protecting the Detector Filaments by Flowing Gas

A carrier gas must be flowing through the detector whenever the detector filaments are turned on; otherwise, the filaments will deteriorate and lose sensitivity. If the gases are shut off, the filaments are automatically turned off after five minutes.

The flowing gas also continuously cleans the plumbing of any water vapor which can enter when the sample tube is removed. Ten cm^3/min is sufficient to perform this function.

Specifying Analysis Temperatures

The guidelines listed here are for all analysis types.

Starting Temperature

Experiments should be started at a temperature that is lower than that at which the reaction begins. If the reaction begins below ambient temperature, you should use the optional CryoCooler.

Maximum Temperature

The experiment's maximum temperature is limited to the temperature at which your sample sinters[1] or melts.

Ramp Rate

The recommended ramp rate is 5-10 °C/min. However, you may wish to use a different ramp rate for various purposes. For example, if you have identified 500 °C as the starting temperature of the reaction being observed, it is more expedient to ramp the temperature rapidly (for example, 50 °C/min.) to a value near 500 °C, then proceed with the experiment at 5-10 ºC/min.

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[1] Sintering is the fusing of small particles (or small features of a sample). Sintering tends to reduce the active surface area.

Performing an Analysis

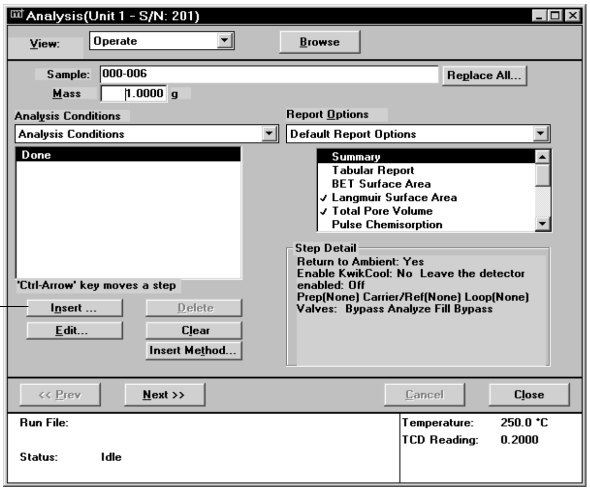
Before starting your analysis, make sure:

- the correct gases are connected and that the Gas Selections dialog (Unit menu) reflects the current gas connections
- the sample has been prepared and the sample tube installed on the analyzer

Appendix J provides tutorials for performing a TPR, TPD, TPO, and Pulse Chemisorption analysis.

Chapter 6 provides a description of the dialogs used for performing an analysis.

1. Select **Unit > Start analysis**; the Sample Analysis dialog is displayed with the Open Sample information dialog positioned on top.
2. Choose a file for your analysis (or accept the default to create a new one) and click **OK**; the Sample analysis dialog containing the parameters of the selected file (or the defaults) is displayed.



Analysis(Unit 1 - S/N: 201)

View: Operate

Sample: 000-006

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

Summary
Tabular Report
BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption

Step Detail
Return to Ambient: Yes
Enable KwikCool: No Leave the detector enabled: Off
Prep(None) Carrier/Ref(None) Loop(None)
Valves: Bypass Analyze Fill Bypass

'Ctrl-Arrow' key moves a step

Run File:

Status: Idle

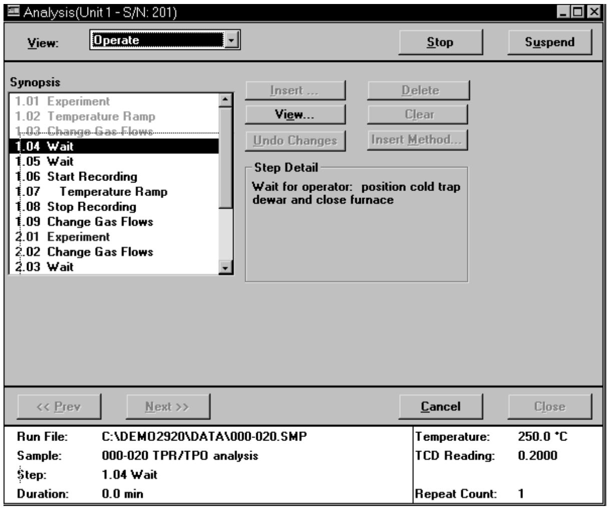
Temperature: 250.0 °C

TCD Reading: 0.2000

This group of push buttons is not displayed on the Basic presentation; you cannot edit parameters when using the Basic mode.

3. Review and verify analysis conditions and report options; edit if necessary.
4. Click **Next**; the second view of the analysis dialog is displayed. From the drop-down list(s), select the calibrations to be associated with each experiment (if any).
5. Click **Next**; specify report output options.
6. Click **Next** to review the safety information.

7. Click **Start** to begin the analysis. A dialog allowing you to monitor the progress of the analysis is displayed.



1.01 Experiment
1.02 Temperature Ramp
1.03 Change Gas Flows
1.04 Wait
1.05 Wait
1.06 Start Recording
1.07 Temperature Ramp
1.08 Stop Recording
1.09 Change Gas Flows
2.01 Experiment
2.02 Change Gas Flows
2.03 Wait

Step Detail: Wait for operator: position cold trap dewar and close furnace

Run File: C:\DEMO2920\DATA\000-020.SMP
Sample: 000-020 TPR/TPO analysis
Step: 1.04 Wait
Duration: 0.0 min
Temperature: 250.0 °C
TCD Reading: 0.2000
Repeat Count: 1

You can also use this dialog to edit any step after the current one. Simply suspend the analysis; this enables the editing push buttons. After editing is completed, resume the analysis.

Listing File Statistics

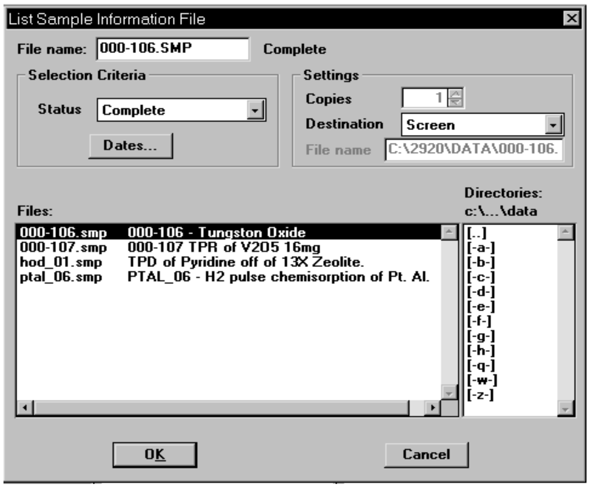
You can generate a list of the following information on one or more sample or parameter files:

- File name
- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- File status (sample information files)

Perform the following steps to generate a list:

1. Select **File > List**.

2. Select the type of file on which you wish information; a dialog similar to the one shown below is displayed.



File name: 000-106.SMP Complete

Status: Complete

Copies: 1

Destination: Screen

File name: C:\2920\DATA\000-106.

Files:

000-106.smp	000-106 - Tungston Oxide
000-107.smp	000-107 TPR of V2O5 16mg
hod_01.smp	TPD of Pyridine off of 13X Zeolite.
ptal_06.smp	PTAL_06 - H2 pulse chemisorption of Pt. Al.

3. From the **Files** list box, choose the desired file(s). If you wish to include all files in the list, leave all files deselected.

4. At the **Destination** field, click on the down arrow and choose an output destination. If you choose File as the destination, enter a name in the **File name** field (or accept the default).

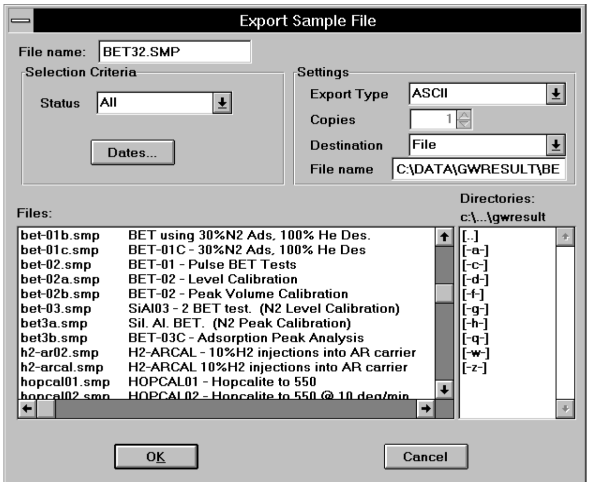
5. Click **OK**; a list for the requested file(s) is sent to the specified destination.

Exporting Data from a Sample Information File

Data from a sample information file can be exported in ASCII or Grams/32 format. ASCII data can then be imported into other applications, such as spreadsheets. Grams/32 data can be imported into the Grams/32 program (a peak-editing program used with earlier versions of the AutoChem software) and manipulated independent of the AutoChem 2920 analysis program. Refer to Appendix D for a description of exported data.

Perform the following steps to export a sample information file:

1. Select **File > Export**; the Export Sample File dialog is displayed.



File name: BET32.SMP

Status: All

Export Type: ASCII

Copies: 1

Destination: File

File name: C:\DATA\GWRESULT\BE

Files:

bet-01b.smp	BET using 30%N2 Ads, 100% He Des.
bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier
hopcal01.smp	HOPCAL01 - Hopcalite to 550
hopcal02.smp	HOPCAL02 - Hopcalite to 550 @ 10 deg/min

Directories: c:\...\gwresult

[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

2. From the **Files** list box, choose the file(s) you wish to export. You can choose multiple files by holding down and clicking on the desired files.

3. From the **Settings** group box:

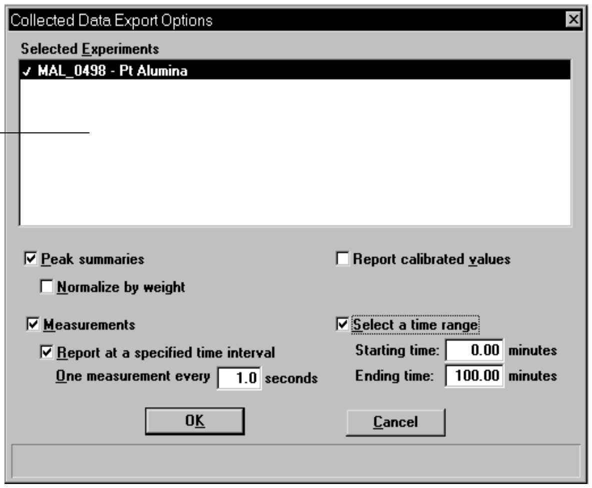
   a. Choose whether you are exporting in ASCII or Grams/32 format.

   b. Choose a destination for your exported file.

      If you choose **File**, enter a name in the **File name** field (or accept the default). If you choose **Printer**, the **Copies** field is enabled allowing you to print up to 4 copies.

You must choose *File* as the destination when exporting in Grams/32 format.

4. Click **OK**; the Collected Data Export Options dialog is displayed.



Selected Experiments
✓ MAL_0498 - Pt Alumina

☑ Peak summaries
☐ Normalize by weight
☑ Measurements
☑ Report at a specified time interval
One measurement every 1.0 seconds
☐ Report calibrated values
☑ Select a time range
Starting time: 0.00 minutes
Ending time: 100.00 minutes

Only the Selected Experiments group box is displayed if data are being exported in the Grams/32 format.

5. Choose the experiments whose data you wish to export. Then select any other desired options. Refer to Chapter 5 for additional information on this dialog.

6. Click **OK**; data are exported to the specified destination.

Creating and Assigning a Calibration File

After you have completed your analysis, you can edit your peaks to create a calibration file and assign the file to your experiment. There are two types of calibration files you can create and assign: a Vapor calibration and a User-Defined calibration. The example in this section shows a User-Defined one.

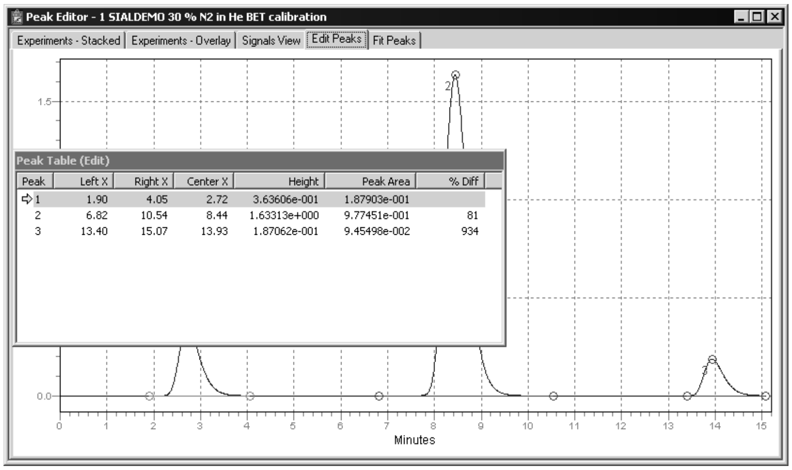
Editing the Peaks

1. Select **Reports** > **Peak Editor** from the main menu.

2. Select the desired sample file and click **OK;** the Peak Editor, Stacked view dialog, is displayed. This view of the dialog displays all calibrations and experiments contained in the file, stacked one after the other.

3. Click a calibration experiment; the name of the experiment displays in the title bar of the dialog.

4. Click the **Edit Peaks** tab; the Edit Peaks dialog and table for the selected calibration are displayed.



Peak Table (Edit)

Peak	Left X	Right X	Center X	Height	Peak Area	% Diff
⇨ 1	1.90	4.05	2.72	3.63606e-001	1.87903e-001	
2	6.82	10.54	8.44	1.63313e+000	9.77451e-001	81
3	13.40	15.07	13.93	1.87062e-001	9.45498e-002	934

Peaks should be in a positive position before integrating. If you have any negative peaks: right-click in the graph area, choose *View, Display* and click the *Invert* option

5. Right-click in the graph area and select **Find All Peaks**; this option finds all peaks and marks them.

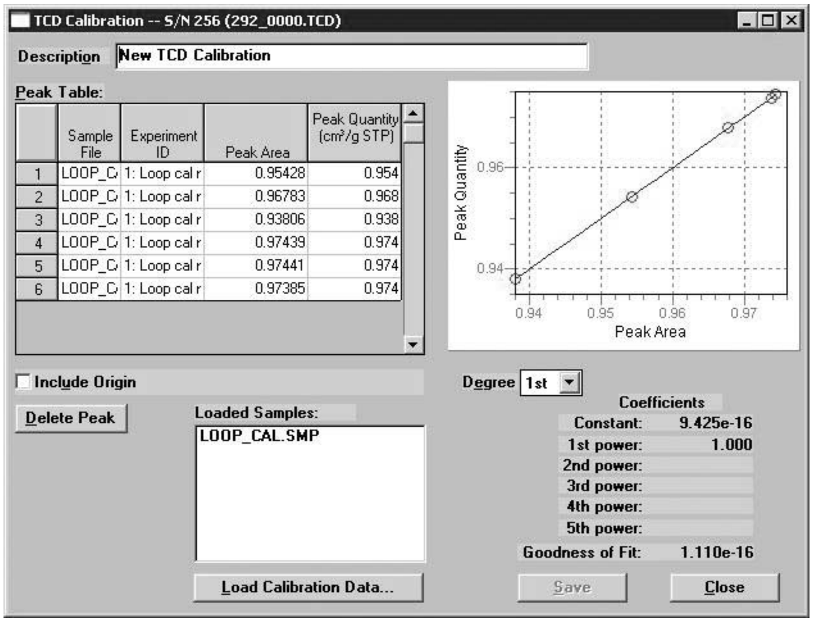
You can further define peaks if desired. Select the peak (the baseline area of the peak displays in green when selected), right-click, and use the *Mark Left Edge* and *Mark Right Edge* options.

6. Right-click and select **Save**.

Creating the Calibration File

After you have defined your calibration peaks, you can create the calibration file.

1. Select **Unit > TCD Calibration >, New**; the TCD Calibration dialog is displayed.



Description: New TCD Calibration

Peak Table:

	Sample File	Experiment ID	Peak Area	Peak Quantity (cm³/g STP)
1	LOOP_C	1: Loop cal r	0.95428	0.954
2	LOOP_C	1: Loop cal r	0.96783	0.968
3	LOOP_C	1: Loop cal r	0.93806	0.938
4	LOOP_C	1: Loop cal r	0.97439	0.974
5	LOOP_C	1: Loop cal r	0.97441	0.974
6	LOOP_C	1: Loop cal r	0.97385	0.974

☐ Include Origin

Loaded Samples: LOOP_CAL.SMP

Degree: 1st

Coefficients

Constant:	9.425e-16
1st power:	1.000
2nd power:	
3rd power:	
4th power:	
5th power:	
Goodness of Fit:	1.110e-16

2. Click **Load Calibration Data**; the Load Calibration Sample File dialog is displayed.

3. Select the sample file containing the calibration data.

4. Enter an intuitive description in the **Description** field. It is important that you use a description that you will easily recognize.

5. Delete from the Peak Table any data not associated with your calibration experiment.

6. Using the formula given below, calculate the peak volume for each peak in the table; enter the results into the appropriate fields.

$$V_{syrinj} \times \frac{273.15}{T_{amb} + 273.15} \times \frac{P_{amb}}{760\ mmHg} = V_{Peak}$$

Example: Calculations for 1 cm^3 injection, ambient temperature of 22 °C, ambient pressure of 760 mmHg:

$$1 \times \frac{273.15}{22 + 273.15} \times \frac{740}{760\ mmHg} = 0.901 cm^3 STP$$

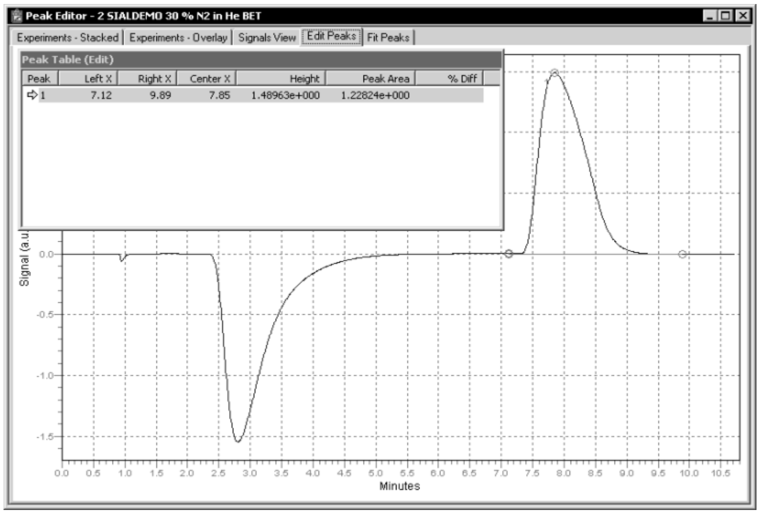
7. Click **Save** to save the file, then **Close** to close the dialog.

Repeat steps 1 through 7 for each gas concentration in the calibration. For example, if you have gas concentrations of 10%, 30%, and 20%, you should mark your calibration peaks and create calibration files for all three concentrations; a total of three files.

Assigning the Calibration File to the Experiment

Now you must assign the calibration to the appropriate experiment.

1. Click back into the Peak Editor dialog and select the **Experiments-Stacked** tab.
2. Click a sample experiment. Remember, you can view the experiment name in the title bar.
3. Click the **Edit Peaks** tab; the Edit Peaks dialog and table for the selected sample experiment are displayed.

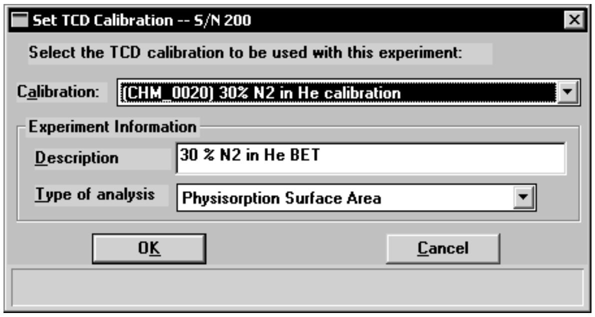


Peak Editor - 2 SIALDEMO 30 % N2 in He BET

Peak Table (Edit)

Peak	Left X	Right X	Center X	Height	Peak Area	% Diff
⇨ 1	7.12	9.89	7.85	1.48963e+000	1.22824e+000	

4. Right-click in the graph area and select **Find All Peaks** from the shortcut menu.
5. You should have only one peak in the table; if multiple peaks are included, delete all but the desorption peak. The desorption peak typically is the last one in the table.
6. Right-click in the graph area and select **Set Calibration** from the shortcut menu; the Set Calibration dialog is displayed.



Set TCD Calibration -- S/N 200

Select the TCD calibration to be used with this experiment:

Calibration: [CHM_0020] 30% N2 in He calibration

Experiment Information

Description: 30 % N2 in He BET

Type of analysis: Physisorption Surface Area

7. Click on the down arrow to the right of the Calibration field and choose the calibration file you created for the concentration of the selected experiment. Be sure that the Experiment and the Calibration match.
8. Right-click in the graph area and select **Save** from the shortcut menu.

5. FILE MENU

The File menu contains options that allow you to manage sample and parameter files.

Description

Listed below are brief descriptions of the File menu options. Detailed descriptions follow this section.

Open	Opens an existing file or creates a new file. When you select this item, a drop-down menu is displayed containing three types of files: sample information, analysis conditions, and report options. Page **5-3**.
Save	Saves the file in the active window. Page **5-54**.
Save As	Allows you to save the file that is in the active window under a different name. It also can be used to save a subset of a sample file as an independent parameter file. Page **5-54**.
Save All	Saves all open files. Page **5-55**.
Close	Closes the file in the active window. Page **5-56**.
Close All	Closes all open files. Page **5-56**.
Print	Prints details of the selected file(s). Page **5-57**.

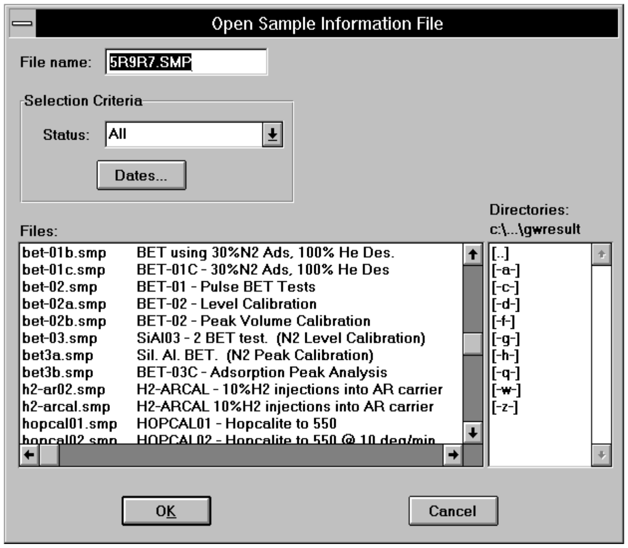
List	Generates a list of certain information for the selected sample file(s) or parameter file(s). Page **5-58**.
Import MS Signal	Allows you to import data collected on a Pfeiffer Vacuum (formerly Balzers) mass spectrometer which uses the Quadstar software. Page **5-59**.
Export	Exports a sample file in ASCII format or in a format compatible with the Grams/32 application (a peak editing program used previously with the AutoChem). Page **5-62**.
Exit	Exits the analysis program. Page **5-65**.

Open

Select Open to create a new file or edit an existing one. The following file types are available:

- Sample information
- Analysis conditions
- Report options

Regardless of which file type you select, a dialog box similar to the one shown below is displayed.



File name: 5R9R7.SMP

Status: All

Files:

bet-01b.smp	BET using 30%N2 Ads, 100% He Des.
bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier
hopcal01.smp	HOPCAL01 - Hopcalite to 550
hopcal02.smp	HOPCAL02 - Hopcalite to 550 @ 10 deg/min

Directories:
c:\...\gwresult

[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

File name	For sample information files, this field contains the next sequenced file name generated by the system. For parameter files (analysis conditions and report options), the file name displayed includes the wild card (*) and the default extension as follows: *.ANC for analysis conditions *.RPO for report options If you are creating a new file, enter a name in the File name field. If you are editing an existing file, select a file from the list.
Files	This window displays the files available in the selected directory for the chosen status. This list includes AutoChem II 2920 files as well as AutoChem 2910 files (if applicable).

Files (*continued*)	If you select an AutoChem 2910 file to use in the AutoChem II 2920 application and it is saved, it will no longer be compatible with the AutoChem 2910 application. Be sure to use the Save as function to save it as a different name if you want the file to remain 2910-compatible.

Refer to **Selecting Files** on page **3-6** for a description of the other fields on this dialog box.

Sample Information

Sample Information files contain information used to control the analysis, as well as collected data. A sample information file consists of four parts:

- Sample information
- Analysis conditions
- Report options
- Collected data (this part does not appear until analysis is complete)

Portions of the sample file can also exist as parameter files separate from the sample information file itself. These parameter files contain frequently used analysis conditions and report options that can be conveniently loaded into a new sample information file.

Sample information files are presented in three different formats, Basic, Advanced, and Restricted. You can select any one of these formats as your default. A convenient toggle button makes it easy to switch between Basic and Advanced formats, regardless of which is selected as your default.

- **Basic Format**
  Presents the entire sample information file in a single dialog box. Basic format makes it easy to select previously created, standard parameter files from convenient drop-down lists.

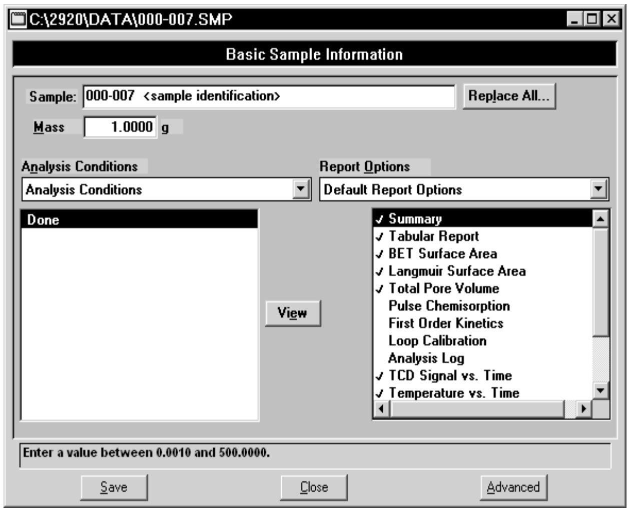
- **Advanced Format**
  Presents all four parts of the sample information file as a group of dialog boxes with index card-type tabs (although the Collected Data tab does not appear until after analysis). Clicking on each tab opens the associated dialog. Use this format to create customized sample information files.

- **Restricted Format**
  Restricted presentation format is identical to Basic presentation format, except it prevents the operator from switching to the Advanced presentation. Restricted presentation limits the operator’s ability to change analysis conditions and report options.

Select **Options** > **Option presentation** to specify your default format.

Basic Presentation

The Basic format displays all parameters of the sample information file on a single dialog. With the Basic format, you can quickly create a sample information file using predefined parameter files. If you wish to view or edit specific parameters, you can switch easily to the Advanced format by clicking **Advanced**.



Sample: 000-007 <sample identification>
Mass 1.0000 g
Analysis Conditions: Analysis Conditions
Report Options: Default Report Options

Done

- ✓ Summary
- ✓ Tabular Report
- ✓ BET Surface Area
- ✓ Langmuir Surface Area
- ✓ Total Pore Volume
- Pulse Chemisorption
- First Order Kinetics
- Loop Calibration
- Analysis Log
- ✓ TCD Signal vs. Time
- ✓ Temperature vs. Time

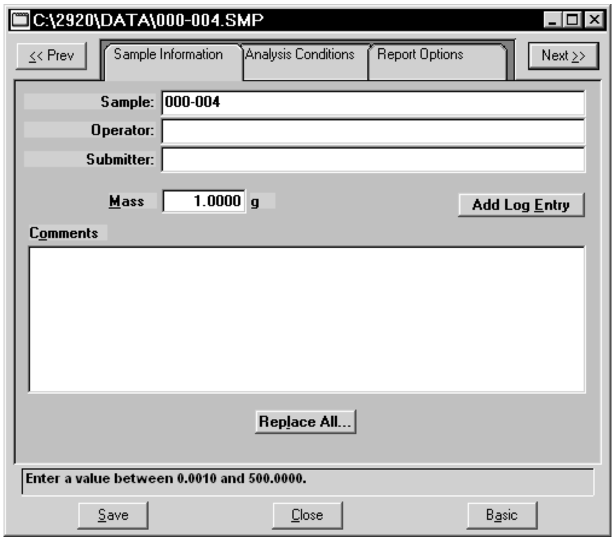
Enter a value between 0.0010 and 500.0000.

Sample	Contains the description of the current sample file. If this is a new file, this field contains the next sequenced description based on the format you specified in Sample defaults (see Chapter 8). You can enter a new description or add to the existing one if desired. *Range:* *50 alphanumeric characters*
Mass	Allows you to enter the mass of the sample.
Analysis Conditions	Displays the name of the current Analysis Conditions file. If this is a new file, this field contains the name of the file you specified as the default. Click on the down arrow to the right of the field to choose a different file. This list includes Analysis Conditions files supplied with the analysis program as well as those you create and save to the directory specified as the Parameter Files directory (Chapter 8). If new files are not saved to the specified directory, they are not included in the list. This list is disabled for files which have been used for an analysis.

View	Allows you to view details about the currently selected step in the analysis conditions set.
Report Options	Displays the name of the current Report Options file. If this is a new file, this field contains the Report options file you specified as the default. Click on the down arrow to the right of the field to choose a different file. This list includes Report Options files supplied with the analysis program as well as those you create and save to the directory specified as the Parameter Files directory (Chapter 8). If new files are not saved to the specified directory, they are not included in the list.
Reports List Box	Displays a list of available reports. The checked reports are the ones requested for the current Report options file. If this is a new file, the checked reports are the ones you specified for the default(s). You can select (or deselect) reports by double-clicking on the desired report. Alternatively, you can highlight the report and press **Spacebar**.
Replace All	Allows you to copy an existing sample file's values into the sample information file you currently have open. A file selection dialog is displayed. Choose the file containing the values you wish to copy, then click **OK**; the values are copied into the new file automatically. You may then edit the values in the new sample file without altering the sample file from which they were copied.
Save	Saves the currently open file.
Close	Closes the currently open file.
Advanced	Switches the presentation format to the Advanced presentation.

Advanced Presentation

The Advanced format displays the sample information file in a manner similar to that of an index card file. Each parameter has its own tab and is accessed by clicking the tab. Or, you can use **Prev** and **Next** to move through the dialogs. This format allows you to create customized sample information files.



C:\2920\DATA\000-004.SMP

Sample: 000-004

Operator:

Submitter:

Mass 1.0000 g

Enter a value between 0.0010 and 500.0000.

The prompts for the **Sample**, **Operator** and **Submitter** fields may be customized by selecting **Sample defaults** from the Options menu. Refer to Chapter 8 for instructions on customizing these prompts.

Sample	Contains the description of the current sample file. If this is a new file, this field contains the next sequenced description based on the format you specified in Sample Defaults (refer to **Sample Defaults** on page **8-7**). You can enter a new description or add to the existing one if desired.
Operator **Submitter**	Displays the Operator and Submitter names (if specified) of the current sample file. If this is a new file, these fields contain the names specified in Sample defaults. You can enter a different name in either (or both) field if desired. You can also edit the prompts for these fields or choose to have them omitted entirely.

Mass	Allows you to enter the mass of the sample.
Add Log Entry	Displays a dialog box allowing you to enter pertinent information relating to the sample. Any information you enter here is printed as part of the Analysis Log report (select in Report Options).
Comments	Use this field to enter any comments about the sample or the planned analysis conditions that you wish to note. Comments entered here are printed in the header of some reports. *Range: Up to 500 alphanumeric characters*
Replace All	Allows you to copy the values for all parameters of an existing sample file into the sample information file you are creating. A file selection dialog is displayed. Choose the file containing the values you wish to copy, then click **OK**; the values are copied into all parameters of the new file automatically. You can then edit the values in the new sample file without altering the sample file from which they were copied.

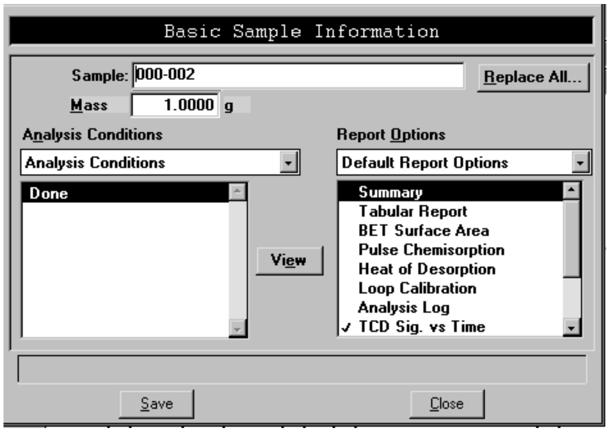
Each of the individual tabbed dialogs that make up the sample information file can also be replaced separately, using Replace that appears on each. Individual Replace buttons replace only the data on the dialog box that is currently shown.

Basic	Switches the presentation format to the Basic presentation.

The Analysis Conditions and Report Options tabs of the Advanced presentation sample information file are described in subsequent sections.

Restricted Presentation

A third format is provided to control access to some portions of the sample information file. Restricted format displays in the same manner as the Basic presentation format.



Sample: 000-002

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

Summary
Tabular Report
BET Surface Area
Pulse Chemisorption
Heat of Desorption
Loop Calibration
Analysis Log
✓ TCD Sig. vs Time

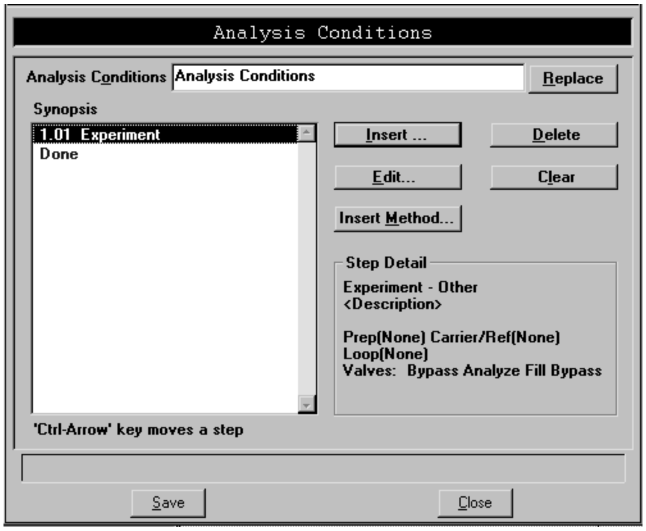
The Restricted format does not allow switching to the Advanced format for editing.

In Restricted presentation format, the **Advanced** button does not appear on the screen, so the operator cannot switch to the Advanced format. This format is also password-protected, preventing the operator from making changes to analysis conditions and report options sets. Chapter 8 provides additional information on this format.

Restricted presentation is ideal for laboratories in which standard analysis procedures are established by a lab manager, while one or more lab technicians actually perform the analyses. The lab manager can create independent analysis conditions and report options files containing standard sets of operating conditions, then the operator(s) can use Restricted mode for daily operations, selecting the standard parameter files from the drop-down lists.

Analysis Conditions

Analysis conditions files specify each step in the analysis, including temperatures, pressures, and gases. You can create an Analysis Conditions file as an independent parameter file or as part of the sample information file. You can also save the analysis conditions portion of the sample information file as a parameter file.



Analysis Conditions: Analysis Conditions

1.01 Experiment
Done

Experiment - Other
<Description>

Prep(None) Carrier/Ref(None)
Loop(None)
Valves: Bypass Analyze Fill Bypass

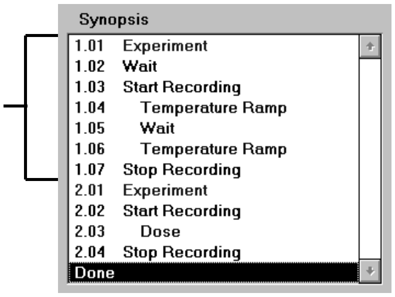
'Ctrl-Arrow' key moves a step

When creating parameter files to display in the drop-down list on the Basic sample information dialog, be sure to save them to the directory specified as the Parameter File directory (explained on page 8-17).

Analysis Conditions	If you are opening an existing analysis conditions file, this field contains the description If you are creating a new analysis conditions file, this field contains the description specified in Sample defaults. You can enter a new description or add to the existing one if desired. Use a description that will help you identify this set of analysis conditions in the future. *Range: 40 alphanumeric characters*
Replace	Allows you to copy values from an existing file to the one you are creating. A dialog box is displayed allowing you to choose the desired file. You can then edit these values if desired; the file you copied them from will not be affected.
Synopsis	Displays a list of the steps the analyzer will follow during the analysis.

An *analysis* is a complete characterization of the sample; in other words, it is all of the *experiments* and steps within the Synopsis. It may consist of up to 99 separate experiments, such as TPR or TPO. Each experiment may consist of up to 1000 *steps*.

Synopsis
(continued)



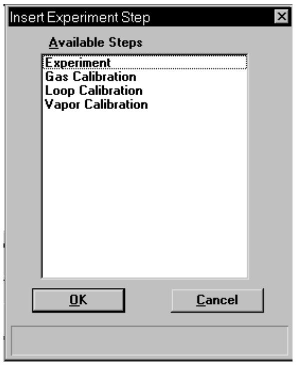
1.01 Experiment
1.02 Wait
1.03 Start Recording
1.04 Temperature Ramp
1.05 Wait
1.06 Temperature Ramp
1.07 Stop Recording
2.01 Experiment
2.02 Start Recording
2.03 Dose
2.04 Stop Recording

Steps within Experiment 1

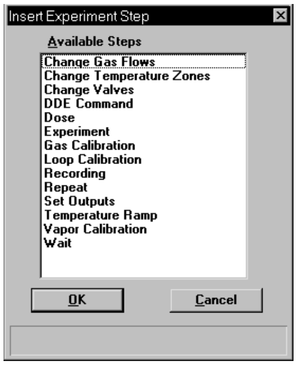
The Synopsis is the key to the AutoChem II 2920's high level of automation. This list enables the software to control and change the gas flow, temperature, and data recording process. It causes the analyzer to progress to the next step, or to wait until specific criteria are met before proceeding. In all, this list provides a step-by-step guide for the analysis.

Insert

Allows you to insert a new experiment or a step within an experiment.



This dialog displays if you are inserting a new experiment.



This dialog displays if you are inserting a step within an experiment.

Insert
(continued)

At some points in the sequence of steps, all steps are available. At other points, certain steps cannot be performed. For example, you cannot start a new experiment inside a **Repeat** loop.

The steps that appear in the list box are those which are currently available. To select an experiment or step to insert: click on it, then click **OK**. A dialog enabling you to specify instructions for the step or experiment is displayed. Each of the specific step and experiment dialogs are discussed in subsequent sections.

The new step is inserted directly before the step that was highlighted when **Insert** was selected. After you insert a **Repeat** or **Record** step, the automatically entered **Stop Repeat** or **Stop Record** step is highlighted. Any steps inserted at that point will occur within the Repeat or Record loop.

You can also insert steps after the analysis has started by suspending the analysis.

You can easily move a step from one position in the list to another. Click on it, then use **Ctrl** + ↑ or **Ctrl** + ↓ to move the highlighted step.

Delete

Deletes the selected item from the synopsis list. If you click this button when an experiment is selected, the entire experiment (including all its steps) is deleted after a confirmation.

Edit

Enables you to edit the selected experiment or step; an associated dialog is displayed.

You can also edit a step or experiment during analysis. However, you must suspend the analysis before any editing can be accomplished. You can edit only the steps or experiments following the current one; all completed steps will be disabled.

After an analysis has been completed, **Edit** displays as **View**. Steps may be viewed but not edited.

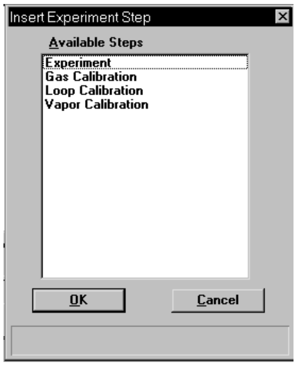
Clear

Clears the list of experiments and steps for this Analysis Conditions dialog. A confirmation message is displayed.

Insert Method	Allows you to insert the entire list of steps from another analysis conditions file. After you click this button, select an analysis conditions file from the list. All the steps in that file are inserted directly above the highlighted step. Inserted methods can be edited without altering the original analysis conditions file(s) from which they were copied. For example, if you have three different standard sample preparation methods that you use under varying circumstances, create an analysis conditions file for each preparation method. Then use **Insert Method** to insert the appropriate preparation method in the analysis conditions file you create when you are ready to analyze a sample. You can also create several analysis conditions files containing individual experiments, then use **Insert Method** to combine them into a comprehensive analysis comprised of these individual analyses.
Step Detail	Displays information about the currently highlighted step in the Synopsis. The information displayed for each step is a summary of the information contained on that step's dialog.

Insert Experiments

An analysis set is created by inserting a series of experiments in the sample file Synopsis. You can insert up to 99 experiments. There are four types of experiments that you can insert in an analysis conditions set. The following dialog is displayed when you choose to insert an experiment:



The first type is the customizable Experiment. You will insert into this Experiment the individual steps which control the reaction(s) in the analysis. When you insert an experiment, you specify the Initial Conditions, then insert individual steps. Each step can be customized in detail.

There are also three specialized experiments which can be inserted:

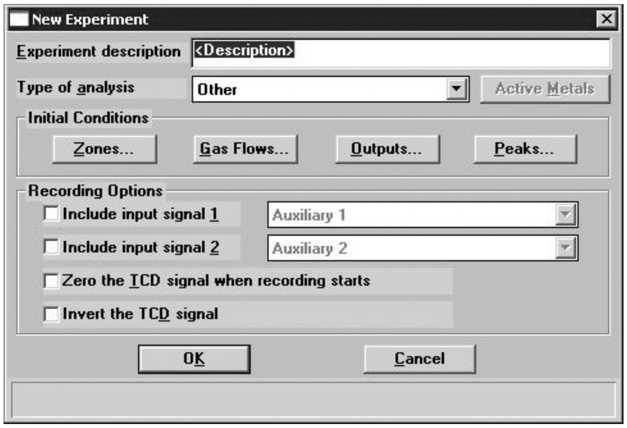
- Loop Calibration
- Gas Calibration
- Vapor Calibration

The sequence of steps in the three specialized experiments cannot be altered.

Data from one experiment (of any of these four types) are not available for editing until the next experiment in the analysis has begun recording.

Experiment

Inserts a basic experiment into the list of steps.



New Experiment

Experiment description: <Description>

Type of analysis: Other

☐ Include input signal 1 — Auxiliary 1

☐ Include input signal 2 — Auxiliary 2

☐ Zero the TCD signal when recording starts

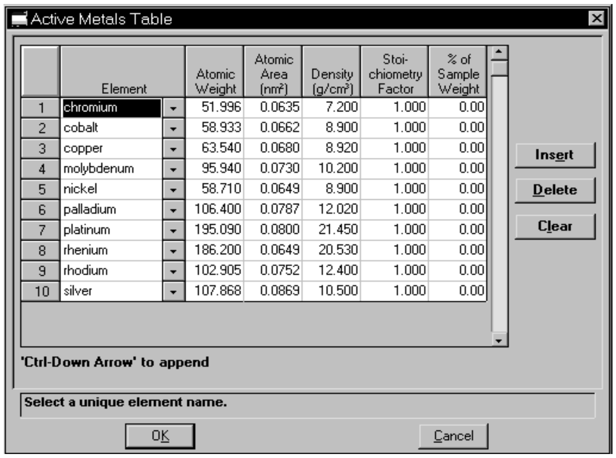
☐ Invert the TCD signal

Experiment description	Allows you to enter a description or identifier for this experiment. The description you enter in this field appears in the legend of the report.
Type of analysis	Select the type of analysis from the drop-down list *Choices: Temperature-Programmed Oxidation* *Temperature-Programmed Reduction* *Temperature-Programmed Desorption* *Physisorption Surface Area* *Pulse Chemisorption* *Other*

It is important to select the correct experiment type because the type is referenced during some other operations. For example, a Pulse Chemisorption report can only be generated for those experiments specifying *Pulse Chemisorption* as the type.

Active Metals

Opens the Active Metals Table dialog, allowing you to specify the percent of sample weight of the active metals in the sample (in the far right column), and the stoichiometry factor.



	Element	Atomic Weight	Atomic Area (nm^2)	Density (g/cm^3)	Stoi-chiometry Factor	% of Sample Weight
1	chromium	51.996	0.0635	7.200	1.000	0.00
2	cobalt	58.933	0.0662	8.900	1.000	0.00
3	copper	63.540	0.0680	8.920	1.000	0.00
4	molybdenum	95.940	0.0730	10.200	1.000	0.00
5	nickel	58.710	0.0649	8.900	1.000	0.00
6	palladium	106.400	0.0787	12.020	1.000	0.00
7	platinum	195.090	0.0800	21.450	1.000	0.00
8	rhenium	186.200	0.0649	20.530	1.000	0.00
9	rhodium	102.905	0.0752	12.400	1.000	0.00
10	silver	107.868	0.0869	10.500	1.000	0.00

'Ctrl-Down Arrow' to append

Select a unique element name.

The contents of this table are copied from the default table on the Options menu. After the sample file is created, however, the contents of this table become specific to the file. Changes made in this table do not affect the default table and changes made to the default table do not affect this table.

Insert

Inserts a new row into the table; the row is inserted above the selected one.

Delete

Deletes the selected row.

Clear

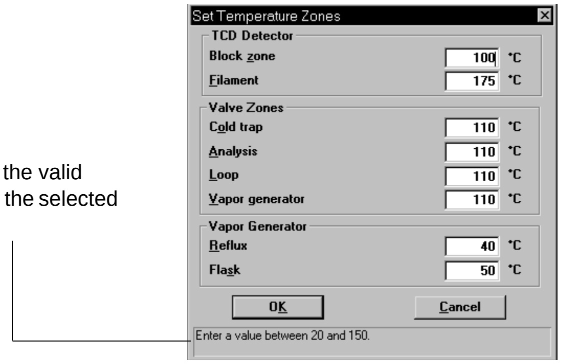
Clears the table of all entries except one; one is required.

Initial Conditions

The push buttons in this group box allow you to specify initial conditions for the analysis.

Zones

Displays the Set Temperature Zones dialog allowing you to set the heat zone temperatures.



Displays the valid range for the selected field.

Set Temperature Zones

TCD Detector
Block zone 100 °C
Filament 175 °C

Valve Zones
Cold trap 110 °C
Analysis 110 °C
Loop 110 °C
Vapor generator 110 °C

Vapor Generator
Reflux 40 °C
Flask 50 °C

Enter a value between 20 and 150.

Valid ranges for the selected field are displayed in the information bar.

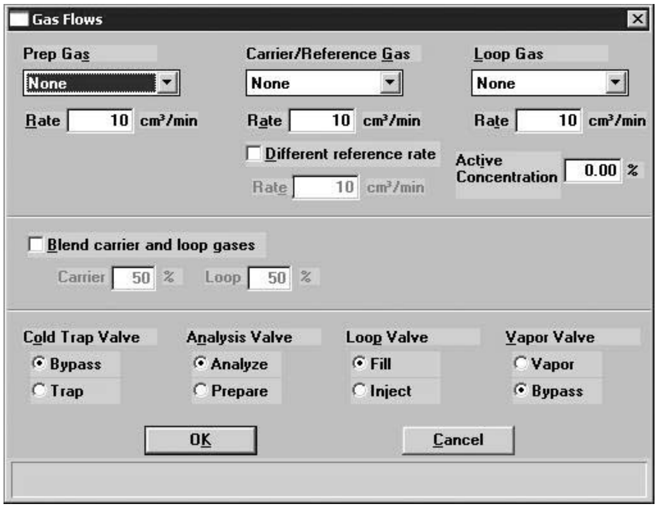
The **Filament** temperature must be set at least 20 ºC higher than the **Block zone** temperature.

The **Reflux** must be at least 10 °C cooler than the **Flask** zone.

If the optional Vapor Generator is not installed when the analysis is started, the Vapor Generator fields are ignored by the instrument when you start the analysis.

Gas Flows

Displays the Gas Flows dialog.



Gas Flows

Prep Gas: None
Rate 10 cm³/min

Carrier/Reference Gas: None
Rate 10 cm³/min
☐ Different reference rate
Rate 10 cm³/min

Loop Gas: None
Rate 10 cm³/min
Active Concentration 0.00 %

☐ Blend carrier and loop gases
Carrier 50 % Loop 50 %

Cold Trap Valve: ● Bypass ○ Trap
Analysis Valve: ● Analyze ○ Prepare
Loop Valve: ● Fill ○ Inject
Vapor Valve: ○ Vapor ● Bypass

Gas Flows
(continued)

This dialog allows you to choose the gases you wish to use for this step, specify the flow rates, specify the percent of the carrier gas mixture which is composed of the reactive, and set the state of the rotary valves.

Prep Gas
Carrier/Reference Gas
Loop Gas

For each set of inlet ports, you may select a gas from the drop-down list.

Rate: *0 to 175* cm^3*/min*

The gases that appear in each drop-down list are those specified in **Gas Defaults** (accessed from the Options menu). Also contained in these lists are two special selections: None and Manual Mixer. Select **None** when no gas is to be flowing (the flow rate is ignored). Select **Manual Mixer** if an external gas blender is to be used (the flow rate displayed in the dialog is ignored and, instead, is controlled by the external blender). As with other selections, the manual mixer must be configured to an inlet port from the Gas Selections dialog of the Unit menu.

When you start an analysis, the system checks to ensure that the selected gases are connected to the appropriate ports. If there is a discrepancy between a gas selected for the current sample file and the gas indicated in the Gas Selections dialog, an error message is displayed when you attempt to begin the analysis.

Make sure the appropriate gases are attached to the appropriate ports, and that the Gas Selections dialog accurately shows which gas is connected to each port.

Different reference rate

The carrier gas flows through two separate paths through the analyzer: through the path that contains the sample tube, and through the reference path. The reference path flows directly from the inlet over the reference detector, making it possible for the detector to detect variations in the gas that traveled through the sample tube (Carrier) path.

The same flow rate is typically used for both the Carrier and Reference Gas. If you wish to use different flows in these two paths, select **Different reference rate**, then specify the flow you wish to use for the reference gas path in the **Rate** field *below* the check box. (The **Rate** field *above* the check box determines the Carrier gas flow rate.)

Active Concentration	Specify the percent of the gas mixture which is composed of the reactive gas versus an inert filler. This value is used for pulse chemisorption data reduction.
Blend carrier and loop gases	Select this option to have the carrier and loop gases blended. Then enter the percentage for the gases. Whichever field you make an entry in, the other field automatically defaults to a percentage totaling 100.
Cold Trap Valve **Analysis Valve** **Loop Valve** **Vapor Valve**	Choose a status for each valve. If you select **Vapor** for the status of the Vapor valve and the Vapor Generator is not attached to the instrument when the analysis is started, an error message notifying you of the discrepancy is displayed.

The following two concerns do not arise when you insert the first Experiment in the Synopsis, but they must be considered when selecting gases for subsequent experiments and for subsequent Change Gas Flows steps.

- Avoid placing incompatible gases in the analyzer in immediate sequence.

  When you change the gas flowing into the analyzer, the new gas enters the analyzer while the previous gas is still present.

  You can “purge” the previous gas before starting to flow the new gas by selecting an inert gas for the initial conditions gas in an experiment step, or by inserting an extra **Change gas flows** step in which you flow an inert gas between the flows of incompatible gases. If you do so, you must also insert a **Wait** step specifying how long to flow the inert gas. You can specify a specific time period or select one of the other **Wait** step criteria. (Each of these steps is described later in this chapter.)

- Changes in the gas cause a brief adjustment of the Mass Flow Controller(s) involved in the change.

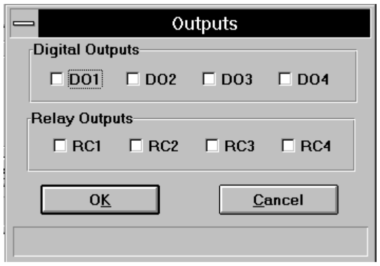
  This adjustment may be detected by the TCD, and may be visible in the data (if data are being recorded when the gas flow is changed).

  Either disregard such disturbances in the data or stop recording before you change the gas flows, then insert a Wait for stable baseline step after the Change gas flows step. Resume recording after a stable baseline is detected. A suggested sequence of steps then might be as follows:

  - Start recording
  - Experiment steps as needed
  - Stop recording
  - Change gas flows

- Wait for stable baseline
- Start recording
- Experiment steps as needed

Outputs Displays the Outputs dialog allowing you to specify the state of the digital and relay outputs.



Outputs

Digital Outputs

☐ DO1 ☐ DO2 ☐ DO3 ☐ DO4

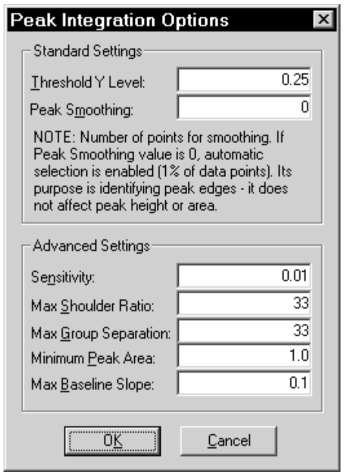
Relay Outputs

☐ RC1 ☐ RC2 ☐ RC3 ☐ RC4

Ports are provided for connecting auxiliary inputs and outputs. For example, the digital outputs can be used to control a mass spectrometer.

Refer to Appendix E, page **E-1** for a complete description of the auxiliary input/output function.

Peaks Allows you to control peak detection during analysis. After analysis, you can further control peak detection using the Peak Editor, described in Chapter 7.



Peak Integration Options

Standard Settings

Threshold Y Level: 0.25

Peak Smoothing: 0

NOTE: Number of points for smoothing. If Peak Smoothing value is 0, automatic selection is enabled (1% of data points). Its purpose is identifying peak edges - it does not affect peak height or area.

Advanced Settings

Sensitivity: 0.01

Max Shoulder Ratio: 33

Max Group Separation: 33

Minimum Peak Area: 1.0

Max Baseline Slope: 0.1

The TCD detects and records all deviations from baseline, but only those which satisfy the criteria established in this dialog are reported as peaks.

The defaults for the parameters on this dialog are usually acceptable. A complete explanation of each parameter on this dialog is contained in Appendix H, page H-1.

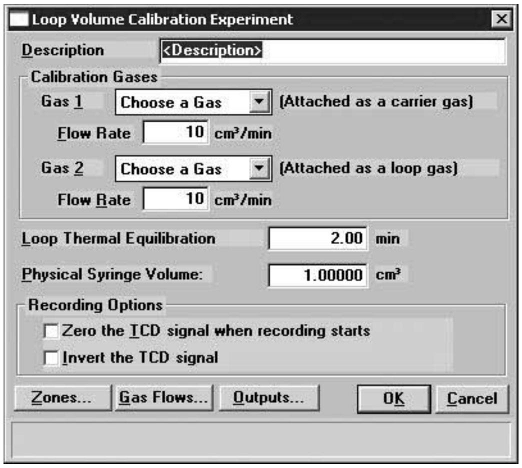
Recording Options

Include input signal [n]	These options allow you to use external electrical input from an auxiliary port, or you can choose to report an instrument-specific parameter from the drop-down list. Refer to Appendix E, page **E-1** for additional information on auxiliary input/outputs.
Zero the TCD signal when recording starts	Allows you to zero the TCD signal automatically the first time recording starts in an experiment.
Invert the TCD signal	Inverts the TCD signal. For example, if you expect to obtain only negative peaks, this option can be used to record the peaks in the positive direction.

Loop Calibration

Loop Calibration enables you to calibrate the gas injection loop. This experiment verifies the exact volume of the loop for use in calculations on analyses in which the loop is used. Sample analysis data yield signal vs. temperature data and peak areas. Associating the sample information file with a loop calibration file makes it possible for the software to convert sample data to volume values.

Appendix J provides a tutorial for performing a loop calibration.


Loop Volume Calibration Experiment

Description: <Description>

Calibration Gases

Gas 1: Choose a Gas (Attached as a carrier gas)

Flow Rate: 10 cm³/min

Gas 2: Choose a Gas (Attached as a loop gas)

Flow Rate: 10 cm³/min

Loop Thermal Equilibration: 2.00 min

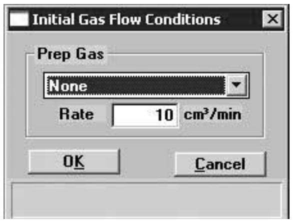
Physical Syringe Volume: 1.00000 cm³

Recording Options

☐ Zero the TCD signal when recording starts

☐ Invert the TCD signal

Description	Enter an identifier for this experiment. It is important to use a descriptive identifier here, in order to easily identify this data when displayed in sample reports.
Calibration Gases	Allow you to select the carrier and loop gases to be used in this calibration. The gases available are those specified in the **Gas defaults** accessed from the **Options** menu.
Flow Rate	Specify the flow rate for the loop and carrier gases. Typically, the default of 10 cm^3/min is appropriate.
Loop thermal equilibration delay	Allows you to specify the duration of the equilibration delay. This delay allows extra time for the instrument to stabilize thermally after the baseline has fully stabilized.
Physical Syringe Volume	Enter the volume of gas to be injected from the syringe you are using. For example: enter 0.45 for a 0.5 cm^3 loop, 0.95 for a 1.0 cm^3 loop, and 4.5 for a 5.0 cm^3 loop.
Zero the TCD signal when recording starts	Select this option to zero the TCD signal automatically the first time recording starts in an experiment.
Invert the TCD signal	Inverts the TCD signal. For example, if you expect to obtain only negative peaks, this option can be used to record the peaks in the positive direction.
Zones **Outputs**	These buttons function in the same manner as those described earlier in **Experiment**.
Gas Flows	Enables you to specify the Preparation gas and its flow rate. This feature is useful if you will have a sample loaded while calibration is occurring.



Prep Gas: None

Rate: 10 cm^3/min

Gas Flows
(continued)

When you start an analysis, the software checks to be sure that the gases you select are connected to the appropriate ports by comparing them to the Gas Selections dialog of the Unit menu. If there is a discrepancy between a selected gas and the gas indicated in the Gas Selections dialog, an error message is displayed when you attempt to begin the analysis.

Make sure the appropriate gases are attached to the appropriate ports, and that the Gas Selections dialog accurately shows which gas is connected to each port.

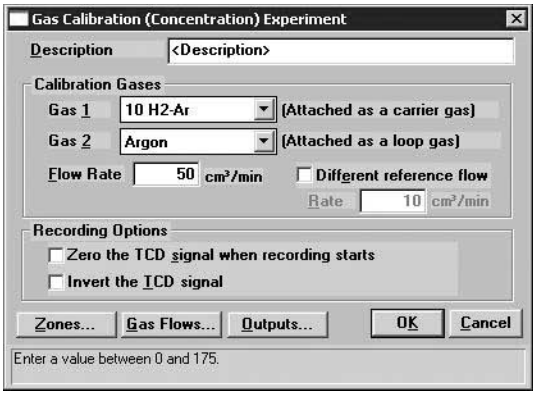
Enter a flow rate in the **Rate** field.

Range: 0 to 175 cm^3*/min.*

Gas Calibration

The Gas Calibration experiment enables you to calibrate the Thermal Conductivity Detector so that peak area data can be converted to volume data. During a Gas Calibration, a series of known gas mixtures flows through the analyzer and the resultant signal readings are recorded. The analyzer can then use these data to calculate the concentrations of unknown mixtures flowing past the detector during subsequent analyses.

Appendix J provides a tutorial for performing a gas calibration (page J-42.)



Description: <Description>

Gas 1: 10 H2-Ar (Attached as a carrier gas)

Gas 2: Argon (Attached as a loop gas)

Flow Rate: 50 cm³/min

☐ Different reference flow

Rate: 10 cm³/min

☐ Zero the TCD signal when recording starts

☐ Invert the TCD signal

Enter a value between 0 and 175.

Description

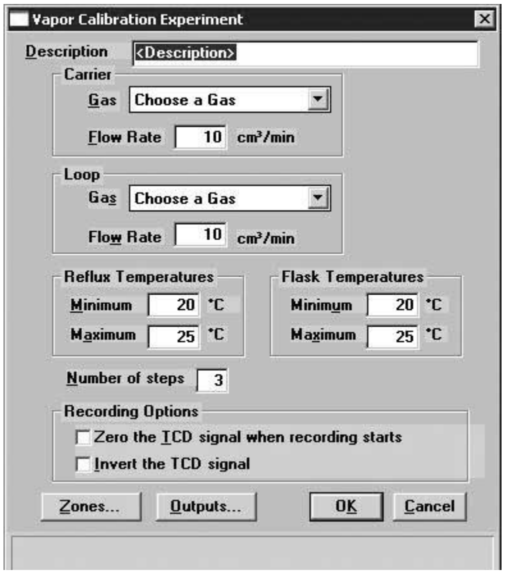
Enter an identifier for this experiment. It is important to use a descriptive identifier here, in order to easily identify this file when it is associated with sample files.

Calibration Gases	These drop-down lists allow you to select the Carrier (**Gas 1**) and Loop (**Gas 2**) gases to be used in this calibration. The gases available are those specified in the **Gas defaults** list on the **Options** menu. Refer to Appendix C for gas combinations.
Flow Rate	This field allows you to specify a flow rate for the calibration gases. Typically, the same flow rate is used for both the Carrier and Reference gas. If you wish to use different flows, select **Different reference flow**. *Range: 0 to 175 cm*^{3}*/min*
Different reference flow	This option allows you to use different flows for the calibration gases. When you select this option, the Rate field directly below is enabled so that you can enter a different flow. This flow is used for the reference gas; the carrier gas will use the flow specified in the **Flow Rate** field (explained previously). The Carrier gas flows through the path that contains the sample tube, and through the reference path. The reference path flows directly from the inlet over the reference detector, making it possible for the detector to detect variations in the gas that traveled through the sample tube (Carrier) path. *Range: 0 to 175 cm*^{3}*/min*
Zero the TCD signal when recording starts	Select this option to zero the TCD signal automatically the first time recording starts in an experiment.
Invert the TCD signal	Select this option to invert the TCD signal. For example, if you expect to obtain only negative peaks, this option can be used to record the peaks in the positive direction.
Zones **Gas Flows** **Outputs**	These buttons function in the same manner as those described earlier in the **Experiment** section beginning on page **5-15**.

Vapor Calibration

A separate calibration of the Thermal Conductivity Detector TCD must be made when vapor is to be used during analysis. The Vapor Calibration experiment enables you to calibrate the TCD so that peak area data can be converted to volume data. During a Vapor Calibration, a series of known vapors at specified temperatures is flowed through the analyzer and the resultant signal readings are recorded. The analyzer can then use this data to calculate the unknown concentrations of vapors flowing past it during subsequent analyses.

Appendix J provides a tutorial for performing a vapor calibration, page J-45.


Vapor Calibration Experiment

Description: <Description>

Carrier
Gas: Choose a Gas
Flow Rate: 10 cm³/min

Loop
Gas: Choose a Gas
Flow Rate: 10 cm³/min

Reflux Temperatures
Minimum: 20 °C
Maximum: 25 °C

Flask Temperatures
Minimum: 20 °C
Maximum: 25 °C

Number of steps: 3

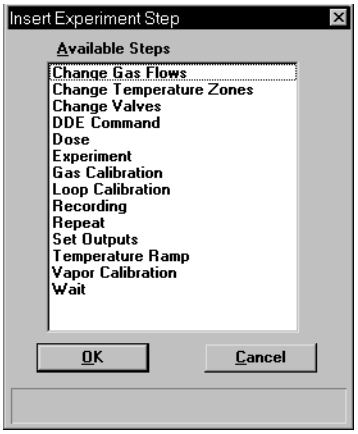
Recording Options
☐ Zero the TCD signal when recording starts
☐ Invert the TCD signal

Description	Enter an identifier for this experiment. It is important to use a descriptive identifier here, in order to easily identify this file when it is associated with sample files.
Carrier group box	Choose the carrier gas you wish to use and enter the rate at which it is to flow. *Range: 0 to 175 cm^3/min*
Loop group box	Choose the loop gas you wish to use and enter the rate at which it is to flow. *Range: 0 to 175 cm^3/min*

Reflux and Flask Temperatures	Enter a minimum (initial) and maximum (final) temperature for each zone. *Range: 20 to 150 °C*
Number of steps	Allows you to enter the number of temperature target adjustments you wish to have performed for the reflux and flask heat zones. Data will be collected for a period of time at each step. The temperature increment is derived using the following formula: $Temperature\ \text{increment} = \frac{T_{max} - T_{min}}{Number\ of\ \text{steps}\ - 1}$ For example, say the minimum temperature is 60, the maximum is 120, and you have entered 5 steps. $\frac{120 - 60}{5 - 1} = 15\ degrees\ between\ each\ step$ The resulting target temperatures will be 60 (the minimum and first step), 75, 90, 105, and 120.
Zero the TCD signal when recording starts	Select this option to zero the TCD signal automatically the first time recording starts in an experiment.
Invert the TCD signal	Select this option to invert the TCD signal. For example, if you expect to obtain only negative peaks, this option can be used to record the peaks in the positive direction.
Zones **Outputs**	These buttons function in the same manner as those described earlier in the **Experiment** section.

Insert Experiment Step

After you have set up the initial conditions for your basic Experiment, the Analysis Conditions dialog is again displayed showing the Experiment inserted. Clicking now allows you to insert a step in the Experiment you just added to the Synopsis. You can insert steps in a wide variety of sequences. You can also use steps repetitively within the Experiment.



Unlike the basic Experiment, specialized experiments are pre-programmed so that you can insert them in your analysis as a single step. There are three specialized experiments for calibration: Loop Calibration, Gas Calibration, and Vapor Calibration. Each of them automatically performs a complex series of actions.

You cannot change the steps included in a specialized experiment, but you can include specialized experiments in analyses that contain other experiments. For example, you can include a Loop Calibration experiment in an analysis that also contains a Pulse Chemisorption experiment.

Change Gas Flows

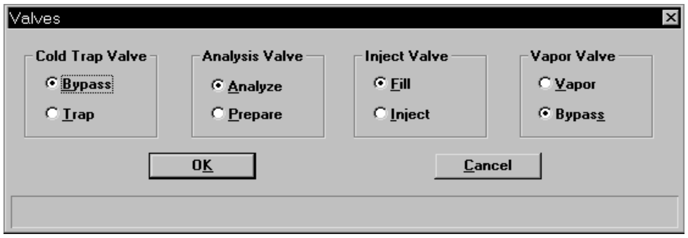
Displays the Gas Flows dialog. This dialog is identical to the one that is opened when **Gas Flows** is selected from the Insert Experiment Step dialog. A complete description of this dialog is found on page 5-17.

Change Temperature Zones

Displays the Set Temperature Zones dialog. This dialog is identical to the one that is opened when **Zones** is selected from the Insert Experiment Step dialog. A complete description of this dialog is found on page 5-17.

Change Valves

Displays the Valves dialog, allowing you to choose the state in which each of the rotary valves is to be placed.



Cold Trap Valve: ● Bypass ○ Trap

Analysis Valve: ● Analyze ○ Prepare

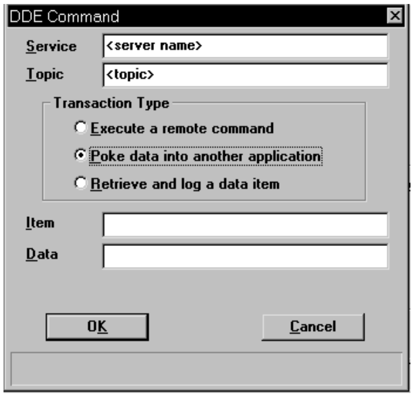
Inject Valve: ● Fill ○ Inject

Vapor Valve: ○ Vapor ● Bypass

If you select **Vapor** for the Vapor Valve and you do not have the Vapor Generator attached, an error message notifying you of the discrepancy is displayed.

DDE Command

The DDE (Dynamic Data Exchange) Command experiment step enables the 2920 application to communicate with another DDE-aware application, such as a mass spectrometer or blender program, running on the computer.



Service: <server name>

Topic: <topic>

Transaction Type:
○ Execute a remote command
● Poke data into another application
○ Retrieve and log a data item

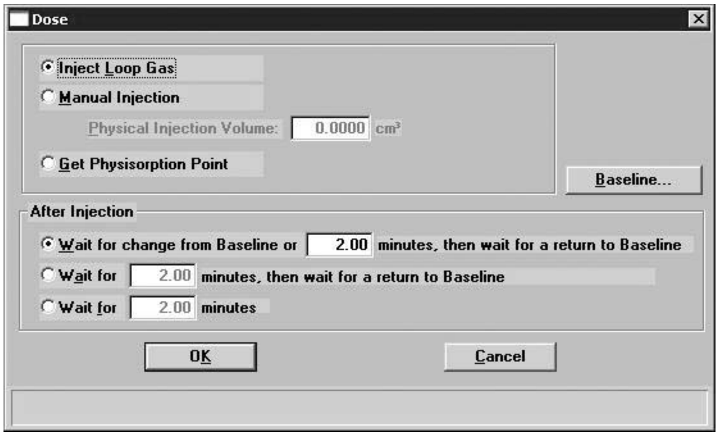
Item:

Data:

Service	Enter the name of the service provided by the DDE-aware application (refer to the DDE server application’s user manual).
Topic	A service will contain one or more topics as described in the application’s user manual. Enter the desired topic.
Transaction Type	Enables you to choose a type of transaction.

Execute a remote command	Select this option to execute the command you enter in the Data field.
Poke data into another application	Select this option to send the text string you enter in the Data field to the specified application.
Retrieve and log a data item	Select this option to add the response to the transaction you specify in the Item field to the analysis log.
Item	If you selected **Retrieve and log a data item** or **Poke data into another application**, enter the name of the transaction (refer to the DDE server application’s user manual).
Data	If you selected **Execute a remote command** or **Poke data into another application**, enter the command or data as a text string.

Dose



● Inject Loop Gas
○ Manual Injection
Physical Injection Volume: 0.0000 cm³
○ Get Physisorption Point

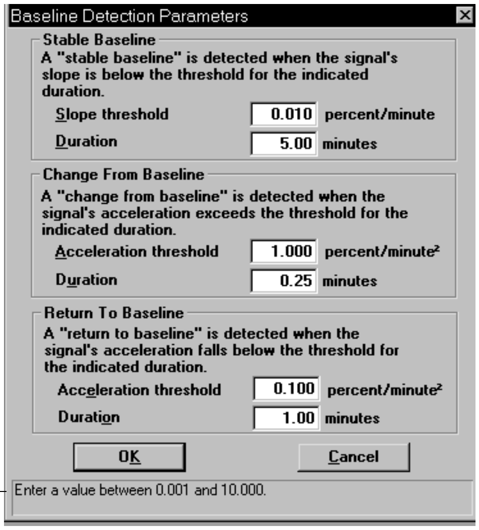
After Injection
● Wait for change from Baseline or 2.00 minutes, then wait for a return to Baseline
○ Wait for 2.00 minutes, then wait for a return to Baseline
○ Wait for 2.00 minutes

When the analysis is performed, pay close attention to the instructions given in prompts that appear on the screen. Perform the prompted steps in the order given.

Dosing Choices	Mark a radio button to select the appropriate dosing method for this experiment step.
Inject Loop Gas	Automatically injects the contents of the loop into the path that leads to the sample. The contents of the loop are pushed out of the loop by the Carrier Gas.
Manual Injection	Prompts you to inject a dose of gas into the septum using a syringe.
Get Physisorption Point	Prompts you through the steps necessary to collect data for a physisorption point. These steps include placing a dewar of liquid nitrogen around the sample tube, waiting for a return to baseline, then replacing the dewar with water at room temperature. You can take repeated points by placing the Dose step within a Repeat Loop (discussed later in this chapter).
After Injection	Allows you to specify the conditions for completion of this step.

Baseline

Click to specify Baseline settings if you have selected a Wait step that depends upon the Baseline.


Baseline Detection Parameters

Stable Baseline

A "stable baseline" is detected when the signal's slope is below the threshold for the indicated duration.

Slope threshold 0.010 percent/minute

Duration 5.00 minutes

Change From Baseline

A "change from baseline" is detected when the signal's acceleration exceeds the threshold for the indicated duration.

Acceleration threshold 1.000 percent/minute^2

Duration 0.25 minutes

Return To Baseline

A "return to baseline" is detected when the signal's acceleration falls below the threshold for the indicated duration.

Acceleration threshold 0.100 percent/minute^2

Duration 1.00 minutes

Enter a value between 0.001 and 10.000.

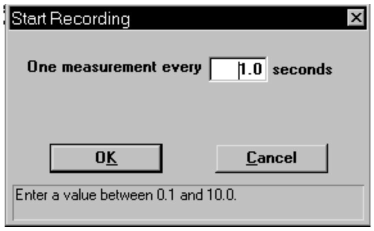
Displays the range for the selected field.

Establish the **Slope** or **Acceleration threshold** and **Duration** for determining what constitutes a **Stable Baseline**, a **Change From Baseline**, and a **Return To Baseline**. These values control whether a particular change in the signal is significant to the current experiment; in other words, they define a stable baseline.

Some Wait steps are contingent upon the values you select here. For example, if your experiment contains a **Wait until Baseline is stable** step, the signal is compared to these values to determine if a stable baseline has been established. Lower slope/acceleration values and longer durations create a more rigorous definition of these factors than higher values and shorter durations. Normally the default values are satisfactory.

The range for each field on this dialog is displayed in the information bar at the bottom of the dialog when the field is selected.

Recording



Enter a value between 0.1 and 10.0.

Allows you to specify how frequently the signal reading is recorded.

Range: 0.1 to 10.0 seconds

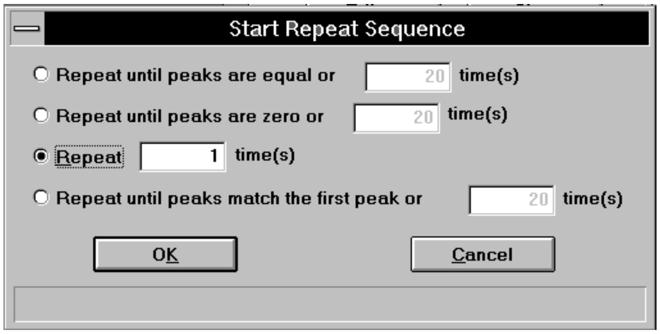
Recommended values are 1.0 for TPR, TPD, and TPO experiments, and 0.2 for pulse chemisorption.

The **Start Recording** dialog starts the recording of the signal for this analysis. A **Stop Recording** step is inserted in the list of steps automatically on the Analysis Conditions dialog when you insert a **Start Recording** step.

If a Start Recording step is immediately followed by a step that prompts an immediate peak, peak data are recorded before any baseline readings can be collected. To collect some baseline data before the first peak in such an experiment, insert a Wait for ____ minutes step after the Start Recording step, but before the step which causes the peak.

Repeat

When you insert a Repeat step in your analysis conditions, the Start Repeat Sequence dialog opens allowing you to specify the duration of the repeat sequence. After you complete the Start Repeat Sequence dialog and click **OK**, a Start Repeat step and a Stop Repeat step are automatically inserted in the list of steps for this analysis. You may insert multiple experiment steps within the Repeat loop.



Start Repeat Sequence

○ Repeat until peaks are equal or 20 time(s)

○ Repeat until peaks are zero or 20 time(s)

● Repeat 1 time(s)

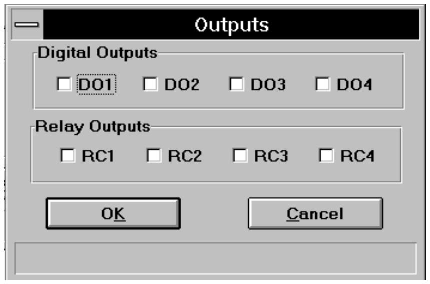
○ Repeat until peaks match the first peak or 20 time(s)

Repeat until peaks are equal or ____ times.	Stops repeating the steps within the loop when the last four peaks of this experiment have equal volumes or when the maximum number of repeats is reached. (Equal means the last four peaks are within 15% of their average, and the average of the last four peaks is larger than the first peak or the average of the last four peaks is larger than $\frac{N+5}{N}$ where N is the number of peaks.) This option is useful when performing H_2 or CO pulse chemisorption on supported metal catalysts.
Repeat until peaks are zero or ____ times.	Stops repeating the steps within the loop when the last four peaks of this experiment have zero volume or when the maximum number of repeats is reached. (Zero volume is defined in the software as four peaks with volumes within 10% of each other and all volumes less than 50% of the first peak.) This option is useful when performing an N_2O decomposition for characterizing copper catalysts.

Repeat _____ times.	Stops repeating the steps within the loop when the specified number of times is reached. *Range: 1 to 200 times*
Repeat until peaks match the first peak or _____times.	Stops repeating the steps within the loop when the peaks match the first peak or when the maximum number of repeats is reached. *Range: 1 to 200 times*

Set Outputs

When this step is selected, the Outputs dialog opens. Select each signal you wish to enable. A complete discussion of the auxiliary input/output options is contained in Appendix E, page **E-1**.



Outputs

Digital Outputs

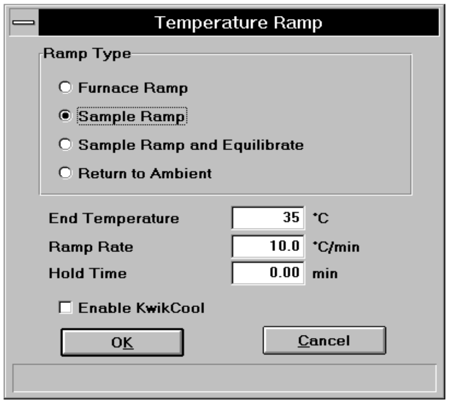
☐ DO1 ☐ DO2 ☐ DO3 ☐ DO4

Relay Outputs

☐ RC1 ☐ RC2 ☐ RC3 ☐ RC4

Temperature Ramp

Insert this step to change the sample temperature.



Temperature Ramp

Ramp Type

○ Furnace Ramp
● Sample Ramp
○ Sample Ramp and Equilibrate
○ Return to Ambient

End Temperature 35 °C
Ramp Rate 10.0 °C/min
Hold Time 0.00 min

☐ Enable KwikCool

Furnace Ramp	Ramps the furnace temperature directly to the End temperature, ignoring the sample temperature.

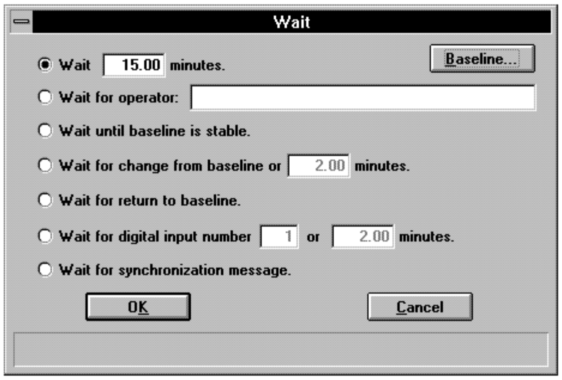
Sample Ramp	Ramps the sample temperature to the End temperature. The actual furnace temperature is adjusted to meet this target.
Sample Ramp with Equilibration	Ramps the sample temperature to the End temperature and waits for it to equilibrate before proceeding.
Return to Ambient	Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature between 14 and 50 °C.
End Temperature	Enter the ending temperature for this ramping procedure. *Range: -120 to 1100 ºC with optional CryoCooler* *Ambient to 1100 °C without* An ending temperature below 20 °C enables the CryoCooler (if installed) automatically. This field is disabled when **Return to Ambient** is selected.
Ramp Rate	Enter the ramp rate for this ramping procedure. Range: 0.1 to 90.0 ºC/min This field is disabled when **Return to Ambient** is selected.
Hold Time	Enter the amount of time to hold the temperature at the conclusion of this step. If Equilibrate is selected, the hold time is not started until the temperature is equilibrated. *Range: 0.00 to 720.00 min* This field is disabled when **Return to Ambient** is selected.

If you are ramping from subambient temperatures and the CryoCooler is installed, the target temperature should be held for a minimum of 15 minutes. This ensures that the entire furnace has reached thermal equilibrium before the ramp begins. Otherwise it may take several minutes for the temperature ramp rate to stabilize.

Enable KwikCool	Select this to use the KwikCool to reduce the furnace temperature more rapidly during cool-down. If this option is checked and the KwikCool is not attached to the analyzer when the analysis is started, an error message is displayed. If the CryoCooler (rather than the KwikCool) is attached, the cool-down operation is performed by the CryoCooler.

Wait

Allows you to specify a waiting routine.



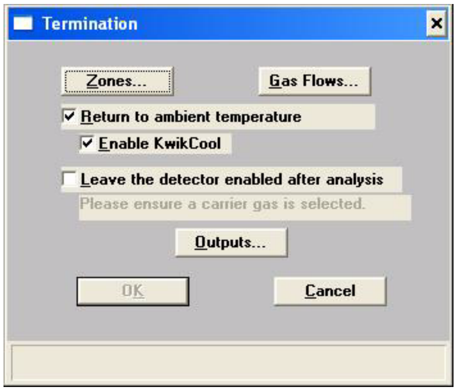
● Wait 15.00 minutes.
○ Wait for operator:
○ Wait until baseline is stable.
○ Wait for change from baseline or 2.00 minutes.
○ Wait for return to baseline.
○ Wait for digital input number 1 or 2.00 minutes.
○ Wait for synchronization message.

Wait ____ minutes.	Specify an exact number of minutes to wait. *Range: 0.01 to 720.00 minutes*
Wait for operator	Enter a description of the operator's task, such as "Fill the dewar, then click **OK**." During an analysis, the message you enter is displayed on the monitor at the appropriate time and the attention LED on the upper front panel will flash or blink. The analysis continues after the operator clicks **OK**. Be sure to specify whether the operator should first perform the task or first click **OK**.
Wait until Baseline is stable.	Waits until the baseline becomes stable. If you choose this option, click **Baseline** to specify the settings.

Wait for change from Baseline or ____ minutes.	Enter the number of minutes to wait, then click **Baseline** to specify the settings. *Range: 0.01 to 720.00 minutes*
Wait for return to Baseline.	Waits for a return to Baseline. If you choose this option, click **Baseline** to specify the settings.
Wait for digital input number ______ or ____ minutes.	Enter the number of the digital input source being awaited, then specify the maximum number of minutes to wait for input. If the digital input is not received before the time elapses, the analysis will continue, and a warning message is displayed. Digital inputs are electrical connections to the right side of the instrument. See Appendix E, page **E-1** for additional information. *Range: Digital input 1 to 4* *0.01 to 720.00 minutes*
Baseline	Click to specify Baseline settings if you have selected a Wait step that depends upon the Baseline. This dialog is described on page **5-31**.
Wait for synchronization message.	Select this option to direct the 2920 software to act as a DDE server. When the 2920 program receives a message from another DDE application (the client), this Wait will be completed and logged into the sample log. The DDE client must use the following parameters in an Execute a Remote Command in order to provide the synchronization message. *Service WIN2920* *Topic Command* *Item Sync*

Termination

This step is automatically inserted in every experiment. The contents of this step are the post-analysis conditions specified using Sample defaults on the Options menu. You can view and/or edit the details of this dialog by clicking on the **Termination** step when it is selected (highlighted); the Termination dialog is displayed.



☑ Return to ambient temperature

☑ Enable KwikCool

☐ Leave the detector enabled after analysis

Please ensure a carrier gas is selected.

Zones	Displays the Set Temperature Zones dialog allowing you to view or edit current temperature zones. Refer to page 5-17 for information on this dialog.
Gas Flows	Displays the Gas Flows dialog allowing you to view or edit current gasses and their flows. Refer to page 5-17 for information on this dialog.
Return sample to ambient temperature	Select this option to have the sample cooled to ambient temperature.
Enable KwikCool	Select this option to enable the KwikCool during the cool-down operation. If the CryoCooler rather than the KwikCool is attached, the cool-down operation is performed by the CryoCooler.
Leave the detector enabled after analysis	Several hours may be required for the instrument to reach thermal stability after the detector is enabled. This option keeps the detector enabled after analysis, allowing the instrument to remain stable. It is not necessary to have gas flowing through the detector, but it is good practice to use an inert gas to continuously purge the system.
Outputs	Displays the Outputs dialog allowing you to view or edit the digital and relay outputs. Refer to page 5-20 for additional information on this dialog.

Report Options

This dialog is used to edit existing report options, or to specify report options for a new file.

When creating parameter files to display in the drop-down list on the Basic sample information dialog, be sure to save them to the directory specified as the Parameter File directory (see Chapter 8).

Report Options: Default Report Options

☑ Show report title: Report Title

☑ Show graphic: Micro.bmp

Width 2.000 in. Height 0.250 in.

Selected Reports

- Summary
- Tabular Report
- BET Surface Area
- ✓ Langmuir Surface Area
- ✓ Total Pore Volume
- Pulse Chemisorption
- First Order Kinetics
- Loop Calibration
- Analysis Log
- ✓ TCD Signal vs. Time
- ✓ Temperature vs. Time

Report options

If you are editing an existing file, this field displays the description or identification of the report options file. This description can be edited if desired.

If this is a new file, this field contains the description specified in Sample defaults. You can enter a new description or add to the existing one if desired. Use a description that will help you identify this set of report options in the future.

Range: 40 alphanumeric characters

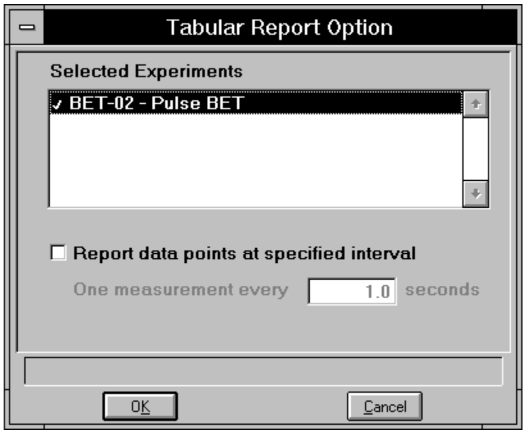
Replace

Allows you to copy values from an existing file to the one you are creating. A dialog is displayed so that you may choose the file containing the values you wish to copy. Since the values are copied into the new file, you can edit them without changing values in the original file.

Show report title	This option allows you to have a heading appear on your report and to enter a title for it. If this is a new file, the title you specified as the default is displayed. Accept the default title or enter a different one.
Show graphic	Select this option to have a graphic (bitmap or enhanced metafile) display above the report title. For example, you may wish to display your company logo. Click **Browse** to select the desired bitmap, then enter the Height and Width in the fields provided. This image can also be edited from the report window.
Selected Reports	Contains a list of available reports. A report is selected when it is preceded with a check mark. Select reports by double-clicking the desired report(s). Alternatively, you can highlight the desired report and press **Spacebar**. Reports are deselected in the same manner. Certain reports can be edited by highlighting the desired report and clicking **Edit**.
Edit	Allows you to edit the highlighted report. Clicking **Edit** opens the appropriate dialog box. The dialog(s) associated with each type of report are described in the following sections. The Summary Report and the Analysis Log cannot be edited.

Tabular Report Option

Clicking **Edit** when Tabular Report is selected opens the following dialog:



Selected Experiments

✓ BET-02 - Pulse BET

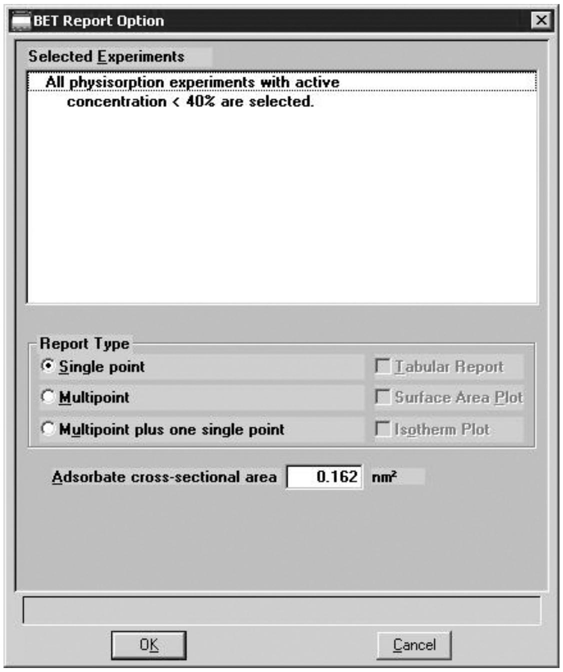
☐ Report data points at specified interval

One measurement every 1.0 seconds

Selected Experiments	All related experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected. If this is a new file, no experiments are listed. Experiments are not included in the list until data are collected.
Report data points at specified interval	Select this option to report data points at a specified interval, then specify the frequency of reported data (the number of seconds between reported data readings). This feature is useful if you anticipate a large amount of data to be collected during the analysis (such as in a lengthy analysis with readings taken every second). If you do not specify an interval for data reporting, all points are reported, regardless of the length of the report.

BET Surface Area Report Option

Clicking **Edit** when BET Surface Area is selected opens the following dialog:



Selected Experiments

All physisorption experiments with active concentration < 40% are selected.

Report Type

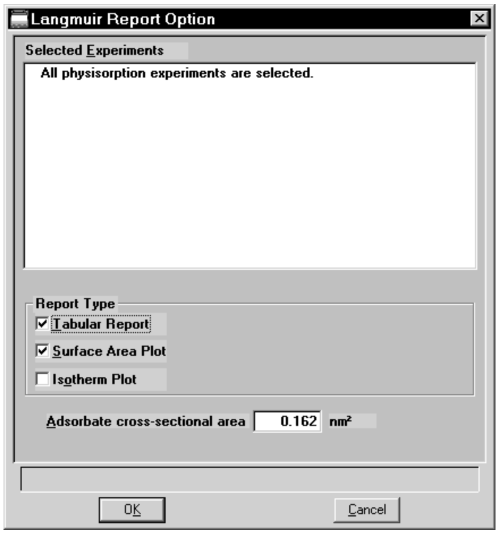
● Single point
○ Multipoint
○ Multipoint plus one single point
☐ Tabular Report
☐ Surface Area Plot
☐ Isotherm Plot

Adsorbate cross-sectional area 0.162 nm²

Selected Experiments	All related experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected. If this is a new file, no experiments are listed. Experiments are not included in the list until BET data are collected.
Report Type	From this group box, choose the type of report(s) desired. If you choose Multipoint or Multipoint plus one single point, the choices on the right side of the group box are enabled. You can choose as many of these options as desired.
Adsorbate cross-sectional area	Enter the appropriate value. The adsorbate cross-section is the area occupied by a single adsorbed molecule on the adsorbent surface.

Langmuir Surface Area Report Option

Clicking **Edit** when Langmuir Surface Area is selected opens the following dialog:



Selected Experiments

All physisorption experiments are selected.

Report Type

☑ Tabular Report

☑ Surface Area Plot

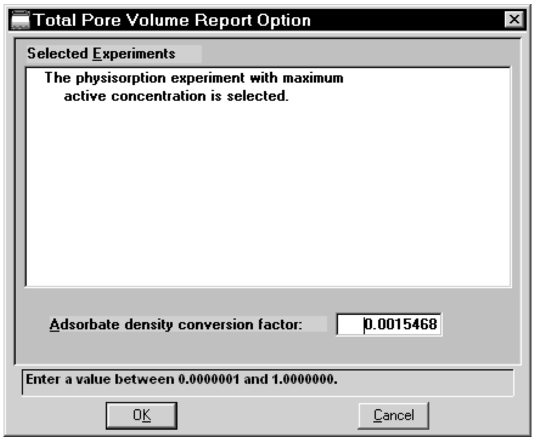
☐ Isotherm Plot

Adsorbate cross-sectional area 0.162 nm²

Selected Experiments	All related experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected. If this is a new file, no experiments are listed. Experiments are not included in the list until data are collected.
Report Type	From this group box, choose the type(s) of reports you wish to generate.
Adsorbate cross-sectional area	Enter the appropriate value. The adsorbate cross-section is the area occupied by a single adsorbed molecule on the adsorbent surface.

Total Pore Volume

Clicking **Edit** when Total Pore Volume is selected opens the following dialog:



The physisorption experiment with maximum active concentration is selected.

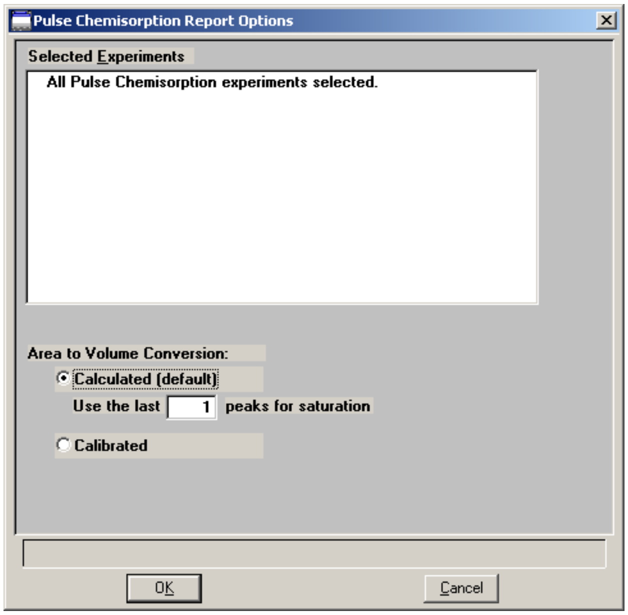
Adsorbate density conversion factor: 0.0015468

Enter a value between 0.0000001 and 1.0000000.

Selected Experiments	All related experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected. If this is a new file, no experiments are listed. Experiments are not included in the list until data are collected.
Adsorbate density conversion factor	Enter the appropriate value for converting the gas volume to liquid volume. This value can be obtained by dividing the gas density by the liquid density at the adsorption temperature. Density conversion factor = $\frac{\rho_{gas}}{\rho_l} \times \frac{T_{BATH}}{T_{STP}}$

Pulse Chemisorption Report Option

Clicking **Edit** when Pulse Chemisorption is selected opens the following dialog:



All Pulse Chemisorption experiments selected.

● Calculated (default)

Use the last 1 peaks for saturation

○ Calibrated

Selected Experiments

All related experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected.

If this is a new file, no experiments are listed. Experiments are not included in the list until related data are collected.

Area to Volume Conversion

Select **Calculated (default)** to use the standard calculation:

$$Volume = \frac{\text{volume injected} \times \text{active concentration}}{\text{area of last peak}}$$

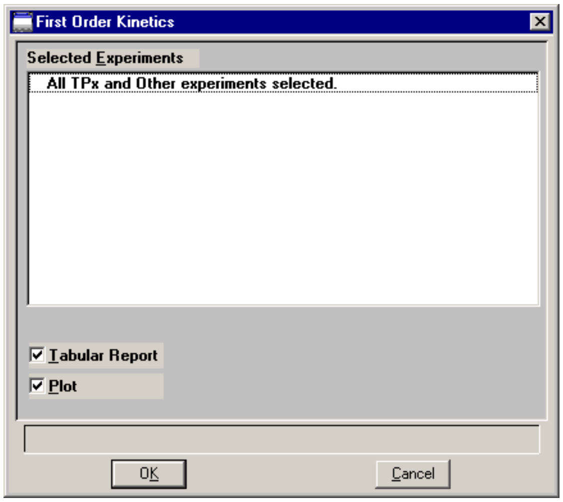
If you select this method, enter the number of peaks to be used for saturation.

This method uses the raw signal; a calibration file is not required.

Select **Calibrated** if greater flexibility is desired. This method enables you to use selected peaks for the conversion. To use this method, a calibration step must be included in the experiment in the sample information file.

First Order Kinetics Report Option

Clicking **Edit** when First Order Kinetics (heat of adsorption) is selected opens the following dialog:



All TPx and Other experiments selected.

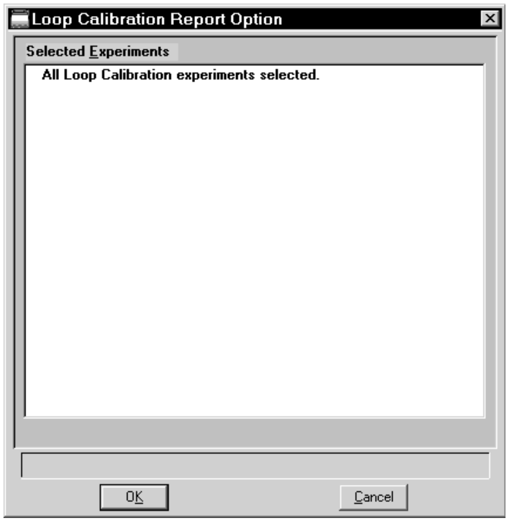
☑ Tabular Report

☑ Plot

Selected Experiments	All related experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected. However, you must include at least three experiments for the first order of kinetics to be calculated. If this is a new file, no experiments are listed. Experiments are not included in the list until related data are collected.
Tabular Report	Select this option to have a tabular (data in a table form) report generated.
Plot	Select this option to have the data plotted in a graphical format

Loop Calibration Report Option

Clicking **Edit** when Loop Calibration is selected opens the following dialog:



All Loop Calibration experiments selected.

Selected Experiments	All loop calibration experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected. If this is a new file, no experiments are listed. Experiments are not included in the list until related data are collected.

Analysis Log Report

When **Analysis Log** is selected, a log of analysis operations is included in the reports. The Analysis Log cannot be edited.

Graph Report Options

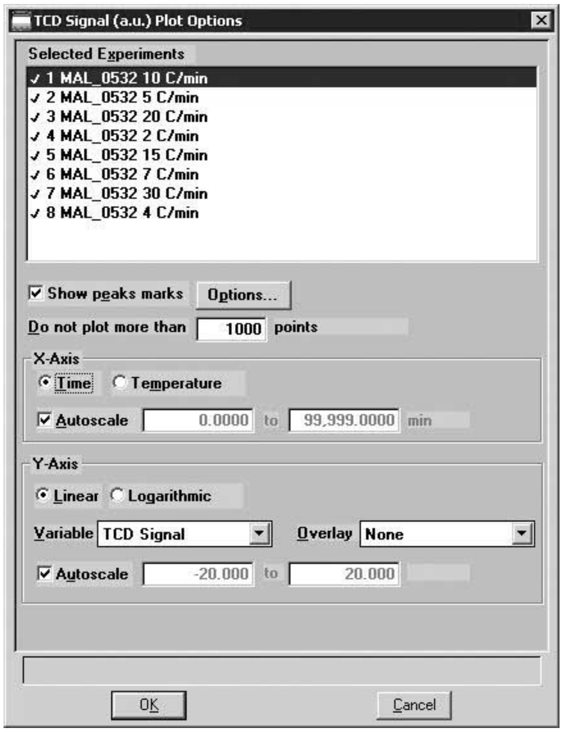
The AutoChem II 2920 program contains 10 user-defined graphs, each definable with regard to the x-axis, y-axis and y-overlay. The graphs are named as follows:

<Y-axis choice> and <Y-axis Overlay Choice> vs. X-axis choice>. For example: TCD Signal and TCD Concentration vs. Time,

Ten default files, which may be edited, are included in the program. They are:

TCD Signal vs. Time
TCD Temperature vs. Time
TCD Signal vs. Temperature
TCD Concentration vs. Time
TCD Concentration vs. Temperature
Input Signal 1 vs. Time
Input Signal 1 vs. Temperature
Input Signal 2 vs. Time
Input Signal 2 vs. Temperature
TCD Signal and TCD Concentration vs. Time

All graph report options are similar and are edited in the same manner. Click **Edit** on a selected graph report; a dialog similar to the following is displayed.



TCD Signal (a.u.) Plot Options

Selected Experiments
✓ 1 MAL_0532 10 C/min
✓ 2 MAL_0532 5 C/min
✓ 3 MAL_0532 20 C/min
✓ 4 MAL_0532 2 C/min
✓ 5 MAL_0532 15 C/min
✓ 6 MAL_0532 7 C/min
✓ 7 MAL_0532 30 C/min
✓ 8 MAL_0532 4 C/min

☑ Show peaks marks
Do not plot more than 1000 points

X-Axis
● Time ○ Temperature
☑ Autoscale 0.0000 to 99,999.0000 min

Y-Axis
● Linear ○ Logarithmic
Variable TCD Signal Overlay None
☑ Autoscale -20.000 to 20.000

Any signal (the detector signal or the 1 or 2 auxiliary signal) may be plotted against time or temperature. Any signal can be overlaid onto the primary signal.

For color output to a monitor or printer, signals are displayed in different colors. For black and white output, different symbols are used.

Selected Experiments

All experiments are selected by default. When the report options file is used in a sample information file, each experiment in the sample file is listed. Any experiment you do not wish to include in the report may be deselected.

If this is a new file, no experiments are listed. Experiments are not included in the list until related data are collected.

Show peak marks

Select this option to:

- display the areas and baselines on the graph
- draw a straight baseline between the selected peaks
- enable the **Peaks** push button, allowing you to specify the peaks to include in the experiment

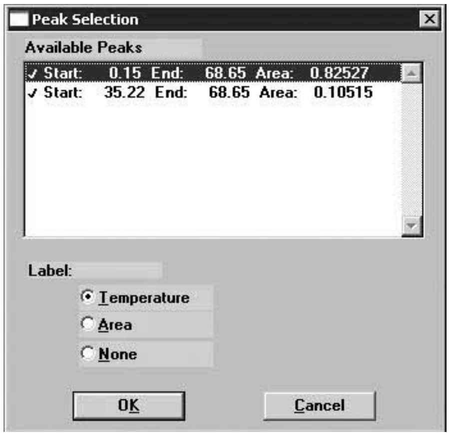
This option is disabled for new files.

Do not plot more than ____ points

Allows you to specify a maximum number of points to plot. Typically, the default of 1000 is sufficient. You can specify up to 8000 if higher resolution and more detail is desired. The speed at which graphs are plotted on some computers may be affected when large numbers of points are requested.

Options

Opens a dialog that contains a list of all the peaks for the results of the experiment highlighted in the **Experiments** list box. All peaks are selected by default; remove the check mark next to any peak you do not wish to include on the graph.



Available Peaks

✓ Start: 0.15 End: 68.65 Area: 0.82527
✓ Start: 35.22 End: 68.65 Area: 0.10515

Label:
● Temperature
○ Area
○ None

The **Label** radio buttons enable you to choose the peak label that will be displayed on graphs.

X-Axis

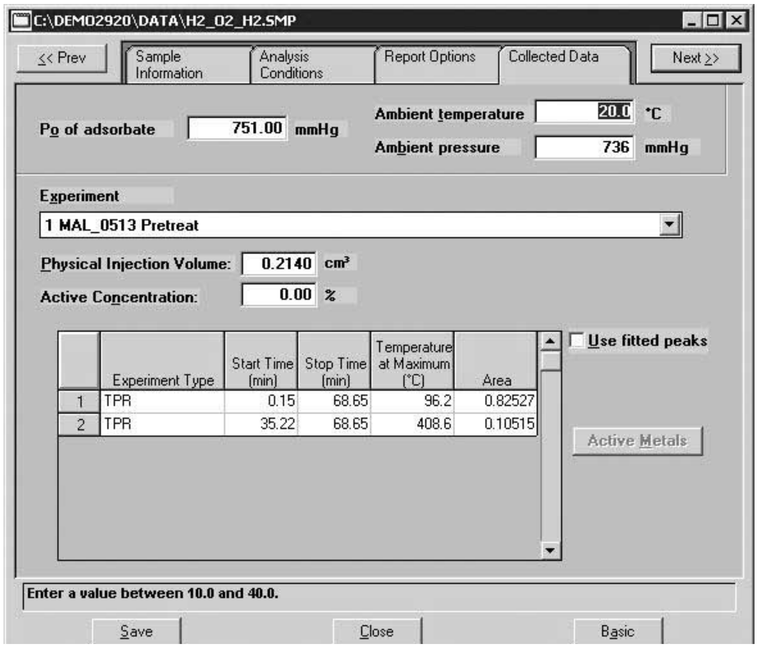
Variable	Specify **Time** or **Temperature** for the X-axis variable.
Autoscale	Select this option to have the X-Axis scaled automatically. As the default, this axis begins at zero, and the system uses the highest value collected during analysis as the ending point. The Axis **From** and **To** fields are disabled if you choose to autoscale data. If you choose not to autoscale data, you must enter from and to values. The value entered in the **To** field must be greater than the value entered in the **From** field.

Y-Axis

Variable	Select a Y-axis variable from the drop-down list.
Overlay	You can use this feature to overlay another variable with the current one. Select a variable from the drop-down list if desired.
Autoscale	Select this option to have the Y-Axis scaled automatically. This axis begins at zero by default; the system uses the highest value collected during analysis as the ending point. The Axis **From** and **To** fields are disabled if you choose to autoscale data. If you choose not to autoscale data, you must enter from and to values. The value entered in the **To** field may be less than the value entered in the **From** field if you plan to invert negative-going peaks.

Collected Data

The tab for Collected Data does not appear on the Advanced dialog presentation until after analysis.



C:\DEMO2920\DATA\H2_O2_H2.SMP

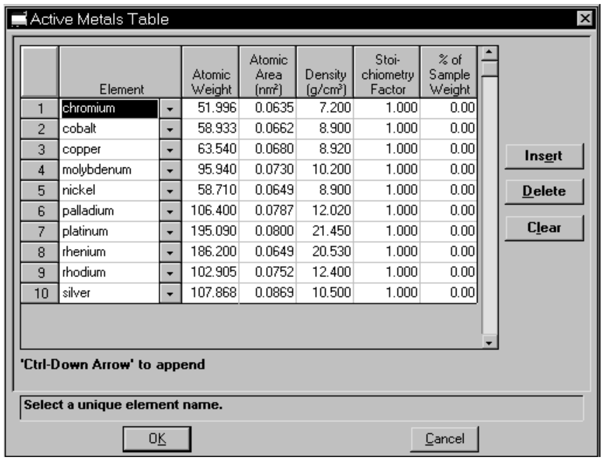
Po of adsorbate: 751.00 mmHg
Ambient temperature: 20.0 °C
Ambient pressure: 736 mmHg
Experiment: 1 MAL_0513 Pretreat
Physical Injection Volume: 0.2140 cm^3
Active Concentration: 0.00 %

	Experiment Type	Start Time (min)	Stop Time (min)	Temperature at Maximum (°C)	Area
1	TPR	0.15	68.65	96.2	0.82527
2	TPR	35.22	68.65	408.6	0.10515

Enter a value between 10.0 and 40.0.

Po of adsorbate	Displays the saturation pressure of the adsorbate.
Ambient temperature	Displays the room temperature.
Ambient pressure	Displays the room pressure.
Experiment	Defaults to the first experiment of the current file. Click the down arrow to choose other experiments in the file.
Physical injection volume	Displays the volume of the syringe used to perform the manual injections.
Active Concentration	Displays the percent of the gas mixture that is composed of the reactive gas versus an inert filler.
Peak table	Displays the peak summaries for the experiment shown in the Experiment field.

Use Fitted points	Select this option to report peak data obtained using the Fit peak option in the Peak Editor (refer to **Peak Editor** on page **7-14**).
Active Metals	Enabled for Pulse Chemisorption reports; displays the Active Metals Table dialog, allowing you to specify the percent of sample weight of the active metals in the sample (in the far right column), and the stoichiometry factor.



	Element	Atomic Weight	Atomic Area (nm^2)	Density (g/cm^3)	Stoi-chiometry Factor	% of Sample Weight
1	chromium	51.996	0.0635	7.200	1.000	0.00
2	cobalt	58.933	0.0662	8.900	1.000	0.00
3	copper	63.540	0.0680	8.920	1.000	0.00
4	molybdenum	95.940	0.0730	10.200	1.000	0.00
5	nickel	58.710	0.0649	8.900	1.000	0.00
6	palladium	106.400	0.0787	12.020	1.000	0.00
7	platinum	195.090	0.0800	21.450	1.000	0.00
8	rhenium	186.200	0.0649	20.530	1.000	0.00
9	rhodium	102.905	0.0752	12.400	1.000	0.00
10	silver	107.868	0.0869	10.500	1.000	0.00

'Ctrl-Down Arrow' to append

Select a unique element name.

The contents of this table are copied from the default table on the Options menu. After this sample file is created, however, the contents of this table become specific to the file. Changes made in this table do not affect the default table and changes made to the default table do not affect this table.

Insert	Inserts a new row into the table; the row is inserted above the selected one.
Delete	Deletes the selected row.
Clear	Clears the table of all entries except one; one is required

Save

Save enables you to save any changes made to the file in the active window. The file is saved under its current name.

Save As

Save As enables you to save the sample file or parameter file in the active window under a new name. Saving the file under a new name allows you to make modifications without changing the original file.

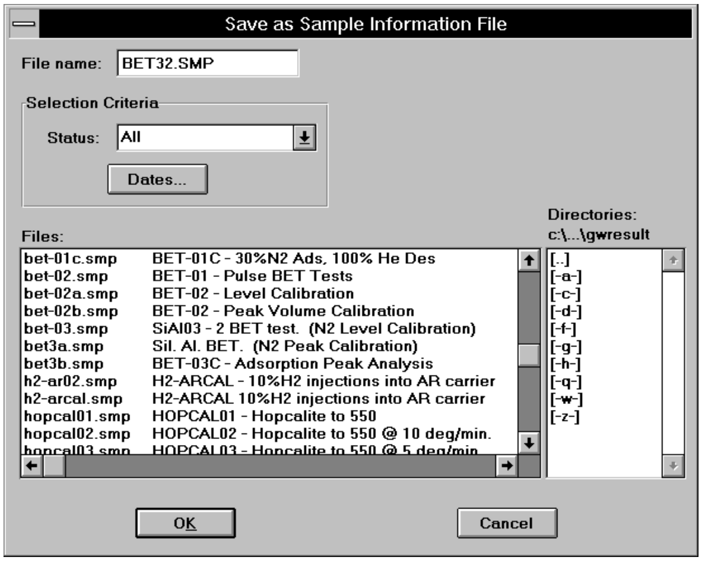
You can save a sample information file as another sample information file or as any parameter file. Parameter files, however, can be saved only as another parameter file of the same type. For example, you can save the analysis conditions portion of a Sample Information file as an analysis conditions file or the report options portion as a report options file. But an analysis conditions file can be saved only as another analysis conditions file.

Saving Parameter Files for Subsequent Use

Save As is a useful tool for making analysis conditions and report options files that were created as part of an Advanced format sample information file available to be copied to other Sample Information files. When the Analysis Conditions window is open, select **Save As**, **Analysis Conditions** (or **Report Options**, if applicable) from the File menu. Be sure to save the file to the directory specified as the Parameter Files directory (see **Parameter Files Directory** on page **8-17**) or it will not display in the drop-down list on the Basic Sample Information file editor. The default directory is PARAMS.

After you use the *Save As* function, the original file is still open, not the new file. To edit the new file, close the original file first, then open the new file.

When you select **Save As** with a sample information file open, three choices are presented: Sample Information, Analysis Conditions, and Report Options. Select the desired file type; a dialog box similar to the one shown below is displayed.



File name: BET32.SMP

Status: All

Files:

bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier
hopcal01.smp	HOPCAL01 - Hopcalite to 550
hopcal02.smp	HOPCAL02 - Hopcalite to 550 @ 10 deg/min.
hopcal03.smp	HOPCAL03 - Hopcalite to 550 @ 5 deg/min.

Directories:
c:\...\gwresult

[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

File name	Enter a name of up to 8 characters or select a file from the **Files** list box. If you select an existing file as the new name, the data contained in that file are overwritten.
Status **Files** **Directories**	These options are explained in **Selecting Files** beginning on page **3-6**.

Save All

Save All enables you to save all open files under their current names. This option provides a faster way to save all open files at one time and avoids having to perform a Save operation on each individual file.

Close

Close enables you to close the file in the active window. The following message is displayed if changes have been made to the file:

(path\file name) has been changed. Save changes before closing?

Yes **No** **Cancel**

Click **Yes** to save the changes and close the dialog box
Click **No** to discard the changes and close the dialog box
Click **Cancel** to allow the file to remain active

Close All

Close All enables you to close all open files under their current names. The following message is displayed for each file in which changes have been made:

(path\file name) has been changed. Save changes before closing?

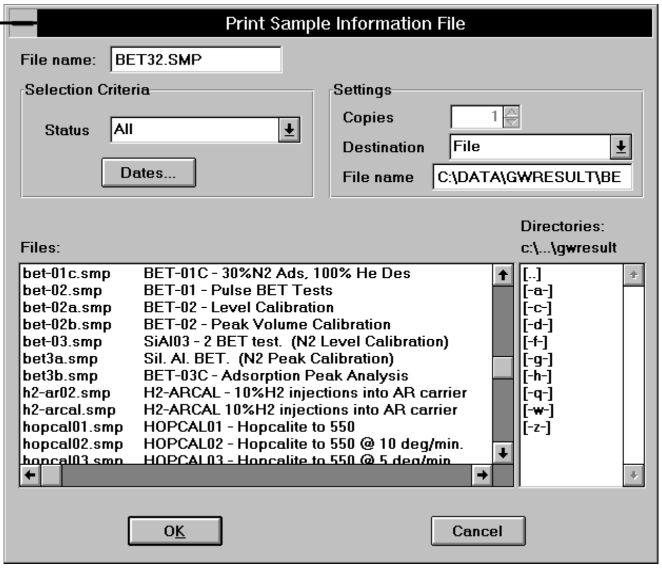
Yes **No** **Cancel**

Click **Yes** to save the changes and close the dialog box
Click **No** to discard the changes and close the dialog box
Click **Cancel** to allow the file to remain active

Print

Print enables you to print details of a file, or multiple files.

When you select **Print**, three choices are presented: **Sample information**, **Analysis conditions**, and **Report options**. Regardless of which file type you select, a dialog similar to the one shown below is displayed.



Displays the type of file requested; in this example, a Sample Information File.

File name: BET32.SMP

Status: All

Copies: 1

Destination: File

File name: C:\DATA\GWRESULT\BE

Directories:
c:\...\gwresult

bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier
hopcal01.smp	HOPCAL01 - Hopcalite to 550
hopcal02.smp	HOPCAL02 - Hopcalite to 550 @ 10 deg/min.
hopcal03.smp	HOPCAL03 - Hopcalite to 550 @ 5 deg/min.

[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

File name	The name of the file you select from the Files: list window is copied to this field. If choosing multiple files, the last name chosen is displayed.
Status **Dates** **Files** **Directories**	These options are explained in **Selecting Files** beginning on page **3-6**.
Settings group box	The Destination field contains a list of the available print destinations.
	If you select **Printer**, the **Copies** field is enabled allowing you to enter up to four copies.
	If you select **File**, the tabular reports of the requested file are converted to a text file which can be viewed with a text editor or other text file manipulation tools. The **File name** field is also enabled allowing you to enter a name (or you can accept the default).

List

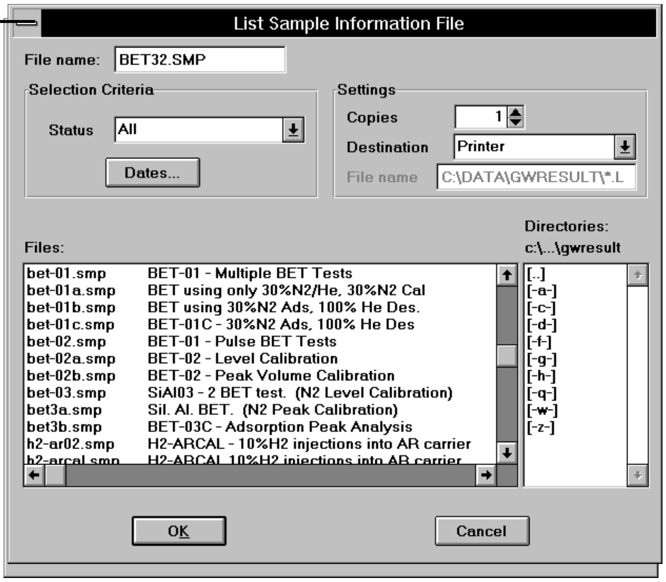
List enables you to generate a listing of the following information on a selected file, or on multiple files:

- File name
- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- File status

When you select **List** from the File menu, three choices are presented: **Sample information**, **Analysis conditions**, and **Report options**.

Regardless of which file type you select, a dialog similar to the one shown below is displayed.

Displays the type of file requested; in this example, a Sample Information File.



File name: BET32.SMP

Status: All

Copies: 1

Destination: Printer

File name: C:\DATA\GWRESULT*.L

Directories: c:\...\gwresult

[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

Files:

bet-01.smp	BET-01 - Multiple BET Tests
bet-01a.smp	BET using only 30%N2/He, 30%N2 Cal
bet-01b.smp	BET using 30%N2 Ads, 100% He Des.
bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier

File name If you select only one file from the Files: list box, the name is copied to this field. If multiple files are selected, the last one selected displays.

You may request a list of multiple files by holding down Ctrl while selecting files. If no files are selected, a list is generated for all files.

Status
Dates
Files
Directories

These options are explained in **Selecting Files** beginning on page **3-6**.

Settings group box

The **Destination** field contains a drop-down list of the available print destinations.

Choices: Screen, Printer, File

If you select **Printer**, the **Copies** field is enabled allowing you to enter up to four copies.

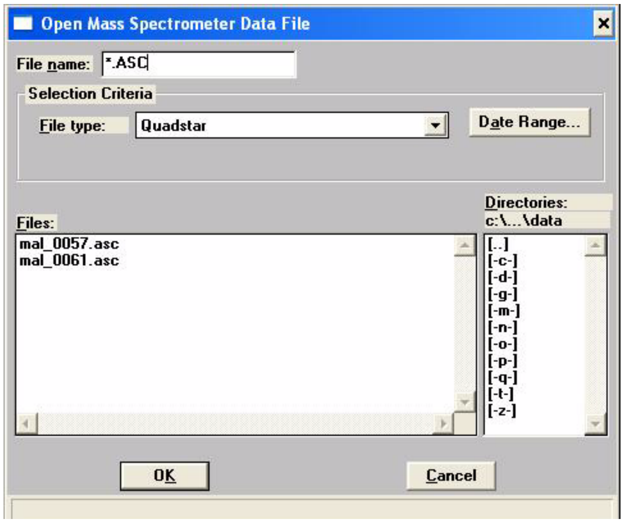
If you select **File**, the **File name** field is enabled allowing you to enter a name (or you can accept the default).

Import MS Signal

Import MS Signal allows you to import data collected by a ThermoStar or MicroStar Mass Spectrometer.

In order to have the analog signals import properly, they must be configured in a certain manner in the mass spectrometer software. Refer to the next page for information on configuring the signals.

Select Import MS Signal from the File menu; the Open Mass Spectrometer Data File dialog is displayed.



File name: *.ASC

File type: Quadstar

Files:
mal_0057.asc
mal_0061.asc

Directories:
c:\...\data
[..]
[-c-]
[-d-]
[-g-]
[-m-]
[-n-]
[-o-]
[-p-]
[-q-]
[-t-]
[-z-]

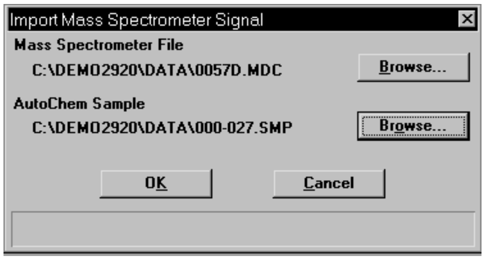
File name

Displays the wildcard (*) with the extension of the selected file type. The file type extensions are:

- MDC (Quadstar)
- ASC (Quadera)
- XLS (TAMS)

File type	Displays a drop-down list of file types.The files types are those produced from the Mass Spectrometer software used with the 2920. The choices are Quadstar, Quadera, or TAMS.
Date Range	Use this push button to limit the files displayed in the **Files** list to a specified date range. Refer to page 3-7 for an explanation of this dialog.
Files list	Displays the files available in the current directory.

After you select the desired file and click **OK**, the Import Mass Spectrometer Signal dialog is displayed.



Mass Spectrometer File
C:\DEMO2920\DATA\0057D.MDC

AutoChem Sample
C:\DEMO2920\DATA\000-027.SMP

Mass Spectrometer File	Displays the mass spectrometer file you selected from the previous dialog to import into the AutoChem program.
AutoChem Sample	Displays the AutoChem sample file into which the mass spectrometer file will be imported. If you have a sample file open, its name is displayed in this field and the data will be imported into this file when you click **OK**. If you do not have a sample file open, the next sequenced file number is displayed in this field. When you click **OK**, the data will automatically import and assume the next sequenced number; you will not be asked if you wish to create the file.
Browse	This push button is provided for each type of file so that you can choose different files from the ones displayed.

Configuring the Analog Signals

Analog signals must be configured **only when using Quadstar software**. If you are using Quadera and TAMS software, this procedure is not required.

The import utility reads both the mass spectrometer data and any analog signals that are collected by the mass spectrometer. The analog signals must be configured for them to be imported properly. This is accomplished through the Parset program.

1. Launch the Parset program using Windows Explorer: double-click on **Parset.exe**.

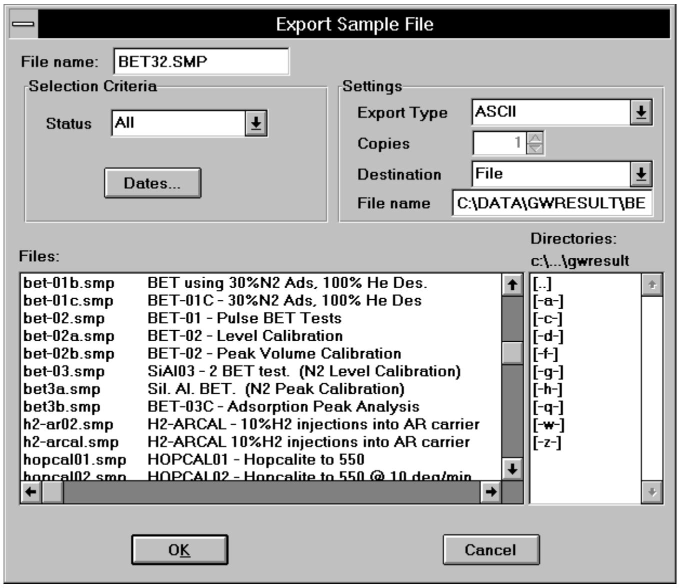
2. Refer to the Instrument Configuration section of the ThermoStar operator's manual and configure the characteristic curves as follows:

   A1 = T
   A0 = TCD

Export

Export allows you to copy data in a sample information file and reformat it in ASCII format or Grams/32 format. ASCII data can be imported into other applications, such as spreadsheets. Grams/32 data can be imported into the Grams/32 program (the peak editing program used in previous versions of the software) and then manipulated without use of the AutoChem II analysis program. You may select multiple files by holding down and clicking the left mouse button once on each file. See Appendix D for a description of exported data.

Select **Export** from the File menu; the Export Sample File dialog is displayed.



File name: BET32.SMP

Status: All

Export Type: ASCII

Copies: 1

Destination: File

File name: C:\DATA\GWRESULT\BE

Files:

bet-01b.smp	BET using 30%N2 Ads, 100% He Des.
bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier
hopcal01.smp	HOPCAL01 - Hopcalite to 550
hopcal02.smp	HOPCAL02 - Hopcalite to 550 @ 10 deg/min

Directories:
c:\...\gwresult

[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

File name	The name of the file you select from the Files: list box is copied to this field.
Status **Dates** **Files** **Directories**	These options are explained in **Selecting Files** beginning on page **3-6**.
Export Type	You can export the data from a 2920 sample information file in an ASCII or Grams/32 format. Choose **ASCII** to create a data file that can be imported into an application such as a spreadsheet program. Choose **Grams/32** to create a data file that can be imported into the Grams/32 program.

Destination

Contains a drop-down list of the available destinations.

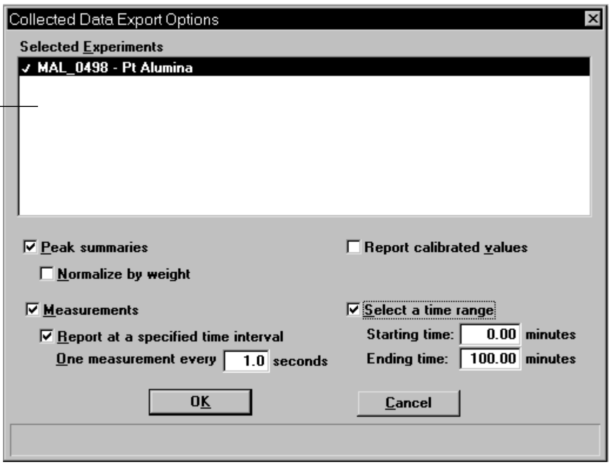
Choices: Screen, Printer, File

A sample information file exported as a Grams/32 type can only be generated to a File destination. When you export a Grams/32 type, five separate files (which can be read only by the Grams/32 software) are generated. Refer to Appendix D for information on the contents of these files.

If you select **Printer**, the **Copies** field is enabled allowing you to enter up to four copies.

If you select **File**, enter a name in the **File name** field of the Settings group box (or accept the default).

When you click **OK**, the Collected Data Export Options dialog is displayed.



Only the Selected Experiments portion of this dialog displays if you choose Grams/32 as the type of Export.

Collected Data Export Options

Selected Experiments

✓ MAL_0498 - Pt Alumina

☑ Peak summaries

☐ Normalize by weight

☐ Report calibrated values

☑ Measurements

☑ Report at a specified time interval

One measurement every 1.0 seconds

☑ Select a time range

Starting time: 0.00 minutes

Ending time: 100.00 minutes

Selected Experiments

All related experiments in the current file are displayed. When the report options file is used in a sample information file, any experiment you do not wish to include in the report may be deselected.

This portion of the dialog is the only field displayed if you are exporting a file as a Grams/32 type.

Peak summaries

Exports a summary of the peaks contained in the selected experiment(s).

Measurements	Selecting this option enables the options in the **Measurement Reporting Options** and **TCD Signal Options** group boxes.
Measurement Reporting Options	Provides options for reporting measurements.
Report measurements at a specified interval	Select this option to report measurements at a specified interval; a field is enabled so that you may enter an interval (the number of seconds between reported data readings). You may wish to use this feature if a large amount of data was collected during the analysis (such as in a lengthy analysis with readings taken every second). If you do not specify an interval for data reporting, points are reported as follows: • Up to 500 lines per experiment if destination is to the **screen** or to a **printer**. • Up to 10,000 total lines if destination is to a file.
Select a time interval to report	Select this option to specify relative starting and ending times of the measurements you wish to export. This feature allows you to specify a region of interest to be extracted from a large data set.
TCD Signal Options	Provides options for reporting the TCD signal.
Report the calibrated data	Exports the calibrated values for the TCD signal if calibration data are available.
Normalize by weight	Divides the TCD signal by the sample weight to correct the signal strength for different sample sizes.

Exit

Exit enables you to exit the AutoChem II analysis program. Select **Exit** from the File menu.

- If a window containing unsaved changes is open when you select **Exit**, the following message is displayed:

  (path\file name) has been changed. Save changes before closing?

  Yes **No** **Cancel**

  Click **Yes** to save the changes and close the dialog box
  Click **No** to discard the changes and close the dialog box
  Click **Cancel** to allow the file to remain active

- If an analysis is in progress, the following message is displayed:

  2459- An instrument is busy. A delay in restarting this application could result in loss of data. Continue with program Exit?

  Yes **No**

  Click **Yes** to exit the analysis program. The analysis continues, but the data are not recorded permanently on the computer's disk drive until you restart the AutoChem II 2920 application. If a loss of power occurs to the AutoChem analyzer before you restart the application, the data will be lost.

  Click **No** to allow the analysis program to remain active and the analysis to finish.

- If a report is in progress or a report window is open, the following message is displayed:

  A report is currently printing. Exiting this application now will cancel the report. Continue with the termination?

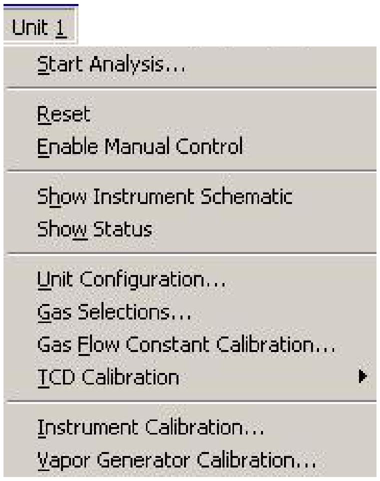
  Yes **No**

  Click **Yes** to cancel the report(s) and exit the analysis program.
  Click **No** to allow the analysis program to remain active.

6. UNIT MENU

The Unit menu contains options that enable you to perform analyses.

Description



Listed below are brief descriptions of the Unit menu options. Detailed descriptions follow this section.

Start Analysis	Opens a series of dialog boxes that enable you to start an analysis. Page **6-3**.
Reset	After confirmation, resets the system to a safe state. Page **6-11**.
Enable Manual Control	Enables you to control various instrument components manually, using the instrument schematic (disabled during an analysis that is not suspended). Page **6-12**.
Show Instrument Schematic	Displays the instrument schematic. Page **6-16**.
Show Status	Displays only the Status Bar of the operational window. Page **6-18**.

Unit Configuration	Displays the hardware configuration, as established when the instrument was installed, including software version, communications ports, and installed options. Page **6-19**. Also allows you to enter the loop (if present) volume.
Gas Selections	Allows you to specify which gases are connected to the analyzer ports. Page **6-20**.
Gas Flow Constant Calibration	Displays a dialog enabling you to obtain the conversion constant of a gas. The conversion constant is used by the Mass Flow Controllers to ensure accurate flow control. Page **6-21**.
TCD Calibration	Displays a series of dialogs allowing you to create, edit, and manage calibration files. Calibration files enable the software to convert sample signal, temperature, and peak area data to volume data. Page **6-22**.
Instrument Calibration	Displays a dialog for calibrating various instrument components. This dialog is used to calibrate the sample thermocouple. Page **6-28**.
Vapor Generator Calibration	Displays a dialog for calibrating various components of the optional Vapor Generator. This option is disabled if the Vapor Generator is not attached to the instrument. Page **6-30**.

Start Analysis

When you select **Start Analysis**, the Analysis dialog is displayed with a file selection dialog positioned on top. You can either select a sample file that was created and saved earlier, or you can create a new sample file now.

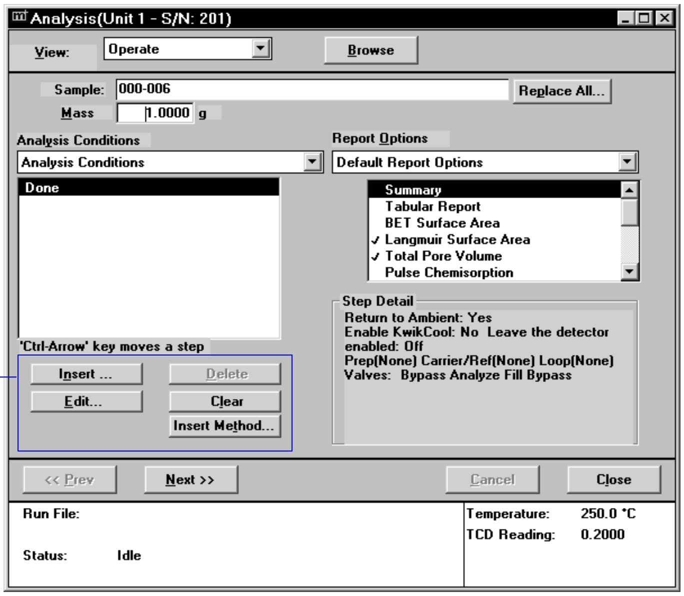
Which option you choose depends on the method you prefer for your laboratory. The analysis process is the same either way. For example, your lab may prefer to create numerous sample files in advance, then analyze the samples when they become available. Alternatively, your lab may prefer to create sample files for each sample as you begin the analysis.

After you choose a sample file and click **OK**, the Analysis dialog becomes totally visible.

If there are any inconsistencies between the sample information file you choose and the way the instrument is configured, a message is displayed instructing you to correct them. For example, if the sample information file specifies *helium* for the Preparation gas, but the Gas Selections dialog indicates that helium is not connected to a Preparation gas port, the software informs you of the discrepancy.

The software is unable to determine if the wrong gas is physically connected to the port. It can only check that the gas you wish to use agrees with the gas you have identified for each port in the Gas Selections dialog. It is important to be sure that the Gas Selections dialog accurately reflects which gas is physically connected to each port.

The appearance of the Analysis dialog depends on whether you are using the Advanced or Basic mode. The same fields appear on both dialogs; however, the Advanced dialog contains additional push buttons that allow you to insert and edit individual steps.



View: Operate

Sample: 000-006

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

Summary
Tabular Report
BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption

'Ctrl-Arrow' key moves a step

Step Detail
Return to Ambient: Yes
Enable KwikCool: No Leave the detector enabled: Off
Prep(None) Carrier/Ref(None) Loop(None)
Valves: Bypass Analyze Fill Bypass

Run File:

Status: Idle

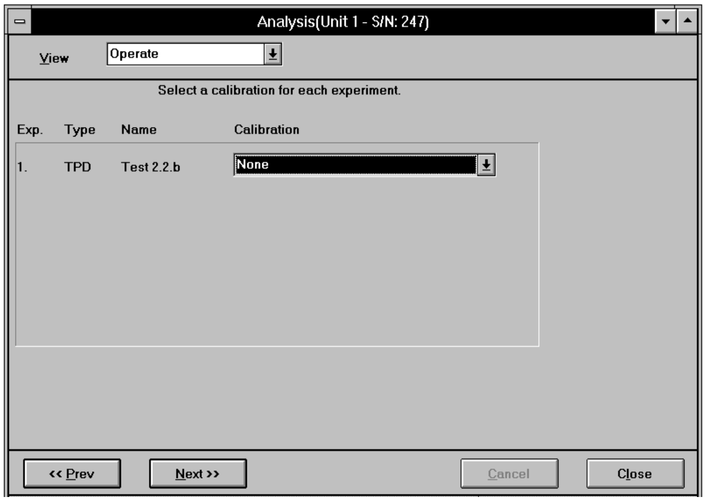
Temperature: 250.0 °C
TCD Reading: 0.2000

This group of push buttons does not display when using the Basic format.

View	Allows you to choose from four views to display in the current window.
Operate	Displays the dialogs associated with the current operation.
Instrument schematic	Displays the instrument schematic. Refer to **Show instrument schematic** on page **6-16** for a description of the components contained on the schematic.
Results	Displays analysis results as they are collected.
Sample log	Displays a log of each step as it is completed, as well as any error messages or manual operations that occur during the analysis.
Sample	Displays the description for the current file.
Mass	Enables you to enter an accurate weight of the sample to be analyzed.
Browse	Allows you to choose a different sample file for the current analysis.

Replace all	Allows you to replace the contents of the current file with those of another one. After the values have been copied into the current file, you may edit them in any way you wish. Editing the values will not affect the file from which they were copied.
Analysis Conditions	Displays the current Analysis Conditions file. This drop-down list contains predefined parameter files, some of which are supplied with the analysis program. For newly created files to display in this list, they must be saved to the directory specified as the parameter files directory (see Chapter 8). Click the down arrow to the right of the field to choose a different file. The window directly below this list contains the conditions specified in the Analysis Conditions file. If you are using the Advanced format for the current analysis, you can edit the conditions using the push buttons provided below the Analysis Conditions window. Refer to page **5-11** for details on these push buttons. You can also edit analysis conditions after the analysis has begun. Click **Suspend** to suspend the analysis; this enables the editing push buttons. You can edit only the steps or experiments following the current one. After editing is complete, resume the analysis.
Report Options	Displays the current Report Options file. This drop-down list contains predefined parameter files, some of which are supplied with the analysis program. For newly created files to display in this list, they must be saved to the directory specified as the parameter files directory (refer to Parameter Files Directory on page **8-17** for additional information). Click the down arrow to the right of the field to choose a different file. The window directly below this list contains the reports requested for the current Report Options file. You can select additional reports or deselect currently selected ones if desired. A report is selected when it is preceded with a check mark. Simply double-click to select (or deselect) a report. Alternatively, you can highlight the report and press the **Spacebar**.

Click **Next** to advance to the second view of the Analysis dialog. This view enables you to choose a TCD calibration for each experiment in this sample's analysis conditions set. A TCD calibration allows the areas of the peaks recorded by the instrument to be converted into volumes. A TCD calibration is not required by all analysis types, nor is it necessary to select the calibration from this dialog. A calibration can be associated with an experiment during peak editing (after analysis has ended).



View: Operate

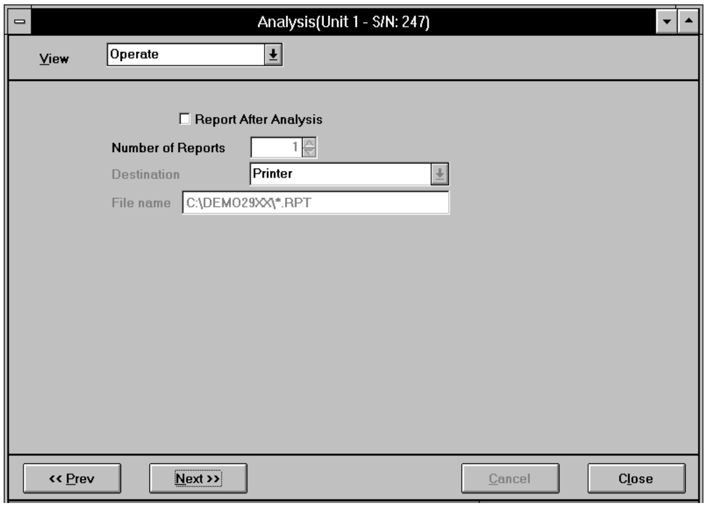
Select a calibration for each experiment.

Exp.	Type	Name	Calibration
1.	TPD	Test 2.2.b	None

The default(s) for the calibration file(s) are those specified using the TCD Calibration Defaults option on the Unit menu (discussed later in this chapter).

The files available in each drop-down list are those available for the instrument.

Click **Next** to advance to the next view of the Analysis dialog. This view enables you to generate reports automatically when the analysis is complete. The reports generated are those selected in the report options portion of the sample information file.



View: Operate

☐ Report After Analysis

Number of Reports: 1

Destination: Printer

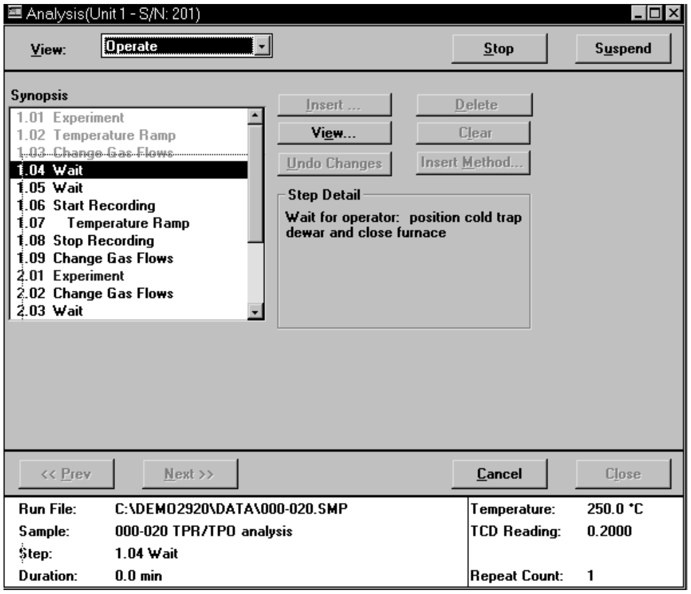
File name: C:\DEMO29XX*.RPT

Report After Analysis	Choose this option to have the reports specified in the sample file generated automatically after the analysis is complete.
Number of Reports	Enabled when **Printer** is selected as the report destination. You can print up to four copies.
Destination	Allows you to choose a destination for the reports *Choices: File, Printer, Screen* If you choose **Screen** for the destination, many options are available for manipulating and customizing your reports.
File name	Enabled when **File** is selected as the report destination, allowing you to enter a file name (or you can accept the default).

Click **Next**; a dialog instructing you to verify the following is displayed:

- Verify that the gas bottles connected to the analyzer are correctly identified in the software.
- Verify in the sample file that the combinations or sequence of gases in the analyzer are not dangerous, and that the sample will not react with any of the gases in a dangerous way.
- Verify that the current method for venting the analyzer’s exhausts is adequate and safe for all products of the analysis.

After you have ascertained that all conditions are safe for the analysis, click **Start** to begin the analysis. The original view of the Analysis window is displayed, except that now it tracks the progress of the analysis.



Synopsis

- 1.01 Experiment
- 1.02 Temperature Ramp
- 1.03 Change Gas Flows
- 1.04 Wait
- 1.05 Wait
- 1.06 Start Recording
- 1.07 Temperature Ramp
- 1.08 Stop Recording
- 1.09 Change Gas Flows
- 2.01 Experiment
- 2.02 Change Gas Flows
- 2.03 Wait

Step Detail

Wait for operator: position cold trap dewar and close furnace

Run File: C:\DEMO2920\DATA\000-020.SMP
Sample: 000-020 TPR/TPO analysis
Step: 1.04 Wait
Duration: 0.0 min
Temperature: 250.0 °C
TCD Reading: 0.2000
Repeat Count: 1

Synopsis	Shows the list of experiment steps in the sample information file. Upcoming steps are shown in blue or black. The current step is highlighted and shown in light blue; completed steps are shown in light green. You may scroll through the list to review the analysis steps. You can also edit steps that have not been started; the analysis must be suspended to do this (see **Suspend** on the following page).
Step Detail	Shows detailed information about the current step.
Stop	Stops operation of the current step and automatically suspends the analysis. Click **Resume** to restart the analysis. The analysis resumes at the beginning of the next step. This button is used to skip the current step. Manual Control is allowed while an analysis is suspended.
Cancel	Cancels the analysis; the current step is not completed. After confirmation, the analysis ends and leaves the analyzer in the current state. The analysis cannot be resumed. To return the analyzer to a safe state automatically, select **Reset** from the **Unit** menu.

Suspend	Suspends the analysis. This button is a toggle button; it reads **Resume** when the analysis is suspended. You may wish to suspend the analysis to edit steps in the list or add steps to the list. Steps can only be added or edited in the portion of the list that has not yet been executed; such editing is available only if you are using Advanced presentation format. The current step cannot be edited. Click **Resume** to continue the analysis after editing has been completed. Manual Control is allowed while an analysis is suspended.

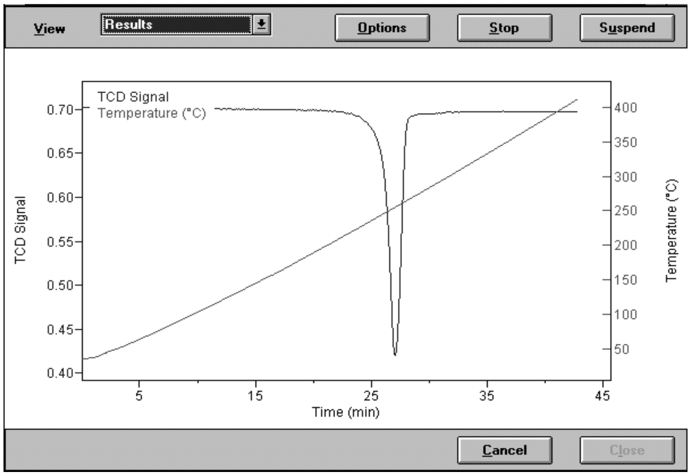
During analysis, analysis conditions can be edited only using this view of the Start Analysis dialogs. Analysis conditions cannot be edited in the sample file during analysis.

There is a very brief pause while the analyzer suspends or resumes the analysis. Do not reclick the button.

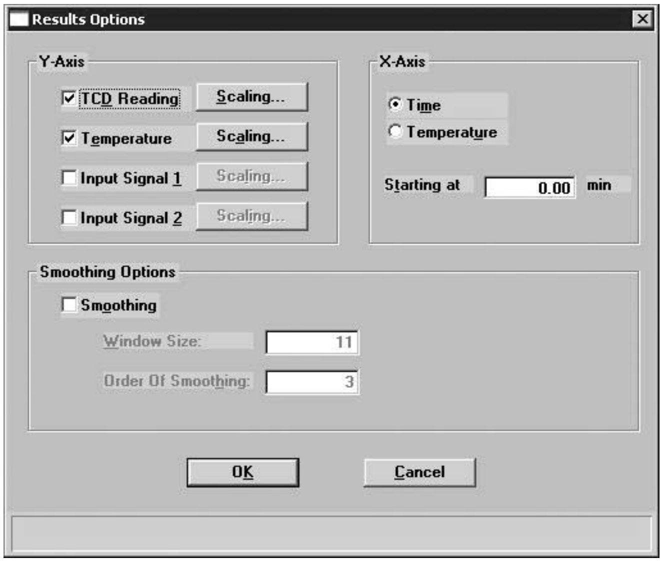
Messages	If you have included an experiment step that requires a message to the user, the message is displayed on the monitor when the appropriate point in the analysis is reached. For example, if you have included a Dose step that takes a BET measurement, you are prompted to change the dewar at the appropriate time. The operator performs the required function, then clicks **OK** to resume analysis.

Viewing the Results

To view data as they are collected, select **Results** from the View drop-down list. Up to four signals may be displayed on the graph: TCD, Sample Temperature, Input Signal 1 and Input Signal 2. The scales for the first two signals are shown on the right and left Y-axes, respectively. If more than two signals are selected, the additional signals are shown without scales.



Click **Options** to customize the presentation of data in the Results view. You can change the axes and select or deselect the data that are presented. If you specify a value for Starting at (X-Axis), only those data collected after that point are shown. If the Starting at value is greater than the experiment duration, then all data are shown.



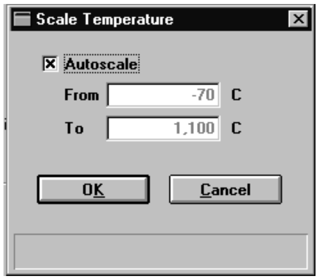
☑ TCD Reading
☑ Temperature
☐ Input Signal 1
☐ Input Signal 2

● Time
○ Temperature

Starting at 0.00 min

☐ Smoothing
Window Size: 11
Order Of Smoothing: 3

Click **Scaling** to adjust the scale of a selected signal. The example shown below is the Scaling dialog associated with the Temperature signal.

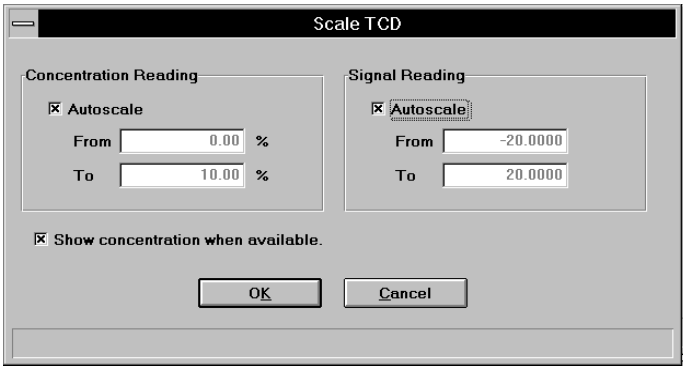

Scale Temperature

☒ Autoscale

From -70 C

To 1,100 C

In addition to allowing you to adjust the scale of the Concentration and Signal Results, the Scale TCD dialog lets you indicate (by checking the box) whether or not you wish to display the Concentration. (The concentration will be displayed if a Gas Concentration calibration has been associated with the experiment.)


Scale TCD

Concentration Reading

☒ Autoscale

From 0.00 %

To 10.00 %

Signal Reading

☒ Autoscale

From -20.0000

To 20.0000

☒ Show concentration when available.

Reset

Use this option to abort the current analysis and return the analyzer to a safe state. The following actions occur:

- All gas flows stop
- The detector is disabled
- The heat zones are reset to 0 °C
- The target furnace temperature is reset to 20 °C

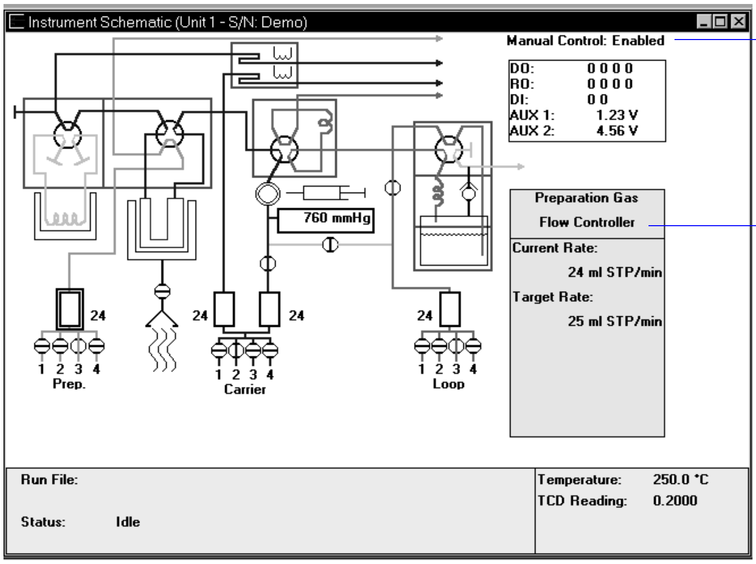
Do not select this option unless you are sure you want to stop the current analysis. The analysis cannot be resumed after **Reset** is selected.

Enable Manual Control

Select Enable manual control to manually control components such as the valves, Mass Flow Controllers, heat zones, etc. If the instrument schematic is not displayed, select **Show Instrument Schematic** from the Unit menu.

Extreme caution should be used when Manual Control is enabled, so that a dangerous condition is not created. If you are not qualified to prevent dangerous conditions (such as volatile combinations of gases), DO NOT attempt to operate the analyzer using Manual Control.

Manual Control cannot be selected during an automatic operation unless the operation is suspended.



Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Preparation Gas
Flow Controller

Current Rate:
24 ml STP/min
Target Rate:
25 ml STP/min

Run File:
Status: Idle

Temperature: 250.0 °C
TCD Reading: 0.2000

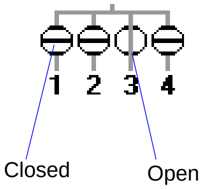
Indicates whether manual control is enabled or disabled.

Indicates the item selected and related statistics.

You can select system components using the mouse or the tab key. A component is selected when it is surrounded by a ruled box. The information window on the right of the schematic indicates the name of the selected component in the upper pane and related statistics in the lower pane.

Most components on the instrument schematic can be manually controlled. These components each have a shortcut menu displaying the operations available for that component. This shortcut menu is accessed by right-clicking on the desired component, or by using the shortcut keys, **Shift F9**.

Gas Valves (Inlet)

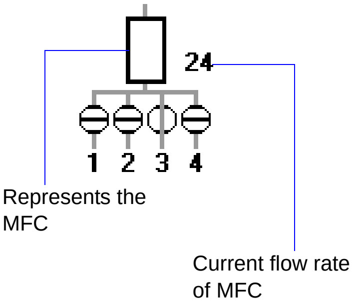


Click a gas valve icon to display (in the yellow window on the right) the name of the gas connected to each of that valve's four ports. The port's state (Open or Closed) is also displayed.

From left to right, the valves control the Preparation gas, the Carrier gas, and the Loop gas.

Flow Gas [n]	Select the gas to flow
Stop Flow	Closes the open valve

Mass Flow Controllers



The Mass Flow Controllers control the flow of gas into the analyzer. Clicking on a MFC icon displays its current flow rate and the target rate for the current experiment. The current flow rate is also shown next to the MFC symbol. If the flow has been turned off (all inlet valves are closed), the target rate shown is the last non-zero target.

From left to right on the display, the MFC's control the Preparation gas, the Reference gas, the Carrier gas, and the Loop gas.

Set Flow Rate
Displays a dialog so that you can enter a flow rate.

Range: *0 to 175* cm^3*/min.*

Stop Flow
Stops the flow of gas through the selected Mass Flow controller.

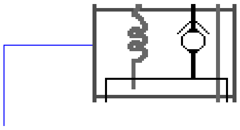
Rotary Valves

The Rotary Valves control the flow of gas through the cold trap, the sample tube, the loop, and the optional Vapor Generator. Each valve has two states. Clicking the valve icon displays its state. Right-clicking the valve icon enables you to change the state of the valve. Click on the desired state.

From left to right, the valves and their states are as follows:

Cold Trap	Bypass, Trap
Analysis	Analyze, Prepare
Loop	Fill, Inject
Vapor (optional)	Vapor, Bypass

Heat Zones



Heat zones are enclosed with a red rectangle. This example shows the Vapor Reflux heat zone.

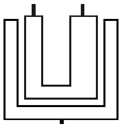
Heat Zones control the temperature of various parts of the analyzer. Component heat zones are enclosed with a red rectangle. Click a Heat Zone icon to display its current flow temperature and the target temperature for the current experiment step.

Right-click, then choose **Set Temperature**, to change the heat zone's target temperature. We recommend using one temperature (100 °C, for example) for all heat zones to ensure a cleaner system.

Range: 20 to 150 ºC for all zones
(except optional Vapor Generator)

From left to right on the display, the Heat Zones control the Cold Trap, the Analysis Valve, Detector, and Loop Valve (and if the Vapor Generator Option is installed, from top to bottom; Vapor Valve, Vapor Reflux, and Vapor Flask temperatures).

Sample Furnace



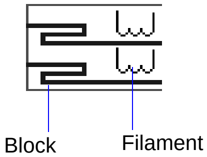
The Sample Furnace controls the temperature of the sample. Click its icon to display the Sample Temperature, the Target Temperature, the Furnace Temperature, and the Ramp Rate.

Right-click, then choose **Set Temperature** to change the Set Point and/or the Ramp Rate.

Set Point Range: -70 to 1100 ºC (with optional CryoCooler);
ambient to 1100 °C (without CryoCooler)

Ramp RateRange: 0.1 to 90.0 ºC/min

Detector

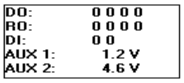


There are actually two areas of the detector icon that can be controlled. Click the filaments (right side) to specify their temperature. Click the other side of the rectangle to control the detector heat zone's temperature. In either case, right-click, then click on Set Temperature to change the temperature. The Filament Temperature must be set at least 20 °C above that of the detector heat zone.

Detector Heat Zone Temperature Range: 20 to 150 ºC

Filament Temperature Range: 20 to 250 ºC

Auxiliary I/O

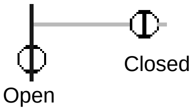


```
DO:      0 0 0 0
RO:      0 0 0 0
DI:      0 0
AUX 1:     1.2 V
AUX 2:     4.6 V
```

The Auxiliary I/O display provides a list of the current auxiliary settings for Digital and Relay Output (DO and RO) and for Digital Input (DI). The current Auxiliary 1 and Auxiliary 2 signals are also displayed, if they were recorded.

A complete discussion of the auxiliary inputs and outputs is contained in Appendix E, page **E-1**.

Calibration Valves



Click a Calibration Valve icon to display its state (Open or Closed). There are three Calibration Valves, lettered, from left to right, B through D. These valves are used by the software during automatic calibration processes.

Generally, it is not necessary for the operator to change the state of the calibration valves.

KwikCool

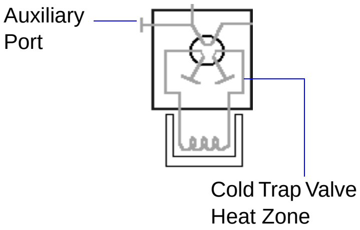
The KwikCool (if installed) icon appears on the schematic below the sample tube. The two states of the KwikCool valve are open or closed.

CryoCooler

The CryoCooler (if installed) icon appears on the schematic below the sample tube. The two states of the CryoCooler valve are open or closed.

The following components on the instrument schematic are for informational purposes only; they cannot be controlled using the manual control function.

Auxiliary Port

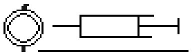


The Auxiliary Port enables you to connect an external detector, such as a mass spectrometer, to the analyzer. It is located on the left side panel as you face the unit. On the instrument schematic, it is located on the left of the Cold Trap Valve Heat zone.

Pressure Transducer

Displays the current pressure in the carrier gas path, upstream from the septum, but downstream from the Mass Flow Controller.

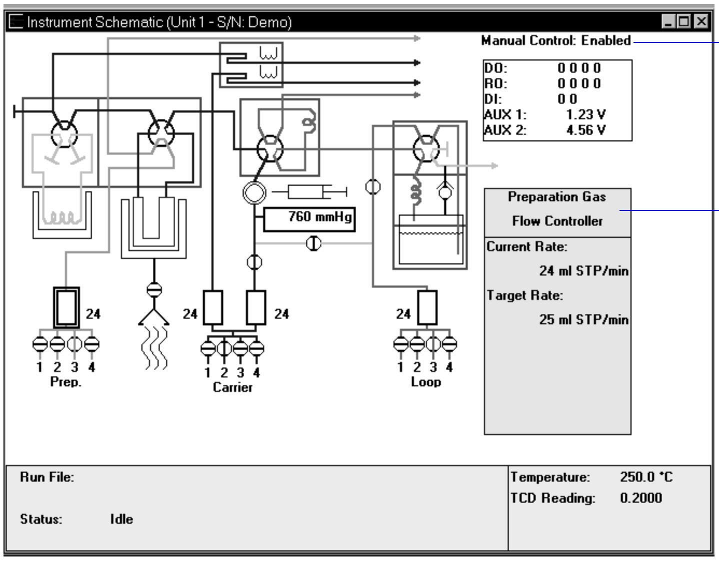
Septum



The septum is used for making manual injections of gas into the gas stream. Refer to Using the Septum on page **4-26**.

Show Instrument Schematic

Select this option to display the instrument schematic. The components of the instrument schematic are described in the previous section, **Enable Manual Control**.



Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Preparation Gas Flow Controller
Current Rate: 24 ml STP/min
Target Rate: 25 ml STP/min

Run File:
Status: Idle
Temperature: 250.0 °C
TCD Reading: 0.2000

Indicates whether manual control is enabled or disabled.

Indicates the item selected and its related statistics.

The instrument schematic is a visual representation of the internal components of the analyzer. Remember that the schematic does not show the components' actual appearance or position. Instead, it provides a simplified graphic illustration.

In automatic mode, the schematic displays information about the instrument's status, as well as providing updated information during an analysis. In Manual Mode, the schematic makes it easy to change the status of various instrument components.

Gas Pathways

The colored lines that lead from the various gas inlets through the analyzer and to the exhaust represent the current flow path. Each gas source is represented by a different color:

Pink	Loop gas
Royal blue	Carrier and Reference gases
Lime green	Preparation gas
Black	Blended gas

When a gas valve is opened or the flow path is changed, it takes several minutes for the new gas to move through the entire system.

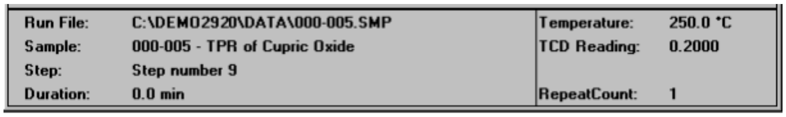
The behavior of the Loop valve is one example. When an analysis includes a loop injection, the instrument schematic shows the path that is available in Fill mode, then in Inject mode. In Fill mode, the analysis gas is shown entering the loop, and excess gas is vented out the exhaust. In Inject mode, the analysis gas is shown bypassing the loop and exiting the analyzer through the exhaust. At that time, the carrier gas path is available from the inlet all the way through the loop, through the sample tube, and past the detector.

When the valve switches to Inject mode, the quantity of analysis gas that is contained in the loop — one exact unit — is pushed out of the loop and through the sample tube by the carrier gas. When the software is recording the TCD signal, the loop injection shows as a peak on the Results view of the Analysis window.

The analysis gas may, in fact, be dissipated greatly as it travels out of the loop and through the sample tube, particularly if the carrier gas flow rate is low. Higher carrier gas flow rates minimize the dispersion of the loop gas.

Show Status

Select this option to display the status bar. The bottom portion of the Analysis dialog sequence displays the instrument's Status Bar. This option allows you to display only the status bar of the operational window.



Run File: C:\DEMO2920\DATA\000-005.SMP
Sample: 000-005 - TPR of Cupric Oxide
Step: Step number 9
Duration: 0.0 min
Temperature: 250.0 °C
TCD Reading: 0.2000
RepeatCount: 1

If more than one instrument is attached to your computer, each instrument's Status Bar is displayed in a different color to help you differentiate the instruments' displays.

The Status Bar displays:

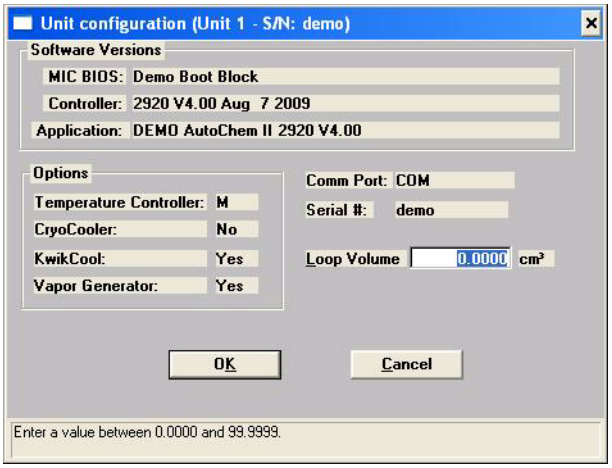
Run File	Gives the name of the sample information file currently being used for analysis. If no file is currently selected, this field remains blank.
Temperature	Gives the sample's temperature, as measured by the sample thermocouple.
TCD Reading	Displays the current detector reading (0.0000 if disabled).

During analysis, the Status Bar also displays:

Sample (Identifier)	Gives the identifier of the sample information file currently being used for analysis. If no file is currently selected, this field remains blank.
Time	Displays the number of seconds that have elapsed since data recording began. This field is blank when no data are being recorded.
Step	Shows the step currently being performed. When no analysis is in progress, *Status: Idle* is displayed instead.
Duration	Shows the amount of time that has elapsed since the current step was started. If the analysis is waiting for a fixed period of time, the time left to wait is shown in parentheses next to the time.
Repeat Count	Shows how many times the current Repeat loop has been entered.

Unit Configuration

Select this option to display the configuration of the instrument’s software, communications ports, and options.



Unit configuration (Unit 1 - S/N: demo)

Software Versions

MIC BIOS: Demo Boot Block

Controller: 2920 V4.00 Aug 7 2009

Application: DEMO AutoChem II 2920 V4.00

Options

Temperature Controller: M

CryoCooler: No

KwikCool: Yes

Vapor Generator: Yes

Comm Port: COM

Serial #: demo

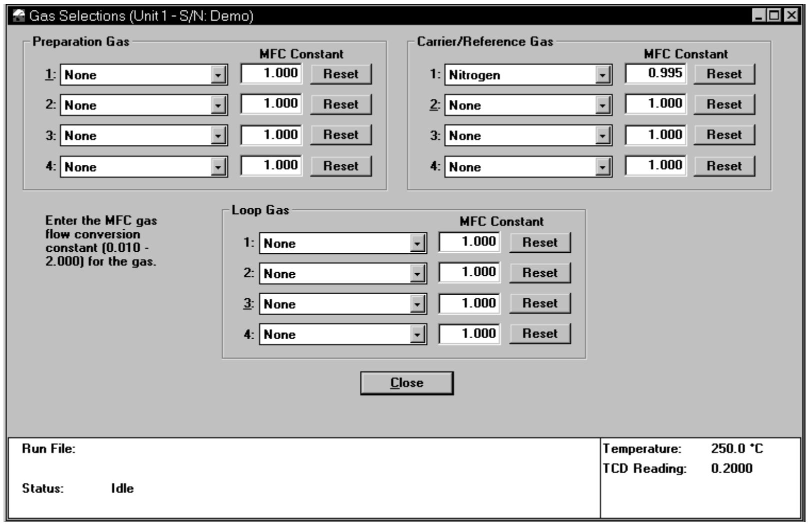
Loop Volume 0.0000 cm³

Enter a value between 0.0000 and 99.9999.

Software Versions **MIC BIOS**	Gives the name and version of the instrument’s internal software.
Controller	Gives the name and version of the instrument’s controller software.
Application	Gives the name and version of the application currently being run on the control module.
Options	Displays the options available for the AutoChem II and whether they are installed or not. For the Temperature Controller: M = Micromeritics, E = Eurotherm
Comm Port	Displays the communications port the instrument is using to communicate with the computer.
Serial #	Displays the instrument’s serial number, as it was entered during installation.
Loop Volume	Allows you to enter the volume of the gas injection loop that is attached to the instrument. Three standard-volume loops are provided with your instrument. The loop volume is determined by performing a loop calibration. Loop calibration is performed by a specialized experiment step (refer to Appendix J, page **J-38**.).

Gas Selections

Select this option to specify which gases are connected to the ports for the preparation, carrier/reference, and loop gases.



Gas Selections (Unit 1 - S/N: Demo)

Preparation Gas

		MFC Constant
1:	None	1.000
2:	None	1.000
3:	None	1.000
4:	None	1.000

Carrier/Reference Gas

		MFC Constant
1:	Nitrogen	0.995
2:	None	1.000
3:	None	1.000
4:	None	1.000

Enter the MFC gas flow conversion constant (0.010 - 2.000) for the gas.

Loop Gas

		MFC Constant
1:	None	1.000
2:	None	1.000
3:	None	1.000
4:	None	1.000

Run File:

Status: Idle

Temperature: 250.0 °C

TCD Reading: 0.2000

Gas Selections cannot be changed during an automatic operation.

This dialog contains a drop-down list of the available gases for each of the 12 available ports. Each gas also has its MFC constant listed.

Identify what gas is attached to each port by selecting that gas name from the drop-down list. If no gas is attached to a given port, select None.

The gases in the drop-down list are those specified in the Gas Defaults table (on the Options menu).

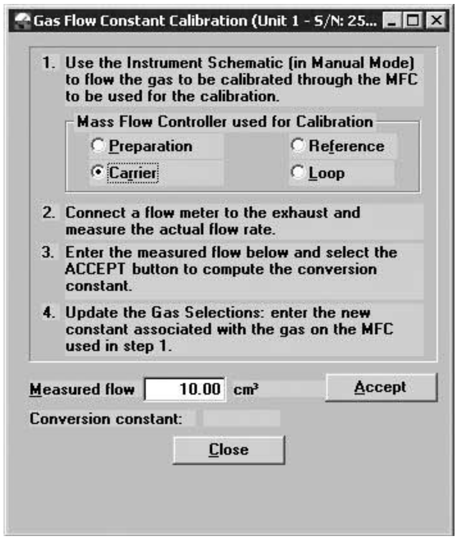
When an analysis is started, the software checks the gases specified in the sample information file against the gases identified in this dialog. If there is a discrepancy between the gas indicated in the sample information file and the gas indicated in the Gas Selections dialog, a message is displayed; analysis cannot begin until the discrepancy is resolved.

The software is unable to determine if the wrong gas is physically connected to the port. It can only check that the gas you wish to use agrees with the gas you have identified for each port in the Gas Selections dialog. It is important to be sure that the Gas Selections dialog accurately reflects which gas is physically connected to each port.

Gas Flow Constant Calibration

The Mass Flow Controllers (MFCs) of your AutoChem II 2920 were calibrated before shipping. Unless you suspect that one of the MFCs is operating improperly, you should use the calculated (factory) values.

Select this option to determine a constant that is used by the analyzer to ensure accurate gas flows through each Mass Flow Controller.



1. Use the Instrument Schematic (in Manual Mode) to flow the gas to be calibrated through the MFC to be used for the calibration.

Mass Flow Controller used for Calibration
○ Preparation ○ Reference
● Carrier ○ Loop

2. Connect a flow meter to the exhaust and measure the actual flow rate.
3. Enter the measured flow below and select the ACCEPT button to compute the conversion constant.
4. Update the Gas Selections: enter the new constant associated with the gas on the MFC used in step 1.

Measured flow 10.00 cm^3

Conversion constant:

A table of the constants for the most commonly used gases is provided in Appendix C. A set of conversion constants is shipped with the analyzer, and is displayed in the Gas Defaults table accessed from the Options menu. In most cases, the defaults provided in the table work well.

If you are using a gas that is not listed in the Gas Defaults table, or if you suspect that one of the MFCs is not functioning properly, calculate a new constant using the instructions and the worksheet provided in Appendix F.

TCD Calibration

Analyses with the AutoChem II 2920 yield data on signal reading, peak area, temperature, and time (plus auxiliary signals, if they were recorded). These data are sufficient for many applications, but in some cases, volume data are desired as well.

In applications where volume data are not needed, it is not necessary to perform a calibration. If volume data are desired, calibration may be performed either before or after the analysis.

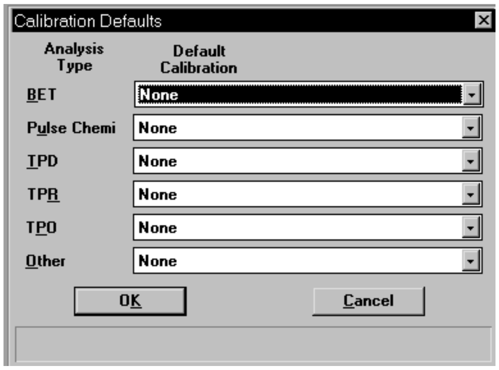
A group of automatic calibration routines are provided in the form of specialized experiment steps. A calibration run is an analysis using one of these experiment steps. The calibration run can be performed before or after the sample analysis. A calibration experiment can be included as a step within your sample analysis, or it can be performed as a separate analysis.

After the sample data and calibration data are collected, the calibration file is associated with the sample file, and the sample data are converted to volume. A single calibration run can be associated with an unlimited number of sample data files. For example, TPR yields peak area and the temperature(s) at which maximum reduction occurs. To obtain the volume of gas uptake, however, you must select a calibration file to associate with the analysis file. Then you can create reports in which the area data are converted to volume data.

You can associate a calibration file with a sample file in three different ways:

- Select **TCD calibration**, **Defaults** on the Unit menu and specify a default calibration file for each experiment type.
- Use the default file specified or choose a different file from the second view of the Start Analysis dialog.
- Click **Set Calibration** on the Peak Editor dialog (described on page 7-14). This choice allows you to select a calibration file that was created after the sample file was used in an analysis.

Defaults



Analysis Type	Default Calibration
BET	None
Pulse Chemi	None
TPD	None
TPR	None
TPO	None
Other	None

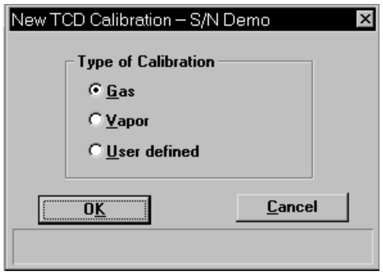
Allows you to specify default calibration files for each type of analysis. An additional default can be specified for analyses you have customized, using the Other drop-down list. Setting default calibrations can save you time when you frequently perform analyses using the same gases.

The calibration files that appear in the lists are those that are available for the instrument.

You will have two opportunities to select a different calibration file before you print a report: during the series of dialogs used to start an analysis (**Start Analysis** from the Unit menu), and using **Set Calibration** on the Peak Editor dialog.

New

Allows you to create a new TCD calibration file; the New TCD Calibration dialog is displayed.

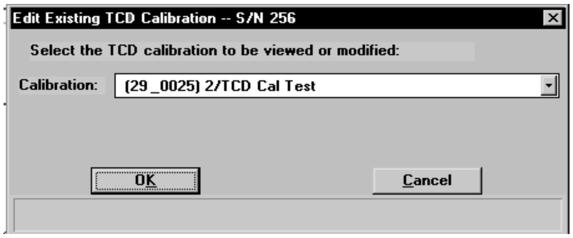


Gas	Enables the analyzer to determine what mixture of gases is flowing past the detector. Use this calibration when the volume of gas uptake is needed.
Vapor	Enables the analyzer to determine the volume of vapor used during the analysis. Use this calibration when the optional Vapor Generator is used in conjunction with the loop.
User defined	Enables the analyzer to convert peak area data to peak volume data for analyses in which the injection loop was used. Use this calibration when the loop volume is needed.

After you click **OK**, a related dialog allowing you to specify the details is displayed. TCD calibration files are used to convert sample data to volume data. Examples of these calibrations are provided in the tutorials (Appendix J).

Open

Allows you to open an existing calibration file for editing; the Edit Existing TCD Calibration dialog is displayed.

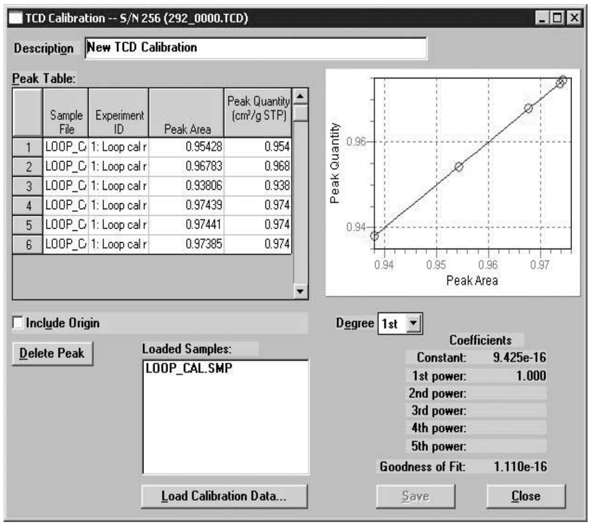


Edit Existing TCD Calibration -- S/N 256

Select the TCD calibration to be viewed or modified:

Calibration: (29 _0025) 2/TCD Cal Test

This dialog contains a drop-down list of existing TCD calibration files. After choosing the desired file, click **OK**; the TCD Calibration dialog is displayed.



TCD Calibration -- S/N 256 (292_0000.TCD)

Description: New TCD Calibration

Peak Table:

	Sample File	Experiment ID	Peak Area	Peak Quantity (cm³/g STP)
1	LOOP_C	1: Loop cal r	0.95428	0.954
2	LOOP_C	1: Loop cal r	0.96783	0.968
3	LOOP_C	1: Loop cal r	0.93806	0.938
4	LOOP_C	1: Loop cal r	0.97439	0.974
5	LOOP_C	1: Loop cal r	0.97441	0.974
6	LOOP_C	1: Loop cal r	0.97385	0.974

☐ Include Origin

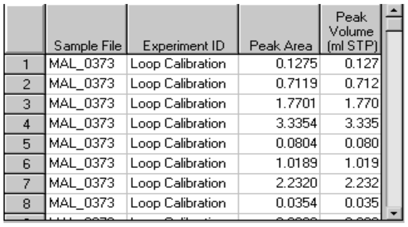
Loaded Samples: LOOP_CAL.SMP

Degree: 1st

Coefficients

Constant:	9.425e-16
1st power:	1.000
2nd power:	
3rd power:	
4th power:	
5th power:	
Goodness of Fit:	1.110e-16

This type of peak table is displayed for a gas concentration calibration.



	Sample File	Experiment ID	Peak Area	Peak Volume (ml STP)
1	MAL_0373	Loop Calibration	0.1275	0.127
2	MAL_0373	Loop Calibration	0.7119	0.712
3	MAL_0373	Loop Calibration	1.7701	1.770
4	MAL_0373	Loop Calibration	3.3354	3.335
5	MAL_0373	Loop Calibration	0.0804	0.080
6	MAL_0373	Loop Calibration	1.0189	1.019
7	MAL_0373	Loop Calibration	2.2320	2.232
8	MAL_0373	Loop Calibration	0.0354	0.035

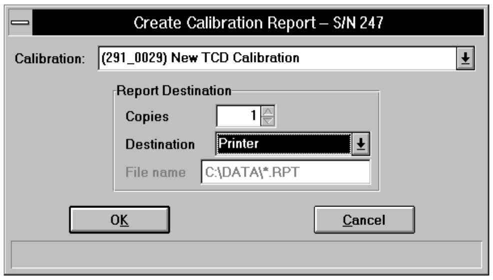
This type of peak table is displayed for a vapor or user-defined calibration.

Peak Table	For a gas calibration, this table displays the sample file number, the experiment identification, the TCD signal, and the percentage of concentration. For a vapor or user-defined calibration, this table displays the sample file number, the experiment identification, the peak area and peak volume. The values in the table are graphically depicted in a plot adjacent to the table.

Loaded Samples	Lists the calibration files currently open.
Delete Peak	Deletes the selected peak.
Load Calibration Data	Allows you to load another calibration file. The calibration file you choose must have been created with the current instrument. If you select a file created on a different instrument, an error message is displayed.
Degree	Allows you to choose a different degree of polynomial; usually the 2nd degree is adequate.
Coefficients	Shows the fit of data to the polynomial.
Description	Displays the name of the current calibration file. This field can also be used to enter a new name when changes have been made.
Save	Enabled when the current file has been edited, allowing you to save the changes. If you wish to save it under a different name, enter a new one in the **Description** field.
Close	Closes the dialog.

Report

Select this option to print a report of a calibration file.



Calibration: (291_0029) New TCD Calibration

Copies 1

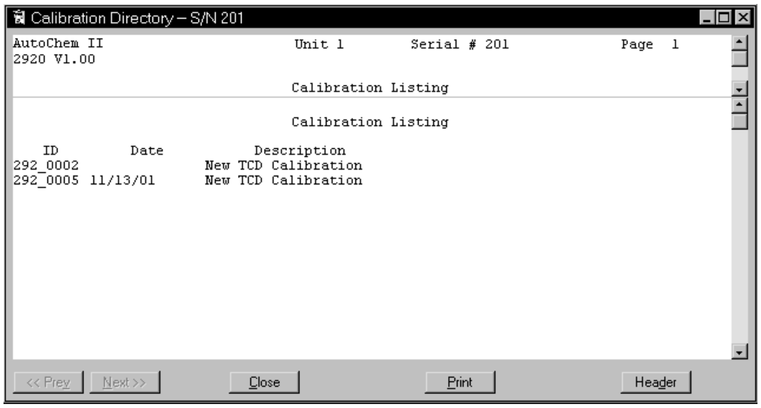
Destination Printer

File name C:\DATA*.RPT

Calibration	From the drop-down list, select the file for which you wish to generate a report.
Report Destination Copies	Enabled if you choose **Printer** as the report destination. You can print up to four copies.
Destination	Allows you to choose a destination for the report. *Choices: File, Printer, Screen*
File name	Enabled if **File** is selected as the report destination, allowing you to enter a file name (or accept the default).

List

Select this option to display a list of the files in the Calibration Directory, including details such as the calibration file's identifier (as entered in the sample information file) and the date the file was created (or last edited).



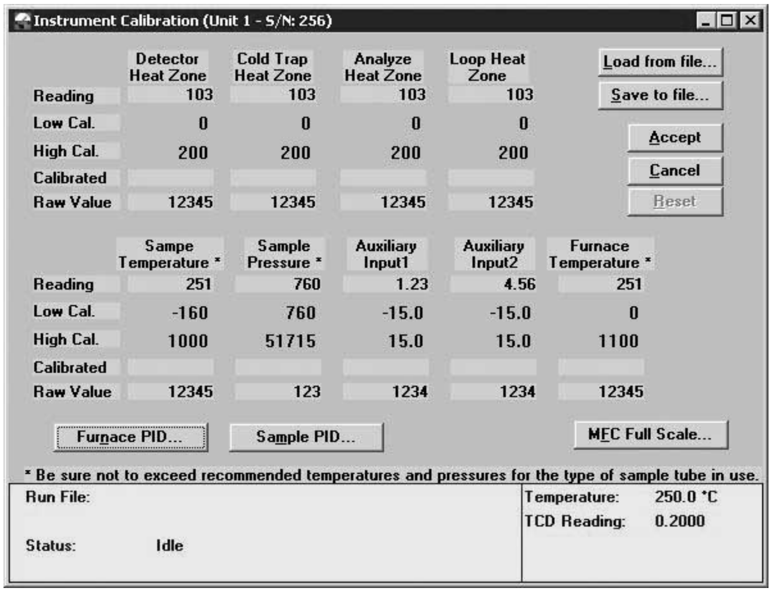
```
AutoChem II                        Unit 1       Serial # 201            Page  1
2920 V1.00

                                   Calibration Listing

                                   Calibration Listing

   ID        Date          Description
292_0002               New TCD Calibration
292_0005  11/13/01     New TCD Calibration
```

Next **Prev**	Allow you to view the next or previous page of the list (if they exist).
Close	Closes the window.
Print	Prints the list.
Header	This toggle button hides/shows the header of this list. If the header is selected, it appears on every page of the list when it is displayed and/or printed.

Instrument Calibration

A calibration of the analyzer is performed before the analyzer is shipped; the correct calibration values for each instrument are shipped with the analyzer. The contents of this calibration file are loaded into the analyzer software during installation.

This option displays the Instrument Calibration dialog.



	Detector Heat Zone	Cold Trap Heat Zone	Analyze Heat Zone	Loop Heat Zone
Reading	103	103	103	103
Low Cal.	0	0	0	0
High Cal.	200	200	200	200
Calibrated				
Raw Value	12345	12345	12345	12345

	Sampe Temperature *	Sample Pressure *	Auxiliary Input1	Auxiliary Input2	Furnace Temperature *
Reading	251	760	1.23	4.56	251
Low Cal.	-160	760	-15.0	-15.0	0
High Cal.	1000	51715	15.0	15.0	1100
Calibrated					
Raw Value	12345	123	1234	1234	12345

* Be sure not to exceed recommended temperatures and pressures for the type of sample tube in use.

Run File:

Status: Idle

Temperature: 250.0 °C

TCD Reading: 0.2000

This dialog shows calibration data for the AutoChem II 2920 components. It also enables you to calibrate the sample thermocouple. Recalibration should be performed only when you have replaced the sample thermocouple or when the analyzer's calibration file (which was installed during initial installation of the software) has been corrupted or lost. Refer to **Recalibrating the Sample Thermocouple** on page **9-18** for instructions for using this dialog to calibrate the sample thermocouple.

You should not attempt to calibrate other components without contacting your Micromeritics service representative.

Do not attempt to use the Calibrate Hardware dialog to calibrate the analyzer unless you are specifically instructed to do so by a Micromeritics service representative.

Load from File	Allows you to load a different calibration file.
Save to File	Allows you to save the current calibration settings to a file. This function is useful in protecting your calibration data. It is wise to save the current settings before attempting a recalibration. Be sure to use the next sequenced file name generated by the system.

Accept	Replaces the existing calibration values with those you have entered in this session. After a confirmation, the software gives you the opportunity to save the existing values. The Calibrate Hardware dialog is closed after you accept the new values.
Cancel	Cancels any changes made to the current calibration settings. A warning message is displayed before the dialog closes.
Reset	Enabled when a change has been made to any field on this dialog. Click this push button to reset any changed value(s) to its last saved state.

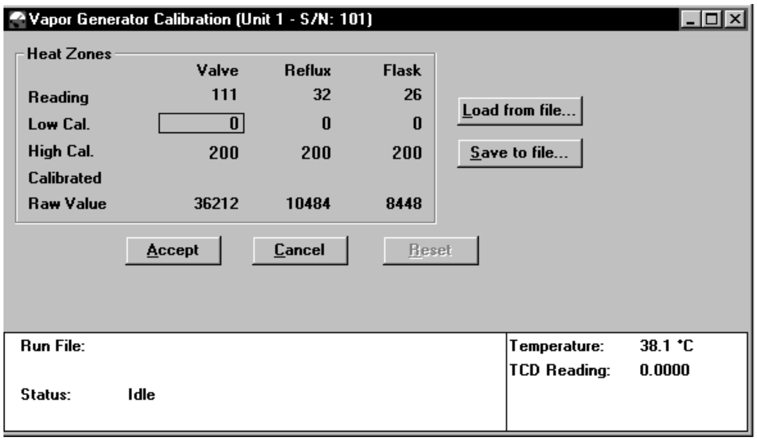
Do not attempt to edit parameters of the following options unless instructed to do so by a Micromeritics service representative.

Furnace PID	Displays the control parameters for the temperature controller. The default values provide linear temperature ramps from ambient to 1100 ºC.
Sample PID	Displays the control parameters for the sample temperature. The proportional and derivative are used. The default values are recommended.
MFC Full Scale	Displays the full scale flow rate for the mass flow controllers. The full scale was determined using H_2 as the calibration gas.

Vapor Generator Calibration

This option allows you to calibrate various components of the optional Vapor Generator. This option is available only if the Vapor Generator is installed on the analyzer. Refer to **Recalibrating the Vapor Generator's Heated Zones** on page 9-20 for instructions on calibrating the vapor generator.

Do not attempt to use the Calibrate Vapor Generator dialog to calibrate the analyzer unless you are specifically instructed to do so by a Micromeritics service representative.



	Valve	Reflux	Flask
Reading	111	32	26
Low Cal.	0	0	0
High Cal.	200	200	200
Calibrated			
Raw Value	36212	10484	8448

Run File:

Status: Idle

Temperature: 38.1 °C

TCD Reading: 0.0000

Load from file	Allows you to load a previously saved calibration file.
Save to file	Allows you to save the current calibration values to a file.
Accept	Accepts the current values and closes the dialog.
Cancel	Cancels any changes made to the current calibration settings. A warning message is displayed before the dialog closes.
Reset	Enabled when a change has been made to any field on this dialog. Click this push button to reset any changed value(s) to its original state.

7. REPORTS MENU

The Reports menu contains options which allow you to start, open, and close reports. This menu also allows you to access the Peak Editor, a flexible and powerful tool for processing results.

This chapter describes the options contained on the Reports menu, as well as a few examples of generated reports.

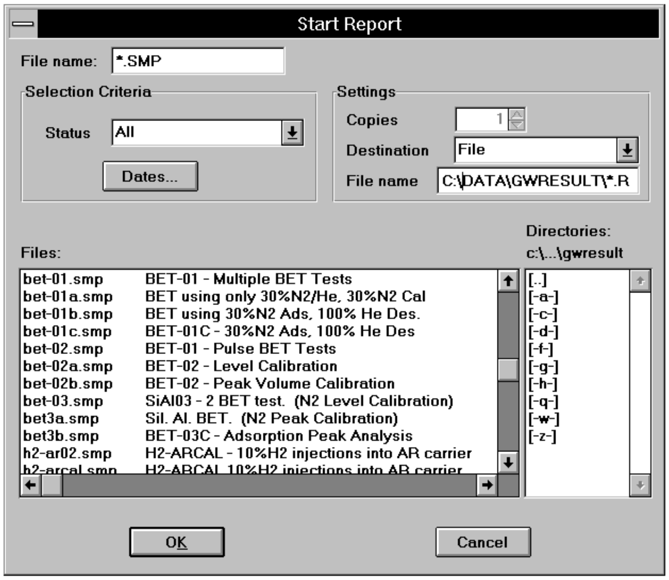
Description

Listed below are brief descriptions of the options contained on the Reports menu. Detailed descriptions follow this section.

Start Report	Allows you to generate a report on a sample which has been analyzed or on one which is being analyzed. Page **7-2**.
Close Reports	Allows you to close all open report windows. Page **7-3**.
Open Report	Allows you to open a saved report. Page **7-4**.
Peak Editor	Allows you to view peak data, combine and overlay results from multiple analyses, change the calibration files selected, and edit the data. Page **7-14**.

Start Report

Select this option to generate a report on a sample analysis. The Start Report dialog is displayed.



File name: *.SMP

Status: All

Copies: 1

Destination: File

File name: C:\DATA\GWRESULT*.R

Directories:
c:\...\gwresult

[..]
[-a-]
[-c-]
[-d-]
[-f-]
[-g-]
[-h-]
[-q-]
[-w-]
[-z-]

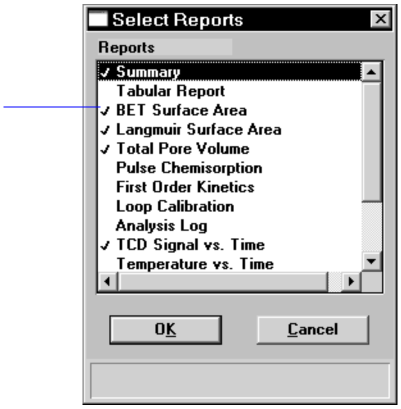
Files:

bet-01.smp	BET-01 - Multiple BET Tests
bet-01a.smp	BET using only 30%N2/He, 30%N2 Cal
bet-01b.smp	BET using 30%N2 Ads, 100% He Des.
bet-01c.smp	BET-01C - 30%N2 Ads, 100% He Des
bet-02.smp	BET-01 - Pulse BET Tests
bet-02a.smp	BET-02 - Level Calibration
bet-02b.smp	BET-02 - Peak Volume Calibration
bet-03.smp	SiAl03 - 2 BET test. (N2 Level Calibration)
bet3a.smp	Sil. Al. BET. (N2 Peak Calibration)
bet3b.smp	BET-03C - Adsorption Peak Analysis
h2-ar02.smp	H2-ARCAL - 10%H2 injections into AR carrier
h2-arcal.smp	H2-ARCAL 10%H2 injections into AR carrier

File name	The name you select from the Files list box is copied to this field. If you select multiple files, the last one selected is displayed. If you have a file open, its name is displayed.
Status	Refer to **“Selecting Files” on page 3-6**.
Dates	Refer to **“Selecting Files” on page 3-6**.
Copies	Enabled when the **Printer** destination is chosen, allowing you to print up to four copies of the selected report(s).
Destination	Displays a drop-down list of output destinations. *Choices: File, Printer, Screen* If you select **Printer**, requested reports are printed to the selected printer. If you select **Screen**, many options are available for manipulating and customizing reports. Refer to **Onscreen Reports** on page 7-5 for manipulation methods of onscreen reports.

Destination *(continued)*	If you select **File**, the tabular reports of the requested file are converted to a text file which can be viewed with a text editor or other text file manipulation tools. Graphical reports cannot be generated to a file.
File name	Enabled when you select **File** as the destination. Allows you to enter a name, or you may accept the default.
Files list box	Displays a list of the available sample files for the choice shown in the **Status** field.
Directories	Displays a list of available drives and directories. The drive and directory last accessed is displayed immediately above the **Directories** list box.

If you choose a single sample file and click **OK**, the Select Reports dialog is displayed so that you may select the desired reports. The reports selected are the ones you specified in the sample file. This dialog allows you to deselect reports or select additional ones. If you choose more than one file, this dialog is not displayed; the reports selected in each file are generated.



A check mark indicates the report is selected.

✓ Summary
Tabular Report
✓ BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption
First Order Kinetics
Loop Calibration
Analysis Log
✓ TCD Signal vs. Time
Temperature vs. Time

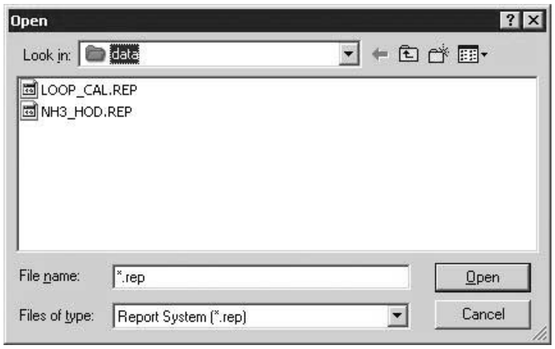
Double-click on the report(s) you wish to select (or deselect). A check mark is placed before each selected report. Pressing the **Spacebar** (when the report is highlighted) performs the same function. After you make your report selection and click **OK**, the requested reports are sent to the specified destination.

Close Reports

This option enables you to close all open report windows at one time. This avoids having to select close on each report window. This option is unavailable if reports are being generated.

Open Report

Select this option to open a saved report. The Open dialog is displayed.



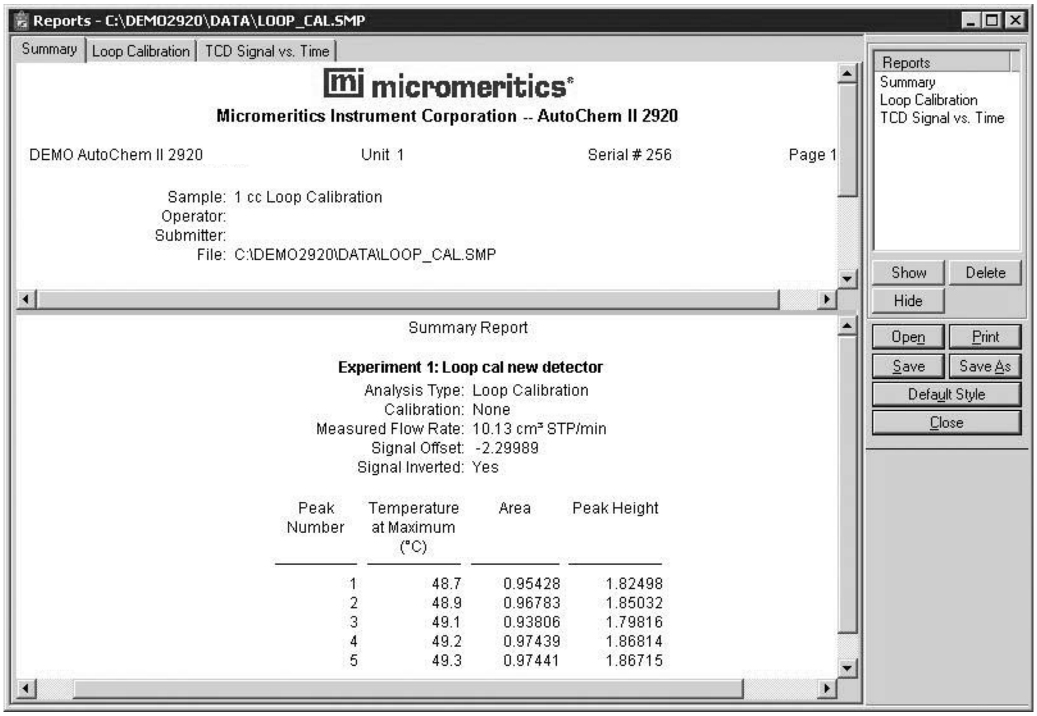
Look in: data

LOOP_CAL.REP
NH3_HOD.REP

File name: *.rep

Files of type: Report System (*.rep)

Select the desired report, then click **Open**. The report is displayed on the screen.



Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 1 Serial # 256 Page 1

Sample: 1 cc Loop Calibration
Operator:
Submitter:
File: C:\DEMO2920\DATA\LOOP_CAL.SMP

Summary Report

Experiment 1: Loop cal new detector

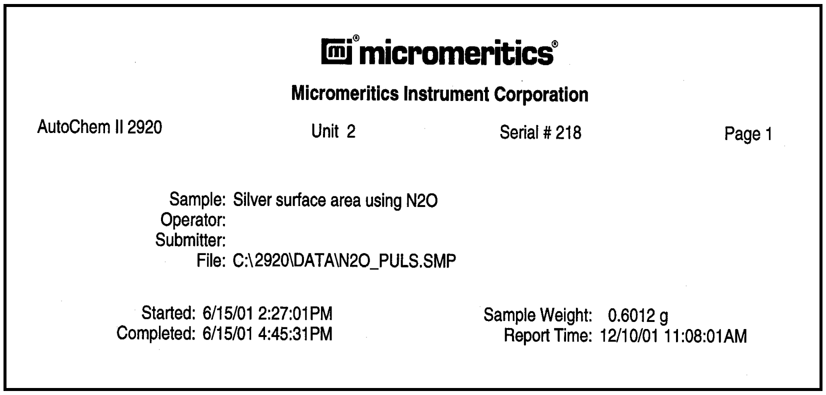
Analysis Type: Loop Calibration
Calibration: None
Measured Flow Rate: 10.13 cm³ STP/min
Signal Offset: -2.29989
Signal Inverted: Yes

Peak Number	Temperature at Maximum (°C)	Area	Peak Height
1	48.7	0.95428	1.82498
2	48.9	0.96783	1.85032
3	49.1	0.93806	1.79816
4	49.2	0.97439	1.86814
5	49.3	0.97441	1.86715

Refer to **“Onscreen Reports” on page 7-5** for an explanation of the commands available for onscreen reports.

Printed Reports

Header



micromeritics

Micromeritics Instrument Corporation

AutoChem II 2920 Unit 2 Serial # 218 Page 1

Sample: Silver surface area using N2O
Operator:
Submitter:
File: C:\2920\DATA\N2O_PULS.SMP

Started: 6/15/01 2:27:01PM
Completed: 6/15/01 4:45:31PM
Sample Weight: 0.6012 g
Report Time: 12/10/01 11:08:01AM

All printed reports (either to the screen or to a printer) contain a header displaying file statistics.

- Tabular and graphical reports contain sample and instrument statistics such as date and time of analysis, analysis conditions, and so forth.

  The headers for these reports also contain notes of any changes to the sample file which occur after analysis.

- Summary report headers contain the same type of information displayed in tabular and graphical reports with the exception of notes.

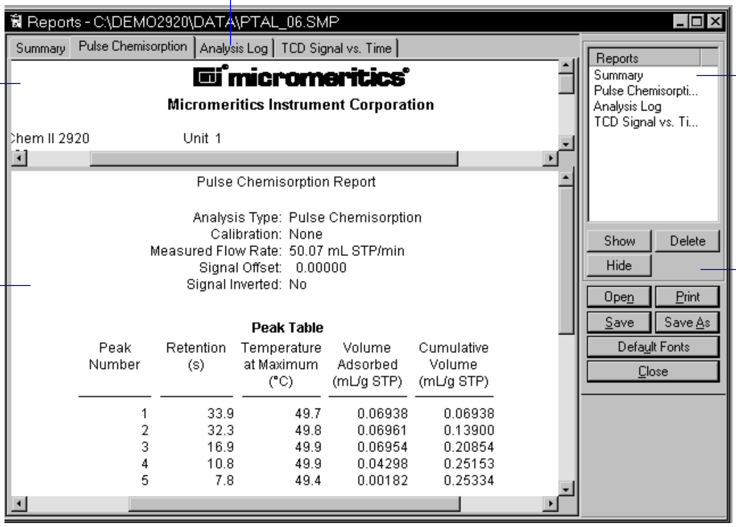
Onscreen Reports

The report window containing onscreen reports provides many options for customizing and manipulating reports:

- a tool bar
- shortcut menu options
- zoom feature
- axis cross hairs

When reports are printed to the screen, they are printed in a window like the one shown below. Each requested report is listed in the Reports window on the tool bar; reports are also indicated by selectable tabs across the top of the report header. To view a specific report, select its tab or select the report in the Reports window and click **Show**.

Tabs display for each type of report you choose to generate.



Micromeritics Instrument Corporation

hem II 2920 Unit 1

Pulse Chemisorption Report

Analysis Type: Pulse Chemisorption
Calibration: None
Measured Flow Rate: 50.07 mL STP/min
Signal Offset: 0.00000
Signal Inverted: No

Peak Table

Peak Number	Retention (s)	Temperature at Maximum (°C)	Volume Adsorbed (mL/g STP)	Cumulative Volume (mL/g STP)
1	33.9	49.7	0.06938	0.06938
2	32.3	49.8	0.06961	0.13900
3	16.9	49.9	0.06954	0.20854
4	10.8	49.9	0.04298	0.25153
5	7.8	49.4	0.00182	0.25334

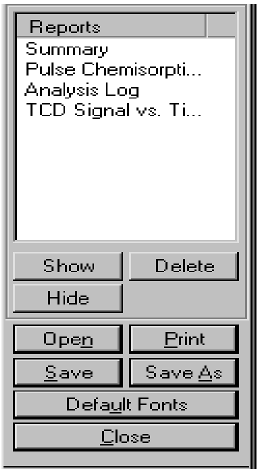
Header

List of reports requested

Displays tabular (or graphical) data.

Options on tool bar

Tool Bar



Reports	Contains a list of all requested reports.
Show	Shows the selected report in the report window. If the report has been hidden, it and its associated tab will become visible.

Delete

Deletes the selected report. A deletion confirmation dialog is displayed since this function cannot be undone. The deleted report(s) will have to be regenerated if deleted in error.

Hide

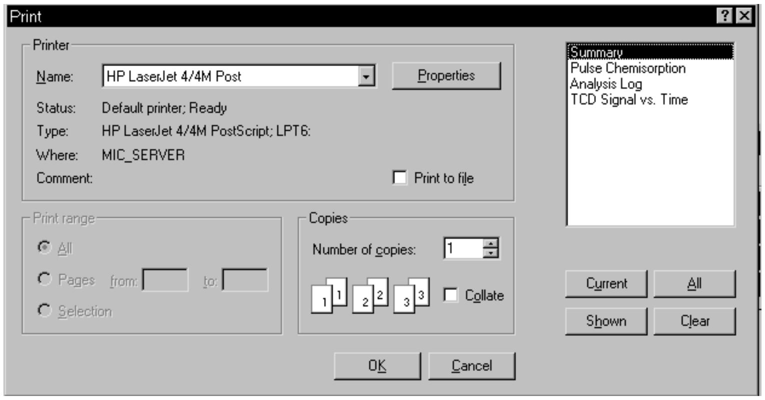
Hides (removes) the selected report from the report window. The report's associated tab is also removed.

Open

Allows you to open a previously saved report file.

Print

Displays a print dialog so that you can choose an appropriate printer for report output. A list of available reports is displayed in the window on the right side of the dialog.



Name: HP LaserJet 4/4M Post
Status: Default printer; Ready
Type: HP LaserJet 4/4M PostScript; LPT6:
Where: MIC_SERVER
Comment:
Number of copies: 1

Summary
Pulse Chemisorption
Analysis Log
TCD Signal vs. Time

For convenience in selecting which reports to print, push buttons are provided beneath the report window. Or, you can make your selection by clicking on the desired reports.

Current selects the report displayed in the report window.

Shown selects only the shown reports; any non-highlighted reports indicate they are hidden. You can still select hidden reports from this window to print.

All selects all reports, including those that may have been hidden.

Clear clears all selections.

Save

Saves all reports of the currently open file in a report format using the same name as the sample file, only with an **rep** extension. If you wish to specify a name and/or specific reports to save, use the **Save As** push button.

Save As

Allows you to save all or specified reports from the currently open file. The push buttons displayed on this dialog perform in the same manner as the print dialog (explained above).

Reports can be saved in three different formats:

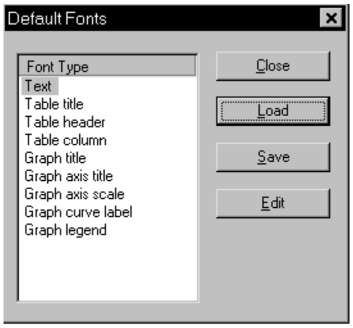
Report system (*.rep): Saved in a format which allows you to reopen the file using the **Open** push button on the Report window tool bar.

Spreadsheet (*.xls): Saved in a format which can be imported into most spreadsheet programs.

Ascii Text (*.txt): Saved in ASCII text which can be imported into programs accepting this type of file.

Default Fonts

Displays the Default Fonts dialog so that you can edit report fonts.



The window on the left lists the items for which the font can be edited.

Click **Edit** to edit the font of a selected item; a font dialog is displayed so that you can specify the desired font and attributes.

Click **Save** to save the changes as the default. If you do not click **Save**, the changes will apply to the current report set only. The next reports will revert to the default font.

Click **Load** to load the default font of a selected item.

Close

Closes the Report window.

If you have multiple reports open, you may wish to use the **Close Reports** option on the **Report** menu. This option allows you to close all open Report windows at one time, without having to click **Close** on each Report window.

Shortcut Menus

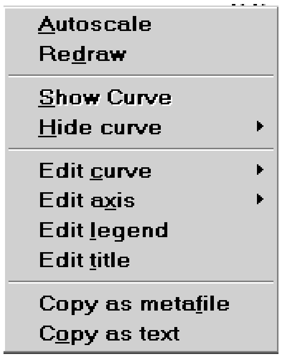
Shortcut menus are accessed when you right-click on the tabular or graphical portion of a report.

For Tabular Reports

Resize column	Displays a dialog so that you can specify the width of the selected column (in inches).
Rename column	Displays a dialog so that you can edit the name of the selected column. Use **Ctrl + Enter** to insert line feeds.
Move column	Allows you to move the location of the selected column to the left or to the right.
Align column	Enables you to right-align, left-align, or center the data in the selected column.
Hide column	Displays a list of all columns, enabling you to select the one you wish to hide.
Show column	Displays a list of all hidden columns, enabling you to select the one you wish to have shown again.
Column font	Displays a Font dialog, allowing you to change font attributes for the tabular data in the current report.
Header font	Displays a Font dialog, allowing you to change font attributes for column headers in the current report.

Edit title	Allows you to edit the table title and font.
Copy table as text	Enables you to copy the entire table (column headers and data) and then insert it into another program. Columns are tab-delimited, allowing easy alignment. Use this option to copy data easily and quickly from the AutoChem program to other applications such as spreadsheet programs.

For Graphs

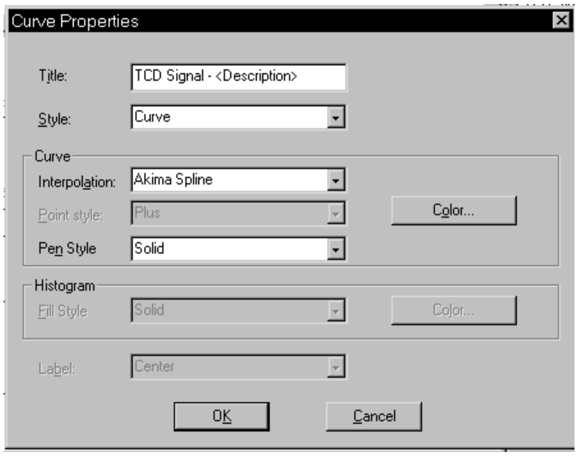


Autoscale	Sets the axis boundaries to autoscale, presenting the signal in full scale.
Redraw	Sets axis boundaries to their original view, allowing easier editing of peaks.

In many cases, the *Autoscale* and *Redraw* options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.

Show curve	Allows you to show curves that have been hidden. This option is disabled (greyed) if no curves have been hidden.
Hide curve	Allows you to hide (remove) unwanted curves.

Edit curve Displays the Curve Properties dialog, allowing you to edit curve properties.

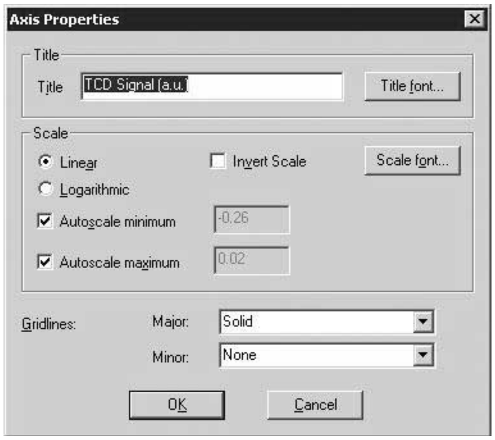


Title: TCD Signal - <Description>
Style: Curve
Interpolation: Akima Spline
Point style: Plus
Pen Style: Solid
Fill Style: Solid
Label: Center

Title	Displays the title of the curve you are editing.
Style	Drop-down list containing styles in which collected data can be displayed. *Choices: Curve, Histogram, Points, Curve and Points*
Curve group box	Contains options for curves and points. You can edit the curve interpolation, the style of curve and/or points, as well as the pen color. The options in this group box are disabled if Histogram is chosen in the **Style** drop-down list.
Histogram group box	Allows you to specify the type of fill as well as the color if Histogram is chosen as the style for collected data.

Edit axis

Displays the Axis Properties dialog, allowing you to edit axis properties.



Title

Title: TCD Signal (a.u.)

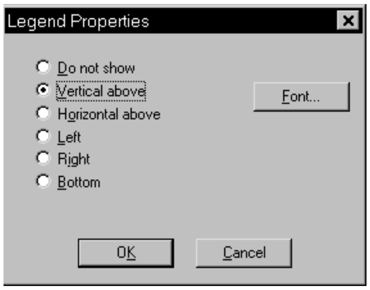
Scale

● Linear
○ Logarithmic
☐ Invert Scale
☑ Autoscale minimum: -0.26
☑ Autoscale maximum: 0.02

Gridlines: Major: Solid
Minor: None

Edit legend

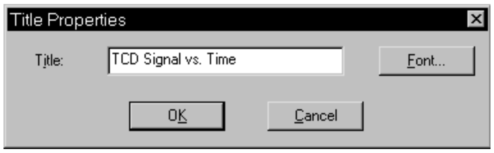
Displays the Legend Properties dialog, allowing you to edit the placement of the legend.



○ Do not show
● Vertical above
○ Horizontal above
○ Left
○ Right
○ Bottom

Edit title

Displays the Title Properties dialog, allowing you to edit the current graph’s title and font.



Title: TCD Signal vs. Time

Copy as metafile

Copies the graph and places it on the clipboard, allowing you to paste it into other applications accepting Windows metafiles.

Use this option to capture graphics for use in reports and presentations.

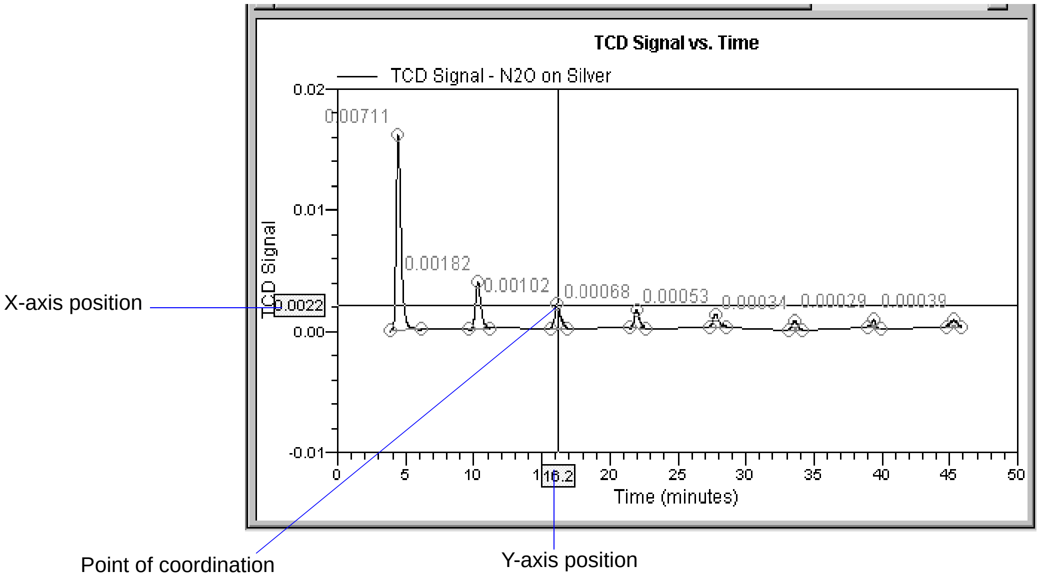
Copy as text	Copies the data used to generate the graph as a series of tab-delimited columns of text.

Zoom Feature

A zoom feature is included with the report system so that you can zoom in to examine fine detail of the distribution. To use this feature, simply hold down the left mouse button and drag the mouse cursor (drawing a box) across the area you wish to view; then release the button. The enlarged area immediately fills the graph area. You can return to the normal view by right-clicking on the graph and selecting Autoscale.

Axis Cross-Hair

A cross-hair function is available so that you can view axis coordinates. To use this feature, simply left-click in the desired area of the graph.

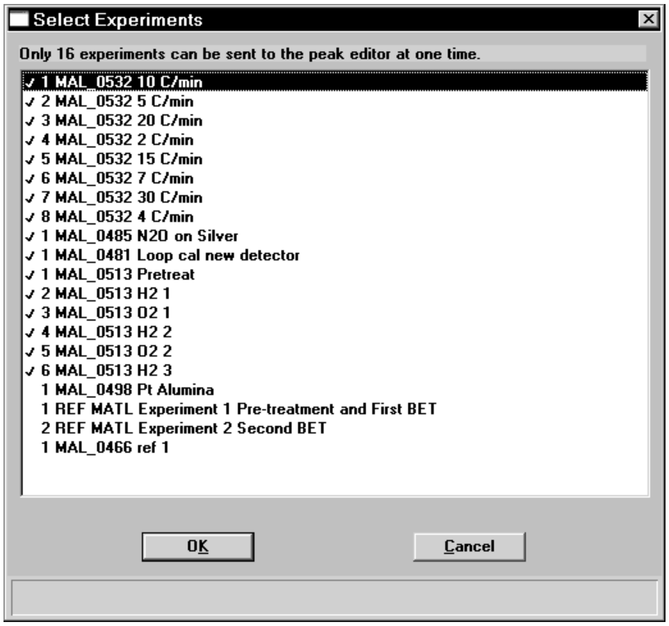


You can remove cross-hair lines and return to the normal view by right-clicking on the graph and selecting **Autoscale** or **Redraw**. You can also click out of the graph area to remove cross-hair lines.

Peak Editor

The Peak Editor allows you to view and edit data from sample files. You can view the peaks in a stacked or overlaid manner. You can also edit the peaks and delete undesirable ones.

When you select the Peak Editor option, the Edit Sample Peaks dialog allowing you to select the desired file(s) is displayed. The fields on this dialog function in the same manner as other dialogs allowing you to select files. The Peak Editor allows you to view up to 16 experiments at one time. If you choose files containing more than 16 experiments, a dialog displaying the experiments available in the chosen files is displayed.



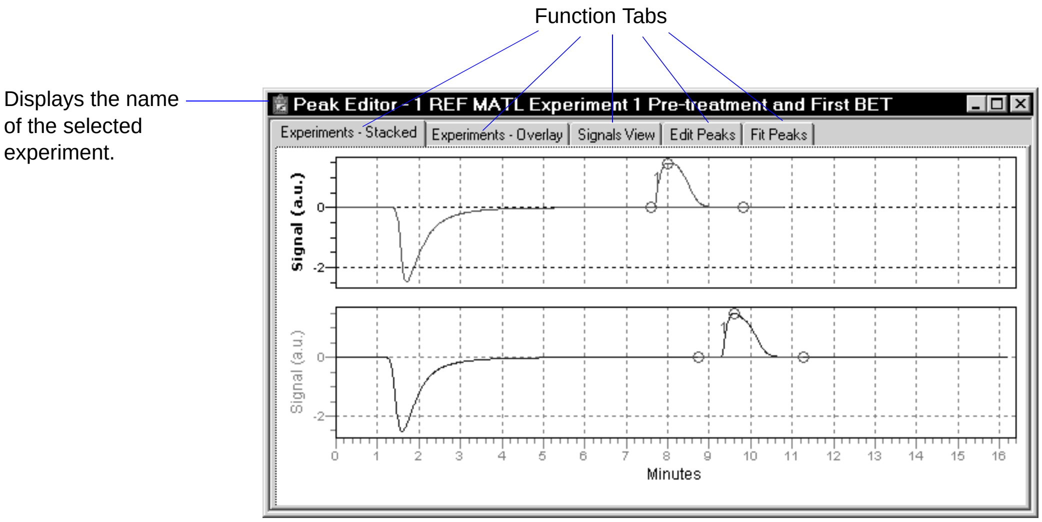
Select Experiments

Only 16 experiments can be sent to the peak editor at one time.

✓ 1 MAL_0532 10 C/min
✓ 2 MAL_0532 5 C/min
✓ 3 MAL_0532 20 C/min
✓ 4 MAL_0532 2 C/min
✓ 5 MAL_0532 15 C/min
✓ 6 MAL_0532 7 C/min
✓ 7 MAL_0532 30 C/min
✓ 8 MAL_0532 4 C/min
✓ 1 MAL_0485 N2O on Silver
✓ 1 MAL_0481 Loop cal new detector
✓ 1 MAL_0513 Pretreat
✓ 2 MAL_0513 H2 1
✓ 3 MAL_0513 O2 1
✓ 4 MAL_0513 H2 2
✓ 5 MAL_0513 O2 2
✓ 6 MAL_0513 H2 3
1 MAL_0498 Pt Alumina
1 REF MATL Experiment 1 Pre-treatment and First BET
2 REF MATL Experiment 2 Second BET
1 MAL_0466 ref 1

This dialog allows you to choose the 16 experiments you wish to view and defaults with the first 16 experiments selected. You may deselect and select as desired. This is accomplished by double-clicking on the file, or by highlighting the file and pressing the space bar (the same method used in selecting reports).

After you select your file(s) or your experiments (whichever is relevant) and click **OK**, the Peak Editor window is displayed.



This window contains five tabs. the first three tabs — **Experiments Stacked**, **Experiments Overlay**, and **Signals View** — provide different views of the data; the last two tabs — **Edit Peaks** and **Fit Peaks** — provide the TCD vs. time (or temperature) and tabular information for the integrated peaks.

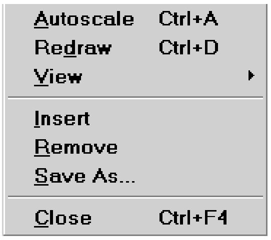
Features of the Peak Editor Window

The Peak Editor window provides the following tools for viewing and editing peaks:

- Shortcut menus
- Shortcut keys
- Zoom in on the TCD signal
- Cross-hairs

Shortcut Menu

A shortcut menu is available for each view of the Peak Editor window. This shortcut menu contains the available functions and is accessed by right-clicking in the graph area. For example, when you right-click in the Experiments view, the following menu is displayed:



The options on the menu displayed for each view are explained in subsequent sections.

Shortcut Keys

Shortcut keys are provided for many of the functions in the Peak Editor. These shortcut keys have been assigned function-relevant characters enabling you to remember them more quickly. For example, **Ctrl+A** is assigned to Autoscale. Refer to the following table for a complete list of the shortcut keys you can use while operating the Peak Editor.

Table 7-1. Shortcut Keys for the Peak Editor

Key(s)	Function
Ctrl + A	Sets the axes to autoscale (full scale view)
Ctrl + D	Returns the Peak Editor window to its original view
Ctrl + M	Shows or Hides Peak Marks
Ctrl + T	Displays the X-axis by Time or Temperature
Ctrl + F	Copies a graph(s) as a Metafile, allowing you to paste it into another application
Ctrl + C	Copies data to the clipboard, allowing you to paste it into another application
Ctrl + N	Advances you to the next peak
Ctrl + P	Returns you to the previous peak
Ctrl + L	Marks the left edge of the peak
Ctrl + R	Marks the right edge of the peak
Ctrl + S	Saves the changes made to the current file

Zoom Feature

A zoom feature is included with the Peak Editor allowing you to zoom in on peaks. This feature functions in the same manner as the zoom for reports; hold down the left mouse button and drag the mouse cursor (drawing a box) across the area you wish to view; then release the button. If you have multiple graphs displayed, the area you define on the selected graph is enlarged for each graph. You can return to the normal view by right-clicking on the graph and selecting Autoscale, or you can use the shortcut keys **Ctrl + D**.

Cross-Hairs

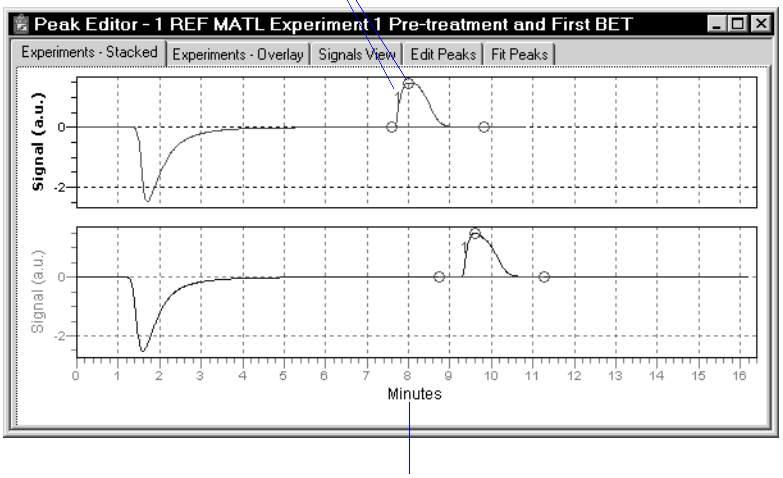
When you click the mouse in the graph area, locator cross-hairs are displayed. The exact values associated with the intersection of the cross-hairs are displayed along the axes. This makes it easy to determine exact values for points on the second Y-axis, or for the point on the time/temperature trace that corresponds with the signal/temperature trace. Refer to **Axis Cross-Hair** on page **7-13** for additional information on cross-hairs.

Experiments

The Experiments View shows curves for all experiments within the selected files. You can display the curves in a stacked fashion or in an overlaid fashion. You can display up to 16 experiments; each experiment is displayed in a different color.

Stacked

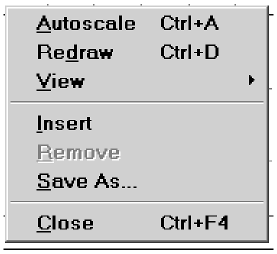
Peak marks can be shown or hidden.



X-axis can be displayed as Time or Temperature.

The Stacked view displays up to 16 experiments, stacked one after the other. In this view, you can select the individual experiments to obtain information. When you select an experiment, its name is displayed in the title bar of the window. Cross-hairs are also displayed to show exact values in the selected area.

When you right-click in the graph area, the following shortcut menu is displayed:



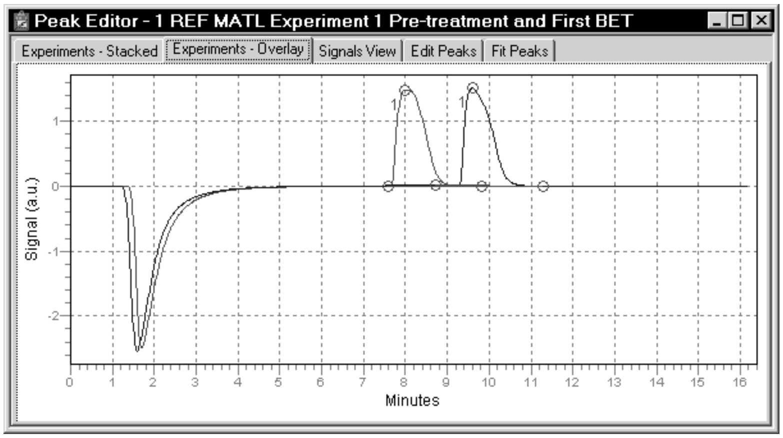
Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1, page 7-16, for a list of the shortcut keys available with the Peak Editor. You may wish to make a copy of this table and keep it close to your keyboard.

Autoscale	Sets the axis boundaries to autoscale, presenting the signal in full scale.
Redraw	Sets axis boundaries to their original view, allowing easier editing of peaks.

In many cases, the Autoscale and Redraw options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.

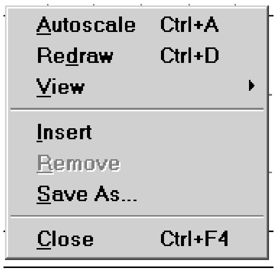
View	Displays a cascading menu allowing you to: • Show or hide the peak marks • Specify X-axis in Time or Temperature • Copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application
Insert	Enables you to insert experiments from another sample file.
Remove	Removes the selected experiment from the Peak editor window; the data are not deleted from the sample file.
Save As	Allows you to save the experiments in the window as a new sample file. For example, if you have a file opened containing five experiments but are only interested in two of them, you can remove the three of no interest and save the file with the remaining two.
Close	Closes the Peak Editor window.

Overlay



The Overlay view displays up to 16 experiments, overlaid on each other. This view does not allow you to select an experiment; you must switch to the Stacked view to select experiments.

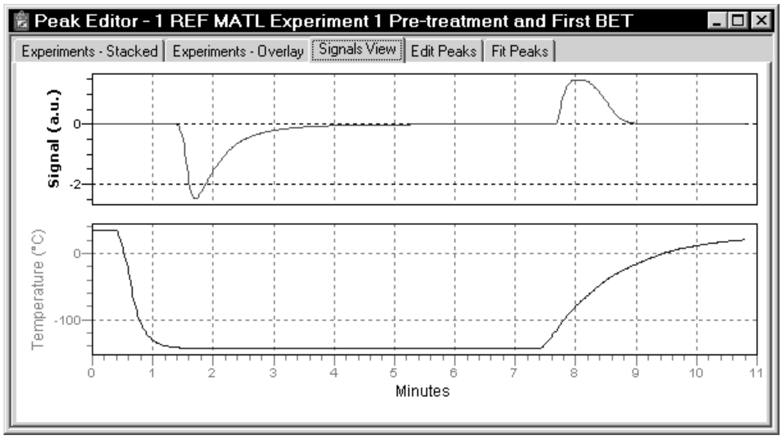
When you right-click in the graph area, the following shortcut menu is displayed:



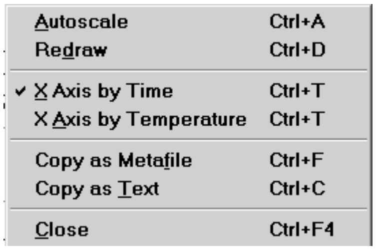
The options on this menu are identical to the ones available in the Stacked view (page 7-17). **Remove** is disabled on this menu because individual experiments cannot be selected in the Overlay view.

Signals View

The Signals view displays the signals for the selected experiement stacked one on top of the other. This allows you to view the relationship of peaks to temperature and auxiliary signal conditions that occurred during data collection.



When you right-click in the graph area of the Signals view, the following shortcut menu is displayed:



Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1 on page 7-16 for a list of the shortcut keys available with the Peak Editor. You may wish to make a copy of this page and keep it close to your keyboard.

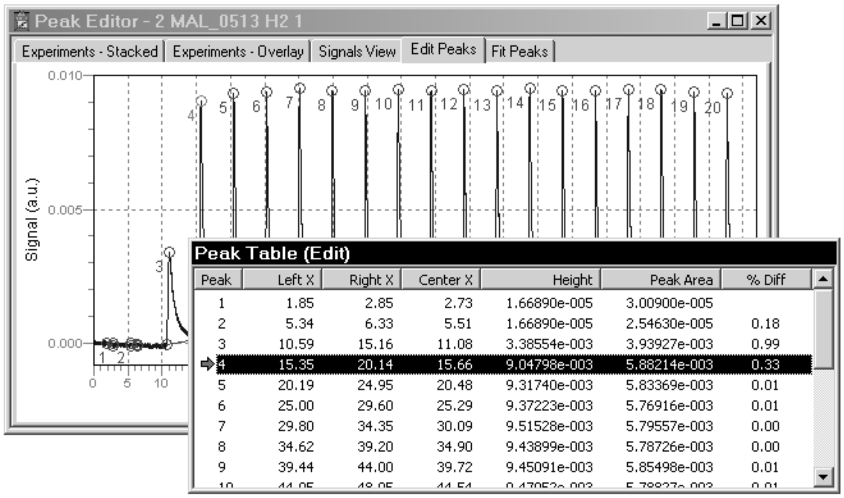
Autoscale	Sets the axis boundaries to autoscale, presenting the signal in full scale.
Redraw	Sets axis boundaries to their original view, allowing easier editing of peaks.

In many cases, the *Autoscale* and *Redraw* options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.

X-Axis	Enables you to display the X-axis by Time or Temperature.
Copy	Enables you to copy the graph area to the clipboard as a metafile or as text, then paste it into another application.
Close	Closes the Peak Editor window.

Edit Peaks

Edit Peaks allows you to edit the peaks of the selected experiment; the Peak Table dialog listing peak information for all peaks of the selected experiment is displayed.



Peak Table (Edit)

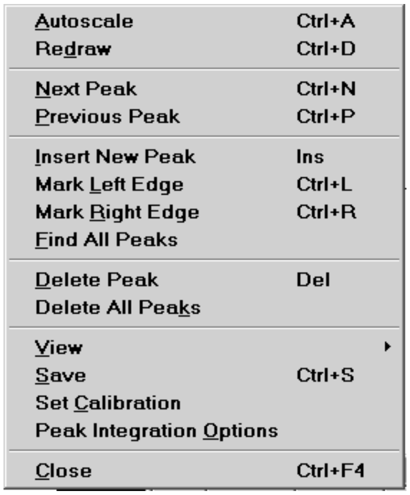
Peak	Left X	Right X	Center X	Height	Peak Area	% Diff
1	1.85	2.85	2.73	1.66890e-005	3.00900e-005	
2	5.34	6.33	5.51	1.66890e-005	2.54630e-005	0.18
3	10.59	15.16	11.08	3.38554e-003	3.93927e-003	0.99
➡ 4	15.35	20.14	15.66	9.04798e-003	5.88214e-003	0.33
5	20.19	24.95	20.48	9.31740e-003	5.83369e-003	0.01
6	25.00	29.60	25.29	9.37223e-003	5.76916e-003	0.01
7	29.80	34.35	30.09	9.51528e-003	5.79557e-003	0.00
8	34.62	39.20	34.90	9.43899e-003	5.78726e-003	0.00
9	39.44	44.00	39.72	9.45091e-003	5.85498e-003	0.01

Use one of the following methods to select a peak:

- Highlight the desired peak in the Peak Table
- Access the shortcut menu and select **Next Peak** or **Previous Peak** until you navigate to the desired peak
- Use **Ctrl + N** (advance to the next peak) or **Ctrl + P** (return to the previous peak) to navigate to the desired peak

When a peak is selected, its peak marks display in a light green color; the peak marks for unselected peaks display in red.

Peaks are edited by accessing functions from the shortcut menu which is displayed when you right-click in the graph area.



Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1 on page 7-16 for a list of the shortcut keys available with the Peak Editor. You may wish to make a copy of this page and keep it close to your keyboard.

Autoscale	Sets the axis boundaries to autoscale, presenting the signal in full scale.
Redraw	Sets axis boundaries to their original view, allowing easier editing of peaks.

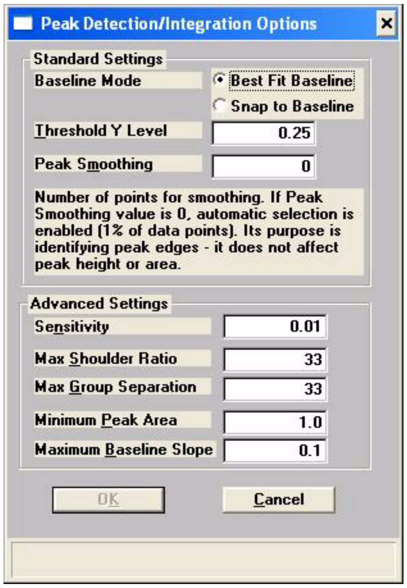
In many cases, the *Autoscale* and *Redraw* options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.

Next Peak	Advances you to the next peak.
Previous Peak	Returns you to the previous peak.
Insert New Peak	Inserts a new peak in a graph. Click in the graph where you want to insert a peak, then select **Insert New Peak**.
Mark Left Edge	A peak is automatically marked by the program; this option allows you to redefine the left edge. Simply place the mouse cursor where you wish to have the left edge marked, right-click, and select this option.

Mark Right Edge	A peak is automatically marked by the program; this option allows you to redefine the right edge. Use the same method used for marking the left edge.
Find All Peaks	This option is used to find all peaks automatically in the Peak Editor window. This is a rapid technique for integrating pulse chemisorption data. This option is most effective for narrow peaks with good baseline separation.
Delete Peak	Removes the selected peak from the peak table. Deletes all peak information for a single peak.
Delete All Peaks	Clears the Peak table of all data and removes all markings from the peaks.
View	Displays a cascading menu allowing you to: • Show the peak table • Show or hide the peak marks • Specify X-axis in Time or Temperature • Enable or disable smoothing, and invert or zero the signal (peak) • Copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application
Save	Saves the peak table to the sample file.
Set Calibration	Allows you to choose a TCD calibration that is appropriate for the current experiment.

Peak Integration Options

Displays the Peak Detection/Integration Options dialog.



Standard Settings

Baseline Mode: ● Best Fit Baseline ○ Snap to Baseline

Threshold Y Level: 0.25

Peak Smoothing: 0

Number of points for smoothing. If Peak Smoothing value is 0, automatic selection is enabled (1% of data points). Its purpose is identifying peak edges - it does not affect peak height or area.

Advanced Settings

Sensitivity: 0.01

Max Shoulder Ratio: 33

Max Group Separation: 33

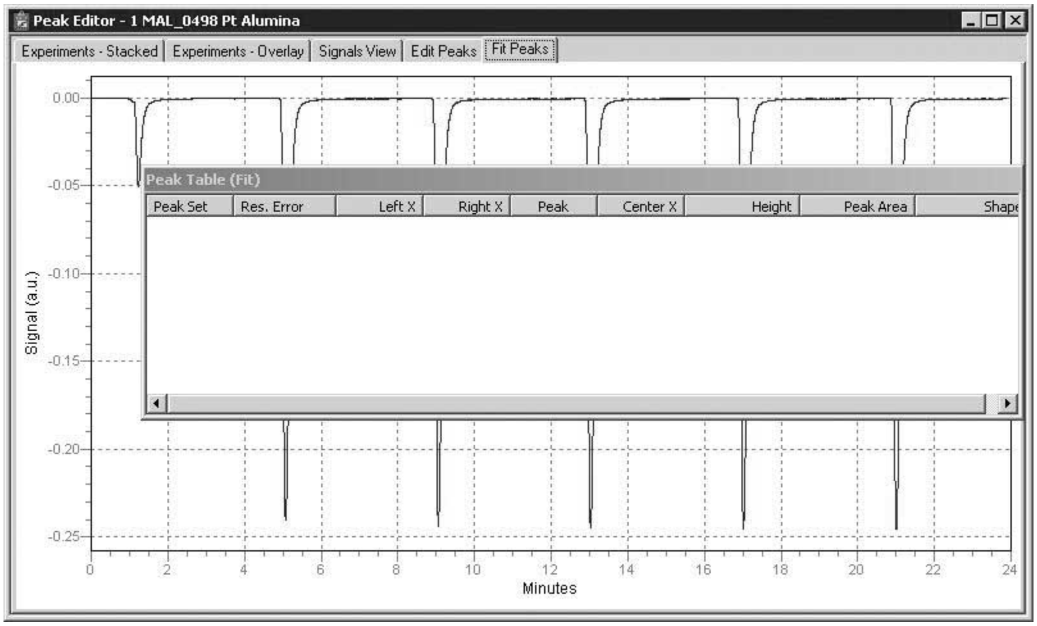
Minimum Peak Area: 1.0

Maximum Baseline Slope: 0.1

This dialog allows you to adjust the parameters enabling you to optimize the results you obtain using the Find All Peaks option. Refer to Appendix H, page **H-1** for additional information on this dialog.

Fit Peaks

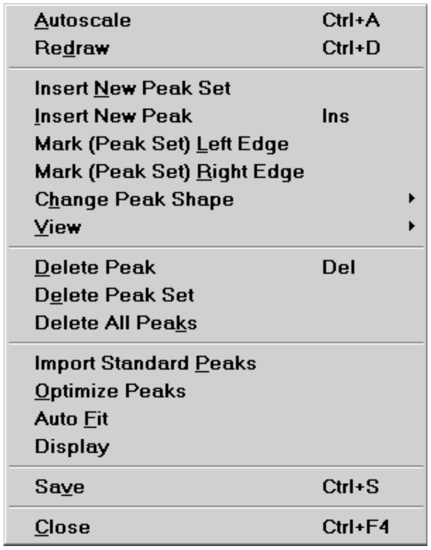
Fit Peaks allows you to separate overlapping peaks by fitting curves to the data, and save them as a new peak table.



Peak Table (Fit)

Peak Set	Res. Error	Left X	Right X	Peak	Center X	Height	Peak Area	Shape

Right-click in the graph area to access the shortcut menu for creating the table.



Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1 on page 7-16 for a list of the shortcut keys available with the Peak Editor. You may wish to make a copy of this page and keep it close to your keyboard.

Autoscale	Sets the axis boundaries to autoscale, presenting the signal in full scale.
Redraw	Sets axis boundaries to their original view, allowing easier editing of peaks.

In many cases, the *Autoscale* and *Redraw* options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.

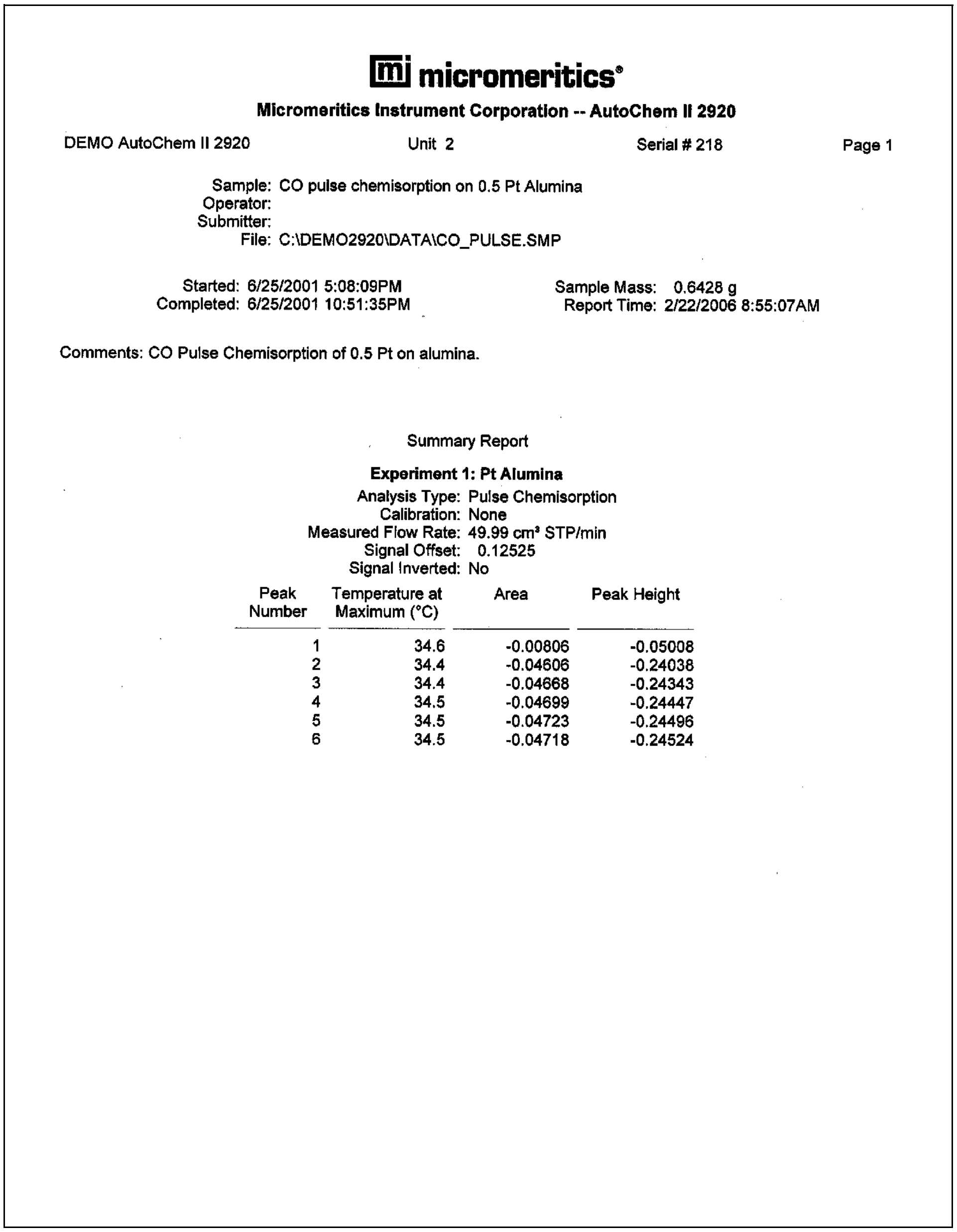
Insert New Peak Set	Enables you to define a region of the TCD signal that contains multiple peaks. The peak fitting and optimization are constrained to this region of the TCD signal. Click in the graph where you want to insert a new peak set, then select **Insert New Peak Set**. After you add the peak set, you can use the commands on the graph shortcut menu to define the peak set (mark edges, for example).

Insert New Peak	Inserts a new peak in a graph. Click in the graph where you want to insert a peak, the select **Insert New Peak**
Mark (Peak Set) Left Edge	Changes the left edge of the peak set.
Mark (Peak Set) Right Edge	Changes the right edge of the peak set.
Change Peak Shape	Allows you to choose the type of peak shape.
Log Normal Skewed	This is a 4-parameter, log-normal shape that allows for skewed peaks, and is the default peak.
Gaussian	This is the traditional symmetrical shape.
View	Displays a cascading menu enabling you to show the peak table or copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application
Delete Peak	Removes the selected peak from the peak set table.
Delete Peak Set	Deletes the selected Peak set from the table.
Delete All Peaks	Removes all peaks and peak-set information from the table.
Import Standard Peaks	Imports the saved peak parameters from the Peak Edit screen and uses them for the initial estimates for the peak set.
Optimize Peaks	Locates the best peak height, width, and skewness after initial peak estimates have been given.
Auto Fit	Locates the best number of peaks and parameters from the Peak Set without user estimates.
Display	Enables you to smooth the signal for display purposes only, and to select or deselect zeroing and signal inversion.
Save	Save the current peak set.
Close	Closes the Peak Editor window.

Report Examples

This section contains examples of some of the types of reports that may be generated by the AutoChem II 2920 analysis program. You can customize portions of some reports, such as the title and the axes of graphs, using the Report Options tab of the sample information file (Advanced format only). If reports are printed to the Screen, there are many ways in which they can be manipulated and customized.

Summary Report



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 2 Serial # 218 Page 1

Sample: CO pulse chemisorption on 0.5 Pt Alumina
Operator:
Submitter:
File: C:\DEMO2920\DATA\CO_PULSE.SMP

Started: 6/25/2001 5:08:09PM
Completed: 6/25/2001 10:51:35PM
Sample Mass: 0.6428 g
Report Time: 2/22/2006 8:55:07AM

Comments: CO Pulse Chemisorption of 0.5 Pt on alumina.

Summary Report

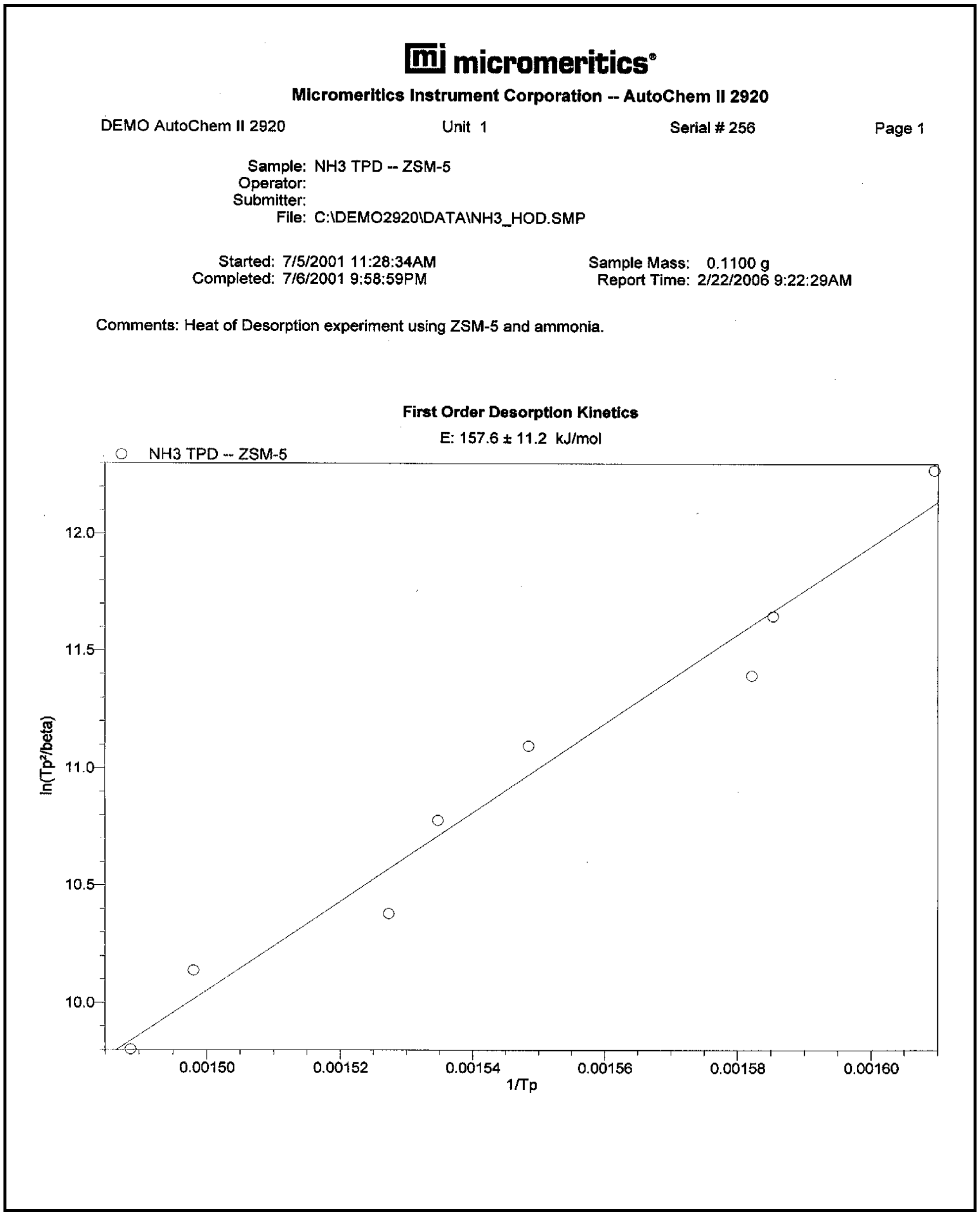
Experiment 1: Pt Alumina

Analysis Type: Pulse Chemisorption
Calibration: None
Measured Flow Rate: 49.99 cm^3 STP/min
Signal Offset: 0.12525
Signal Inverted: No

Peak Number	Temperature at Maximum (°C)	Area	Peak Height
1	34.6	-0.00806	-0.05008
2	34.4	-0.04606	-0.24038
3	34.4	-0.04668	-0.24343
4	34.5	-0.04699	-0.24447
5	34.5	-0.04723	-0.24496
6	34.5	-0.04718	-0.24524

TPD Analysis

First Order Kinetics



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 1 Serial # 256 Page 1

Sample: NH3 TPD -- ZSM-5
Operator:
Submitter:
File: C:\DEMO2920\DATA\NH3_HOD.SMP

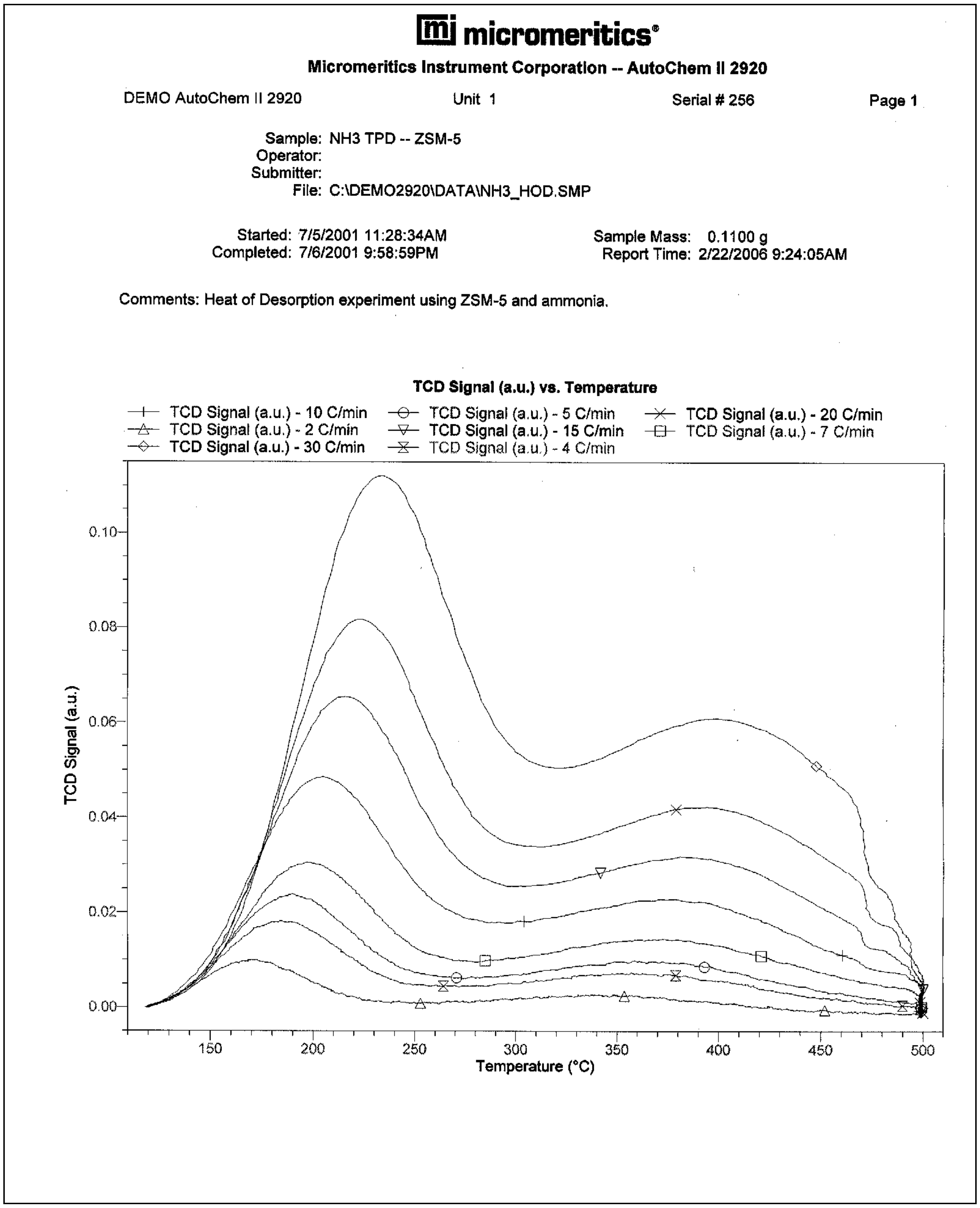
Started: 7/5/2001 11:28:34AM
Completed: 7/6/2001 9:58:59PM
Sample Mass: 0.1100 g
Report Time: 2/22/2006 9:22:29AM

Comments: Heat of Desorption experiment using ZSM-5 and ammonia.

First Order Desorption Kinetics

E: 157.6 ± 11.2 kJ/mol

TCD Signal vs. Temperature



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 1 **Serial # 256** **Page 1**

Sample: NH3 TPD -- ZSM-5
Operator:
Submitter:
File: C:\DEMO2920\DATA\NH3_HOD.SMP

Started: 7/5/2001 11:28:34AM
Completed: 7/6/2001 9:58:59PM

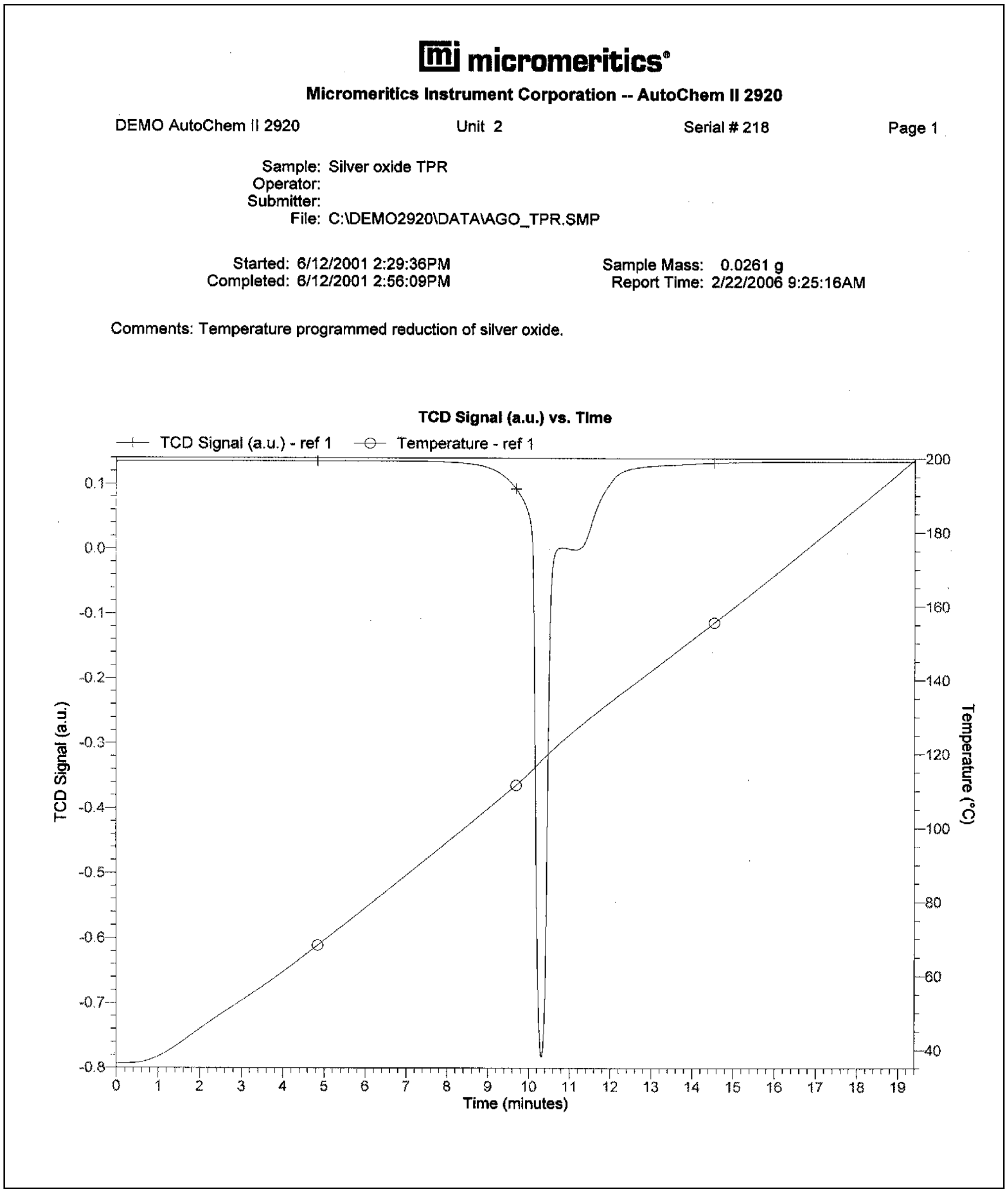
Sample Mass: 0.1100 g
Report Time: 2/22/2006 9:24:05AM

Comments: Heat of Desorption experiment using ZSM-5 and ammonia.

TCD Signal (a.u.) vs. Temperature

TPR Analysis

TCD Signal vs. Time



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 2 Serial # 218 Page 1

Sample: Silver oxide TPR
Operator:
Submitter:
File: C:\DEMO2920\DATA\AGO_TPR.SMP

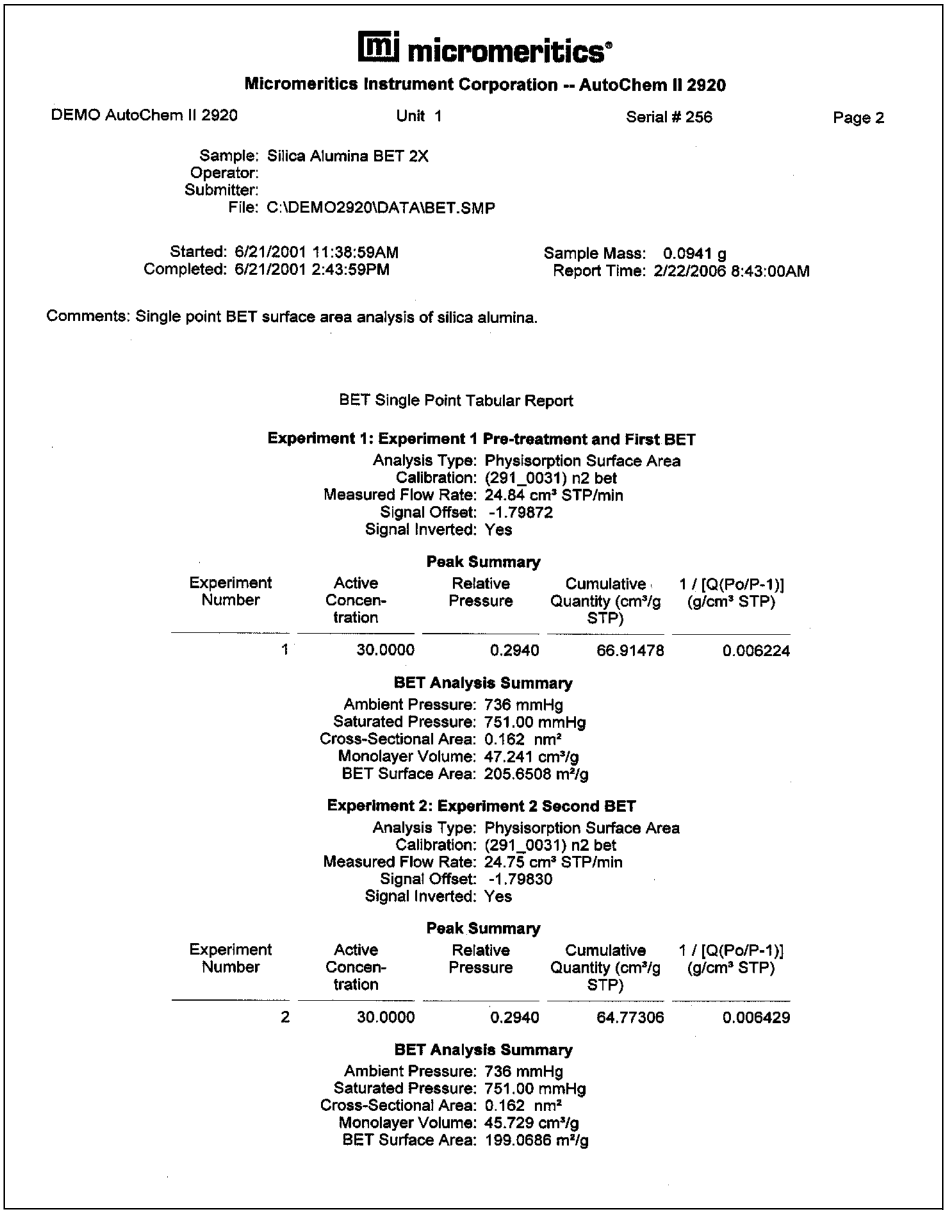
Started: 6/12/2001 2:29:36PM
Completed: 6/12/2001 2:56:09PM

Sample Mass: 0.0261 g
Report Time: 2/22/2006 9:25:16AM

Comments: Temperature programmed reduction of silver oxide.

TCD Signal (a.u.) vs. Time

BET Surface Area



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 1 Serial # 256 Page 2

Sample: Silica Alumina BET 2X
Operator:
Submitter:
File: C:\DEMO2920\DATA\BET.SMP

Started: 6/21/2001 11:38:59AM
Completed: 6/21/2001 2:43:59PM

Sample Mass: 0.0941 g
Report Time: 2/22/2006 8:43:00AM

Comments: Single point BET surface area analysis of silica alumina.

BET Single Point Tabular Report

Experiment 1: Experiment 1 Pre-treatment and First BET

Analysis Type: Physisorption Surface Area
Calibration: (291_0031) n2 bet
Measured Flow Rate: 24.84 cm^3 STP/min
Signal Offset: -1.79872
Signal Inverted: Yes

Peak Summary

Experiment Number	Active Concentration	Relative Pressure	Cumulative Quantity (cm^3/g STP)	1 / [Q(Po/P-1)] (g/cm^3 STP)
1	30.0000	0.2940	66.91478	0.006224

BET Analysis Summary

Ambient Pressure: 736 mmHg
Saturated Pressure: 751.00 mmHg
Cross-Sectional Area: 0.162 nm^2
Monolayer Volume: 47.241 cm^3/g
BET Surface Area: 205.6508 m^2/g

Experiment 2: Experiment 2 Second BET

Analysis Type: Physisorption Surface Area
Calibration: (291_0031) n2 bet
Measured Flow Rate: 24.75 cm^3 STP/min
Signal Offset: -1.79830
Signal Inverted: Yes

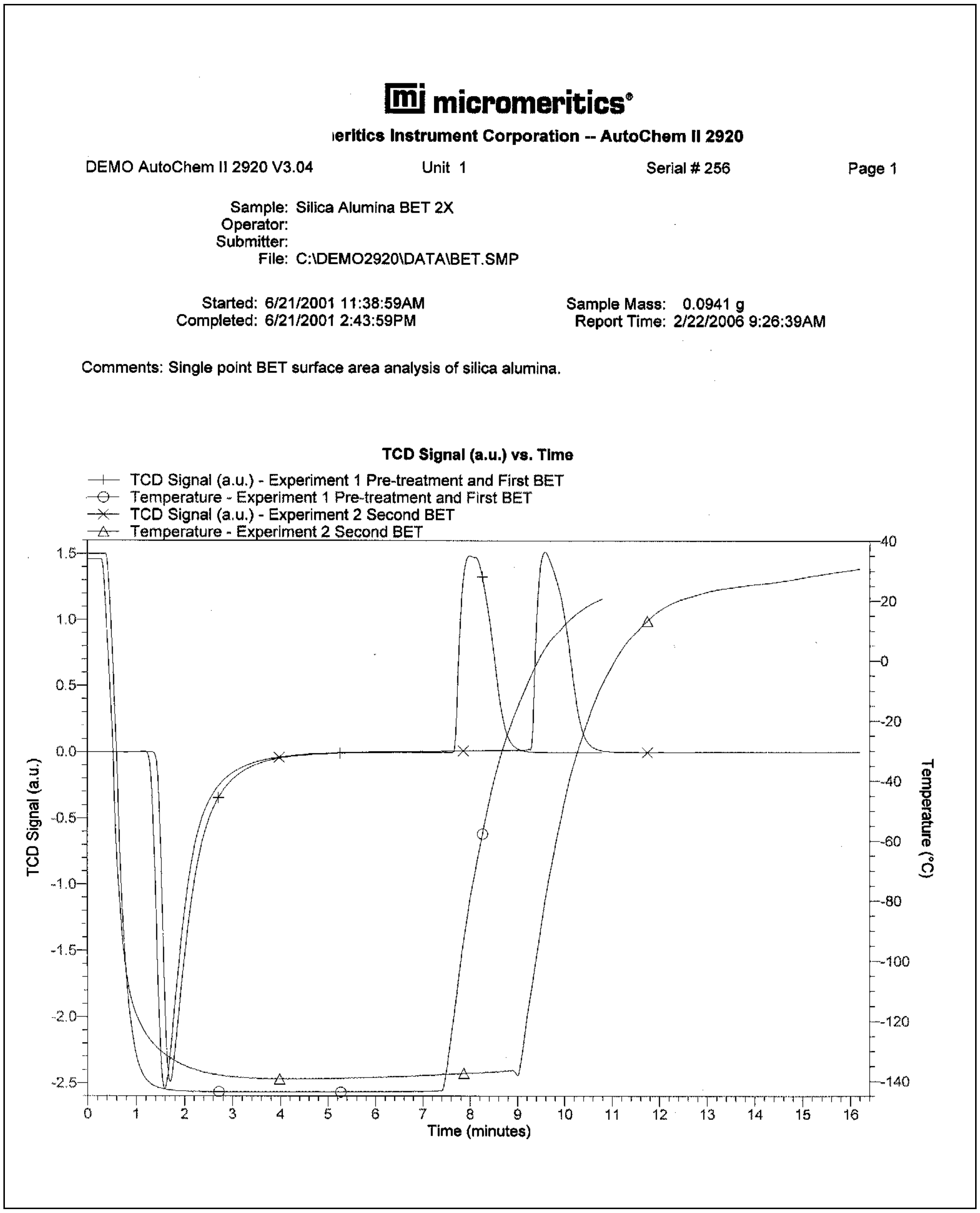
Peak Summary

Experiment Number	Active Concentration	Relative Pressure	Cumulative Quantity (cm^3/g STP)	1 / [Q(Po/P-1)] (g/cm^3 STP)
2	30.0000	0.2940	64.77306	0.006429

BET Analysis Summary

Ambient Pressure: 736 mmHg
Saturated Pressure: 751.00 mmHg
Cross-Sectional Area: 0.162 nm^2
Monolayer Volume: 45.729 cm^3/g
BET Surface Area: 199.0686 m^2/g

TCD Signal vs. Time



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DEMO AutoChem II 2920 V3.04 Unit 1 Serial # 256 Page 1

Sample: Silica Alumina BET 2X
Operator:
Submitter:
File: C:\DEMO2920\DATA\BET.SMP

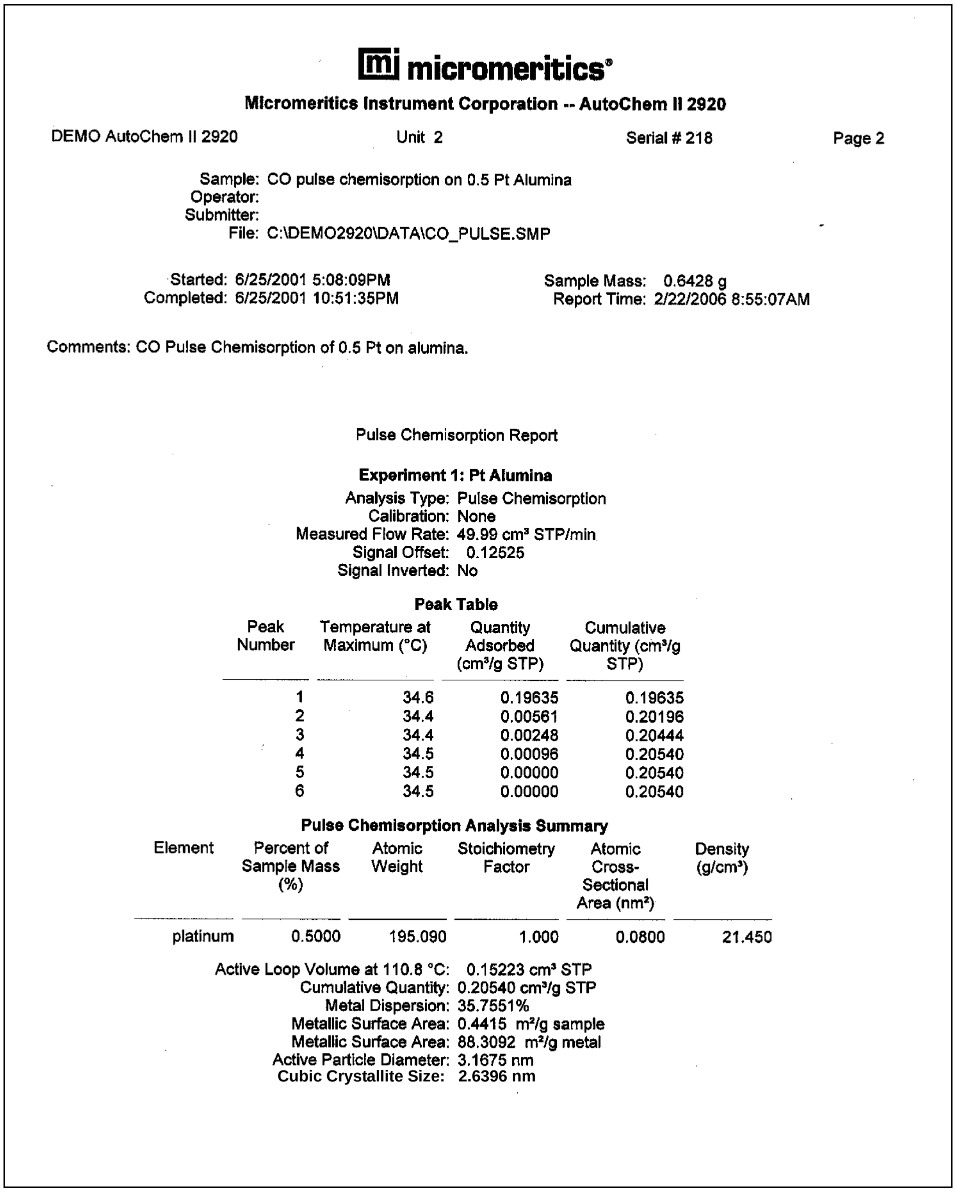
Started: 6/21/2001 11:38:59AM
Completed: 6/21/2001 2:43:59PM

Sample Mass: 0.0941 g
Report Time: 2/22/2006 9:26:39AM

Comments: Single point BET surface area analysis of silica alumina.

TCD Signal (a.u.) vs. Time

Pulse Chemisorption



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 2 Serial # 218 Page 2

Sample: CO pulse chemisorption on 0.5 Pt Alumina
Operator:
Submitter:
File: C:\DEMO2920\DATA\CO_PULSE.SMP

Started: 6/25/2001 5:08:09PM
Completed: 6/25/2001 10:51:35PM
Sample Mass: 0.6428 g
Report Time: 2/22/2006 8:55:07AM

Comments: CO Pulse Chemisorption of 0.5 Pt on alumina.

Pulse Chemisorption Report

Experiment 1: Pt Alumina

Analysis Type: Pulse Chemisorption
Calibration: None
Measured Flow Rate: 49.99 cm³ STP/min
Signal Offset: 0.12525
Signal Inverted: No

Peak Table

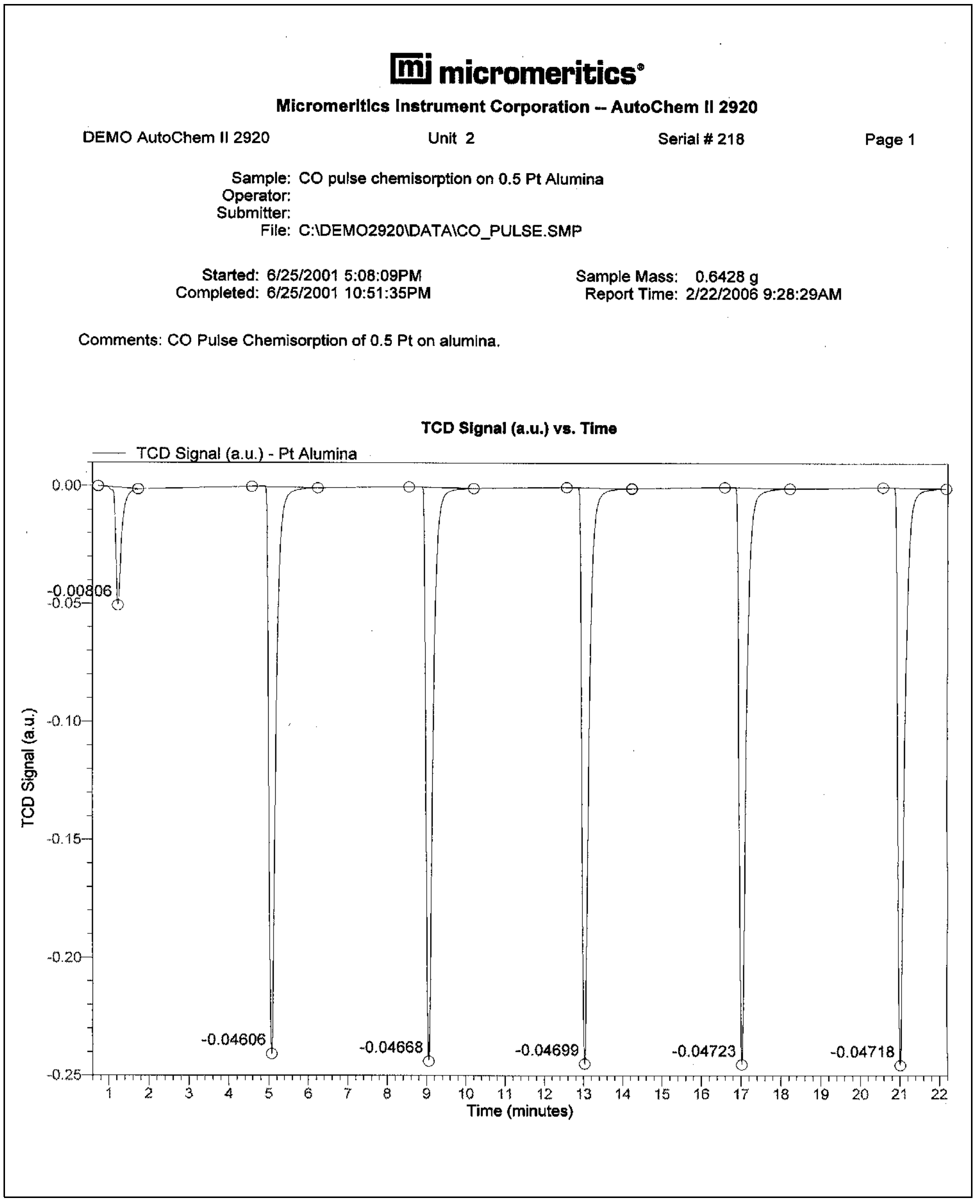
Peak Number	Temperature at Maximum (°C)	Quantity Adsorbed (cm³/g STP)	Cumulative Quantity (cm³/g STP)
1	34.6	0.19635	0.19635
2	34.4	0.00561	0.20196
3	34.4	0.00248	0.20444
4	34.5	0.00096	0.20540
5	34.5	0.00000	0.20540
6	34.5	0.00000	0.20540

Pulse Chemisorption Analysis Summary

Element	Percent of Sample Mass (%)	Atomic Weight	Stoichiometry Factor	Atomic Cross-Sectional Area (nm²)	Density (g/cm³)
platinum	0.5000	195.090	1.000	0.0800	21.450

Active Loop Volume at 110.8 °C: 0.15223 cm³ STP
Cumulative Quantity: 0.20540 cm³/g STP
Metal Dispersion: 35.7551%
Metallic Surface Area: 0.4415 m²/g sample
Metallic Surface Area: 88.3092 m²/g metal
Active Particle Diameter: 3.1675 nm
Cubic Crystallite Size: 2.6396 nm

TCD Signal vs. Time



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 2 Serial # 218 Page 1

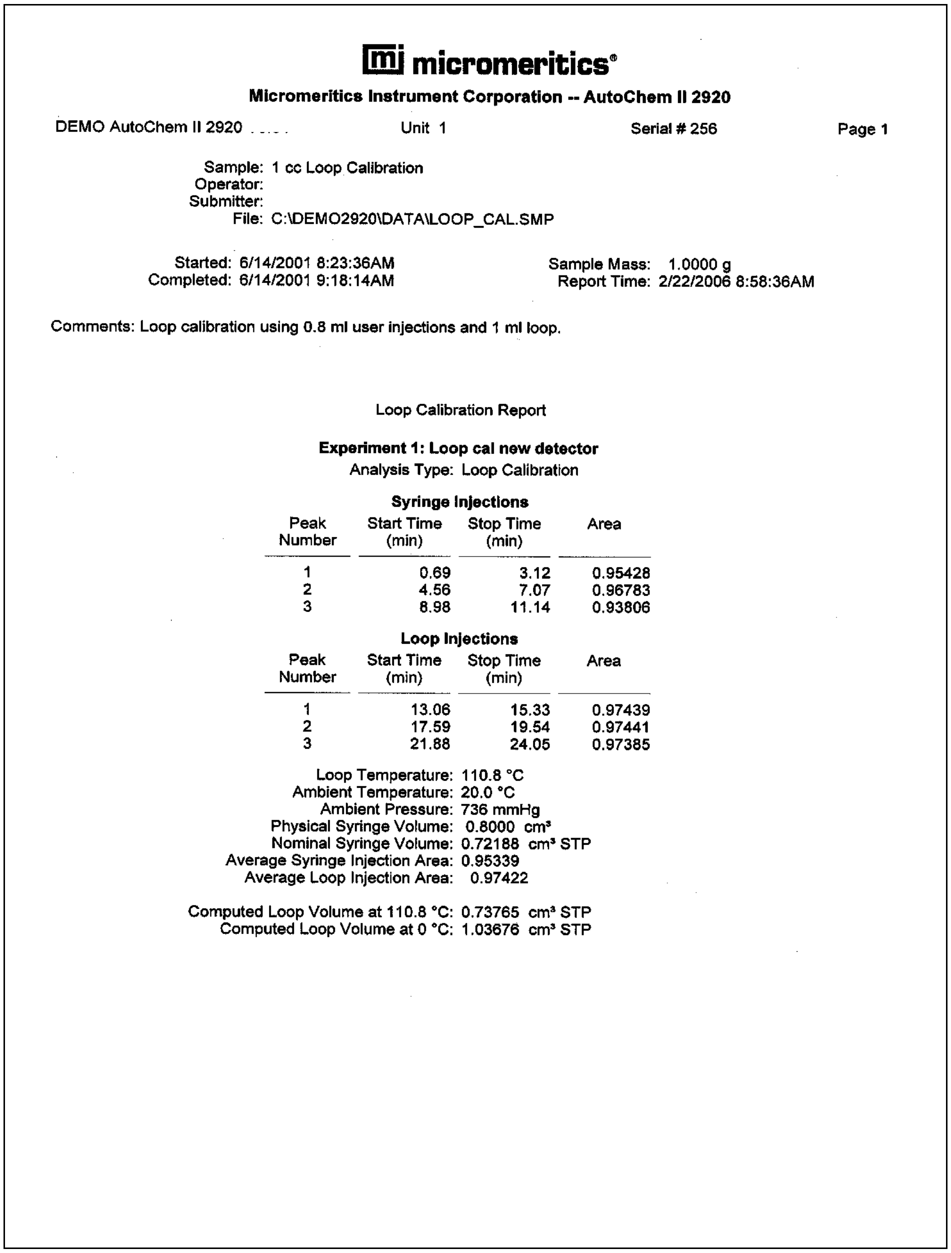
Sample: CO pulse chemisorption on 0.5 Pt Alumina
Operator:
Submitter:
File: C:\DEMO2920\DATA\CO_PULSE.SMP

Started: 6/25/2001 5:08:09PM
Completed: 6/25/2001 10:51:35PM
Sample Mass: 0.6428 g
Report Time: 2/22/2006 9:28:29AM

Comments: CO Pulse Chemisorption of 0.5 Pt on alumina.

TCD Signal (a.u.) vs. Time

Loop Calibration



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Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 1 **Serial # 256** **Page 1**

Sample: 1 cc Loop Calibration
Operator:
Submitter:
File: C:\DEMO2920\DATA\LOOP_CAL.SMP

Started: 6/14/2001 8:23:36AM
Completed: 6/14/2001 9:18:14AM
Sample Mass: 1.0000 g
Report Time: 2/22/2006 8:58:36AM

Comments: Loop calibration using 0.8 ml user injections and 1 ml loop.

Loop Calibration Report

Experiment 1: Loop cal new detector

Analysis Type: Loop Calibration

Syringe Injections

Peak Number	Start Time (min)	Stop Time (min)	Area
1	0.69	3.12	0.95428
2	4.56	7.07	0.96783
3	8.98	11.14	0.93806

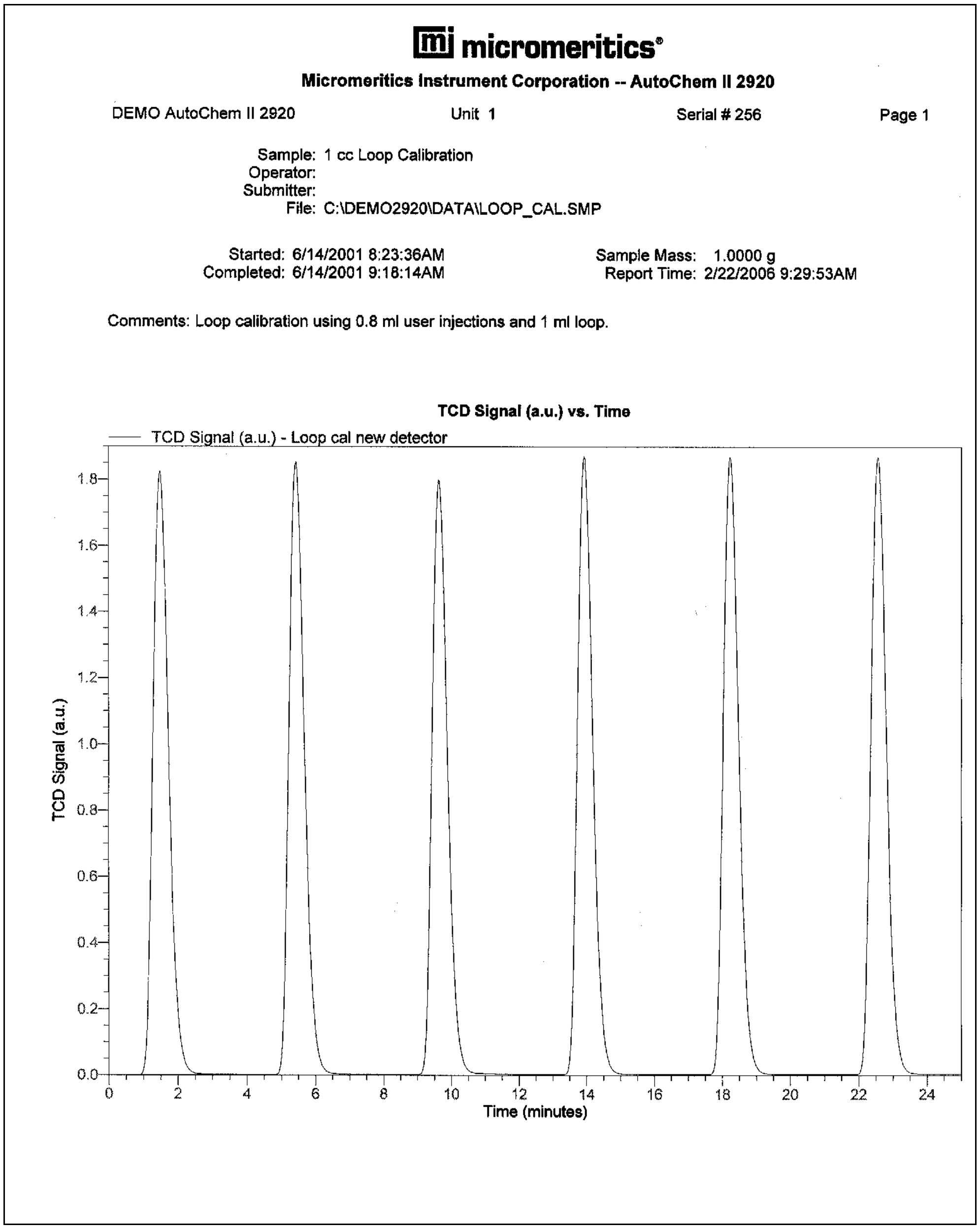
Loop Injections

Peak Number	Start Time (min)	Stop Time (min)	Area
1	13.06	15.33	0.97439
2	17.59	19.54	0.97441
3	21.88	24.05	0.97385

Loop Temperature: 110.8 °C
Ambient Temperature: 20.0 °C
Ambient Pressure: 736 mmHg
Physical Syringe Volume: 0.8000 cm^3
Nominal Syringe Volume: 0.72188 cm^3 STP
Average Syringe Injection Area: 0.95339
Average Loop Injection Area: 0.97422

Computed Loop Volume at 110.8 °C: 0.73765 cm^3 STP
Computed Loop Volume at 0 °C: 1.03676 cm^3 STP

TCD Signal vs. Time



micromeritics®

Micromeritics Instrument Corporation -- AutoChem II 2920

DEMO AutoChem II 2920 Unit 1 Serial # 256 Page 1

Sample: 1 cc Loop Calibration
Operator:
Submitter:
File: C:\DEMO2920\DATA\LOOP_CAL.SMP

Started: 6/14/2001 8:23:36AM
Completed: 6/14/2001 9:18:14AM
Sample Mass: 1.0000 g
Report Time: 2/22/2006 9:29:53AM

Comments: Loop calibration using 0.8 ml user injections and 1 ml loop.

TCD Signal (a.u.) vs. Time

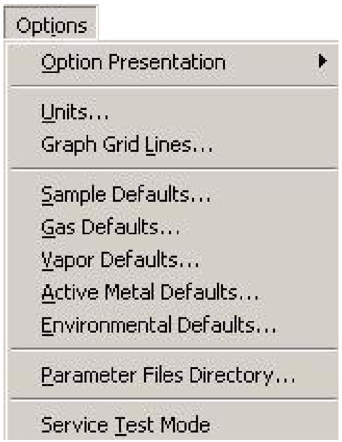
8. OPTIONS MENU

The Options menu enables you to configure the system to suit your laboratory's requirements. With the options on this menu, you can:

- Specify data presentation modes
- Specify units of measurement
- Specify the grid layout of graphical reports
- Select a default for the format of the sample information file, as well as for its values
- Specify or edit program defaults
- Enter environmental conditions
- Specify a directory for predefined parameter files

Description

Listed below are brief descriptions of the Options menu contents. Detailed descriptions are found in this chapter.



Option Presentation	Allows you to set the default for the sample information file presentation format. Choose between Basic, Advanced, or Restricted format. Page **8-3**.
Units	Allows you to select the units of measurement for quantity and pressure data presented on the screen and in printed reports. Page **8-5**.
Graph Grid Lines	Allows you to specify the way in which graph lines are shown on the screen and in printed reports. Page **8-6**.

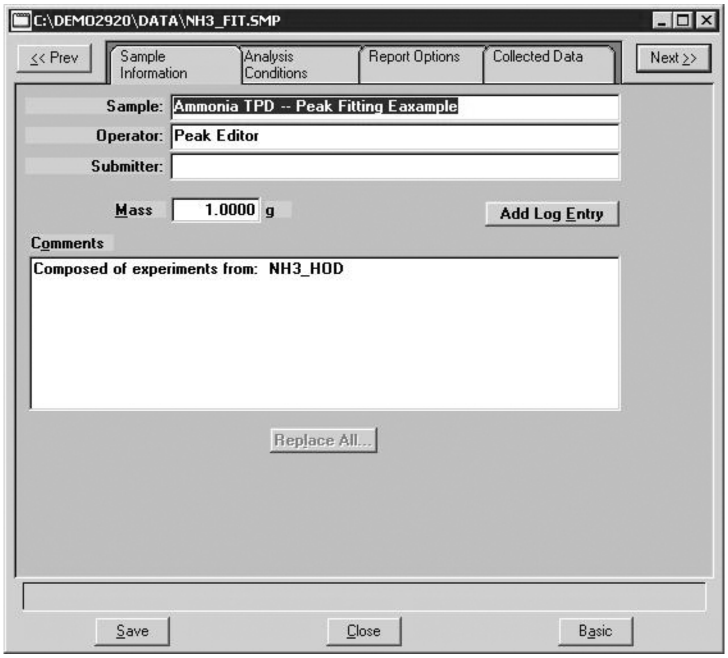
Sample Defaults	Allows you to specify defaults for the parameters contained in the sample information file. Page **8-7**.
Gas Defaults	Provides a table defining characteristics of gases used for analysis, to be used in calculations. Page **8-13**.
Vapor Defaults	Allows you to enter the Antoine constants for vapors and/or edit vapor defaults. Page **8-14**.
Active Metal Defaults	Provides a table defining characteristics of metals contained in samples, to be used in calculations**.** Page **8-15**.
Environmental Defaults	Allows you to enter the current environmental conditions, so that their effect on analyses can be accounted for in calculations. Page **8-16**.
Parameter File Directory	Allows you to specify a directory for predefined parameter files displayed in the drop-down lists of the Basic sample information dialog. Page **8-17**.
Service Test Mode	Provides password-protected access to certain functions of the analyzer to Micromeritics-trained service personnel only. Do not attempt to access Service Test Mode unless you are specifically directed to do so by a Micromeritics service representative. Page **8-18**.

Option Presentation

Sample information file dialogs may be presented in three formats: Advanced, Basic, and Restricted.

Advanced Format

Presents all four parts of the sample information file as a group of dialog boxes with index card-type tabs (the Collected data tab does not appear until after analysis). Clicking on each tab opens an associated dialog.



C:\DEMO2920\DATA\NH3_FIT.SMP

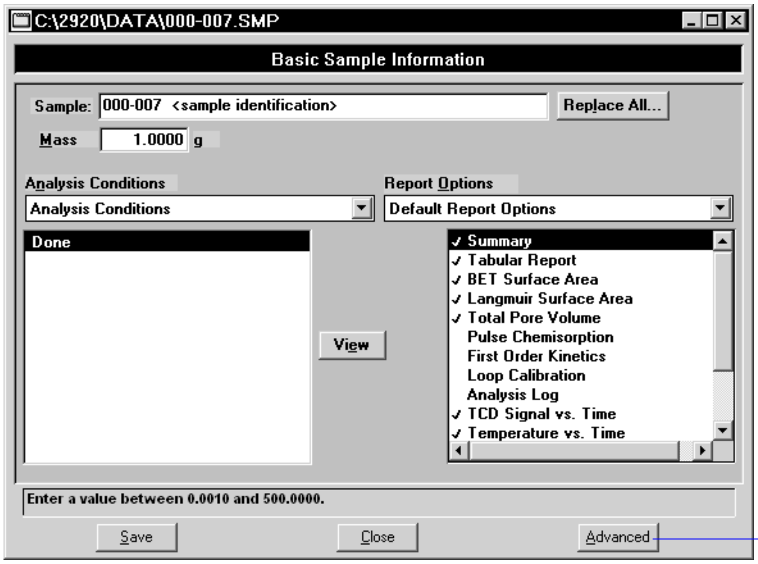
Sample: Ammonia TPD -- Peak Fitting Eaxample
Operator: Peak Editor
Submitter:
Mass 1.0000 g
Comments
Composed of experiments from: NH3_HOD

Basic and Advanced format files provide a toggle button for conveniently switching back and forth between them, regardless of which format is specified as the default.

Use this format when you wish to edit the parameter files and create customized Sample Information files, rather than using standard parameter files. Just click on the tab of the parameter you wish to edit, or use **Next** and **Prev** to navigate among the dialogs.

Basic Format

Presents the entire sample information file in a single dialog box.



C:\2920\DATA\000-007.SMP

Basic Sample Information

Sample: 000-007 <sample identification>

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

✓ Summary
✓ Tabular Report
✓ BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption
First Order Kinetics
Loop Calibration
Analysis Log
✓ TCD Signal vs. Time
✓ Temperature vs. Time

Enter a value between 0.0010 and 500.0000.

Not available in the Restricted format.

Basic format makes it easy to select previously defined parameter files from convenient drop-down lists. If editing is required, you can easily switch to the **Advanced** format by clicking **Advanced**.

Restricted Format

Restricted format is identical to the Basic format, except that certain menu options are disabled. You also cannot switch to the Advanced format. This format is password-protected and typically is used in laboratories where analysis conditions must remain constant, for example, in the pharmaceutical industry.

Option Presentation Password

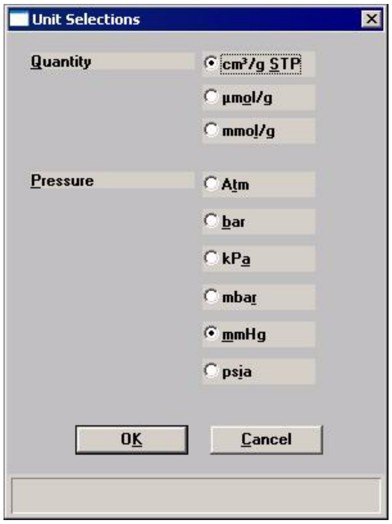
Please set a new password.

****

You can enter any password (up to 31 characters) to enable the Restricted format. The same password must be used to exit the Restricted format. For example, if you enter “password” to enable the restricted format, then you must enter “password” to exit. If you forget the password, open the system INI file and navigate to the Private section. The password is shown immediately following “OptionPresentationPassword.” Make a note of the password, exit the INI file, and enter the password where requested. Deleting the password from the INI file will not disable the Restricted mode; you must enter the password using the Password dialog to exit the Restricted format.

Units

This option enables you to select the units of measurement for the quantities and pressures displayed on the screen or in printed reports.



Quantity
● cm³/g STP
○ µmol/g
○ mmol/g

Pressure
○ Atm
○ bar
○ kPa
○ mbar
● mmHg
○ psia

Quantity

Choose one of the following for quantity data presentation:

cm^3/g STP
µmol/g
mmol/g

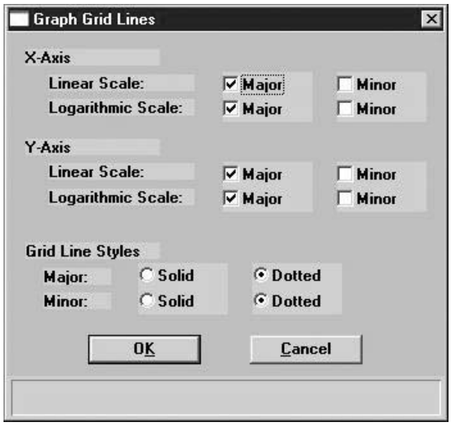
Pressure

Choose one of the following for pressure data presentation:

Atm
bar
kPa
mbar
mmHg
psia

Graph Grid Lines

Graph Grid Lines enables you specify how you wish graph lines to be displayed on screen and in printed reports; the Graph Grid Lines dialog is displayed.



X-Axis
Linear Scale: ☑ Major ☐ Minor
Logarithmic Scale: ☑ Major ☐ Minor

Y-Axis
Linear Scale: ☑ Major ☐ Minor
Logarithmic Scale: ☑ Major ☐ Minor

Grid Line Styles
Major: ○ Solid ● Dotted
Minor: ○ Solid ● Dotted

X-Axis **Y-Axis**	Enables you to choose **Major** and/or **Minor** lines to display in printed reports for the Logarithmic and Linear scales. If you deselect these items (remove the check marks), your report will not display grid lines.
Grid Line Styles	Allows you to choose the type of grid line to display if grid lines are being shown.

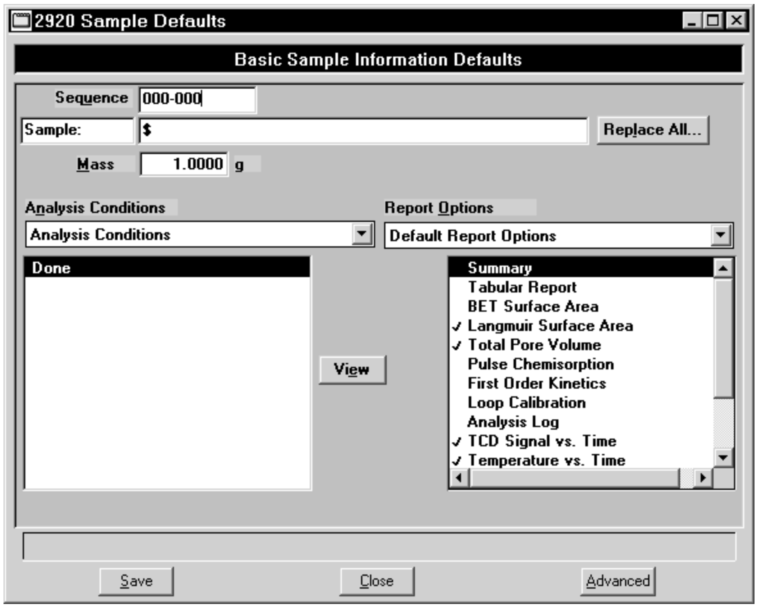
Sample Defaults

This option allows you to specify default parameters for the dialogs used to create sample information files.

Sample defaults are the values you see in the sample information editors when you create a new sample file. For efficiency, it is best to specify defaults for materials you most commonly analyze. You can always edit the values in the sample file when it is created.

The dialog used for specifying default parameters is presented in the same format you have selected as your default for sample information files. Sample defaults cannot be specified using the Restricted format.

Basic



Sequence 000-000

Sample: $

Mass 1.0000 g

Analysis Conditions: Analysis Conditions

Done

Report Options: Default Report Options

- Summary
- Tabular Report
- BET Surface Area
- ✓ Langmuir Surface Area
- ✓ Total Pore Volume
- Pulse Chemisorption
- First Order Kinetics
- Loop Calibration
- Analysis Log
- ✓ TCD Signal vs. Time
- ✓ Temperature vs. Time

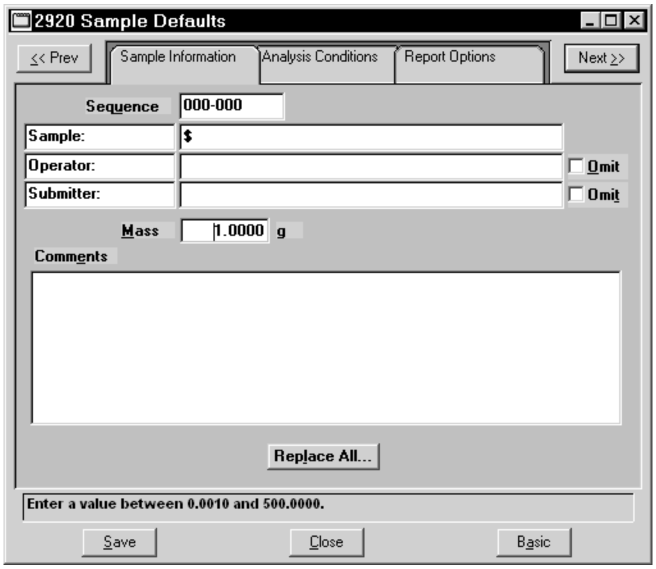
Sequence	Allows you to specify a default sequence for the file name. The number you specify is incrementally sequenced each time you create a file. It is the number that appears in the File name field when you select **File > Open > Sample Information**. • Use numbers, letters, or other printable characters, such as dashes. • Include at least three numbers • Use up to eight characters. • Do not use characters such as * or ?. Refer to your system operating manual for more information.

Sample	Allows you to enter an additional identification that provides more information than the sequence alone. In the field on the left, you can edit the *prompt* for the sample identification. For example, you may prefer to use **Test** or **Material**. The maximum number of characters is 20. In the field on the right, you can specify a format for the sample identification. • Use numbers, letters, or other printable characters, such as dashes. • Include the automatically generated file name as part of the identification by using the $ symbol where you want the sequence number to appear. For example, if the sequence number is 000-001, enter the sample identification as follows: **Lab #25 - $** The resulting sample identification for the first Sample Information file would be: **Lab #25 - 000-001** and for the second one: **Lab #25 - 000-002**, and so on.
Replace All	Click this button to copy the values from an existing sample information file into the default file you are creating. A dialog is displayed so that you can choose the file containing the values you wish to copy. Click **OK**; the values are copied into the defaults automatically. You may then edit the values in the default file without altering the sample information file from which they were copied.
Mass	Enter a value for the sample mass. This value can be edited in the sample file if needed.

Analysis Conditions	From the drop-down list, choose a default analysis conditions file. The list contains the analysis conditions files included with the software, as well as any you have created and stored in the parameter file directory (selected on the Options menu). Each step in the analysis is listed in the Synopsis and its parameters can be viewed by clicking **View**.
Report Options	From the drop-down list, choose a default report options file. The list contains the report options files included with the software, as well as any you have created.
Report Options List Box	Contains the reports available for the report options file you selected as the default. Select the report(s) you wish to include as your default(s).
Save	Saves the defaults you have selected.
Close	Closes the dialog. If there are any unsaved changes, you are given the option to save them.
Advanced	Changes the presentation of the Sample Defaults dialog to the Advanced presentation.

Advanced

When you select Sample Defaults while your system is set to Advanced format, the Sample Defaults dialog is displayed in the Advanced format.



Sequence 000-000
Sample: $
Operator:
Submitter:
Mass 1.0000 g

Enter a value between 0.0010 and 500.0000.

Sequence

Allows you to specify a default sequence for the file name. The number you specify is incrementally sequenced each time you create a file. It is the number that appears in the File name field when you select **File > Open > Sample Information**.

- Use numbers, letters, or other printable characters, such as dashes.
- Include at least three numbers
- Use up to eight characters.
- Do not use characters such as * or ?. Refer to your system operating manual for more information.

Sample

Allows you to enter an additional identification that provides more information than the sequence alone.

In the field on the left, you can edit the *prompt* for the sample identification. For example, you may prefer to use **Test** or **Material**.

Sample
(continued)

In the field on the right, you can specify a format for the sample identification.

- Use numbers, letters, or other printable characters, such as dashes.
- Include the automatically generated file name as part of the identification by using the $ symbol where you want the sequence number to appear.

  For example, if the sequence number is 000-001, enter the sample identification as follows:

  Lab #25 - $

  The resulting sample identification for the first Sample Information file would be:

  Lab #25 - 000-001

  and for the second one:

  Lab #25 - 000-002, and so on.

Operator
Submitter

These fields contain the name (or other identification) of the operator performing the analysis, and the department, customer, etc., that submitted the sample for analysis.

In the fields on the left, you can change the prompts.

In the fields on the right, you can specify the default names or titles.

- Include the automatically generated file name as part of the identification by using the symbol $ where you want the sequence number to appear. Refer to the example in the previous section.
- Select **Omit** to prevent the fields from appearing on the sample information dialog.

Mass

Enter a value for the default sample mass. You can edit this value in the sample file if needed.

Comments	Enables you to enter comments concerning the sample or its conditions. The information you enter in this window displays in the report header
Replace All	Click this button to copy the values from an existing sample information file into the default file you are creating. A dialog is displayed so that you can choose the file containing the values you wish to copy. Click **OK**; the values are copied into the defaults automatically. You may then edit the values in the default file without altering the sample information file from which they were copied.

Analysis Conditions Defaults

The default settings for analysis conditions files are specified using the Advanced format sample information file defaults, regardless of whether you plan to create independent analysis conditions files, or complete analysis conditions dialogs as part of the sample information file.

Select **Options** > **Sample Defaults**; the Advanced presentation of the 2920 Defaults dialog is displayed; click the Analysis Conditions tab. The fields on the sample defaults dialog are identical to the ones on the Advanced format sample file, which is described on page **5-10**.

If you repeatedly use the same sample preparation techniques, then vary the analysis parameters, you may wish to make your default analysis conditions the preparation steps alone. Then the preparation steps will always be in place when you open a new sample information file, ready for you to add analysis steps as needed.

When the first experiment is inserted into an analysis conditions file, its initial values are set by the first experiment step in the default analysis conditions. Including a single “default” experiment step in your default sample file will ensure that new experiments are properly initialized.

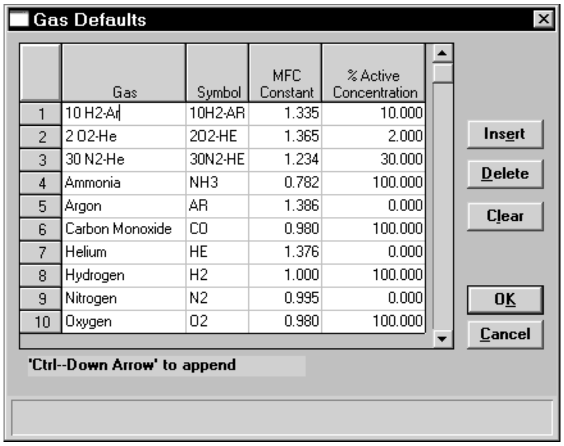
Report Options Defaults

The default settings for report options files are set using the Advanced format sample information file defaults, regardless of whether you plan to create independent report options files, or complete report options tabs as part of the sample information file.

Select **Options** > **Sample Defaults**; the Advanced presentation of the 2920 Defaults dialog is displayed; click the Report Options tab. The fields on the sample defaults dialog are identical to the ones on the Advanced format sample file, which is described on page **5-39**.

Gas Defaults

In order to characterize the gases used in an analysis, a symbol and a Mass Flow Controller (MFC) Constant must be defined. A table is provided, containing default values for the following:



	Gas	Symbol	MFC Constant	% Active Concentration
1	10 H2-Ar	10H2-AR	1.335	10.000
2	2 O2-He	2O2-HE	1.365	2.000
3	30 N2-He	30N2-HE	1.234	30.000
4	Ammonia	NH3	0.782	100.000
5	Argon	AR	1.386	0.000
6	Carbon Monoxide	CO	0.980	100.000
7	Helium	HE	1.376	0.000
8	Hydrogen	H2	1.000	100.000
9	Nitrogen	N2	0.995	0.000
10	Oxygen	O2	0.980	100.000

'Ctrl--Down Arrow' to append

Symbol	Lists the standard symbol (mnemonic) for each gas.
MFC Constant	Contains the MFC Conversion Constant for each gas listed. This constant allows the MFC to control the gas flow precisely. If this number is incorrect, the actual flow rate for that gas differs from the rate reported. **This value defaults to 1 when a new gas is listed**. You must enter the correct gas constant. A table of MFC conversion constants is provided in Appendix C. If the gas you are using is not listed in the table, or if you are using a mixture of gases, you can determine the correct MFC Constant using the instructions in Appendix F.

In general, the MFC conversion constants provided with the analyzer are accurate and should be used. If you suspect that a MFC is not functioning properly, you may wish to calibrate the MFC.

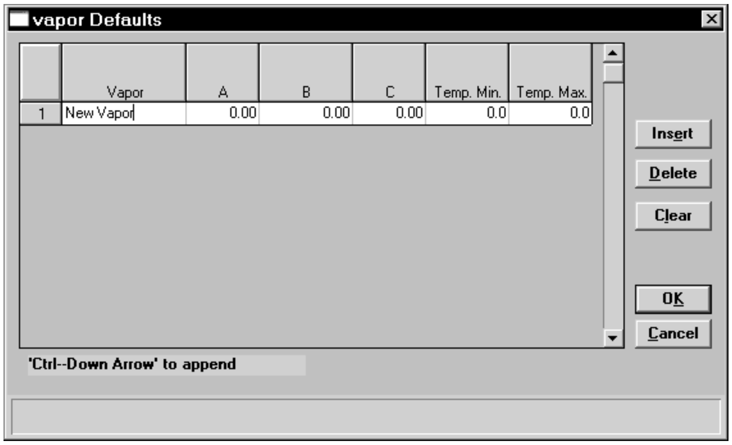
% Active Concentration	Displays the percentage of the gas that is active.

You can change any one of the values in the table by highlighting it and entering the value you prefer. You can also edit the gases listed using the following push buttons:

Insert	Inserts a row in the table in which you may enter a new gas and its values. The new row is inserted before the row in which the cursor is located.
Delete	Deletes from the table the row in which the cursor is located. No confirmation is provided; therefore, do not click **Delete** unless you are certain this is what you wish to do.
Clear	Clears all but one entry of the table, after confirmation. One entry is required.

Vapor Defaults

This option allows you to specify defaults for vapors when used in an analysis.



	Vapor	A	B	C	Temp. Min.	Temp. Max.
1	New Vapor	0.00	0.00	0.00	0.0	0.0

'Ctrl--Down Arrow' to append

Name	Lists the name of the vapor.
A, B, C	These columns enable you to enter the Antoine constants used in the vapor TCD calibration.
Temp. Min/Temp. Max	Enables you to enter a temperature range (in K) in which the constants are valid.
Insert	Inserts a row before the selected row, allowing you to add a new vapor to the table.

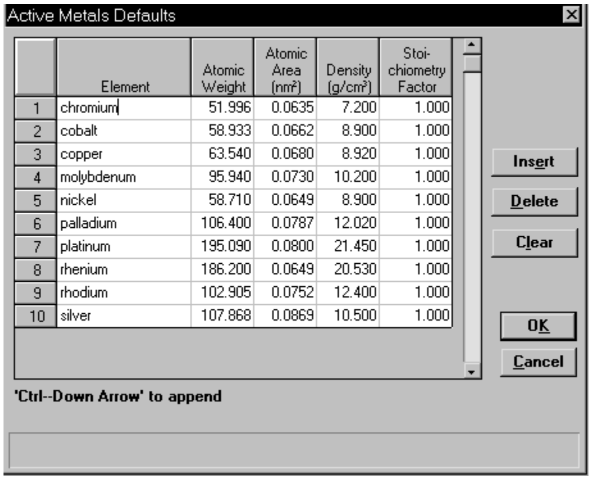
Delete	Deletes the selected vapor from the table; no confirmation is provided.
Clear	Clears all but one entry from the table; one entry is required.

Active Metals Defaults

In order to characterize the active metals in a sample, certain properties of the metals must be defined.

The values defined in this table are the defaults used when a new entry is inserted into the metal table of the Pulse Chemisorption report options. Changing this table does not affect the tables within the report options files.

A table containing default values for the following properties is provided:



	Element	Atomic Weight	Atomic Area (nm²)	Density (g/cm³)	Stoi-chiometry Factor
1	chromium	51.996	0.0635	7.200	1.000
2	cobalt	58.933	0.0662	8.900	1.000
3	copper	63.540	0.0680	8.920	1.000
4	molybdenum	95.940	0.0730	10.200	1.000
5	nickel	58.710	0.0649	8.900	1.000
6	palladium	106.400	0.0787	12.020	1.000
7	platinum	195.090	0.0800	21.450	1.000
8	rhenium	186.200	0.0649	20.530	1.000
9	rhodium	102.905	0.0752	12.400	1.000
10	silver	107.868	0.0869	10.500	1.000

'Ctrl--Down Arrow' to append

Atomic Weight	The atomic weight of the element. *Range:* *1.0000 to 500.0000*
Atomic Area	The atomic cross-sectional area of the element. *Range:* *0.0000 to 9.9999* nm^2
Density	The density of the element. *Range:* *0.0000 to 99.9999* g/cm^3

Stoichiometry Factor	A factor which expresses the ratio between the number of active metal atoms and the number of adsorbate molecules. This number varies, based on the adsorbate used. The most common value should be entered into the defaults table. Within the Pulse Chemisorption report options; the value for a specific analysis can be set. *Range: 0.001 to 100.000*

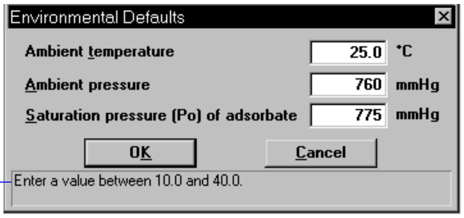
You can change any of these values by highlighting it, then entering the value you prefer. You can also edit the elements listed using the following buttons:

Insert	Inserts a row in the table in which you may enter a new gas and its values. The new row is inserted before the row in which the cursor is located.
Delete	Deletes from the table the row in which the cursor is located. No confirmation is provided; therefore, do not click **Delete** unless you are certain this is what you wish to do.
Clear	Clears all but one entry of the table, after confirmation. One entry is required.

Environmental Defaults

To ensure accurate loop calibration and BET surface area data, you should enter current environmental conditions every day, or whenever there is a significant change in one or more of the environmental conditions. These values are used in some calculations to account for the effect of environmental conditions on the analysis. When you select this option, the Environmental Defaults dialog opens.

As an analysis is started, these parameters are saved in the Collected Data portion of the sample information file. (Collected Data can be viewed in the sample file in Advanced format only.)



Ambient temperature 25.0 °C
Ambient pressure 760 mmHg
Saturation pressure (Po) of adsorbate 775 mmHg
Enter a value between 10.0 and 40.0.

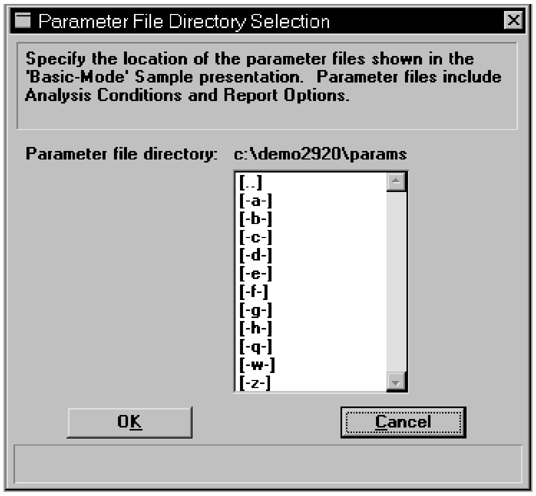
Displays the valid range for the selected field.

Valid ranges for the selected field are shown in the information bar on the dialog.

Ambient temperature	Enter the temperature of the room in which the analyzer is located.
Ambient pressure	Enter the atmospheric pressure of the room in which the analyzer is located.
Saturation pressure (Po) of adsorbate	Enter the saturation pressure of the adsorbate gas in the room in which the analyzer is located. Disregard this field if the saturation pressure of the adsorbate gas is not pertinent to your analysis.

Parameter Files Directory

When you select this option, the Parameter File Directory Selection dialog is displayed.



Parameter File Directory Selection

Specify the location of the parameter files shown in the 'Basic-Mode' Sample presentation. Parameter files include Analysis Conditions and Report Options.

Parameter file directory: c:\demo2920\params

This option allows you to choose which analysis conditions and report options files (parameter files) display in the drop-down lists on the Basic format sample information dialog. You do so by selecting a directory. Parameter files located in the specified directory appear in the drop-down lists; files located in other directories do not.

For this reason, it is important that whenever you create an independent parameter file, you save it in the correct directory. The destination directory is specified on the Open Analysis Conditions or Open Report Options dialog when you first begin to create the parameter file. To be sure that the parameter files you create are included in the drop-down list, make sure that the directory they are saved to matches the directory specified using this option.

Service Test Mode

Various service tests are included in the AutoChem II 2920 operating program. These tests can be performed only with the assistance of a trained Micromeritics service representative. When you select **Service Test Mode** from the Options menu, a dialog box prompting you to enter a password is displayed. This password is coded to change on a regular basis and is known only by your service representative. You will not be able to perform these tests without his guidance.

9. TROUBLESHOOTING AND MAINTENANCE

This chapter describes potential operational problems and their solutions, and maintenance procedures. If further assistance is needed after following the procedures in this chapter, contact a Micromeritics service representative.

Troubleshooting

Operating problems encountered with the analyzer are usually easily corrected. Typical problems and corrective actions are described in the following table.

Table 9-1. Potential Operational Problems

What Happened	Why	What To Do
Unit does not work when power switch is turned on.	Power cord not fully inserted at one of the ends.	Insert power plug firmly into outlet socket; insert unit connector into power connector opening.
	No power at outlet.	Plug in lamp or small appliance to test outlet. If there is no power, contact electrician.
	Plug prongs bent so that contact not made at outlet.	Gently move power plug at outlet while watching the status LEDs on the front panel. If at least one LED becomes illuminated, have an electrician replace the outlet or the plug.
	Power cord damaged.	Have electrician check cord using a test meter. Replace cord if defective.
	Loose internal connection or broken wire.	Call a Micromeritics Service Representative for repair or replacement.
Liquid nitrogen boils away too quickly to complete an analysis.	Dewar damaged.	Replace dewar.
	Lengthy analysis.	Refill dewar during analysis.
Specified temperature not reached or not maintained.	Thermocouple or other internal component is damaged or disconnected.	Call the appropriate service personnel.

Table 9-1. Potential Operational Problems (continued)

What Happened	Why	What To Do
Gas drained from tank.	Leaks in the gas line connection.	Replace the gas tank; then pressurize the system. Close, then open the cylinder valve. If the needle on the pressure gauge on the gas cylinder jumps abruptly, a leak in the gas line connection may be indicated. Check all gas line connections.
Too much heat supplied to analyzer.	Thermocouple or other internal component is damaged or disconnected.	Call the appropriate service personnel.
A stable TCD baseline cannot be maintained.	TCD filaments are contaminated or need to be replaced.	Refer to **Replacing the Thermal Conductivity Detector (TCD)** to clean the TCD filaments or replace the TCD.
	Possible leak in Reference/carrier path.	Perform leak test in the Reference/carrier path. Refer to **Performing a Leak Test**.
	Faulty RTD in one of the temperature zones.	Access manual mode and check for temperature stability in the temperature zones.
Data collection results in very high or very low peaks that are inconsistent with previous experience.	TCD filaments are contaminated or need to be replaced.	Refer to **Replacing the Thermal Conductivity Detector (TCD)** to clean the TCD filaments or replace the TCD.
	A different (lower) TCD temperature is being used.	Repeat the analysis using a higher temperature.
During data collection, a TCD signal of zero is recorded, or no peaks are seen.	TCD filaments are contaminated or need to be replaced.	Refer to **Replacing the Thermal Conductivity Detector (TCD)** to clean the TCD filaments or replace the TCD.
	TCD temperature was not reset after a run automatically suspended.	Be sure to re-enable the TCD after all run suspensions.

Maintenance Schedule

Use the following table to determine when maintenance procedures should be performed.

Procedure	Location of Instructions (Page)	3 Months 75 runs	6 Months 150 runs	9 Months 225 runs	12 Months 300 runs
Clean cold trap	**9-4**	U/I/A	U/I/A	U/I/A	F
Change septum	**9-6**		U/R		F
Change loop	**9-7**		U/I/A		F
Replace TCD	**9-10**		U/I/A		F
Adjust sample thermocouple	**4-11**		U/I/A		F
Replace sample tube filter	**9-13**		U/R		F/R
Clean analyzer	**9-15**		U/I/A		F
Clean foam air filter	**9-16**				F
Clean dewars	**4-26**	U/I/A	U/I/A	U/I/A	F
Change sample O-rings	**4-21**				F/R
Perform leak check	**9-24**		U/A		F
Check temperature stability	F				F
Check sample thermocouple calibration	**9-18**				F/A
Perform calibration run	F				F

U = User procedure
F = Factory/Micromeritics Service procedure
I = Inspect
R = Replace
A = As necessary

Maintenance Procedures

Changing or Cleaning the Cold Trap or Delay Path

Because the cold trap removes condensibles from the gas stream, it will require occasional cleaning. The delay path (used for BET analyses) also attaches to the cold trap port fittings. If you need to change from one attachment to the other, follow the steps below, but substitute the appropriate attachment during reassembly.

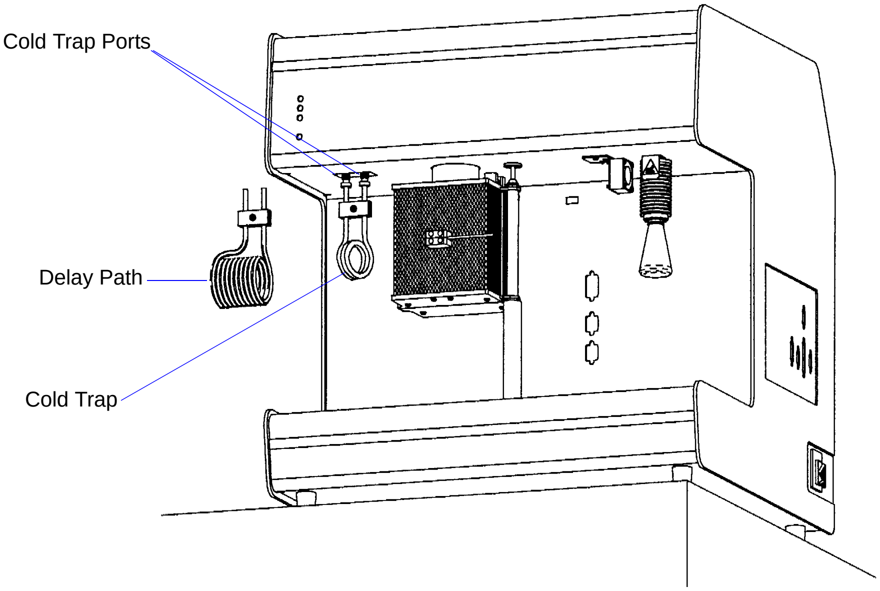


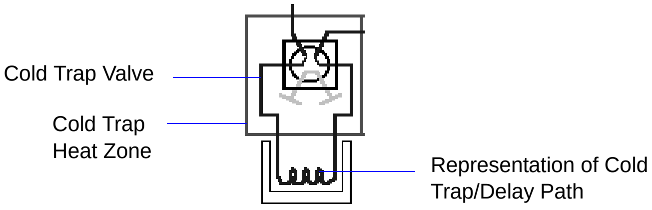
Figure 9-1. Location of Cold Trap or Delay Path

To clean the cold trap, follow the steps below.

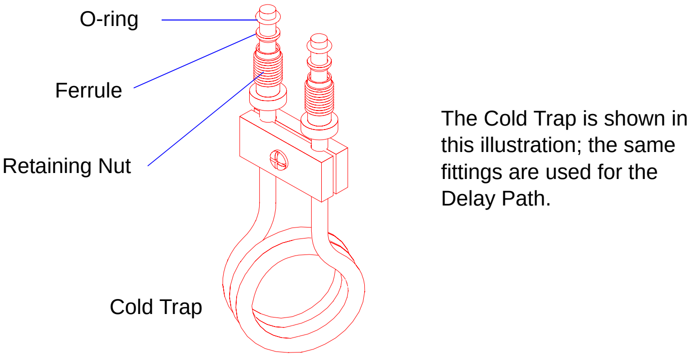
1. If a dewar is around the cold trap, raise the dewar slightly, move the dewar support out of the way, and remove the dewar. Allow the cold trap to warm to room temperature.

Depending upon the state of the analyzer, the cold trap fittings may be hot. Use caution. If necessary, wear cotton gloves to protect your hands.

2. From the Unit menu, select **Show instrument schematic**, then select **Enable manual control**. Right-click on the cold trap valve (the detail from the schematic is shown below) and choose **Bypass**.



3. Loosen the retaining nuts on the cold trap port fittings. (These can be unscrewed using your fingers.)



The Cold Trap is shown in this illustration; the same fittings are used for the Delay Path.

4. Remove the cold trap (or delay path), nuts, ferrules, and O-rings.

5. Clean the cold trap (or delay path) by flushing it with isopropyl alcohol (IPA). Dry the trap in an oven or by blowing compressed air or nitrogen through it before replacing it.

Use only Kalrez^{TM} O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.

6. Inspect the O-rings for cracks or other damage that might cause leaks. If the O-rings are cracked or damaged, replace them before reinstalling the cold trap (or delay path). (Extra O-rings are shipped with the analyzer. Refer to Chapter 10 for ordering information.) Otherwise, clean the O-rings you removed, dry them, and reinstall them.

7. Place the retaining nuts, ferrules, and O-rings on the cold trap (or delay path).

Do not over tighten the retaining nuts. Doing so may damage the O-rings. Finger-tight is sufficient.

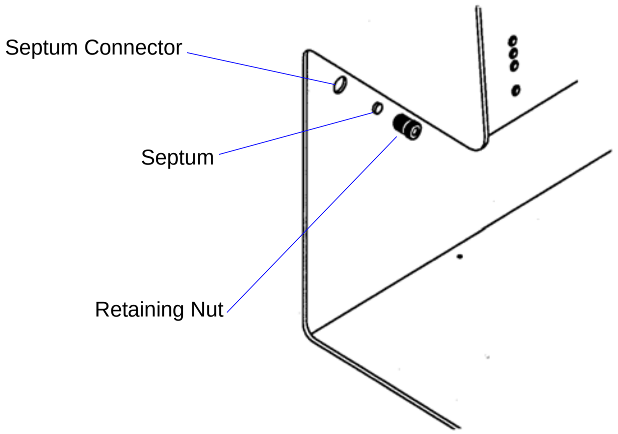
8. Hold the cold trap (or delay path) in place and tighten the retaining nuts. Tighten the nuts only enough to hold the cold trap securely in place and prevent leaks.

Changing the Septum

Because the septum is pierced each time you perform an analysis requiring an injection, it will eventually leak. When the analyzer septum begins to leak, replace it with a new septum.

To replace the septum, follow the steps below.

1. Turn the knurled septum retaining nut on the front panel counterclockwise to loosen it.
2. When the mounting nut is free, remove it with the old septum.
3. Remove the old septum from the retaining nut.



4. Install a new septum using the original retaining nut.

Do not use a wrench to tighten the septum retaining nut. Do not overtighten the retaining nut. Doing so may damage the septum or the fitting.

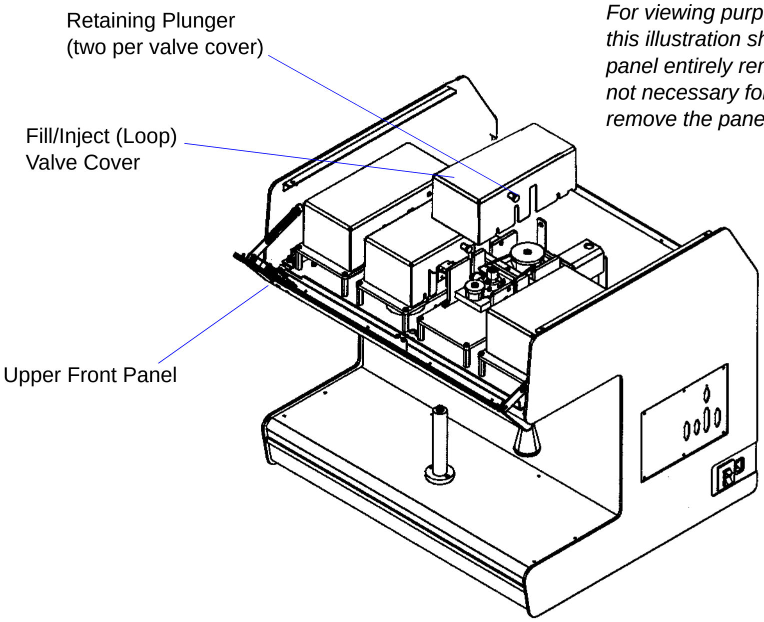
5. Use your fingers to turn the septum retaining nut clockwise until it is tight. You will feel resistance as the septum is squeezed, and then the nut will be difficult to tighten further.

Changing the Injection Loop

1. Make sure that no heat is being applied to the analyzer. If necessary, enable manual control and set the target temperature for the heat zones to ambient. Wait for the analyzer to cool down.

2. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle. Then, using the handle, open the top panel. The top panel is hinged to allow easy access to its components; ***it is not necessary to remove the top panel***.

   Pull down the upper front panel.



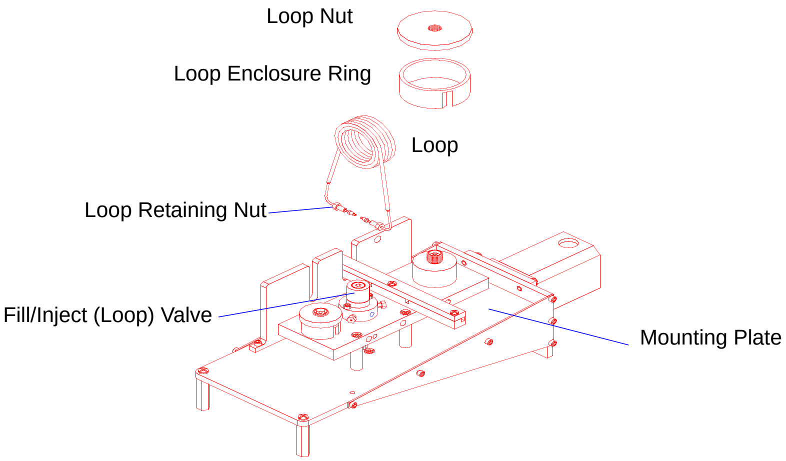
For viewing purposes only, this illustration shows the top panel entirely removed. It is not necessary for you to remove the panel.

Depending upon the state of the analyzer, the internal components may be hot. Use caution.

3. The Fill/Inject (Loop) valve cover is held in place by two retaining plungers. Remove the valve cover by pulling the plungers outward until the Fill/Inject valve cover is released. Pull the cover up and out.

4. Remove the insulation from Fill/Inject valve (not shown).

5. Unscrew the loop nut and lay it aside. It may be necessary to loosen the screw in the middle of the assembly in order to free the loop nut.



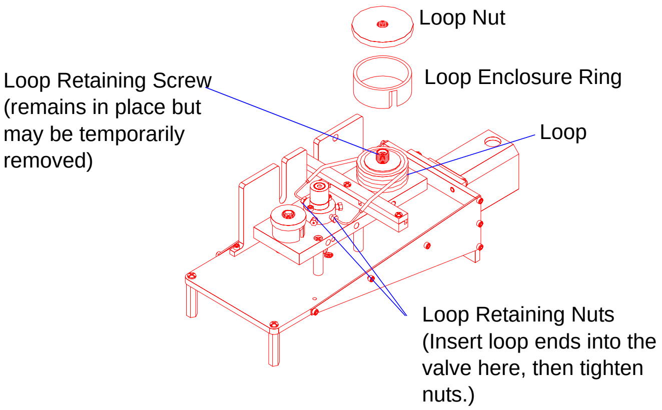
6. Use a 1/4-in. open-end wrench to loosen the nuts on the injection loop.

7. Pull up and remove the injection loop and enclosure ring from the mounting plate.

Make sure the body of the loop is positioned in the temperature-controlled enclosure of the loop valve. This orientation keeps the temperature (and volume) of the loop constant. Failure to keep the full loop at a constant temperature may cause variations in the quantity of gas contained in the loop.

8. Install the replacement injection loop by placing the ends of the loop into the sides of the valve and turning the nuts finger-tight.

9. Push the replacement loop down to contact the mounting plate and place the loop enclosure ring firmly into place.



10. Screw the loop nut down finger-tight. Tighten the screw in middle of the assembly if it was loosened earlier.

11. Tighten the loop nuts securely, using a 1/4-in. open-end wrench.

12. Check the injection loop for leaks. Refer to Checking the Analyzer for Leaks later in this chapter. If you detect a leak, the injection loop is probably not installed properly. Repeat the installation and check for leaks again.

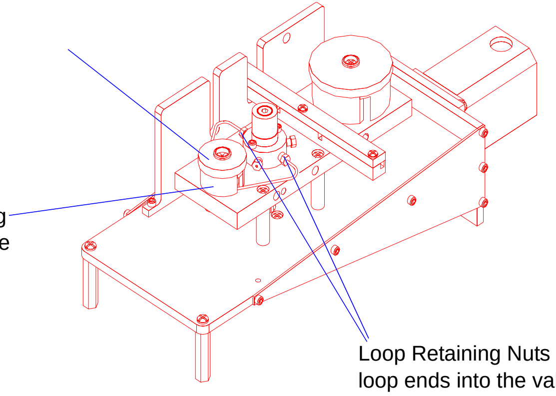
13. After you have checked for leaks, move the insulation back into position and reinstall the Fill/Inject valve cover.

14. Close the top panel and the lower front panel.

15. After installing a new injection loop, it is necessary to calibrate the loop. Follow the instructions for performing a loop calibration in Appendix J.

    Then update the loop volume recorded on the Unit Configuration dialog. This dialog is accessed from the Unit menu.

The smaller loops connect to the same openings in the valve, but they rest in a different position than the larger loop, as shown below.



Both the 0.5-cm^3 and the 1.0-cm^3 loops are located in this position when installed on the analyzer.

Loop Nut

Loop Enclosure Ring (the loop is inside the enclosure ring.)

Loop Retaining Nuts (Insert loop ends into the valve here, then tighten nuts.)

Replacing the Thermal Conductivity Detector (TCD) Assembly

Because the filaments in the thermal conductivity detector assembly are heated to high temperatures and may be exposed to corrosive gases, they will eventually fail. Any of the following problems may indicate that the TCD filaments need to be replaced:

- A stable TCD baseline cannot be maintained.
- Data collection results in very high or very low peaks that are inconsistent with previous experience.
- During a data collection, no TCD signal is displayed on the main display, or the software indicates that no TCD signal is present.

In some instances, contamination of the thermal conductivity detector filaments will produce the symptoms described above. To clean the filaments, set the filament zone to 150 °C and flow an inert gas through the analyzer for several hours. If this action does not correct the problem, perform the following steps to replace the thermal conductivity detector assembly.

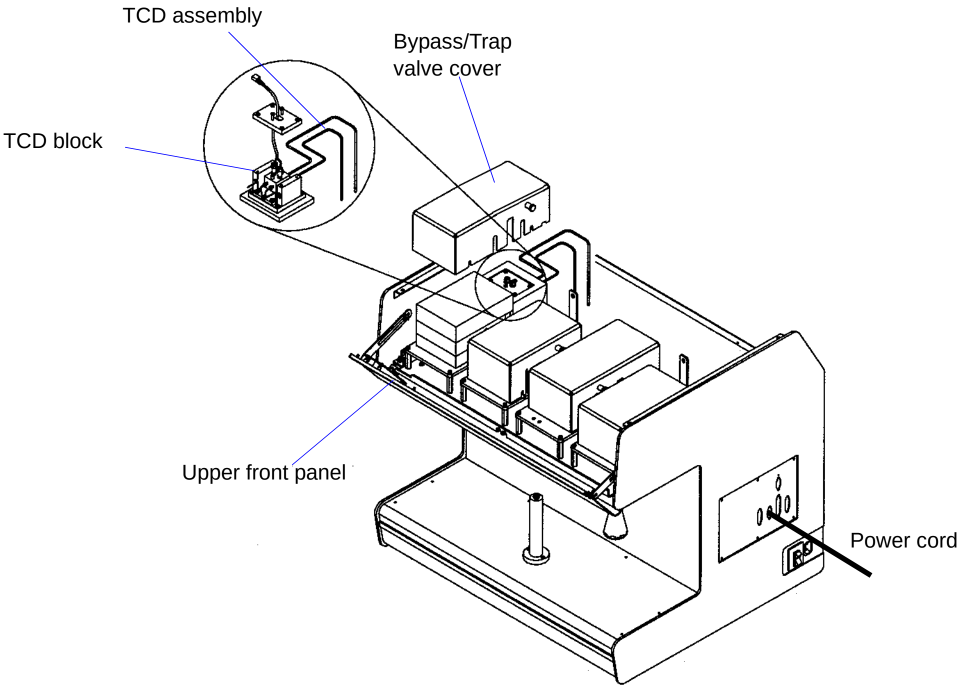
1. Make sure that no heat is being applied to the analyzer. If necessary, enable manual control and set the target temperature for the heat zones to ambient. Wait for the analyzer to cool down.

2. Turn instrument power switch off but do NOT disconnect the power cord from the analyzer.

The power cord must remain connected to maintain an electrical ground connection to the analyzer.

3. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle. Then, using the handle, open the top panel. The top panel is hinged to allow easy access to its components; ***it is not necessary to remove the top panel***.

   Pull down the upper front panel.



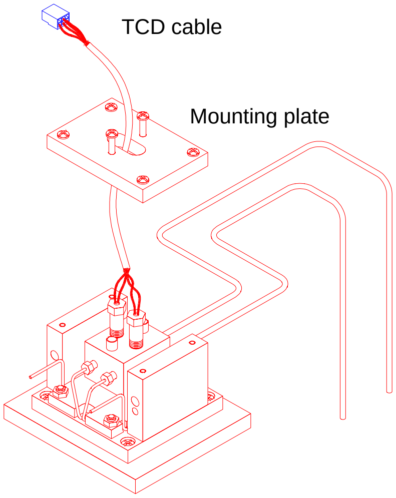
4. Pull the Bypass/Trap valve cover retaining plungers outward until the Bypass/Trap valve cover is released. Pull the cover up and out.

5. Remove the insulation from the rear of the Bypass/Trap valve and locate the TCD assembly (not shown).

The thermal conductivity detector assembly and nearby components may be hot. Make sure you allow the TCD to cool before removing it. Use the cotton gloves provided with the analyzer to protect your hands.

6. Loosen all four fittings for the stainless steel tubing. Label each piece of tubing for reconnection.

7. Unplug the TCD assembly cable from the card cage motherboard and cut the plastic cable ties that retain the various cables. This frees the cables so that you can remove the TCD and its cable.



8. Use a screwdriver to remove the two TCD mounting screws from the yoke plate. Lift the TCD assembly out of the analyzer.

9. Install the new TCD assembly in the analyzer. Reconnect the tubing at all four fittings and reconnect the cable to the card cage motherboard.

10. Reinstall the insulation in the rear of Bypass/Trap valve over the TCD assembly.

11. Reinstall the Bypass/Trap valve cover and allow the retaining plungers to snap into place.

12. Close the top panel and the upper front panel.

13. Turn the instrument power switch ON.

14. Adjust the filament temperature to 150 °C and select an inert carrier gas to flow through the TCD. Set the TCD Zone temperature to 110 °C.

15. Allow the TCD baseline to stabilize. You can observe the TCD reading from the instrument’s Status Bar. (Display the Status Bar by selecting it from the Unit menu.) Stabilization may take several hours.

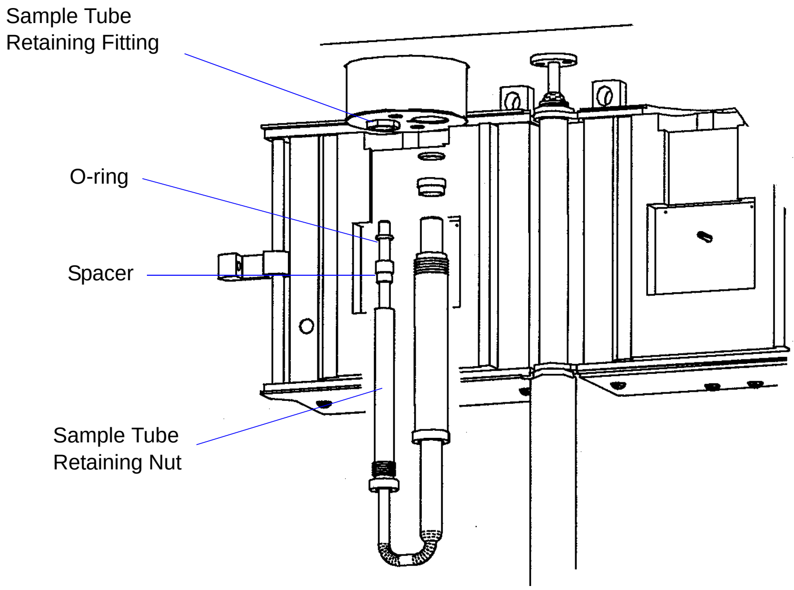
Changing the Analysis Port (Sample Tube) Filter

A frit filter is used to protect the analyzer internal components from contaminants that may be forced out of the sample tube during an analysis. However, as the analyzer is used, the filter may gradually become clogged with trapped particles. If the filter does become blocked, it must be removed and cleaned or replaced. One indication of a clogged filter is a sample pressure greatly exceeding atmospheric pressure (displayed on the instrument schematic). The nominal sample pressure reading should be a few mmHg above atmospheric pressure.

To change the analysis port filter, follow the steps below:

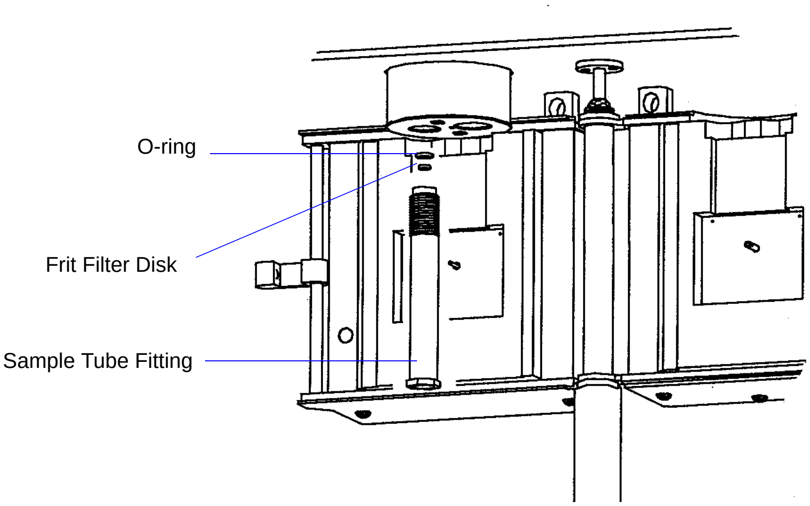
1. Loosen the analysis port fittings while holding the sample tube.

The sample tube and nearby components may be hot. Make sure you allow the sample tube to cool before removing it. Use the cotton gloves provided with the analyzer to protect your hands.



2. Carefully remove the sample tube by unscrewing the retaining nuts. Be careful not to allow the nuts to slide down onto the curved portion of the sample tube or the tube may break.

3. Remove the left (smaller) sample tube fitting (it has a tube within a tube).



4. Use a small diameter probe to remove the O-ring and frit filter disk from the sample tube fitting.
5. Install a new (or cleaned) analysis port filter in the sample tube fitting. (Extra frit filters are shipped with the analyzer. Refer to Chapter 10 for ordering information.)

Use only Kalrez O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt or decompose.

6. Inspect the O-ring for cracks or cuts. If the O-ring is damaged, replace it with a new O-ring. (Extra Kalrez O-rings are shipped with the analyzer. Refer to Chapter 10 for ordering information.) Otherwise, clean the O-ring you removed, dry it, and place it in the sample tube fitting. Make sure the O-ring is securely in place.
7. Place the left sample tube fitting back into position and tighten it slightly.
8. Reinstall the right sample tube fitting and sample tube, then tighten both fittings.

Cleaning the Analyzer

The exterior casing of the analyzer may be cleaned using a clean cloth, dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution.

Do not immerse the analyzer or the power cord in any liquids. Doing so could result in electrical shock to personnel or damage to the unit.

Do not allow liquid to penetrate the casing of the analyzer. Doing so could result in damage to the unit.

Cleaning the Vapor Generator

If the optional vapor generator is installed, it is necessary to clean it before changing the liquid you use in it. To clean the Vapor Generator:

1. Fill the flask with Isopropyl Alcohol (IPA).

2. Install the flask on the analyzer

3. Flow an inert gas through the Vapor Generator and analyzer for approximately 20 minutes.

Such cleaning can be performed using Manual Control. Instructions for using Manual Control are contained in Chapter 6.

Cleaning the Dewar

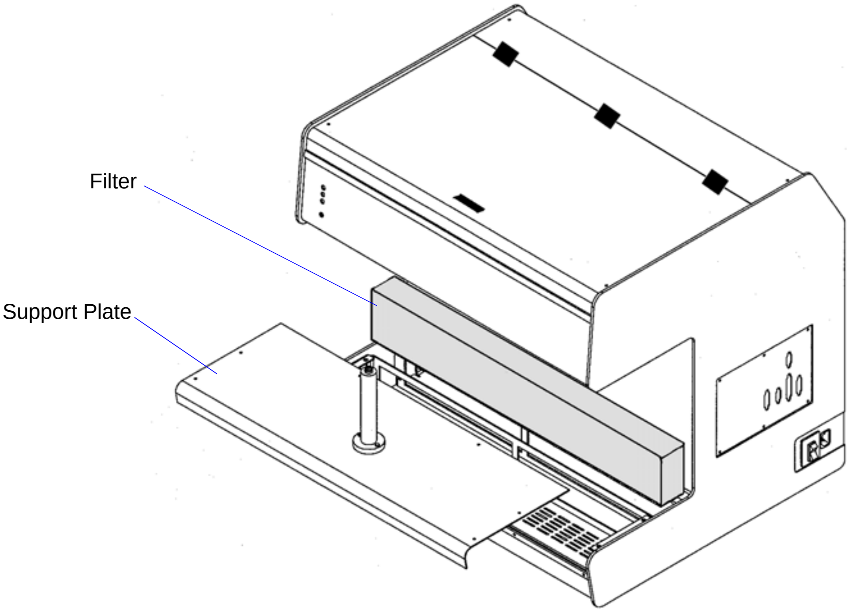
The dewar must be cleaned between uses. Empty the dewar, then rinse it with water. Make sure that it is dry before the next use.

Be sure to observe dewar precautions when cleaning the dewar. Refer to **Precautions** on page **4-24**.

Changing or Cleaning the Foam Air Filter

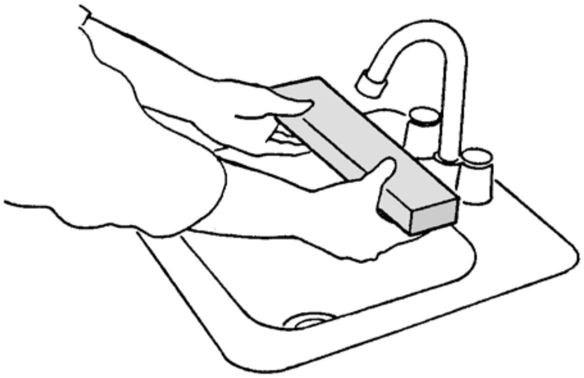
The air filter in the AutoChem II analyzer should be cleaned every 30 days (more frequently in environments with high levels of dust). If, after cleaning, you determine that the filter is degrading, it should be replaced.

1. Click **Show instrument schematic** from the Unit menu to display the instrument schematic. Click the Analysis valve and ensure that it is in the Prepare position. (If it is not, enable manual control and place it in the Prepare position.)
2. Remove the furnace, the cold trap (or delay path), the sample tube, and any other parts or accessories (such as the optional Vapor Generator or a dewar) from the front of the analyzer.
3. Remove the screws that hold the stainless steel support plate in place.



4. Hold the furnace support post and lift the support plate up and away from the analyzer.
5. Pull the filter up and out of the analyzer.

6. Rinse the filter with clean water and allow it to dry completely.



7. If the filter is in good condition, place it back inside the instrument. If the filter needs to be replaced, obtain a new filter and place it inside the instrument.
8. Place the support plate back into position and reinstall its retaining screws.
9. Reinstall the furnace, cold trap (or delay path), sample tube, etc.

Adjusting or Replacing the Sample Thermocouple

The sample thermocouple is designed to pass either through the analysis port and into the sample tube or through an adjacent opening to rest along the side of the sample tube.

Because the sample thermocouple is heated to high temperatures, it will eventually fail and must be replaced. Two indications that the sample thermocouple may need to be replaced are unreasonably high sample temperature readings or erratic sample temperature readings.

To replace the thermocouple, follow the instructions for **Changing the Position of the Thermocouple** on page **4-11**. Instead of moving the thermocouple from one position to the other, disconnect the used thermocouple from the cable that attaches it to the analyzer. Attach the new thermocouple to the cable and place it in the appropriate opening.

After you replace the sample thermocouple, you must calibrate it using the instructions in the following section.

Recalibrating the Sample Thermocouple

A complete calibration file is created specifically for each analyzer before the analyzer is shipped. This calibration file is installed during initial software installation (Chapter 2). You may need to recalibrate the sample thermocouple if you replace the thermocouple or if the original calibration file is corrupted or missing.

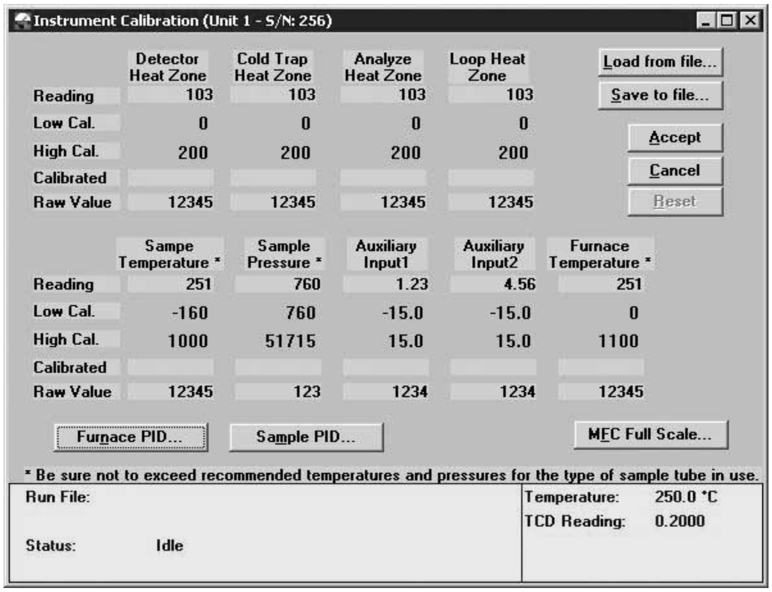
Do not attempt to calibrate anything other than the sample thermocouple without prior approval from a qualified Micromeritics Service Representative.

Use the following instructions to recalibrate the sample thermocouple.

Before beginning, make sure:

- The analyzer and software are turned on.
- You are familiar with manually controlling the analyzer.
- All gas flows through the analyzer are stopped.
- All heat zones are stabilized at ambient temperature.
- The furnace is open and the sample tube is removed (the thermocouple is bare and exposed).
- A traceable thermocouple meter and a water bath (ambient temperature) to immerse the sample thermocouple are available.

1. Select **Unit > Instrument Calibration**; the Instrument Calibration dialog opens.



	Detector Heat Zone	Cold Trap Heat Zone	Analyze Heat Zone	Loop Heat Zone
Reading	103	103	103	103
Low Cal.	0	0	0	0
High Cal.	200	200	200	200
Calibrated				
Raw Value	12345	12345	12345	12345

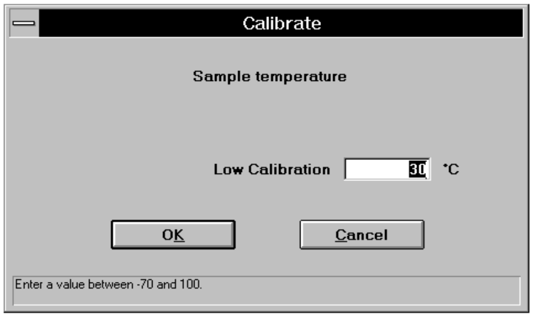
	Sampe Temperature *	Sample Pressure *	Auxiliary Input1	Auxiliary Input2	Furnace Temperature *
Reading	251	760	1.23	4.56	251
Low Cal.	-160	760	-15.0	-15.0	0
High Cal.	1000	51715	15.0	15.0	1100
Calibrated					
Raw Value	12345	123	1234	1234	12345

* Be sure not to exceed recommended temperatures and pressures for the type of sample tube in use.

Run File:
Status: Idle
Temperature: 250.0 °C
TCD Reading: 0.2000

2. Place the water bath around the thermocouple so that the lowest 25 mm (1 inch) is immersed in the water. Place the thermocouple meter (reference thermocouple) parallel to the sample thermocouple so that both thermocouples are at the same temperature.

3. After the reference thermocouple temperature stabilizes, enter its reading on the Calibrate dialog. To do so, click the **Low Cal** field, then press the space bar. The Calibrate dialog is displayed. Enter the value in the **Low Calibration** field, then click **OK**.



Sample temperature

Low Calibration 30 °C

Enter a value between -70 and 100.

4. Remove the water bath. Move the tip of the reference thermocouple so that it touches the sample thermocouple tip, yet allows the furnace to be closed.

5. Use manual control of the sample furnace to set the furnace target temperature to 1000 °C. Use a ramp rate of 50 °C/min. (Refer to **Enable Manual Control** on page **6-12**.)

6. Observe the instrument schematic to ensure that both the furnace temperature and sample temperature are increasing.

7. After the furnace and sample temperature have stabilized, read the value from the reference thermocouple. Enter its reading on the Calibrate dialog. To do so, click on the **High Cal** field, then press the space bar. The Calibrate dialog is displayed. Enter the value in the **High Calibration** field, then click **OK**.

8. Use manual control of the furnace to return its temperature to ambient.

9. On the Instrument Calibration dialog, click **Accept** to save the new calibration values. The software then displays dialogs that allow you to verify this change and make a backup copy of the values you are replacing. If desired, make a backup copy by clicking **Yes** then specifying a file name.

Recalibrating the Vapor Generator’s Heated Zones

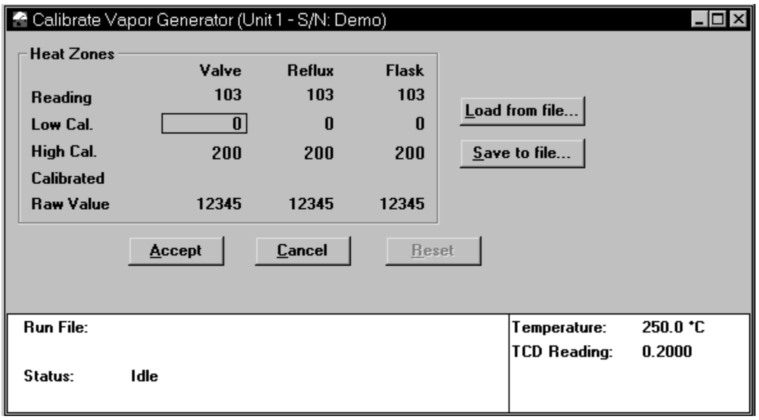
The Vapor Generator is an option that is initially calibrated by Micromeritics personnel, either at the factory or on-site during installation. If the factory values become corrupted for any reason, use the following instructions to recalibrate the Vapor Generator’s heated zones. This process consists of taking temperature readings of the three heat zones (valve, flask, and reflux) at ambient, then near the maximum temperature allowed. These temperature readings are entered in the software and the Vapor Generator is calibrated automatically.

Do not attempt to calibrate the Vapor Generator unless you are specifically instructed to do so by a Micromeritics Service Representative.

Before beginning, make sure:

- The analyzer and software are turned on.
- You are familiar with manually controlling the analyzer.
- All heat zones to be calibrated are stabilized at ambient temperature.
- A traceable thermocouple meter is available.
- The flask is filled with water and installed on the port.
- The heating mantle is in place (but it may be unzipped) and the connector is plugged in.

1. Select **Unit > Vapor Generator Calibration**; the Calibrate Vapor Generator dialog is displayed. (In this example, the default values are shown, not the calibrated values.)



Calibrate Vapor Generator (Unit 1 - S/N: Demo)

Heat Zones

	Valve	Reflux	Flask
Reading	103	103	103
Low Cal.	0	0	0
High Cal.	200	200	200
Calibrated			
Raw Value	12345	12345	12345

Run File:

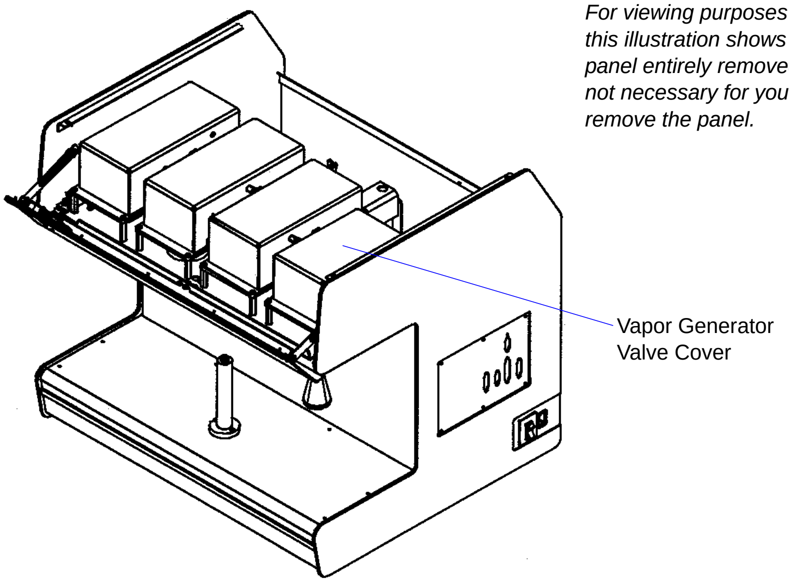
Status: Idle

Temperature: 250.0 °C

TCD Reading: 0.2000

2. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle. Then, using the handle, open the top panel. The top panel is hinged to allow easy access to its components; ***it is not necessary to remove the top panel***.

   Pull down the upper front panel.



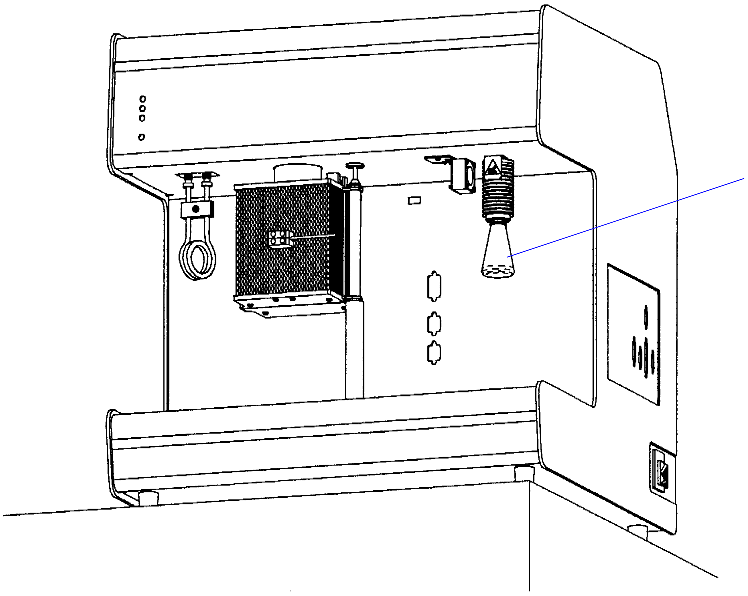
For viewing purposes only, this illustration shows the top panel entirely removed. It is not necessary for you to remove the panel.

3. Take the ambient temperature readings for the three zones as instructed below. For each zone, enter the ambient temperature reading in the Calibrate dialog. To do so, click on the **Low Cal** field, then press the space bar. The Calibrate dialog is displayed. Enter the value in the **Low Calibration** field, then click **OK**. Repeat for each zone.

   a. **Valve zone**: Place the thermocouple probe into the small hole in the valve cover. After the thermocouple meter reading stabilizes, enter it in the Calibrate dialog.

   b. **Reflux zone**: Remove the valve cover. The RTD probe is located directly above the reflux zone and in front of the valve. It is held in place by a nut and ferrule. Before removing the probe, you must measure it or mark it so that you can place it back at the same height. Remove the probe, immerse the RTD probe and thermocouple probe in cold water. When the temperature reading stabilizes, enter it enter it in the Calibrate dialog.

   c. **Flask zone**: place the thermocouple probe next to the Resistance Temperature Device (RTD), which is located on the floor of the heating mantle. Zip the mantle closed to hold the probe in place. When the thermocouple meter reading has stabilized, enter it in the Calibrate dialog.



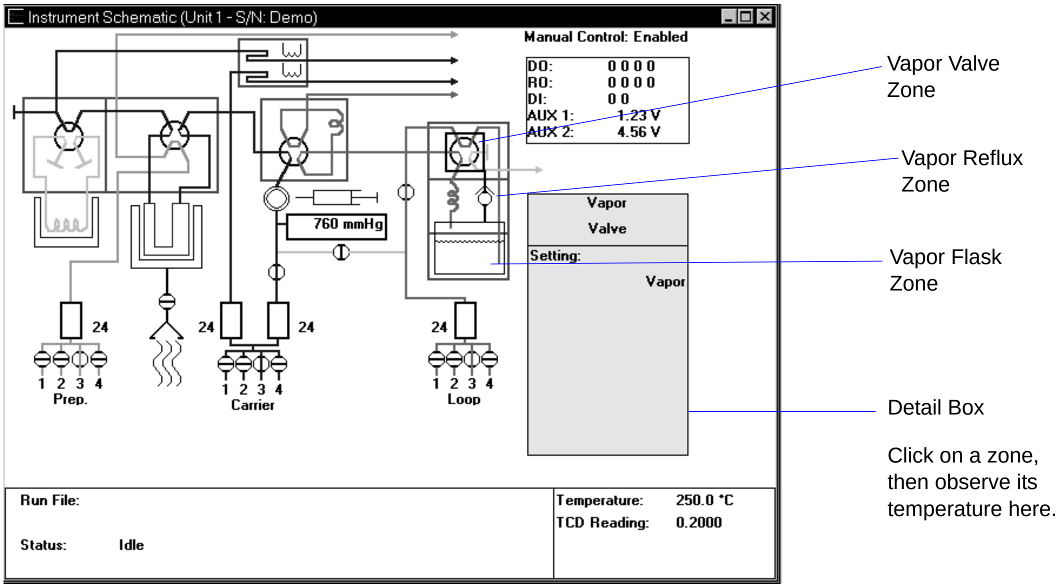
Install the flask, filled with water. Zip the heating mantle and plug in the connector.

4. The reflux zone probe must be calibrated at a high temperature, around 70 to 100 °C. Use hot water or another heat source to heat the reflux zone probe and the reference thermocouple probe simultaneously. When the temperature stabilizes, record the temperature from the reference thermocouple meter. On the Calibrate dialog, enter the new **High Calibration** value for the reflux zone. Click **OK**.

5. Reinstall the reflux zone probe. Replace the Vapor Generator valve cover.

6. Use manual control to set the target temperature for each of the other Vapor Generator heat zones. (For complete instructions on using manual control, see Chapter 6.)

   - Set the valve zone at 110 °C.
   - Set the flask zone at 90 °C.
   - Set the reflux zone at 80 °C, although it needs no further calibration.



Instrument Schematic (Unit 1 - S/N: Demo)

Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Vapor Valve

Setting:
Vapor

Run File:

Status: Idle

Temperature: 250.0 °C
TCD Reading: 0.2000

Vapor Valve Zone

Vapor Reflux Zone

Vapor Flask Zone

Detail Box

Click on a zone, then observe its temperature here.

7. Insert the reference thermocouple into the small access hole in the valve cover.

8. Observe the detail box on the instrument schematic (shown above) to ensure that the temperature of each zone is increasing.

9. After the temperature of each zone has stabilized, read the value from the reference thermocouple probe, using the locations described earlier. Enter these readings on the Calibrate dialog in the **High Calibration** fields. To do so, click on one of the **High Cal** fields, then press the space bar. The Calibrate dialog is displayed. Enter the value, then click **OK**. Repeat for each zone.

10. Use manual control to return the zone temperatures to ambient. Replace the valve cover.

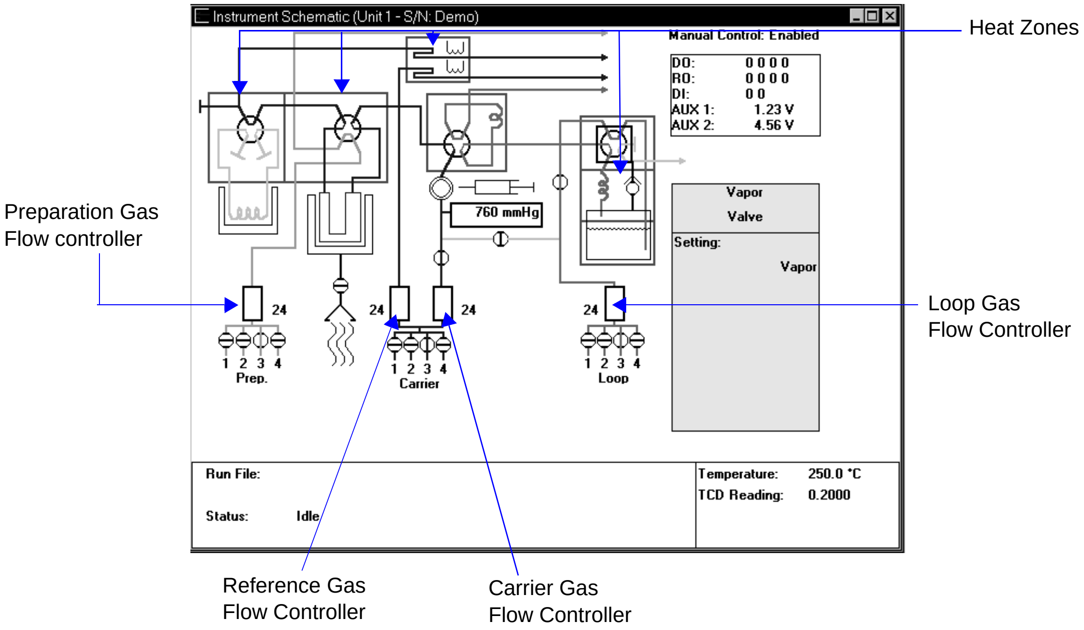
11. On the Calibrate Vapor Generator dialog, click **Accept** to save the new calibration values. The software then displays dialogs that allow you to verify this change and make a backup copy of the values you are replacing. If desired, make a backup copy by clicking **Yes** then specifying a file name.

12. Replace the analyzer’s top panel.

Performing a Leak Test

Set Up

1. Select **Unit > Enable Manual Mode**, then select **Unit > Show Instrument Schematic**.



Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Setting: Vapor

Run File:

Status: Idle

Temperature: 250.0 °C
TCD Reading: 0.2000

2. Set all heat zones to ambient temperature.
3. Set all four Flow Controllers to 50 cm^3/min.
4. Allow the analyzer to cool to room temperature.

This test must be done in the order detailed in this document to test fully the AutoChem. Any flow reading other than zero indicates a leak in the gas flow path being tested.

Preparation Gas Path Tests

1. Right-click the analysis valve and choose **Analyze**.
2. Right-click the Prep. valves and select **Flow Gas [n] (He)**.
3. Plug the preparation gas exhaust outlet.
4. Observe the Preparation Gas Flow Controller reading as it slowly drops to zero.

A flow reading of zero indicates the flow path between the Preparation Gas Flow Controller and the preparation exhaust does not leak.

This tests all fitting connections in the gas flow path and part of the analysis valve.

5. Any flow reading other than zero indicates a leak in the gas flow path being tested.
6. To fix a leak that may be indicated on the Preparation Gas Flow Controller, tighten the fittings in the gas flow path and observe the reading for any decrease that would indicate that the leak has stopped.

Any leak detected in the remaining steps of this procedure may be fixed by using the same process mentioned in Step 6.

7. Right-click the Preparation Gas Flow Controller and choose **Stop Flow**. This will close the helium valve.
8. Remove the plug from the preparation gas exhaust outlet.

Reference Gas Path Tests

1. Valve positions need not be preset for this test.
2. Right-click the Carrier valves and choose Flow Gas [n] (He) through the Reference Gas Flow Controller.
3. Plug the reference gas exhaust outlet.
4. Observe the Reference Gas Flow Controller reading as it slowly drops to zero.
5. A flow reading of zero indicates the flow path between the Reference Gas Flow Controller and the reference gas exhaust does not leak.

   This tests all fitting connections in the gas flow path and the reference side of the thermal conductivity detector.

   Any flow reading other than zero indicates a leak in the gas flow path being tested.
6. Right-click the Reference Gas Flow Controller and select **Stop Flow**. This will close the helium valve.
7. Remove the plug from the reference gas exhaust outlet plug in place.

Carrier Gas Path Tests

1. Right-click the Cold Trap Valve and select **Bypass**.

2. Right-click the Analysis Valve and select **Prepare**.

3. Right-click the Loop Valve and select **Fill**.

4. Turn the pressure control knob on the helium gas regulator to deliver less than 5 psi.

5. Right-click the Carrier Valves and choose **Flow Gas [n] (He)**.

6. Plug the analysis gas exhaust outlet.

7. Right-click on Calibration Valve B and select **Close Valve**, then repeat the step for Calibration Valve C.

8. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero.

9. A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and Calibration Valve B does not leak.

10. Right-click Calibration Valve B and select **Open Valve**.

11. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

    A flow reading of zero indicates the flow path between the carrier MFC and the carrier exhaust outlet does not leak.

    This tests all fitting connections in the gas flow path and part of the analysis valve, part of the bypass valve, the transducer, the septum, part of the inject valve, and the analysis side of the thermal conductivity detector.

12. Right-click the Loop Valve and select **Inject**.

13. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

    A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and the carrier gas exhaust outlet does not leak.

    This tests all fitting connections in the gas flow path, the remaining parts of the Loop Valve, and the loop.

14. Install an empty sample tube on the sample port.

15. Right-click the Analysis Valve and select **Analyze**.

16. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

    A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and the carrier gas exhaust outlet does not leak.

    This tests all fitting connections in the gas flow path, the sample tube, and the remaining parts of the Analysis Valve.

17. Install a cold trap (or a delay path) on the cold trap port.
18. Right-click the Cold Trap Valve and select **Trap**.
19. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

    A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and the carrier gas exhaust outlet does not leak.

    This tests all fitting connections in the gas flow path, the remaining parts of the Cold Trap Valve, and the cold trap.

20. Right-click the Carrier Valves and select **Stop Flow**. This will close the helium valve.
21. Remove the plug from the analysis exhaust outlet.
22. Reset the pressure on the Helium gas regulator to its original pressure.

Vapor Generator (If installed) Gas Path Tests

1. Right-click the Loop Valve and select **Inject**.
2. Right-click the Vapor Valve and select **Bypass**.
3. Right-click Calibration Valve C and select **Close Valve**, then repeat the step for Calibration Valve D.
4. Plug the loop gas exhaust outlet.
5. Plug the vapor generator gas exhaust outlet.
6. Right-click the Loop Gas Valves and choose **Flow Gas [n] (He)** (or other appropriate gas) through the Loop Gas Flow Controller.
7. Observe the Loop Gas Flow Controller reading as it slowly drops to zero.

    A flow reading of zero indicates the flow path between the Loop Gas Flow Controller and Calibration Valves C and D does not leak.

    This tests all fitting connections in the small section of the gas flow path to Calibration Valves C and D.

8. Right-click Calibration Valve D and select **Open Valve**.
9. Observe the Loop Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

   A flow reading of zero indicates the flow path between the Loop Gas Flow Controller and the loop gas exhaust outlet does not leak.

   This tests all fitting connections in the gas flow path, portions of the vapor generator valve, and the tubing up to the inject valve.
10. Install an empty flask onto the vapor generator port.
11. Right-click the Vapor Valve and select **Vapor**.
12. Observe the Loop Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

    A flow reading of zero indicates the flow path between the Loop Gas Flow Controller and the loop gas exhaust does not leak.

    This tests all fitting connections in the gas flow path, the remaining portions of the vapor generator valve, and the relief valve in the vapor generator.
13. Right-click the Loop Gas Flow Controller and select Stop Flow. This will close the gas valve.
14. Remove all exhaust plugs.

Loop Gas Path Tests (Vapor Generator not installed)

1. Right-click the Vapor Valve and select **Inject**.
2. Right-click Calibration Valve C and select **Close Valve**, then repeat the step for Calibration Valve D.
3. Plug the loop gas exhaust outlet.
4. Right-click the Loop Gas Valves and choose Flow Gas [n] (He) (or other appropriate gas) through the Loop Gas Flow Controller.
5. Observe the Loop Gas Flow Controller reading as it slowly drops to zero.

   A flow reading of zero indicates the flow path between the Loop Gas Flow Controller and Calibration Valves C and D does not leak.

   This tests all fitting connections in the small section of the gas flow path to Calibration Valves C and D.
6. Right-click Calibration Valve D and select **Open Valve**.

7. Observe the Loop Gas flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

   A flow reading of zero indicates the flow path between the Loop Gas Flow Controller and the loop gas exhaust outlet does not leak.

   This tests all fitting connections in the gas flow path, Calibration Valves C and D, and the tubing to the Loop Valve.

10. ORDERING INFORMATION

The AutoChem II 2920 Analyzer components and accessories can be ordered by:

- Telephone: 770/662-3636
- Email: customer.service@micromeritics.com
- Internet: www.micromeritics.com

When ordering, please use the information provided below.

Part Number	Item and Description
292-42803-00	Operator's Manual, AutoChem II 2920 Analyzer
008-42001-00	*Analytical Methods in Fine Particle Technology*, a comprehensive resource of information on particle analysis. This hardbound text describes theory, instrumentation, and applications pertaining to chemisorption and temperature-programmed reactions, as well as other analytical methods used in the characterization of catalysts and other materials.
291-34005-00	KwikCool; provides rapid cooling of the furnace, decreasing analysis time and increasing throughput.
291-34012-00	Vapor Generator; allows a carrier gas to take vapor generated from liquid at up to 100 ºC
291-33005-00	CryoCooler; allows furnace system to hold sample at approximately -120 °C, then ramp upwards. Includes benchtop dewar.
021-00000-00	Model 021 LN_2 transfer system, for easy filling of sample dewars; includes 47-L Dewar, mobile platform, and pump with 100-cm discharge line
291-25911-00	Cold trap assembly, stainless steel
290-25845-00	Delay path assembly (for BET analysis), copper
290-25846-00	Gas inlet line assembly; includes copper tubing, with ferrules and nuts (1/8-in. male fitting to connect to analyzer on one end, 1/8-in. female fitting to connect to regulator on opposite end), 6 ft (1.8 m) in length
290-25446-01	Same as 290-42846-00, except 16 ft (5 m) in length
201-25818-00	Gas inlet line assembly; includes stainless steel tubing, with ferrules and nuts (1/8-in. male fitting to connect to analyzer on one end, 1/8-in. female fitting to connect to regulator on opposite end), 6 ft (1.8 m) in length
201-25818-01	Same as 201-25818-00, except 16 ft (5 m) in length

Part Number	Item and Description
930-54603-00	Brush, narrow; for cleaning sample tubes
004-54609-00	Brush, wide; for cleaning sample tubes
291-31801-00	Cover, half, for cold trap dewar. (Two needed per dewar.)
004-61064-00	Dewar, 600 cm^3
004-62102-00	Rotor for 6-port rotary valves
004/25940/00	Ferrule, one piece, 1/8-in. PEEK, for the quartz thermocouple sheath, which mounts through a 1/8-in. fitting
004/25938/00	Ferrule, one piece, 1/16-in. ID, for mounting the sample thermocouple in the same 1/8-in. fitting as the quartz thermocouple sheath
004-25913-00	Ferrule Set (front and rear), 1/8 in., PEEK polymer
291-25815-00	Ferrule, for sample tube, 6 mm
291-25818-00	Ferrule, for sample tube, 12 mm
004-27041-00	Filter Disk, fritted, 20 μm SS, 1/4-in. diameter x 1/16-in. thick, for sample exhaust port
290-29800-00	Flowmeter, bubble, 10 cm^3
004-16247-00	Fluid, gas bubble
004-32187-00	Gloves, cotton flannel, pair
290-62600-00	Injection loop, 0.5 cm^3 nominal, stainless steel
290-62601-00	Injection loop, 1.0 cm^3 nominal, stainless steel
290-62602-00	Injection loop, 5.0 cm^3 nominal, stainless steel, silver-solder joints in loop
004-25678-00	O-ring, -009 Kalrez™, for sample tube outlet side
004-25465-00	O-ring, -010 Kalrez, for sample port
004-25474-00	O-ring, -013 Kalrez, for sample tube inlet
004-32164-01	Quartz Wool, 4-in. x 6-in. bag
004-25549-00	Reducer, 1/8 in. tube x 1/4 in. tube, for regulators
004-16836-00	Reference Material, silver oxide
290-25842-00	Septum, external gas-sampling assembly
280-32800-00	Support, sample weighing
004-61602-00	Syringe, 1 cm^3

Part Number	Item and Description
004-61604-00	Syringe, 10 cm^3
004-61606-00	Syringe, 0.1 cm^3
291-53904-00	Thermocouple, sample, pre-shaped for analyzer
291-61702-00	Sample tube, quartz
004-32004-00	Stopper for sample tube inlet
004-32604-08	Cap, plastic, for sample tube exhaust
292-32801-00	Protective shelf mat
004-62230-32	Gas regulator, 2-stage, CGA 320 fitting, 30 psig (CO_2)
004-62230-35	Gas regulator, 2-stage, CGA 350 fitting, 30 psig (CO, H_2)
004-62230-54	Gas regulator, 2-stage, CGA 540 fitting, 30 psig (O_2)
004-62230-58	Gas regulator, 2-stage, CGA 580 fitting, 30 psig (N_2,He, Ar. Kr)
004-62230-59	Gas regulator, 2-stage, CGA 590 fitting, 30 psig (ultrapure air)
004-62200-58	Gas regulator, 2-stage, CGA 580 fitting, 100 psig (N_2, He, Ar, Kr)
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator (includes fittings and instructions)
004-54805-00	Extraction Tool, for inserting quartz wool into or for removing from sample tubes

A. FORMS

Determining the Sample Mass

Complete instructions for determining the mass of your sample and loading it into the sample tube are located in Chapter 4 beginning on page **4-10**. This worksheet provides a format for recording the values necessary to calculate the sample's mass. A space is also provided for recording the sample mass after analysis.

Some operators prefer to use the sample's mass after the analysis; this is particularly desirable when a significant amount of sample preparation occurs in situ during the analysis. If you want the after-analysis value for the mass to be used in calculations, determine the after analysis sample mass, then open the sample information file and enter the after analysis mass in the Sample mass field before editing or reporting data for the analysis.

In some cases, the difference in the sample's mass recorded after the analysis versus that recorded before the analysis may be significant in assessing the results.

Sample identification:____

		________
1	Empty sample tube mass (including quartz wool and stoppers):	________
2	Sample tube plus sample mass:	________
3	Sample mass (Step 2 - Step 1):	________
4	Sample tube plus sample mass after analysis (optional):	________
5	Sample mass after analysis (Step 4 - Step 1) (optional)	________

Manual Injections

When a series of manual injections is performed, it may be useful to list the planned injection volumes, then check each one off immediately after it is performed.

Injection Volume	Check when completed	Comments
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

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Current Gas Connections

Use this form to record the gases currently connected to the analyzer. You may wish to post the completed form on or near the analyzer.

This form may help you make sure that the correct gas for each port is identified in the software. Complete the form, then open the Gas Selections dialog from the Unit menu. Verify that the correct gas is selected for each port. If you are using the optional Gas Blender, you can also record the gases connected to each blender port.

Unit number (S/N): ___________________________________

Date:____

Operator: ____

Preparation Gas	**Carrier/Reference Gas**	**Loop Gas**

B. CALCULATIONS

Volume at STP

Volume at standard reduction of gas data to Standard Temperature Pressure (STP) conditions (273.13K and 760 mmHg).

$$V_{STP} = V_a\left(\frac{273.15}{273.15 + T_a}\right) \times \left(\frac{P_a}{760\ mmHg}\right)$$

V_{STP} = Volume at STP, cm^3
V_a = Volume at ambient temperature and pressure, cm^3
T_a = Ambient temperature, °C
P_a = Ambient pressure, mmHg

Injection Loop Calibration

These equations are used during calibration of a dose loop

$$V_{S_{STP}} = V_S\left(\frac{273.15}{273.15 + T_a}\right) \times \left(\frac{P_a}{760\ mmHg}\right)$$

$$V_l = \left(\frac{A_l \times V_{S_{STP}}}{A_S}\right)$$

V_{SSTP} = Volume of syringe at STP, cm^3
V_S = Physical volume of syringe, cm^3
T_a = Ambient temperature, °C
P_a = Ambient pressure, mmHg
V_l = Effective loop volume (at loop temperature), cm^3
A_l = Average peak area of loop injections
A_S = Average peak area of syringe injections

Injection Loop Volume – Temperature Change

This equation is used to correct the apparent loop volume when the temperature of the loop heat zone is changed.

$$V_n = V_o\left(\frac{T_o + 273.15}{T_n + 273.15}\right)$$

V_n = New Effective Loop Volume, cm^3
V_o = Old Loop Volume, cm^3
T_o = Old Loop Temperature, °C
T_n = New Loop Temperature, °C

Calibration Error (Goodness of Fit)

This equation is a means to determine whether the calibration curve is acceptable.

$$E_{\%} = \left[\sum_{i=0}^{N}\frac{(Q_{m_i} - Q_{c_i})^2}{(N-1)}\right]^{\frac{1}{2}}$$

$E_{\%}$ = Percent error
Q_m = Quantity measured
Q_c = Quantity calculated
N = Number of measurements

Percent Dispersion

This equation is used in a Pulse Chemisorption analysis to calculate the percent dispersion. Some understanding of the chemistry is required to select the proper stoichiometric factor and gram molecular weight.

$$PD = 100\left(\frac{V_S \times SF_{calc}}{SW \times 22414}\right)GMW_{calc}$$

PD = Percent dispersion
V_s = Volume sorbed (cm^3 at STP)
SF_{calc} = Calculated stoichiometry factor
SW = Sample weight (g)
GMW_{calc} = Gram Molecular Weight (g/g-mole)

BET Surface Area

For each included point i, relative pressure P/Po_i and specific volume adsorbed V_{STPi} are calculated for a single point BET. Then the BET transformation B_i is calculated:

$$B_i = \frac{\frac{P}{Po_i}}{\left(1 - \frac{P}{Po_i}\right) V_{STP_i}}$$

where B_i is in units of g/cm^3 STP.

A linear least squares fit is performed on the (B_i, P/Po_i) paris where B_i is the dependent variable and P/Po_i is the independent variable. The following are calculated:

Slope, S, g/cm^3 STP
Y-intercept, Y_{INT}, g/cm^3 STP
Error of the slope, S_{ERR}, g/cm^3 STP
Error of the y-intercept, YI_{ERR} g/cm^3 STP
Correlation coefficient, C_C

Using the results of the above calculations, the following are calculated:

BET Surface Area (SA_{BET} m$_2$/g):

$$SA_{BET} = \frac{CSA \times (6.023 \times 10^{23})}{(22414\ cm^3\ STP) \times (10^{18}\ nm^2/m^2) \times (S + Y_{INT})}$$

CSA = adsorbate molecular cross-sectional area (nm$_2$)

BET C value:

$$C = \frac{S + Y_{INT}}{Y_{INT}}$$

Volume of monolayer (cm^3/g STP):

$$V_M = \frac{1}{C \times Y_{INT}} = \frac{1}{S + Y_{INT}}$$

Error of the BET Surface Area (m^2/g):

$$BET_{ERR} = \frac{SA_{BET} \times (S_{ERR}{}^2 + YI_{ERR}{}^2)^{0.5}}{Y_{INT} + S}$$

Langmuir Surface Area

For each included point i, pressure P_i and specific volume adsorbed $VSTP_i$ are calculated as for a single point BET. Then the Langmuir transformation L_i is calculated:

$$L_i = \frac{P_i}{V_{STP_i}}$$

where L_i is in units of g/cm^3 STP.

A linear least squares fit is performed on the (L_i, P) pairs where L_i is the dependent variable and P is the independent variable. The following are calculated:

Slope, S, g/cm^3 STP
Y-intercept, Y_{INT}, g/cm^3 STP
Error of the slope, S_{ERR}, g/cm^3 STP
Error of the y-intercept, YI_{ERR} g/cm^3 STP
Correlation coefficient, C_C

Using the results of the above calculations, the following are calculated:

Langmuir Surface Area (SA_{LAN} m^2/g):

$$SA_{LAN} = \frac{CSA \times (6.023 \times 10^{23})}{(22414\ cm^3\ STP) \times (10^{18}\ nm^2/m^2) \times S}$$

Volume of monolayer (cm^3/g STP):

$$V_M = \frac{1}{S}$$

Langmuir b value:

$$b = (Y_{INT})(V_M)$$

Error of the Langmuir Surface Area (m^2/g):

$$LAN_{ERR} = \frac{SA_{LAN} \times S_{ERR}}{S}$$

Total Pore Volume

Total pore volume V_{POR} at a selected physisorption point is calculated:

$$V_{POR} = (V_{STP})(DCF)$$

DCF = adsorbate density conversion factor

Volume Sorbed

This equation is for loop or syringe injections to calculate volume removed from the injections by the sample.

$$V_S = NV_i - V_{na}$$

V_s = Volume sorbed, cm^3
N = Number of injections
V_i = Volume per injection, cm^3
V_{na} = Total volume not sorbed, cm^3

Peak Area Volume

This is the basic data reduction equation. It converts the area under a peak to cm^3 of gas.

$$V_{pa} = A_p \times F_c$$

V_{pa} = Peak area volume, cm^3
A_p = Peak area
F_c = Calibration factor

Stoichiometry Factor

The calculated stoichiometry factor is a weighted average. It is dependent on both the individual stoichiometry factor and number of moles of each active metal.

$$SF_{calc} = \left[\left(\frac{F_1 \times SF_1}{W_{atomic1}}\right) + \left(\frac{F_2 \times SF_2}{W_{atomic2}}\right) + \ldots + \left(\frac{F_N \times SF_N}{W_{atomicN}}\right)\right] \cdot GMW_{calc}$$

SF_{calc} = Calculated stoichiometry factor
SF_N = Stoichiometry factor for metal N
F_1 = Fraction of sample weight for metal N
$W_{atomic1}$ = Gram molecular weight of first metal (g/g-mole)
GMW_{calc} = Gram molecular weight (g/g-mole)

Gram Molecular Weight

The calculated Gram Molecular Weight is a weighted average of the number of moles of each active metal.

$$GMW_{calc} = \frac{1}{\left(\frac{F_1}{W_{atomic1}}\right) + \left(\frac{F_2}{W_{atomic2}}\right) + \ldots + \frac{F_N}{W_{atomicN}}}$$

GMW_{calc} = Gram molecular weight (g/g-mole)
F_N = Fraction of sample weight for metal N
$W_{atomicN}$ = Gram Molecular Weight of first metal (g/g-mole)

Cross-Sectional Area

$$SA_{calc} = \left[\left(\frac{F_1 \times SA_1}{W_{atomic1}}\right) + \left(\frac{F_2 \times SA_2}{W_{atomic2}}\right) + \ldots + \left(\frac{F_N \times SA_N}{W_{atomicN}}\right)\right] \cdot GMW_{calc}$$

SA_{calc} = Calculated cross-sectional area
F_N = Fraction of sample weight for metal N
SA_N = Cross-sectional area for metal N
$W_{atomicN}$ = Gram molecular weight of first metal (g/g-mole)

Metal Density

$$D_{calc} = \left[\left(\frac{F_1 \times D_1}{W_{atomic1}}\right) + \left(\frac{F_2 \times D_2}{W_{atomic2}}\right) + \ldots + \left(\frac{F_N \times D_N}{W_{atomicN}}\right)\right] \cdot GMW_{calc}$$

D_{calc} = Calculated density
F_N = Fraction of sample weight for metal N
D_N = Density for metal N
$W_{atomicN}$ = Gram Molecular Weight of first metal (g/g-mole)

Metallic Surface Area

This equation calculates the effective metallic surface area per gram of sample.

$$SA_{Metallic} = \left(\frac{V_S}{SW \times 22414}\right) \times (SF_{calc}) \times (6.123 \times 10^{23}) \times (SA_{calc})$$

$SA_{Metallic}$ = Metallic surface area (m2/g of metal)
V_s = Volume sorbed (cm^3 at STP)
SF_{calc} = Calculated stoichiometry factor
SA_{calc} = Calculated specific surface area
SW = Sample weight (g)

Active Particle Size

This equation calculates the active particle size.

$$APS = \frac{6}{(D_{calc}) \times \left(\frac{W_S}{GMW_{calc}}\right) \times (6.023 \times 10^{23}) \times (SA_{calc})}$$

APS = Active particle size
D_{calc} = Calculated metal density (g/cm^3)
W_s = Sample weight, g
GMW_{calc} = Gram molecular weight (g/g-mole)
SA_{calc} = Calculated specific surface area (per gram of metal)

First Order Kinetics

This series of steps calculates the First Order Kinetics (Heat of Desorption). At least two experiments must be run — commonly 3 experiments are run. The ramp rate must be different for each run. The data are plotted and the slope determined.

The general equation is:

$$\ln\left(\frac{\beta}{T_p{}^2}\right) = -\frac{E_d}{RT_p} + \ln\left(\frac{E_d A}{RC}\right)$$

Determine the slope s by plotting 2 ln T_p - ln β *vs.* $\frac{1}{T_p}$ then determine E_d using:

$$E_d = s \times R$$

β = Ramp rate, degrees/min
E_d = Heat of desorption, K joules/(mole K)
R = Gas constant
T_p = Temperature at peak max
s = Slope
A = The quantity adsorbed at saturation
C = A constant related to the desorption rate

Vapor Generator

The partial pressure of the vapor is estimated using the Antoine equation:

$$\ln p_v = A - \frac{B}{T + C}$$

p_v = Partial pressure of vapor at reflux temperature in mmHg
A = Antoine coefficient A
B = Antoine coefficient B
C = Antoine coefficient C
T = Temperature in Kelvin

The following references contain Antoine constants:

- R. Reid *et.al.*, *The Properties of Gases and Liquids*, McGraw-Hill (1987)
- J. Dean, *Lange's Handbook of Chemistry*, McGraw-Hill (1999)

Use the appropriate format of the Antoine equation as given by R. Reid or J. Dean. Some authors prefer to use temperature in Celsius rather than Kelvin and, similarly, $\log_{10}(P_v)$ rather than $\ln(P_v)$.

The fraction of vapor can then be calculated using:

$$y_v = \frac{P_v}{P}$$

y_v = Fraction of vapor at the reflux temperature
P = Ambient pressure

The volume of vapor for each peak in the vapor calibration is determined from:

$$V_v = Y_v \times V_l \times \frac{273.15}{t_v + 273.15}$$

V_v = Vapor volume at reflux temperature
V_l = Loop volume at STP
t_v = Reflux temperature in °C

C. GAS INFORMATION

Table C-1. Relative Thermal Conductivity of Gases

Name	Chemical Formula	Conductivity (Relative to Air)
Air		1.00
Ammonia	NH_3	0.92
Argon	Ar	0.68
Butane	C_4H_{10}	0.60
Carbon Dioxide	CO_2	0.62
Carbon Monoxide	CO	0.97
Ethane	C_2H_6	0.79
Helium	He	5.84
Hydrogen	H_2	7.07
Krypton	Kr	0.37
Methane	CH_4	1.29
Neon	Ne	1.87
Nitric Oxide	NO	0.99
Nitrogen	N_2	1.00
Nitrogen Dioxide	NO_2 or N_2O_4	1.51
Nitrous Oxide	N_2O	0.65
Oxygen	O_2	1.02
Sulfur Dioxide	SO_2	0.38
Water Vapor	H_2O	0.67

Table C-2. Typical Gases Used with the AutoChem 2920 Analyzer

Test	Gases			Flow Rate (cm^3/min)	Other
	Preparation	Carrier	Loop		
TPR Experiment	Argon	10% H_2 in Argon	N/A	50	
Calibration	N/A	10% H_2 in Argon	Argon	50	TCD Level Calibration
TPD Ammonia	Helium or 15% NH_3 in Helium	Helium	N/A	50	
Calibration	N/A	Helium	NH_3 in Helium	50	TCD Level Calibration
TPD Pyridine	Helium	Helium	Helium	50	Pyridine in Vapor Generator
Calibration	N/A	Helium	Helium	50	User-Defined Pyridine in Vapor Generator
TPD Hydrogen	10% H_2 in Argon	Argon	N/A	50	Calibration
Calibration	N/A	Argon	10% H_2 in Argon	50	TCD Level Calibration
TPD Oxygen	10% O_2 in Helium	Helium	N/A	50	
Calibration	N/A	Helium	10% O_2 in Helium	50	TCD Level Calibration
TPO Experiment	Helium	10% O_2 in Helium	N/A	50	
Calibration	N/A	10% O_2 in Helium	Helium	50	TCD Level Calibration
H_2 Pulse Chemisorption	10% H_2 in Argon	Argon	10% H_2 in Argon	50	
CO Pulse Chemisorption	10% H_2 in Argon	Helium	10% CO in Helium	50	
Calibration	Not required				
BET Surface Area	Helium	30% N_2 in Helium	N/A	50	
Calibration	N/A	30% N_2 in Helium	N/A	50	User-Defined Manual injections of N_2 (0.5, 1.0, 1.5, and 2.0 cm^3)

Table C-3. Conversion Constants for the Mass Flow Controllers (MFCs)

Gas	**Symbol**	**MFC Conversion Constant (H_2 = 1.000)**
Acetylene	C_2H_2	0.6535
Air (mixture)		0.9901
Allene	C_3H4	0.4752
Ammonia	NH3	0.7822
Argon	Ar	1.3861
Arsine	A_5H_3	0.7525
Boron Trichloride	BCl_3	0.4356
Boron Trifluoride	BF_3	0.5743
Bromine Pentafluoride	BrF_5	0.2871
Bromine Trifluoride	BrF_3	0.4356
Butane	C_4H_{10}	0.2871
Butene	C_4H_8	0.3267
Carbon Dioxide	CO_2	0.7723
Carbon Monoxide	CO	0.9802
Carbon Tetracloride	CCl_4	0.3465
Carbon Tetrafluoride	CF_4	0.4752
Carbonyl Fluoride	COF_2	0.2673
Carbonyl Sulfide	COS	0.6733
Chlorine	Cl_2	0.8218
Chloroform	$CHCl_3$	0.4356
Chlorine Trifluoride	ClF_3	0.4257
Cyanogen	C_2N_2	0.4950
Cyclopropane	C_3H_6	0.5050
Deuterium	D_2	0.9901
Diborane	B_2H_6	0.5446
Dichlorosilane	SiH_2Cl_2	0.4356
Dimethylamine	$(CH_3)_2NH$	0.6634
Dimethylether	$(CH_3)_2O$	0.5842
Ethane	C_2H_6	0.5446
Ethyl Chloride	C_2H_5Cl	0.2871
Ethylene	C_2H_4	0.6139
Ethylene Oxide	C_2H_4O	0.5842

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Fluorine	F_2	0.9208
Fluroform	CHF_3	0.5644
Freon 11	CCl_3F	0.3762
Freon 12	CCl_3F2	0.3861
Freon 13	$CClF_3$	0.4257
Freon 13 B1	$CBrF_3$	0.4059
Freon 14	CF_4	0.4703
Freon 21	$CHCl_2F$	0.4554
Freon 22	$CHClF_2$	0.5050
Freon 23	CHF_3	0.5644
Freon 113	$CCl_2F\text{-}CClF_2$	0.2277
Freon 114	$CCl_2F_4\text{-}CClF_2$	0.2554
Freon 115	$CClF_2\text{-}CF_3$	0.2713
Freon 116	$CF_3\text{-}CF_3$	0.2277
Germane	GeH_4	0.6436
Helium	He	1.3762
Hexamethyldisizane	HMDS	0.1386
Hydrogen	H_2	1.0000
Hydrogen Bromide	HBr	0.9703
Hydrogen Chloride (Dry)	HCl	0.9802
Hydrogen Fluoride	HF	0.9901
Hydrogen Iodide	HI	0.9505
Hydrogen Selenide	H_2Se	0.8317
Hydrogen Sulfide	H_2S	0.8416
Isobutane	C_4H_{10}	0.3069
Isobutylene	C_4H_8	0.3366
Krypton	Kr	1.3762
Methane	CH_4	0.8020
Methylamine	CH_3NH_2	0.5644
Methyl Bromide	CH_3Br	0.6436
Methyl Chloride	CH_3Cl	0.6832
Methyl Fluoride	CH_3F	0.7525
Methyl Mercaptan	CH_4S	0.5842
Neon	Ne	1.3861

Gas	**Symbol**	**MFC Conversion Constant (H_2 = 1.000)**
Nitric Oxide	NO	0.9901
Nitrogen	N_2	0.9950
Nitrogen Dioxide	NO_2	0.7525
Nitrogen Trioxide	N_2O_3	0.4356
Nitrogen Trifluoride	NF_3	0.5446
Nitrous Oxide	N_2O	0.7426
Oxygen	O_2	0.9802
Ozone	O_3	0.7327
Pentaborane	B_5Hg	0.2871
n Pentane	C_5H1_2	0.2376
Perchloryl Fluoride	ClO_3F	0.4455
Phosgene	$COCl_2$	0.5050
Phosphine	PH_3	0.7822
Phosphorous Pentafluoride	PF_5	0.3465
Propane	C_3H_8	0.3861
Propylene (Propene)	C_3H_6	0.4653
Silane	SiH_4	0.6733
Silicon Tetrachloride	$SiCl_4$	0.3168
Silicon Tetrafluoride	SiF_4	0.3960
Sulfur Dioxide	SO_2	0.7228
Sulfur Hexafluoride	SF_6	0.2970
Trichlorosilane	$SiCHl_3$	0.3267
Trimethylamine	$(CH_3)_3N$	0.3168
Tungsten Hexafluoride	WF_6	0.2871
Uranium Hexafluoride	UF_6	0.2178
Vinyl Bromide	C_2H_3Br	0.5248
Vinyl Chloride	C_2H_3Cl	0.5347
Vinyl Fluoride	C_2H_3F	0.5743
Xenon	Xe	1.3762

D. FORMAT OF EXPORTED DATA

Data collected using the AutoChem II 2920 analyzer can be exported to a file for use in other applications. Data may be exported as an ASCII or Grams/32 (a peak-editing program used in earlier versions of the AutoChem application) format. Instructions for exporting data are contained in Chapter 4.

ASCII Format

Data are exported in the following format when ASCII is chosen as the export type:

Header:

<sample id>
<operator>
<submitter>
<sample mass>
<instrument serial number>
<ambient temperature>
<ambient pressure>
<Po of adsorbate>

After the Header is exported, the remaining data are determined by whether **Peak Summaries** or **Measurements** was specified in the report options set.

- For **Peak Summaries**, the peaks for each selected experiment in the file are summarized. Data are exported as follows:

  <experiment number>, <start time (minutes)>, <stop time (minutes)>

- For **Each Peak:**

  <experiment type>, <start time (minutes)>, <stop time (minutes)>, <temperature at maximum (°C)>, <area>

  <experiment type> is one of the following: TPD", :TPR", "TPO", "Physisorption", "Chemisorption", "Level Cal.", "Vapor Cal.", "Loop Cal.", "Other"

- **For Measurements**, the data points for each checked experiment in the file are reported. Data are exported as follows:

  <experiment number>, <start time (minutes)>, <stop time (minutes)>

For **Each Point:**
<time (minutes)>, <temperature (°C)>, <TCD signal>, <event flag>, <input signal 1>, <Input signal 2>

The TCD signal may be either calibrated or not calibrated based upon the selections made when the export operation was performed.

The *event flag* is either 1 or 0. The state of the event flag switches when a pulse chemisorption dose is started.

Grams/32

When Grams/32 is chosen as the export type, five files are created for each experiment selected for export. The data in these files can be read only by the Grams/32 software. Table D-1 lists the names and contents of these files.

Table D-1. Grams/32 Export File Names

File Name	Contents
xxxnn.CGM	Calibrated TCD signal vs. time (same as xxxSGnn.SPC when no calibration is specified)
xxxSGnn.SPC	Uncalibrated TCD signal vs. time
xxxTMnn.SPC	Sample temperature vs. time
xxxT1nn.SPC	Auxiliary signal one vs. time
xxxT2nn.SPC	Auxiliary signal two vs. time

Where **xxx** is the first three characters of the input sample file name and nn is the number of the experiment within the sample.

For example, the *second* experiment of the sample file **ABC.SMP** would be exported into the following Grams/32 files:

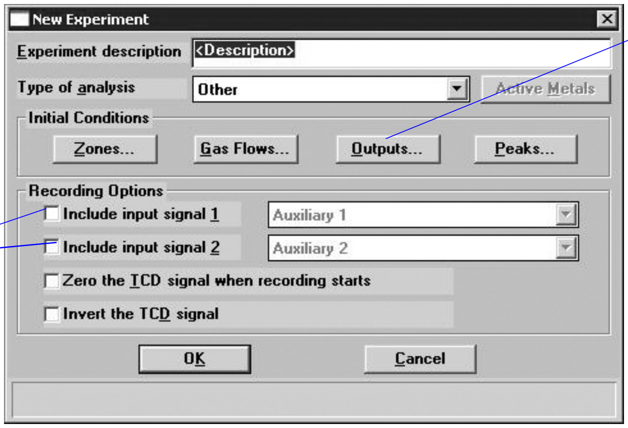
ABC02.CGM
ABCSG02.SPC
ABCTM02.SPC
ABCT102.SPC
ABCT202.SPC

E. AUXILIARY INPUTS AND OUTPUTS

You can connect analog and digital signals to the AutoChem II 2920 analyzer. Two connectors are available on the analyzer's right side panel; both are industry-standard "D" shell connectors. You must supply a shielded cable for any auxiliary signal you connect to the analyzer. All cables must use male pins in the connectors. The analog connector uses a 15-pin body; the digital connector uses a 25-pin body. The cable shield must connect to a metal shell "D" connector housing.

Software

Analog inputs are enabled by marking one or both check boxes on the Insert Experiment dialog; this dialog is opened from the Analysis Conditions dialog of the sample information file (Advanced format only). Click **Insert**, then select **Experiment**. After an auxiliary input is enabled, it remains enabled for the duration of the experiment. Because an analysis can be comprised of many experiments, an input can be disabled or enabled at various points in the analysis, as determined by the selection on the New Experiment dialogs.



Experiment description: <Description>

Type of analysis: Other

☐ Include input signal 1 — Auxiliary 1

☐ Include input signal 2 — Auxiliary 2

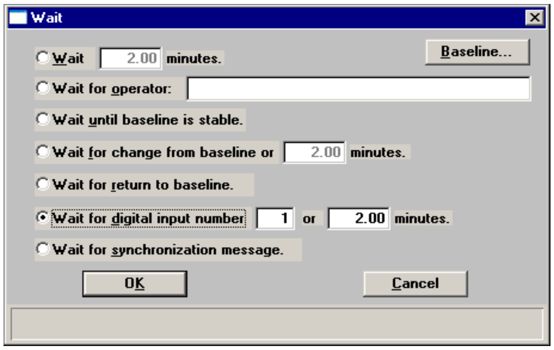
☐ Zero the TCD signal when recording starts

☐ Invert the TCD signal

Click this button to set the analog and digital outputs.

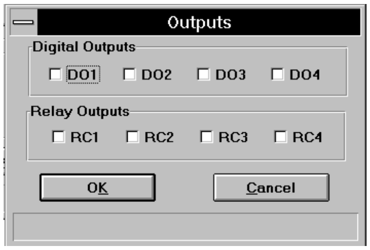
Mark one or both boxes to enable the analog

Digital inputs are controlled by inserting a **Wait** step in the analysis. On the Wait dialog (shown below), mark the radio button next to Wait for digital input number. Specify **1** or **2** to indicate the desired input signal. Enter a number of minutes to wait; if the desired signal is not received before that amount of time elapses, the analysis continues without the input.



○ Wait 2.00 minutes.
○ Wait for operator:
○ Wait until baseline is stable.
○ Wait for change from baseline or 2.00 minutes.
○ Wait for return to baseline.
● Wait for digital input number 1 or 2.00 minutes.
○ Wait for synchronization message.

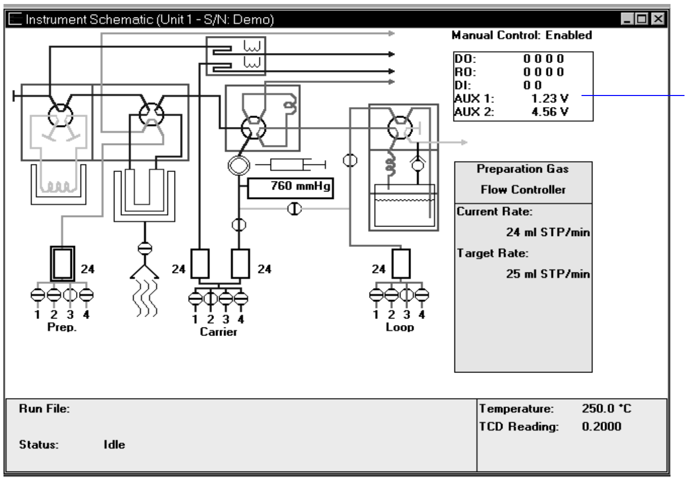
Analog and digital outputs are enabled by marking the applicable check box(es) on the Outputs dialog. The Outputs dialog is available when you are setting the initial conditions for an experiment (by clicking the **Outputs** button on the New Experiment dialog), or at many other points in the analysis (by inserting a Set Outputs step in the Synopsis). The state of the auxiliary outputs can therefore be changed frequently throughout the analysis.



Digital Outputs
☐ DO1 ☐ DO2 ☐ DO3 ☐ DO4

Relay Outputs
☐ RC1 ☐ RC2 ☐ RC3 ☐ RC4

The state of the auxiliary inputs and outputs is displayed on the instrument schematic. Auxiliary signals can be incorporated in reports (overlaid over other data in graphs), in the Results view of the Analysis dialog (during analysis), and in the Peak Editor.



Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Preparation Gas Flow Controller
Current Rate: 24 ml STP/min
Target Rate: 25 ml STP/min

Run File:
Status: Idle

Temperature: 250.0 °C
TCD Reading: 0.2000

Auxiliary status display

In the Auxiliary Status Display on the instrument schematic, a zero or a one is displayed to show the state of each digital output (DO), relay output (RO), and digital input (DI). Refer to **Digital** on page **E-4** for additional information.

The voltage is displayed for each analog input (AUX 1 and AUX 2).

Analog

The 15-pin analog connector provides two output signals and two input signals.

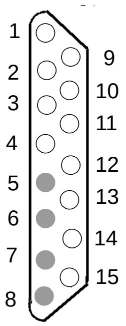
Outputs

The two output signals are:

- The TCD signal, which could be as high as 12 volts (positive or negative)
- The sample temperature signal, which is a buffered voltage from the sample thermocouple. The voltage ranges from –0.365 V at –40 °C, 0 at 0 °C, to 10.88 V at 110 °C. The signal follows the type-K thermocouple nonlinear characteristic.

The signals are available at these pins:

TCD (+)	7
TCD (–)	5
Temp (+)	8
Temp (–)	6

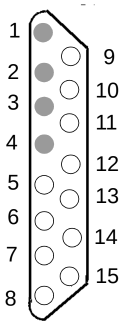


Inputs

The inputs are available for connection to other detectors, such as a mass spectrometer. The voltage range is –15 to 15 volts. The software can record these signals at the same time as it records TCD and sample thermocouple data.

The signals connect to these pins:

Auxiliary input 1 (+)	4
Auxiliary input 1 (–)	3
Auxiliary input 2 (+)	2
Auxiliary input 2 (–)	1



All other pins in the 15-pin connector are floating (not connected). You should connect the metal shell of your connector to chassis ground and the cable shield to maintain shielding of the signals.

Digital

The 25-pin digital connector provides floating signals to control and monitor external hardware, such as auxiliary detectors or chart recorders.

The outputs consist of four sets of latching relay contacts and four opto-coupled photo transistors. The inputs are two optically isolated photo couplers.

The software sets these as logic 1 or logic 0, as defined by the analysis step. They are displayed on the instrument schematic.

A logic 1 causes the relay to connect the “common” contact to the “normally open” contact. The relay will retain this condition even when power to the instrument is disconnected. A logic 0 causes the relay to connect the “common” contact to the “normally closed” contact.

A logic 1 to the opto couple outputs causes the NPN photo transistor to be turned on. A logic 0 turns the NPN photo transistor off.

The two opto-coupled inputs are read by the instrument as logic 1 (when current flows through the opto coupler photo diodes) or logic 0 (when no current is flowing).

Electrical Specifications

Relays

Maximum voltage: 250 VAC, 48 VDC

Maximum switching current: 4A at 250 VAC; 3A at 30 VDC

Maximum switching current: 100 mA at 100 VDC

Opto-coupled outputs

Maximum voltage, V_{CE} = 70 VDC

Maximum reverse voltage, V_{CE} = 7 VDC

Maximum current = 10 mA

Opto-coupled inputs

Maximum reverse voltage = 3 v

Nominal forward voltage = 1.3 v at 10 mA

Minimum current required to be recognized by the 2920 analyzer = 1 mA

Digital Connector Pin Designations

Pin	Type	Function
1	Relay 1	normally closed
2	Relay 1	common
3	Relay 1	normally open
4	Relay 2	normally closed
5	Relay 2	common
6	Relay 2	normally open
7	Relay 3	normally closed
8	Relay 3	common
9	Relay 3	normally open
10	Relay 4	normally closed
11	Relay 4	common
12	Relay 4	normally open
13	Not used	Not used
14	Opto output 1	collector
15	Opto output 1	emitter
16	Opto output 2	collector
17	Opto output 2	emitter
18	Opto output 3	collector
19	Opto output 3	emitter
20	Opto output 4	collector
21	Opto output 4	emitter
22	Opto input 1	anode (+)
23	Opto input 1	cathode (-)
24	Opto input 2	anode (+)
25	Opto input 2	cathode (-)

F. CALIBRATING THE MASS FLOW CONTROLLERS FOR EACH GAS

The AutoChem II 2920 Analyzer uses Mass Flow Controllers (MFCs) to control the flow of gases. These MFCs require a conversion constant for each gas or gas mixture, to compensate for variations in gas flows resulting from variations in the gases' properties. Default MFC conversion constants are provided for a wide variety of gases is contained in the software. You can view this table by selecting **Options > Gas Defaults**. A more complete list of gases and their conversion constants can be found on page **C-3** in Appendix C.

In most cases, using the default MFC conversion constant yields accurate data. You may wish to determine a new conversion constant if:

- You are using a unique gas mixture
- The gas you wish to use is not included in the table
- You require a higher precision calibration of the MFCs for a given gas
- Unexpected analysis data lead you to believe that the default value is in error

Gas Mixtures

To determine the conversion constant for a gas mixture, you must first obtain the conversion constant for each gas in the mixture. For each gas, you may either use the default conversion constant or determine a new constant using the method described in this appendix. A combination of default and calculated constants may be used.

After a constant is obtained for each gas in the mixture, use the formula on the worksheet at the end of this appendix to determine the constant for the mixture.

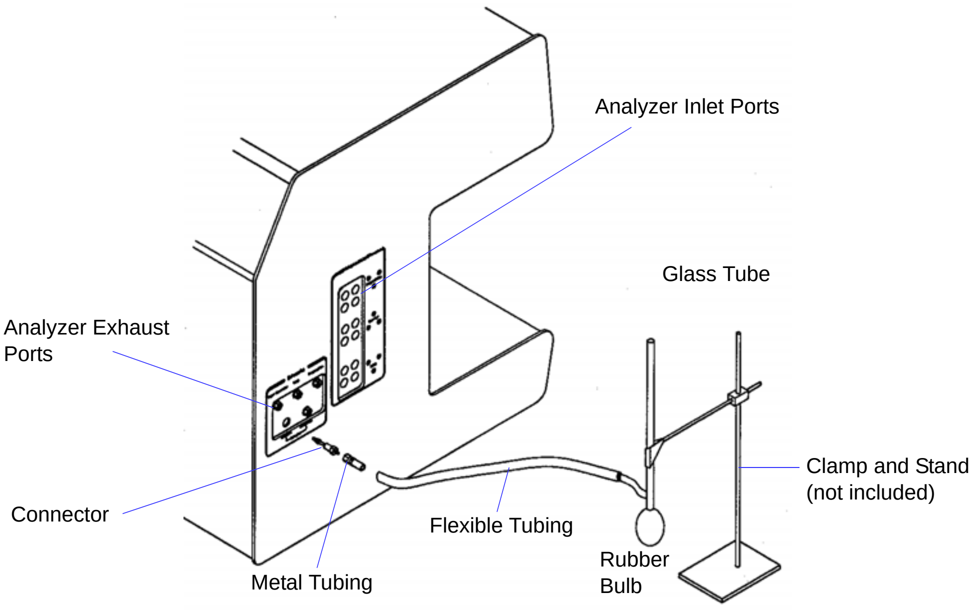
Flow Measurement

An external device that measures the flow of gas from the analyzer's exhaust is used during MFC calibration. A soap bubble burette is shipped with your instrument. For even more precise calibration, you may choose to use another type of flow meter; follow these instructions for obtaining a new conversion constant, substituting your flow meter for the bubble burette.

Installing the Soap Bubble Burette

These instructions are for assembling and using the burette supplied by Micromeritics. If you are using another type of flow meter, follow the meter manufacturer's assembly and operation instructions.

1. Carefully unwrap the glass tube and the rubber bulb.
2. Attach one end of the flexible tubing to the side arm of the glass tube. Attach the metal tubing provided with the bubble burette to the other end of the flexible tubing.



3. Locate the exhaust port that corresponds to the MFC you are using for this calibration. Remove external plumbing from that port and attach the bubble burette using the connector provided with the burette. For example, if you are using the Carrier Flow Controller, attach the bubble burette to the carrier gas exhaust port.
4. Fill the rubber bulb with leak detector fluid. An 8-oz. bottle of the fluid is included in the accessory kit. Attach the bulb to the bottom of the glass tube.

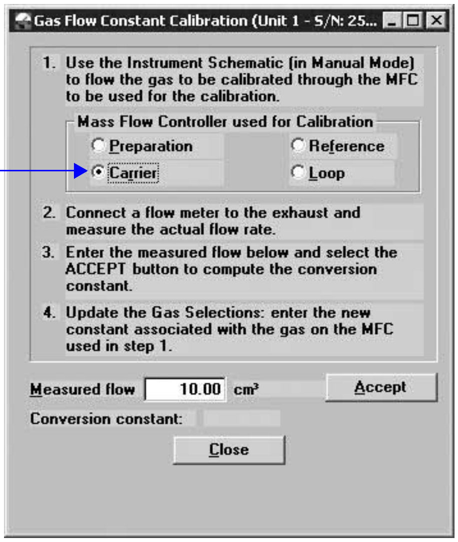
The soap bubble burette must be held in an upright position. An example of a stand and clamp is shown in the illustration above.

Determining the Constant for Individual Gases

1. Make a copy of the Gas Flow Constant Calibration Worksheet (at the end of this appendix). Use the worksheet to complete the following procedure.

2. If the gas for which you wish to determine a calibration factor is not attached to an inlet port, attach it before proceeding. Use an inlet port for the MFC you plan to use during this calibration. For example, if you plan to use the carrier MFC, attach the gas to one of the carrier inlet ports.

3. From the Unit menu, select Gas flow constant calibration. On the dialog box, mark a radio button to indicate which MFC you plan to use. The MFCs are identical, so you should use the most convenient one.



1. Use the Instrument Schematic (in Manual Mode) to flow the gas to be calibrated through the MFC to be used for the calibration.

2. Connect a flow meter to the exhaust and measure the actual flow rate.

3. Enter the measured flow below and select the ACCEPT button to compute the conversion constant.

4. Update the Gas Selections: enter the new constant associated with the gas on the MFC used in step 1.

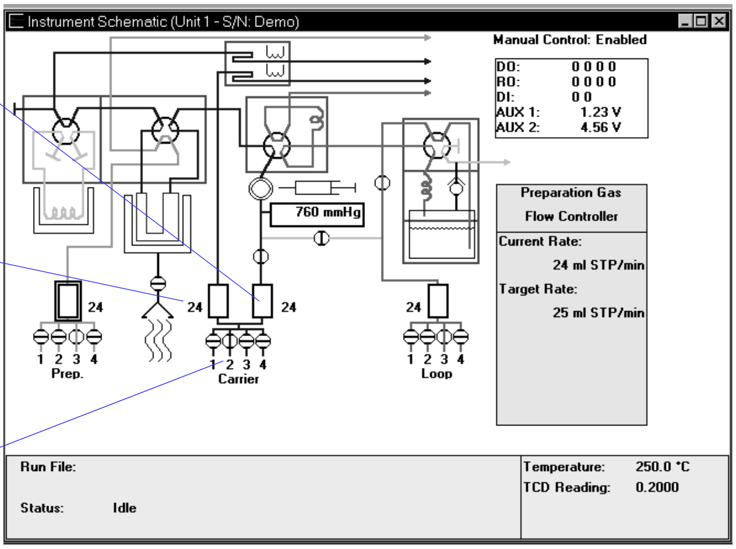
Measured flow 10.00 cm³

Conversion constant:

In this example, the carrier gas MFC is used.

4. Flow the gas to be calibrated through the appropriate Mass Flow Controller by following these steps:

   a. Select **Unit > Show instrument schematic**. Then, select **Unit > Enable manual control**. (The manual control function is explained on page **6-12**.)



Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Current Rate: 24 ml STP/min
Target Rate: 25 ml STP/min

Run File:
Status: Idle

Temperature: 250.0 °C
TCD Reading: 0.2000

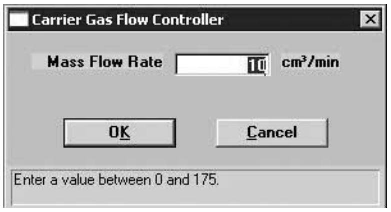
In this example, the carrier gas MFC will be used to obtain a calibration factor for the new gas.

The current flow rate is displayed beside the MFC icon.

In this example, the new gas is connected to carrier gas inlet port 2. Inlet valve 2 is opened to create a gas flow through the carrier MFC.

b. On the instrument schematic, place the cursor over the icon that represents the inlet valves for the Flow Controller you are using. Click the right mouse button and select the Flow Gas (number) option for the inlet valve to which the new gas is connected.

c. Place the cursor over the icon representing the Mass Flow Controller you wish to use. Click the right mouse button, then select **Set Flow Rate**. A window opens allowing you to specify a flow rate.



Mass Flow Rate 10 cm³/min

Enter a value between 0 and 175.

Be sure to use a rate that can be verified by the flow meter you plan to use. Record the flow rate on line 1 of the worksheet. Click **OK** to close the dialog.

5. Make sure a flow meter is connected to the appropriate exhaust port. Measure the actual flow rate in standard cm^3/min and record the flow rate on line 2 of the worksheet.

   If you are using the bubble burette shipped with your analyzer, observe a bubble as it rises through the glass tube. The lines on the glass tube indicate the beginning and ending points for measuring the progress of a bubble through the tube. Use a stopwatch to measure the amount of time that elapses from the moment the bubble passes the lower mark on the tube until the moment it passes the higher mark on the tube.

6. Convert the measured flow to Standard Temperature and Pressure (STP) conditions. The formula is provided on the worksheet; record the value on line 3.

7. Enter the measured flow rate at STP on the Gas Flow Constant Calibration dialog. Click **Accept** to calculate a conversion constant for this gas. Record the value on line 5.

8. Open the Gas Defaults table (on the Options Menu) and update it to include the new gas and its conversion constant. (If you have used this process to obtain a new constant for an existing gas in the table, update the conversion constant for that gas.)

The gas you just added can then be selected for any port on the Gas Selections Dialog. If you plan to leave this gas connected to the port, be sure to change the gas selection for that port to the correct name.

Gas Flow Constant Calibration Worksheet

Follow the instructions for calibrating the analyzer’s Mass Flow Controllers for a given gas, using this form to record measured values and perform calculations included in this process. Instructions are provided for single (individual) gases and gas mixtures.

Single Gases

MFC being used (optional):

____ Preparation

____ Reference

____ Carrier

____ Analysis (Loop)

Gas:________________________________

1. MFC flow rate (as set on instrument schematic): __________
2. Measured flow rate (using an external flow meter): __________
3. Convert the flow rate to Standard Temperature and Pressure (STP) using the following formula:

   $$\text{Rate at STP} = \text{Rate measured (cm}^3\text{) x } \frac{273.15\ K}{273.15\ K + room\ temp\ °C} \times \frac{atmospheric\ pressure}{760\ mmHg}$$

   Measured rate at STP: __________
4. Enter the value from # 3 in the Measured Flow field of the Gas Flow Constant Calibration dialog, then click **Accept**.
5. The new MFC Conversion Constant (displayed at the bottom of the Gas Flow Constant Calibration dialog) is: __________
6. Enter the new Conversion Constant for this gas (# 5) on the Gas Defaults table, which is accessed from the Options menu.

Gas Mixtures

Obtain a conversion constant for each gas in the mixture. Use the conversion constant provided in the **Gas Defaults** table (on the Options menu) or in Table C-3 on page **C-3**, or use the instructions above to obtain a new constant. Record the names and constants for each gas in the mixture in the table provided in this section. Use the following formula to calculate the conversion constant for the gas mixture.

$$M = \frac{1}{\left[\frac{P_1}{F_1 \times 100}\right] + \left[\frac{P_2}{F_2 \times 100}\right] + \ldots + \left[\frac{P_n}{F_n \times 100}\right]}$$

M = the mixture conversion constant
P = the percentage of gas n in the mixture, expressed as a whole number (example: for 15%, use 15, not .15)
F = the conversion constant (factor) for gas n (From the Gas Defaults Table or from step 5 on this worksheet)

Enter the gas mixture in the Gas Defaults table; use M as the conversion constant.

Gas Name (n)	Conversion Constant (F)
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	
11.	
12.	
13.	
14.	
15.	
16.	
17.	
18.	
19.	
20.	

Mixture name:______________________ Constant (M):_____________________

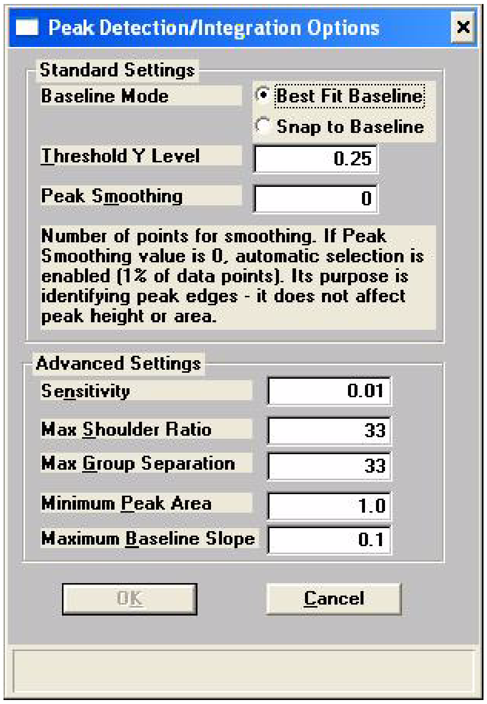
G. ATOMIC WEIGHTS AND CROSS-SECTIONAL AREAS FOR SELECTED METALS

Metal	Symbol	Atomic Weight (g/mole)	Cross-Sectional Area (sq nm)
chromium	Cr	51.996	0.0635
cobalt	Co	58.933	0.0662
copper	Cu	63.546	0.0680
gold	Au	196.967	0.08696
hafnium	Hf	178.490	0.0862
iridium	Ir	192.220	0.0769
iron	Fe	55.847	0.0613
manganese	Mn	54.938	0.0714
molybdenum	Mo	95.940	0.0730
nickel	Ni	58.710	0.0649
niobium	Nb	92.906	0.0806
osmium	Os	190.220	0.0629
palladium	Pd	106.400	0.0787
platinum	Pt	195.090	0.0800
rhenium	Re	186.207	0.0649
rhodium	Rh	102.906	0.0752
ruthenium	Ru	101.070	0.0613
silver	Ag	107.868	0.0869
tantalum	Ta	180.947	0.0800
thorium	Th	232.038	0.1350
titanium	Ti	47.900	0.0741
tungsten	W	183.850	0.0741
vanadium	V	50.942	0.0680
zirconium	Zr	91.220	0.0877

H. PEAK DETECTION/INTEGRATION OPTIONS

Peak detection parameters can be customized using the Peak Detection/Integration Options dialog. If you customize this dialog when you create the sample file, you can control peak detection during analysis. You can also control peak detection after analysis by customizing this dialog in the Peak Editor. The defaults for the parameters on this dialog are usually acceptable, but they may be changed if you wish.

- **To open this dialog while you are creating the sample file**:
  With the sample file open in Advanced presentation, click on the Analysis Conditions tab. Click **Insert**, then select **Experiment**. Click **Peaks**.
- **To open the dialog while you are in Peak Editor**:
  Select the **Edit Peaks** tab. Then right-click in the graph area and choose **Peak Integration Options**.


Peak Detection/Integration Options

Standard Settings

Baseline Mode: ● Best Fit Baseline ○ Snap to Baseline

Threshold Y Level: 0.25

Peak Smoothing: 0

Number of points for smoothing. If Peak Smoothing value is 0, automatic selection is enabled (1% of data points). Its purpose is identifying peak edges - it does not affect peak height or area.

Advanced Settings

Sensitivity: 0.01

Max Shoulder Ratio: 33

Max Group Separation: 33

Minimum Peak Area: 1.0

Maximum Baseline Slope: 0.1

The TCD detects and records all deviations from baseline, but only those which satisfy the criteria established in this dialog are reported as peaks.

Peak detection is accomplished through a combination of noise, height, and area thresholds.

Baseline Mode

This feature affects how the Find All Peaks function works.

If **Best Fit Baseline** is selected, the bottom of the peaks is placed in the baseline that best describes the signal outside the range of the peaks. This assumes a linear baseline between the beginning and the end of the peak.

If **Snap to Baseline** is selected, the bottom of the peaks is moved to the signal recorded, not the best fit baseline between the peaks.

Threshold Y Level

Sets the minimum height for peaks to be identified and included in the peak table. This value is expressed in terms of the trace’s Y-axis units. Use a value of 0 (zero) to include all peaks.

Smoothing

This feature allows the software to average the points before using them, so that noise spikes are ignored. Specify the number of points you wish the software to average into a single value during the peak picking process.

The smoothing parameter can be turned off by setting the value at **1** or **0** (zero). A setting of **1** disables smoothing, and the peak edges are interpolated to the best X-axis value. A setting of **0** also disables smoothing, but the peak edges (the points where the peak begins and ends) are not interpolated. Instead, the nearest data point is used as the peak edge.

Sensitivity

Sets the noise rejection level for identifying the peaks in a trace. Use a value from -100 to 100%. For example, a sensitivity level of 5% means that 5% of all local maxima in the trace are greater than the noise and are, therefore to be considered as peaks. A value of 100% identifies all local maxima (or minima for traces with transmission Y units) as peaks. One possible exception to note is the combination of other rejection parameters (such as Minimum Peak Area and Threshold Y Level) which can reject peaks even when the sensitivity is set to 100%.

The sensitivity can also be set to negative values to define a specific noise level (in Y units) for peak rejection. For example, a sensitivity setting of -2.5% sets the noise rejection to 2.5 V. This means that maxima with an amplitude of 2.5 V or less will be considered as baseline noise instead of as peaks. (As opposed to the Threshold Y Level rejection parameter which eliminates refined peaks by using their height above the baseline.)

A setting of **0** (zero) automatically sets a default noise level for the trace.

Max Shoulder Ratio

There can also be “shoulder peaks” (also called combination peaks) within a group. (For a discussion of peak groups, see the description of Max Group Separation below.) Shoulders are usually small peaks that are overlapped on the front or the tail of a larger peak. These peaks can also be called “leaders” and “followers,” respectively. As with baseline groups, the areas of these peaks can be calculated incorrectly. If the larger “parent” peak has a long tail with a much smaller peak riding on it, most of the area under the trace belongs to the parent peak. However, if the area of these peaks was determined using baseline grouping, the smaller peak would be calculated by using vertical drop lines at the edges. This would give the parent peak too little area, and the rider peak too much.

The software can detect these shoulder peaks. The areas of shoulder peaks are calculated by drawing a skimmed baseline from the leading edge to the trailing edge. Either an exponential or a straight skim line can be used. The skim type is specified by a secondary method parameter (see the Grams/32 manual Appendix or the Method Editor) and the default is exponential skimming. The remaining area between the shoulder peak baseline and the group baseline is considered to be part of the parent peak.

The **Max Shoulder Ratio** parameter is used to specify whether the peaks that are overlapped in the front or the tail of much larger peaks should be identified as shoulder peaks. To use shoulder peak detection, use a non-zero value for the **Max Shoulder Ratio** parameter. After a baseline group has been identified, the software looks for peaks within the group that satisfy the following shoulder peak criteria:

Shoulders must have a significantly higher Y value at one edge than the other. More importantly, the height of the shoulder above the common value must be much smaller than the height of the “parent” (larger) peak above the same valley. It must be smaller by the **Max Shoulder Ratio** setting. For example, a setting of 33 implies that shoulders must be smaller than 33% of their parents in terms of height above the common valley. The areas for shoulder peaks are calculated by drawing a skimmed baseline from the left edge to the right edge of the peak. The remaining area between the shoulder peak baseline and the group common baseline is considered to be part of the parent peak.

Shoulder peaks can only be calculated within a group of peaks. (See the description of peak groups in the section below on **Max Group Separation**.) If the **Max Group Separation** parameter is set to **0** (no groups), a **Max Shoulder Ratio** parameter value is not used. Use a value of zero to specify no shoulder peak detection.

A value of 33% works well with most data. Use a setting of zero to treat shoulder peaks with a perpendicular drop to the common group baseline instead of a skimmed baseline.

If the software detects unwanted baseline noise peaks, try increasing the Sensitivity setting. Conversely, if some peaks are not detected, decrease the value.

Max Group Separation

The software normally calculates peak areas by drawing a “valley-to-valley” baseline from the leading edge to the trailing edge of every identified peak. However, in many traces, the valleys between peaks do not always drop back to the original baseline. If a valley-to-valley baseline is used for this type of peak, the calculated area does not accurately reflect the true area under the peak.

The software provides a parameter that allows the calculation of “Baseline Groups.” A group of peaks is defined by a common baseline that extends from the leading edge of the first peak in the group to the trailing edge of the last. The areas of grouped peaks are calculated by dropping vertical lines from the peak edges down to the group baseline. (Note there may also be “Report Groups” defined by the method and assigned group letters from A through Z. Unlike these Baseline Groups, the Report Groups need not be next to one another. The two types of groups are not related.)

The Max Group Separation parameter is used to determine which peaks in a trace have a common baseline. When using peak grouping, the software compares the width (actually double the largest half width) of every identified peak in a trace to the width of the following peak. The Max Group Separation parameter specifies a percentage of the smallest of these two widths in X units. If the edges of two adjacent peaks differ by less than this value, the two peaks constitute a group and are given a common baseline. For example, if two adjacent peaks in the trace have largest half widths of 1 and 1.5 respectively, and the Max Group Separation parameter is set at 20%, then a difference of less than 0.4 X units between the adjacent edges of these peaks would make them a group with a common baseline. If the same two peaks have adjacent edges that are greater than 0.4 X units apart, they do not define a group, and each peak has its own separate baseline.

The areas for grouped peaks are calculated by drawing imaginary vertical lines from the peak edges to the common baseline. Any peaks that share common edges are automatically considered a group and are given a common baseline for any Max Group Separation setting greater than 0. To specify no peak grouping (each identified peak has its own baseline), use a setting of **0**. A value of 33% for this parameter works well with most data. Use a setting of zero to force all baselines to be drawn from peak valley to valley.

Minimum Peak Area

This parameter sets the minimum area required for refined, processed peaks to be recognized, identified, and included in the peak table. Any peak with a calculated peak area smaller than the current setting is not detected. Values for this parameter are expressed in terms of the trace X-axis units multiplied by the Y-axis units (e.g., millivolt-minutes).

Setting the Sensitivity parameter to large values (greater than 20%) can cause noise “spikes” (or “dips”) on the sides of major peaks to be identified as peaks themselves. If the areas of the peaks are smaller than the Minimum Peak Area parameter, major peaks in the trace may not be identified at all. Exercise caution when using high Sensitivity settings with the Minimum Peak Area parameter.

If peaks are not detected by the software, lower the setting with a smaller value. You must adjust this parameter in conjunction with the Peak Sensitivity and/or Threshold Y parameters above. Peak rejection is accomplished through a combination of peak height and peak area rejection parameters. If the software is not detecting your peak(s) of interest, you probably need to decrease both parameters.

I. ERROR MESSAGES

Error messages are listed numerically. If the **Action** response instructs you to contact your service representative, record the error message and make backup copies of any files involved in the operation.

The 1000-series error messages (used primarily for software testing) are not included in this appendix. These errors should not occur during normal operation. If you receive a 1000 series error message or an error message not listed in this appendix: record the error message, make backup copies of any files involved, then contact your service representative.

2200 Series

2201- Cannot execute report subsystem.

2202- Cannot execute Peak Editor subsystem.

Cause: The software for the report (or Peak Editor) subsystem has been corrupted and cannot be started.

Action: Reinstall the AutoChem software.

2400 Series

2401- FATAL ERROR: (error message)

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your Micromeritics service representative if you continue to receive this message.

2430- Error accessing file (file name), error code = [n]

Cause A: Hard disk may be damaged.

Action A: Contact your service representative.

Cause B: A software error occurred when the file was accessed.

Action B: Contact your service representative.

Cause C: The name specified contains one or more invalid characters.

Action C: Enter a valid file name; do not use special characters such as * or ?.

2431- Error writing file (file name), error code = [n].

Cause: The hard disk does not have enough space left to perform the operation.

Action: Copy files not used regularly from the hard disk to a CD or network drive, delete them from the hard disk, and then try the operation again.

2432- Invalid response from MMI ‘File_READ’ request.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error message.

2433- New entries have been found in this directory. Refresh the directory information?

Cause: Several files (sample information, analysis conditions, or report options) have been added to this directory by some function other than the analysis program.

Action: Select **Yes** to update the directory information with data form each new file. This operation may take a minute.

Select **No** if you do not want to spend the time updating the directory information. This option may be feasible if a large number of files have been copied into the directory and you know the name of the file you wish to access.

2434- File (file name) - Subset # [n] wrote wrong amount of data.

Cause:	An internal processing and/or hardware error has occurred.
Action:	Contact your service representative if you continue to receive this error message.

2436- Path specification (path name) is invalid.

Cause:	You entered an invalid path name and/or extension.
Action:	Type a valid path name (including the proper extension) and press **Enter**.

2437- File (file name) does not exist.

Cause:	You entered a file specification that does not exist.
Action:	Enter an existing file specification or select a file name from the list box.

2438- Disk drive (letter): is inaccessible.

2439- Could not register file.

2440- Subset not found.

2441- Seek within file failed.

2442- Had header in subset file.

2443- Subset owner denied access.

2444- Not a valid file format.

2445- Subset wrote the wrong amount of data.

2446- Error reading data.

2447- Error writing data.

Cause: An unexpected error occurred when you tried to access a data file.

Action: Contact your service representative.

2448- File directory (path name) is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.

Action: The installation directory will be substituted. The next time you open a file, use the Directories list to move to the correct directory.

2449- This field does not contain a valid file specification.

Cause: You entered an invalid file name.

Action: See the description of file naming conventions in your operating system or Windows manual and reenter the name.

2450- Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause: You are in the process of initiating an automatic analysis (an analysis in which sample files are created using the defaults) while editing the defaults.

Action: Finish your edit session of the defaults and close the dialog. Then restart the automatic analysis.

2452- Attempt to write MICATTR.DIR in read only mode. (file name)

2453- Attempt to append MICATTR.DIR in read only mode. (file name)

Cause: The Read-Only attribute is turned on in the application’s MICATTR.DIR file (this file exists in each folder containing sample or parameter files).

Action: Use Windows Explorer to access the folder containing the MICATTR.DIR file and disable the Read-Only option.

2454- Too many selections for a print-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

2455- Too many selections for an export-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause:	You selected too many files for this operation.
Action:	Select only the number of files specified in the message.

2456- Insufficient file handles available. Application cannot continue.

Cause:	You have more than 50 files open at the same time.
Action:	Refer to the manual for your operating system and set the limit for open files to 50 or greater.

2457- Results cannot be displayed. More than (number) windows are currently displaying or printing results.

Cause:	You have too many windows open in the application.
Action:	Close some of the open windows.

2458- An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause:	You are attempting to exit the application while the instrument is performing a critical operation. This operation must be completed before the application can be stopped.
Action:	Wait a short time and attempt to stop the application again.

2459- An instrument is busy. A delay in restarting this application could result in loss of data. Continue with program Exit? (Yes, No)

Cause:	You are attempting to exit the application while an analysis is in progress. While this is possible, the data collected while the application is inactive will not be permanently recorded until the application is restarted. A power failure to the instrument could cause some data to be lost. Continued data collection may also exhaust the memory of the controller.
Action:	If you are not concerned with the potential for loss of data should a power failure occur, select **Yes** to continue; otherwise select **No**.

2460- Fatal Communications error on (Unit n - S/N: nnnn)

Cause: There was a fatal error in the serial communications between the application and the interface controller (the software and the instrument). All displays for that instrument will be closed.

Action: Check that the interface controller is connected to the control module on the communications port configured in the Setup program. Stop the 2920 software and then restart it. If this error persists, contact your Micromeritics Service Representative.

2461-No active instruments. The application will unconditionally terminate.

Cause: At least one instrument must be active for the application to operate. The initialization of all of the instruments configured with the Setup program has failed. The application stops.

Action A: Usually this message is preceded by another message giving the reason for the instrument’s failure to initialize. See the instructions for that message.

Action B: Ensure that the instrument is attached to the computer on the communications port configured with the Setup program. Verify that the instrument has the power switch in the ON position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the instrument, contact your Micromeritics Service Representative.

2471- Unit n - S/N: nnnn has an invalid communications port specified. It cannot be initialized.

Cause: The communications port associated with this unit was not valid.

Action: Run the Setup program and install the unit on a valid port.

2474- Unit n - S/N: nnnn communications port specified in the program control files is already in use. Unit cannot initialize.

Cause:	The communications port associated with this instrument is in use by some other program in the system.
Action:	Close the other program to release the port. Restart the 2920 software.

2475- Unit n - S/N: nnnn communications port specified in the program control files cannot be accessed. Unit cannot initialize.

2476- Unit n - S/N: nnnn startup failed. Cannot initialize the communications port.

Cause:	The communications port associated with this unit was not valid.
Action:	Run the Setup program, specifying a valid port for this unit, when you are prompted to do so.

2477- Unit n - S/N: nnnn did not properly initialize.

Cause:	The software was unable to initialize this instrument; this is usually caused by one of the conditions listed in the error messages above.
Action:	Correct the problem as described above, then restart the application.

2478- Error copying sequential data segment.

Cause: An internal processing and/or hardware error occurred while accessing a portion of a sample file.

Action: Confirm that the media being accessed does not contain errors; for example, you may wish to use a utility such as ScanDisk.

Contact your service representative if you continue to receive this message.

2479- Unit n - S/N: nnnn The instrument is busy performing an operation of which this application is unaware. Do you want to cancel?

Cause: During initialization, the instrument was found to be analyzing, but the analysis program has no record of it.

Action: Click **Yes** to cancel the analysis and synchronize the analysis program with the instrument's status. Click **No** to abort the initialization and allow the analyzer to continue the analysis.

2480- File <name> cannot be analyzed. It is currently being edited.

Cause: You are trying to start an analysis using a file that is open for editing.

Action: Finish editing the file, save and close it, then start the analysis.

2481- Error accessing the sample information file <name>.

Cause: An unexplained error prevents you from accessing this file.

Action: The hard disk drive may be corrupt. Run diagnositics.

2482- File cannot be opened for writing. It is already in use.

Cause: You attempted to open a file that is currently being used (either by this application or another).

Action: Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all windows, one of which may contain this file).

2483- An analysis cannot be performed on (file name). It is open for editing and contains errors.

Cause: You are trying to start an analysis using a file that contains errors and is open for editing.

Action: Finish editing this file, save and close it, then start the analysis.

2484- The edit session for <name> must be saved before the analysis. Save changes and proceed with the analysis? (yes, no)

Cause: You are trying to start an analysis using a file that contains unsaved changes and is open for editing.

Action: Select **Yes** to save the changes and proceed with the analysis; select **No** to cancel the analysis and continue editing the Sample Information file.

2485- The service test file has an invalid status and cannot be used for this analysis.

Cause: The selected service test file has a status other than *No Analysis*.

Action: Select a different service test file or create a new one and use **Replace All** to copy parameters from the file you originally selected.

2486- Could not construct (name) report type. Program will terminate.

2487- Could not start report generator. Error code (number). Program will terminate.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this message.

2488- File (file name) cannot be opened for editing. It is already in use.

Cause: The file you specified is already open for editing.

Action: Check the windows list to locate the other edit session.

2489- File (file name) cannot be opened for writing. It is already in use.

Cause: The file you specified in a **Save As** operation is already open for edit.

Action: Select a different file for the **Save As** operation.

2490- No ‘.INI’ file present. Application will terminate.

Cause: The ASCII (.INI) file containing initialization and system options information cannot be found. The .INI file may have become corrupted. The ASAP 2920 cannot be run without this file.

Action: Uninstall, then reinstall the ASAP 2920 application as described below. When you uninstall the application, only the application files are deleted; the 2920 data files remain intact.

a. From the Windows Start menu, select **Settings** > **Control Panel** > **Add or Remove Programs**.

b. Select **AutoChem II 2920**, then click **Remove**.

c. Insert the 2920 Setup CD in the CD drive and reinstall the 2920 application (refer to **“Installing the Software” on page 2-18**).

d. Start the 2920 application.

2491- Highlighted fields contain errors. Please correct the errors before closing.

Cause: The highlighted fields contain invalid entries. You will not be able to close the dialog box until you correct the errors.

Action: Check the entries, correct the errors, and close the dialog box.

2492- This field’s entry is invalid.

Cause: The highlighted field contains an invalid entry.

Action: Check the entry and correct the error.

2493- An entry is required for this field.

Cause: This field contains an invalid entry.

Action: Enter or select an appropriate value.

2494- Value is out of the valid range.

Cause: The value you entered in the highlighted field is outside the valid range of values.

Action: Check the entry and enter or select a value within the indicated range. The valid range is shown in the information bar across the bottom of the dialog.

2495- Value is out of the valid range. Enter a value between (value) and (value).

Cause: The value you entered in the highlighted field is outside the valid range of values.

Action: Check the entry and enter a value between the range indicated.

2496- Invalid number.

Cause: The number you entered in the highlighted field is invalid.

Action: Check the entry and enter or select a valid number.

2497- This field contains an invalid character.

Cause: You entered an invalid character in the highlighted field.

Action: Check the entry and enter valid characters.

2498- The requested change to the Sample's status is invalid at this time.

Cause:	A request to change the file's status (for example, from automatically collected to manually entered data) could not be done.
Action:	Contact your service representative if you continue to receive this message. Record the name of the sample file in which the problem occurred.

2499- Sequence number must contain at least 3 digits.

Cause:	You tried to enter a sequence number that did not contain at least three digits.
Action:	Enter a sequence number that contains at least three digits.

2500 Series

2500- All sample files that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause:	No more sample information files can be created using the currently specified file sequence number.
Action:	Select **Options > Sample defaults** from the main menu and enter a new sequence number.

2501- System resources have reached a dangerously low level. Please close some windows to avoid loss of data.

Cause:	A large number of windows is open and consuming the system resources available to all applications.
Action:	Close any unneeded windows to free system resources. Contact your service representative if you continue to receive this error message.

2502- Error writing to file (name) during print. Error code: (number).

Cause:	An error occurred in the file being written to during a print operation.
Action:	Ensure that there is sufficient space on the drive containing the file.

2503- Error converting file (name). Could not create DIO intermediate file.

Cause A:	Insufficient space is available on the hard disk. The DIO file is placed in the directory specified by the TEMP environment variable.
Action A:	Determine if there is sufficient space on the drive where the TEMP directory is located.
Cause B:	An internal processing and/or hardware error has occurred.
Action B:	Contact your service representative.

2504- Cannot create output file for sample (name).

Cause: Insufficient space may be available on the hard disk.

Action: Ensure that sufficient space is available. Contact your service representative if you continue to receive this message.

2505- Error Logger cannot be initialized. Error code (number). Program will exit.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative.

2506- (sample file) Output device (name) is not installed. Printing cannot be accomplished.

Cause: The selected output device is not installed.

Action: Select a different output device in the System Configuration dialog. Install the device using the Control Panel, Printers operation.

2508- (sample file) Overlay file (name) was not found. It will not be included in the reports.

Cause: The specified overlay file could not be found.

Action: Ensure that the file specified as an overlay does exist.

2509- (sample file) Error opening file (name): (error). Reports cannot be produced.

Cause: An error occurred while the program was opening a file necessary to the report operation.

Action: Use the name given in the error message to investigate. Contact your service representative if you continue to receive this error message.

2510- (sample file) Error parsing reports from file (name). Reports cannot be produced.

Cause A:	One or more data entry fields in the sample file may contain an invalid character (such as a single quote or double quotes).
Action A:	Review the data entry fields (for example, the Sample field) and remove the invalid character.
Cause B:	The system was unable to create the usual temporary files during the report, possibly due to insufficient disk space.
Action B:	Check the space available on the hard disk.
Cause C:	An internal processing error occurred.
Action C:	Contact your service representative.

2511- Print job (name) has been cancelled due to insufficient disk space. Delete unnecessary files and restart the report.

Cause:	The disk drive does not have enough space for the temporary file required by the Windows Print Manager. Therefore, printing of the requested report has been canceled.
Action:	Delete unnecessary files form the disk. You will require at least five megabytes of free space for normal operation.

2512- Print job (name) has been canceled.

Cause:	The requested print job was canceled at your request.
Action:	None required.

2513- Unable to read the calibration file (file name).

Cause:	The calibration file selected for loading is not valid or cannot be read.
Action:	Confirm the media containing the alleged calibration file has no problems.

2514- Unable to write the calibration file (file name).

Cause: An attempt to Save calibration data has failed due to possible media problems.

Action A: Confirm the media containing the alleged calibration file has no problems.

Action B: Choose an alternate media to Save the calibration data.

2515- Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

Cause: You are in the process of performing a calibration.

Action: Calibration operations should only be done under the direction of qualified service personnel.

2516- Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?

Cause: You have completed a calibration procedure.

Action: Save the calibration data to a file.

2517- Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?

Cause: You have completed a calibration procedure, but you have not accepted the new calibration data.

Action: If the calibration operation was successful, click **Accept**.

2521- Unable to program controller.

Cause: A hardware malfunction has occurred.

Action: Contact your local service representative.

2522- Invalid controller application file.

Cause:	The application's control file has been corrupted or deleted.
Action:	Reinstall the AutoChem program.

2523- Programming the controller failed.

2524- CRC check failed on programming controller.

2525- Unknown error programming controller.

2526- Controller download was not successful.

2527- Controller CRC error on boot block.

2528- Controller DRAM error.

2529- Controller Com1: error.

2530- Controller Com2: error.

2531- Controller debug port error.

Cause:	An internal processing and/or hardware error has occurred.
Action:	Contact your service representative if you continue to receive this message.

2532- The instrument contains a different software version. Do you want to reset it?

Cause:	The application has discovered that a different version of software is already running on the instrument.
Action:	If there is no chance that some instrument other than the 2920 is connected to the instrument, select **Yes** to reset the instrument and download the proper software. Otherwise, select **No** to leave the instrument unchanged.

2533- Mass initialization failed.

Cause:	The instrument initialization found that the instrument hardware configuration did not meet the minimum requirements.
Action:	Contact your service representative.

2534- Error opening file (name) for printing. Error code: (number)

Cause: An error occurred in the selected file for print output.

Action: Ensure that sufficient space is available on the drive containing the file.

4200 Series

4200- At least two experiments are needed for this report.

Cause: The report options for this report do not include the selection of at least two experiments.

Action: Open the sample file and choose at least two experiments for this report.

4202- Null connection, unable to start Transaction.

4203- Advise for unknown transaction received.

4204- Unable to create DDE conversation <number>.

4205- Unable to create DDE transaction <number>.

4206- Callback for unknown transaction received.

4207- DDE error number <number>.

Cause: A DDE operation was not properly executed.

Action: Ensure that the other application is loaded and in a state which supports DDE operations. If the problem persists, exit all applications and Windows, then restart the applications.

Cause: The DDE command was not specified correctly.

Action: Review the DDE server application manual for the specific command and enter it correctly in the DDE command step. If the problem persists, exit all applications and Windows, then restart the applications.

4208- An error occurred while loading the application control information. Data entry cannot be performed. (Code #)

Cause: An error occurred accessing the control information disk file required by this application.

Action: The disk drive may have failed or be corrupt. Run diagnostics on the disk drive.

4209- New steps cannot be inserted in the synopsis at any point that precedes the step that is currently being executed.

Cause: The analysis has already progressed past the point at which you attempted to insert a step; therefore the step would not be performed.

Action: Insert the new analysis step after the step that is currently being executed.

4210- No new steps may be inserted. Analysis is complete.

Cause: The analysis for this sample file has ended. You cannot insert a step in a sample file whose analysis is complete.

Action: The analysis conditions for a completed sample cannot be changed. You can, however, create a new sample file and use the **Replace** button to copy the values from the completed file. Then, add the desired step to the new sample file. A new sample can be analyzed using the new (edited) sample file.

4213- This analysis can only be executed when Service Test mode is enabled.

Cause: You have attempted to analyze a sample using an analysis conditions set which may only be used under the supervision of an authorized Micromeritics Service Representative. These analysis conditions are provided to assist in the diagnosis of certain instrument problems.

Action: Contact your Micromeritics Service Representative for advice on your current problem.

4214- This analysis exceeds the maximum allowed length.

Cause: The current analysis conditions script contains the maximum number of allowed steps (1000). You cannot add another step.

Action: Divide the analysis steps into two or more sample information files. Allow the sample tube to remain undisturbed on the analyzer while you repeatedly start analyses using these sample information files in sequence. When you divide the steps between sample information files, plan your divisions so that the ending conditions of one file and the initial experiment conditions of the next file facilitate the continuing analysis. The data from the various sample information files can be integrated using the Peak Editor, since multiple sample information files can be edited at once. Note that the maximum number of experiments that can be loaded into Peak Editor at one time (regardless of the number of sample files) is 16.

4219- At least one experiment must be selected and at least one box checked.

Cause: You are attempting to export the collected data from a Sample file, but not all of the required fields on this dialog box have been completed correctly.

Action: Select at least one experiment to be exported and check either **Peak summaries**, **Measurements**, or both.

4220- Unexpected I/O error accessing data for an experiment in sample <name>.

Cause: An unexpected disk error occurred while you were attempting to access experiment data.

Action: The hard disk drive may have failed or may be corrupted. Run diagnostics.

4221- Cannot append an experiment to Sample file <name>. Maximum size may be exceeded.

Cause: You selected **Save As** from the experiment editor and elected to append the loaded experiments to an existing file. Either the existing Sample file already contains the maximum number of allowed experiments or the hard disk is full.

Action: Verify that at least 20 megabytes of disk space are available on the target disk drive and that the target Sample file contains fewer than 20 experiments.

4222- The Active Metal Table Options dialog box does not specify a stoichiometry factor for element <name>. One (1.0) was substituted for this value in the calculations.

Cause: The Pulse Chemisorption report was selected, and requires a valid stoichiometry factor for calculations. The Active Metal Table does not contain a valid stoichiometry factor for the specified element.

Action: Open the sample file in Advanced presentation. Click on the Report Options tab. Select the Pulse Chemisorption report, then click the **Edit** button. Edit the Active Metal Table for this file, specifying a valid stoichiometry factor for the metal named in the error message. Save and close the file, then print the report.

4223- No element in the Active Metals Table Options has a `% of Sample Weight' value greater than zero.

Cause: The Pulse Chemisorption report was selected. None of the metals in the Active Metal Table are designated as being part of this sample.

Action: Edit the Active Metal Table in the Pulse Chemisorption report options and specify a `% of Sample Weight' greater than zero for at least one metal.

4224- The first peak is larger than the last. This report assumes inverse chemisorption.

Cause: This message appears on the Pulse Chemisorption Report when an inverse chemisorption experiment is reported.

Action: None required; informational only. If this experiment is not actually an inverse chemisorption experiment, it is not appropriate for this type of data reduction.

4225- No reports were selected.

Cause: A report was requested, but there are no reports selected in the sample information file.

Action: Open the sample information file and edit the report options. Make sure that at least one report type is selected (double-click on a report to select it; a check mark appears next to each selected report).

4226- No experiments were selected for inclusion in this report.

Cause: The report type that was selected for printing requires that you specify which experiments should be included in the report. No experiments were selected for this report.

Action: Open the sample file in advanced presentation. Click on Report Options, then on the selected report type. Click **Edit** and choose the experiments you wish to include in this report. If none of the experiments within the Sample file are applicable to this type of report, deselect the report type from the list shown on the Report Options dialog.

4227- The data requested on this report are not available.

Cause: The data requested on a report, such as the Auxiliary 1 signal, are not contained within any of the selected experiments.

Action: Open the sample file in advanced presentation. Click the Report Options tab, then deselect this report type. Or, click the report type, then click the

button. When the editing dialog for this report type opens, change the options so that they request data contained within the file.

4228- This sample does not contain an experiment which can be used in a <name> calibration.

Cause: You are attempting to load a sample which does not have valid peaks for this type of calibration into the calibration editor. A **Gas Concentration calibration** requires a TCD Calibration experiment. A **Spread Peaks calibration** requires a Vapor Calibration experiment. A **User Defined Peaks calibration** will accept any experiment type with valid peaks defined.

Action: Select a sample file that contains the appropriate experiment type.

4229- The calibration data for experiment <name> may already be loaded. You may delete duplicates after loading.

Cause: Before you can reload the peak information for a sample file into the TCD Calibration dialog, you must first delete all previously loaded peaks.

Action: Delete all peaks from the dialog, then try loading the sample file again.

4230- The destination of a Grams/32 export must be a file.

Cause: You selected a destination other than File for exporting in the Grams/32 format.

Action: Perform the export operation again and choose File as the destination. You must use a File destination to export Grams/32 data.

4231- An error occured exporting experiment <number> from the sample '<file name>'.

Cause: You were attempting to export data to the Grams/32 application when an error occurred. Either the disk file required by the export could not be created or the named experiment contains invalid data.

Action: Ensure that sufficient disk space (at least 20 MB) to contain the exported file is available. Also examine the selected experiment with the Peak Editor to ensure that it contains valid data.

4232- At least two peaks are required to determine heat of desorption.

Cause: The heat of desorption is determined by fitting data obtained at different temperature ramp rates. At least two data points must be present for the fit to be performed.

Action: Repeat the analysis with at least two experiments that have different ramp rates. Refer to Appendix J, **TUTORIALS** for a tutorial on performing a TPD analysis.

4233- Default mass spectrometer file directory (path name) is invalid. Resetting to the installation directory.

Cause:	A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.
Action:	The installation directory will be substituted. The next time you open a file, use the Directories list to move to the correct directory.

4234- File (name) is not a mass spectrometer data file.

Cause:	You selected a file that does not contain appropriate mass spectrometer data.
Action:	Choose a file that contains data collected by the Pfeiffer Vacuum (formerly Balzers) mass spectrometer using the QuadStar software.

4235- The mass spectrometer file (file name) cannot be opened for reading.

Cause:	A file-access error occurred when you attempted to access the mass spectrometer file.
Action:	Ensure that you have selected a valid file name, and that you have the required permissions to read mass spectrometer files.

4236- The calibration data file (name) was not created with this instrument.

Cause:	You attempted to load a calibration file containing data collected on an instrument different from the current one.
Action:	Choose a sample file containing calibration data that were collected on the current instrument.

4237- Peak processing cannot be performed. The mass spectrometer data cannot be imported.

Cause:	The mass spectrometer data did not include identifiable peaks.
Action:	Select a different mass spectrometer file.

4238- Peak Editor cannot be started.

Cause: You attempted to open the Peak Editor, but one of its components is missing.

Action: Reinstall the software.

4239- A calibration from instrument S/N <number> is required to calibrate experiment <name>.

Cause: In order to associate a calibration file with a Sample file, both files' analyses must have been performed on the same instrument. The instrument must be connected to the computer for its calibration files to be available.

Action: Ensure that the instrument with the designated serial number is connected to the computer and is active.

4240- Error writing calibration information. The calibration of experiment <name> could not be changed.

Cause: The calibration information for an experiment could not be changed because of an error writing to the disk file.

Action: The hard disk drive may have failed or it may be corrupt. Run diagnostics. Check that no other application has write access to the sample information file.

4241- TCD Calibration is required for the calculations of this report.

Cause: This report requires that TCD calibration be applied to the sample file.

Action: BET, Langmuir, and Total Pore Volume reports require inclusion of a TCD calibration. Use the Edit Peak view of the Peak Editor to assign a TCD calibration to the file.

4242- No valid vapors available.

Cause: You are performing a vapor calibration and the peak table contains at least one entry in which the reflux temperature is not in the temperature range of any item in the vapor defaults.

Action A: Check the vapor defaults and see if the vapor you wish to use is listed; if so, ensure that its temperature range is correct.

Action B: If the values in vapor defaults are correct, delete the peaks in the peak table which have reflux temperatures outside the range of all items in the vapor defaults table.

4243- Ambient Temperature should be > 10 ºC.

Cause: The value for the ambient temperature is out of range; it is possible that the sample file has been corrupted.

Action: Call your Micromeritics service representative.

4244- Average Syringe Area should be > 0.

Cause: There is no peak data for the Loop calibration.

Action: Using the Peak Editor, edit the peaks, inserting new peaks if possible.

4245- The peak table contains [nn] peaks. Exactly 6 peaks are required for loop calibration.

Cause: The number of peaks for the Loop calibrating is less than six; six are required.

Action: Using the Peak Editor, edit the peaks. Insert enough peaks to total six.

4375- Cannot insert peak with negative height.

Cause:	You attempted to insert a peak below the peak set’s baseline.
Action:	Insert the new peak above the peak set’s baseline and/or adjust the baseline.

4376- Auto Fit cannot be executed unless a valid peak-set is selected.

Cause A:	You attempted to auto-fit a peak set, but there is no peak set current active.
Action A:	Click on the desired peak or peak set in the peak-fit table to select a peak sest.
Cause B:	You attempted to auto-fit, but there are no peak sets in the experiment.
Action B:	Insert a new peak set into the experiment. Then choose Auto Fit to find all peaks in the set.

4377- This operation cannot be performed on a negative peak set.

Cause A:	You attempted to insert a peak in a negative peak set.
Cause B:	You attempted to move the boundaries of a negative peak set.
Cause C:	You attempted to change the shape of a peak found within a negative peak set.
Cause D:	You attempted to optimize the peaks of a negative peak set.
Cause E	You attempted to auto-fit the peaks of a negative peak set.
Action:	Invert the graph to make the peak set positive, then repeat the operation.

6200 Series

6209- File cannot be opened for writing. It is already in use.

Cause: You are attempting to access a file that is in use by some other part of the application.

Action: Close the window or application which is currently using the desired file and try again.

6219- Warning: the instrument (Unit <n> - S/N: nnn) is uncalibrated.

Cause: During initialization, the instrument determined that no calibration data exists for this instrument.

Action: Before performing any analysis or experiment, load the calibration information from the factory-supplied disk.

6221- Unit - S/N:nnn has an invalid status/calibration file and cannot be initialized.

Cause: The status/calibration file, which tracks the status of the instrument, cannot be read by the AutoChem software.

Action: Reinstall the AutoChem software to make sure that the file is installed correctly.

6222- The Vapor Generator for the instrument (Unit <n> - S/N: nnn) is uncalibrated.

Cause: During initialization, the instrument determined that no calibration data exists for this instrument.

Action: Before performing any analysis or experiment, load the calibration information from the factory-supplied disk.

6224- Unit - S/N:nnn has an invalid status/calibration file and cannot be initialized.

Cause: The AutoChem software cannot read the file that tracks the instrument's status.

Action: Reinstall the AutoChem software to make sure that the file is set up correctly.

6225 - No Vapor Generator calibration is available on Unit n - S/N: nnn.

6226- No instrument calibration is available on Unit n - S/N: nnn.

Cause:	The unit could not be initialized because no factory-supplied calibration file has been loaded.
Action:	Load the factory-supplied calibration file.

6231- The sample has an invalid status and cannot be used for this analysis.

Cause:	Only sample files with a status of **No Analysis** can be used for analysis. The sample file you have selected has a status other than **No Analysis**.
Action:	Select a sample file with a status of **No Analysis**.

6232- Warning: The furnace is open, and sample/furnace temperature is above ambient. Do you want to continue? (Yes, No)

Cause:	The sample's temperature (or that of the furnace) was above ambient, and/or the furnace was open when you attempted to start the analysis.
Action:	If the furnace/temperature condition is correct, click **Yes**. Otherwise, click **No**, correct the problem, then restart the analysis.

6233- Warning: The furnace is open. Do you want to continue? (Yes, No)

Cause A:	You tried to start an analysis with the furnace door open, or the furnace is not connected to the analyzer.
Action A:	If the furnace condition is correct, click **Yes**. Otherwise click **No**, correct the problem, and restart the analysis.
Cause B:	You tried to start an analysis with the furnace door closed, but the interlock switch inside the furnace has failed.
Action B:	Contact a Micromeritics Service Representative.

6234- Warning: The sample/furnace temperature is above ambient. Do you want to continue? Yes, No

Cause:	The sample's temperature (or that of the furnace) was above ambient when you attempted to start the analysis.
Action:	If the sample's temperature is correct, click **Yes**. Otherwise, click **No**, correct the problem, and restart the analysis.

6264- Warning: The CryoCooler needs to be re-filled. Do you want to continue? (yes, no)

Cause:	You are starting an analysis with a low level of liquid in the CryoCooler.
Action:	Select **Yes** to proceed with the analysis if the CryoCooler is not required. Select **No**, refill the CryoCooler, and restart the analysis if the CryoCooler is needed for this analysis.

6267- Inter-application message cannot be delivered. Select Continue to resume analysis.

6268- Analysis paused. An inter-application message could not be delivered.

6269- Analysis paused. An inter-application message failed. The Receiver was busy.

6270- Analysis paused. An inter-application message failed. The Receiver refused the request.

6271- Analysis paused. An inter-application message failed for an unknown reason.

Cause A: A DDE operation was not properly executed.

Action A: Ensure that the other application is loaded and in a state which supports DDE operations. If the problem persists, exit all applications and Windows, then restart the applications.

Cause B: The DDE command was not specified correctly.

Action B: Review the DDE server application manual for the specific command and enter it correctly in the DDE command step. If the problem persists, exit all applications and Windows, then restart the applications.

6273- Warning. For the chosen total flow rate and blending proportions, the carrier and/or loop MFC will be set to a flow rate less than 10 cm^3/min. Blending performance may not be acceptable. Do you wish to keep these settings? (Yes, No)

Cause: You requested blending of the carrier and loop gasses, but the proportions of the gasses and the flow rate of the mixture chosen caused at least one of the MFCs to be set to a rate less than 10 cm^3/min. The accuracy of the MFC at such low flow rates may be unacceptable for some applications.

Action: Click **Yes** if reduced accuracy is acceptable or click **No** and change the proportions or total flow rate.

6275- Instrument (Unit, S/N) initialization failure (code)

Cause: An internal error has occurred.

Action: Record the contents of the error message, then contact your Micromeritics service representative.

6400 Series

6400- Warning. The flow sensed by the carrier flow sensor is below 5 cm^3/min.

Cause: The gas flow in the analysis path carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the analysis gas Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6401- Warning. The flow sensed by the reference flow sensor is below 5 cm^3/min.

Cause: The gas flow in the reference gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the reference Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6402- Warning. The flow of the carrier Mass Flow Controller is below 5 cm^3/min.

Cause: The gas flow in the carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the carrier Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6403- Warning. The flow of the reference Mass Flow Controller is below 5 cm^3/min.

Cause: The gas flow in the reference gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the reference Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6404- Warning. The flow of the loop Mass Flow Controller is below 5 cm3/min.

Cause: The gas flow in the analysis gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the analysis Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6405- Warning. The flow of the carrier Mass Flow Controller differs from the target flow by 10% or more.

Cause: The gas flow in the carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the carrier Mass Flow Controller is set for a flow greater than 5 cm^3/min, that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6406- Warning. The flow of the reference Mass Flow Controller differs from the target by 10% or more.

Cause: The gas flow in the reference gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the reference Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6407- Warning. The flow of the loop Mass Flow Controller differs from the target by 10% or more.

Cause: The gas flow in the loop gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the loop Mass Flow Controller is set for a flow greater than 5 cm^3/min, that one of its valves is open, and that the gas supply to the open valve is operating correctly.

6408- Warning. The detector heat zone has been reporting an invalid temperature (reading) for one minute.

6409-Warning. The loop heat zone has been reporting an invalid temperature (reading) for one minute.

6410- Warning. The analysis heat zone has been reporting an invalid temperature (reading) for one minute.

6411- Warning. The cold trap heat zone has been reporting an invalid temperature (reading) for one minute.

6412- Warning. The vapor valve heat zone has been reporting an invalid temperature (reading) for one minute.

6413- Warning. The vapor flask heat zone has been reporting an invalid temperature (reading) for one minute.

6414- Warning. The vapor reflux heat zone has been reporting an invalid temperature (reading) for one minute.

Cause A: Heater cable is loose or disconnected.

Action A: Connect the heater cable.

Cause B: There is a failure in the hardware or electronics.

Action B: Establish an Idle state in the instrument, turn the instrument power OFF, then turn it back ON. Allow the software to be initialized. If the problem recurs after the software has been active for about one minute, contact your Micromeritics Service Representative.

6415- Warning. The furnace thermocouple is not connected.

Cause A: The cable connecting the furnace thermocouple to the front panel of the instrument has been disconnected.

Action A: Connect the cable (the connector is shaped so that it can only go in one way).

Cause B: There is a failure in the hardware or electronics.

Action B: Contact your Micromeritics Service Representative.

6416- Warning. The sample thermocouple is not connected or operating properly.

Cause:	Either the sample thermocouple is not connected, or it is not operating properly.
Action:	Remove the top cover of the instrument and inspect the sample thermocouple cable to insure that it is properly connected. If everything appears correct, substitute a different thermocouple. If the problem persists, there is a failure in the hardware or electronics. Contact a Micromeritics Service Representative.

6417- Warning. System pressure has been above the allowed limit for more than 10 seconds.

6418- Warning. System pressure has been above the tolerance level for more than one minute.

Cause:	Gas flow is restricted at some point in the system.
Action:	Examine the instrument schematic display to determine the current flow path from the carrier Mass Flow Controller, then examine the instrument's components that correspond to the flow path. Use Manual Control to identify the location of the restriction. A typical source of blockage is a sample that is too tightly packed in the tube. Clear the restriction, if possible. If necessary, contact a Micromeritics Service Representative.

6419- Warning. Manual Control of the furnace is not supported when the Sample Temperature system is active.

Cause:	The operator has attempted a Manual Control operation during an automatic analysis. The current step involves the sample temperature system.
Action:	Do not attempt manual operations during an analysis. If you wish to alter the sample temperature system manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

6420- Warning. Manual Control of a flow-related item is not supported when the Flow Management system is active.

Cause:	You have attempted a Manual Control operation during an automatic analysis. The current step involves a flow-related item.
Action:	Do not attempt manual operations during an analysis. If you wish to alter a flow-related item manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

6421- Warning. Manual Control of a TCD-related item is not supported when the Signal Management system is active.

Cause:	You have attempted a Manual Control operation during an automatic analysis. The current step involves a TCD-related item.
Action:	Do not attempt manual operations during an analysis. If you wish to alter a TCD-related item manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

6422- Warning. Manual Control of a heat zone is not supported when the Signal Management system is active.

Cause:	You have attempted a Manual Control operation during an automatic analysis. The current step involves a heat zone.
Action:	Do not attempt manual operations during an analysis. If you wish to alter a heat zone manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

6423- Error. Operation suspended. The flow sensed by the carrier flow sensor is below 5 cm^3/min.

Cause:	The gas flow in the analysis gas plumbing is not adequate. Either it is blocked, or the gas source (a Mass Flow Controller, its associated valves, and gas supplies) is not activated.
Action:	Ensure that the analysis Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly. If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6424- Error. Operation suspended. The flow sensed by the Reference Flow sensor is below 5 cm^3/min.

Cause:	The gas flow in the reference gas plumbing is inadequate. Either it is blocked, or the gas source (a Mass Flow Controller, its associated valves, and gas supplies) is not activated.
Action:	Ensure that the reference Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly. If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6425- Error. Operation suspended. The flow of the carrier Mass Flow Controller is below 5 cm^3/min.

Cause: The gas flow in the carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the carrier Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6426- Error. Operation suspended. The flow of the reference Mass Flow Controller is below 5 cm^3/min.

Cause: The gas flow in the reference gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the reference Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6427- Error. Operation suspended. The flow of the prep Mass Flow Controller differs from the target by 10% or more.

Cause: The gas flow in the prep plumbing is inadequate due to either a blockage or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the prep Mass Flow Controller is set for a flow, that one of its associated valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6428- Error. Operation suspended. The flow of the carrier Mass Flow Controller differs from the target by 10% or more.

Cause: The gas flow in the carrier plumbing is inadequate due to either a blockage or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the carrier Mass Flow Controller is set for a flow, that one of its associated valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6429- Error. Operation suspended. The flow of the reference Mass Flow Controller differs from the target by 10% or more.

Cause: The gas flow in the reference gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the reference Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6430- Error. Operation suspended. The flow of the loop Mass Flow Controller differs from the target by 10% or more.

Cause: The gas flow in the loop gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

Action: Ensure that the loop Mass Flow Controller is set for a flow greater than 5 cm^3/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6431- Error. Operation suspended. The detector heat zone has been reporting an invalid temperature (reading) for one minute.

6432- Error. Operation suspended. The loop heat zone has been reporting an invalid temperature (reading) for one minute.

6433- Error. Operation suspended. The analysis heat zone has been reporting an invalid temperature (reading) for one minute.

6434- Error. Operation suspended. The cold trap heat zone has been reporting an invalid temperature (reading) for one minute.

6435- Error. Operation suspended. The vapor valve heat zone has been reporting an invalid temperature (reading) for one minute.

6436- Error. Operation suspended. The vapor flask heat zone has been reporting an invalid temperature (reading) for one minute.

6437- Error. Operation suspended. The vapor reflux heat zone has been reporting an invalid temperature (reading) for one minute.

Cause A: Heater cable is loose or disconnected.

Action A: Connect the heater cable.

Cause B: A failure has occurred in the hardware or electronics.

Action B: Establish an Idle state in the instrument, turn the instrument power OFF, then turn it back ON. Allow the controller software to be initialized. If the problem recurs after the software has been active for about one minute, contact your Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must reestablish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

Cause C: The heat zone is calibrated incorrectly.

Action C: Contact your Micromeritics Service Representative.

6438- Error. Operation suspended. The furnace thermocouple is not connected.

Cause A: The cable connecting the furnace thermocouple to the front panel of the instrument has been disconnected.

Action A: Connect the cable (the connector is shaped so that it cannot be improperly inserted).

Cause B: There is a failure in the hardware or electronics.

Action B: Contact your Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6439- Error. Operation suspended. The sample thermocouple is not connected or operating properly.

Cause: Either the sample thermocouple is improperly connected or it is not functioning properly.

Action: Remove the top cover of the instrument and inspect the sample thermocouple cable to ensure that it is properly connected. If the connect appears correct, substitute a different thermocouple. If the problem persists, there is a failure in the hardware or electronics. Contact a Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the TCD heat zone and set the temperature to the correct value.

6440- Error. Operation suspended. System pressure has been above the allowed limit for more than 10 seconds.

Cause: Gas flow has been restricted at some point in the system.

Action: Examine the instrument schematic display to determine the current flow path from the carrier Mass Flow Controller, then examine the instrument’s components that correspond to the flow path. Use Manual Control to identify the location of the restriction. A typical source of blockage is a sample that is too tightly packed in the tube. Clear the restriction, if possible. If necessary, contact a Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6441- Error. Operation suspended. An invalid instruction has been encountered (code # at # in experiment # after step #).

Cause: A software error has occurred.

Action: Carefully record the entire text of this error message and the name of the sample file. Contact your Micromeritics Service Representative.

6442- Error. Operation suspended. After 200 attempts, the instrument has failed to detect two consecutive peaks of equal area.

6443- Error. Operation suspended. After 200 attempts, the instrument has failed to detect two consecutive peaks of zero area.

Cause: The sample tube contains little or no sample, or the sample is not reacting with the analysis gas.

Action: If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6444- Error. Operation suspended. A signal wait time-out has occurred.

Cause: The time allowed for a Wait step expired before the condition(s) of the Wait step were met.

Action: Examine the analysis conditions for this analysis to determine if sufficient time was allowed for the conditions of this Wait step to be met. Determine if the analysis should proceed. You may either resume the analysis or cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6445- Error. Operation suspended. A sample temperature operation time-out has occurred.

Cause: The time allowed for a sample temperature operation to be complete expired before the target temperature was reached. The control software calculates a time limit based upon the ramp rate and the difference between the current temperature and the target temperature.

Action: Examine the current sample temperature. If it is acceptable for the purposes of the current analysis, you may choose to resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6446- Error. Operation suspended. A gas flow operation time-out has occurred.

Cause: Stable gas flow in a Mass Flow Controller was not achieved within two minutes.

Action: Ensure that the gas supply for the open valve associated with the Mass Flow Controller is set to the correct pressure. Use Manual Control to detect the cause of the problem.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6447- Error. Operation suspended. An invalid parameter has been encountered (code # #).

6448- Error. Operation suspended. The requested script (ID = #) is not located in the instrument.

6449- Error. Operation suspended. There is insufficient room for the script (ID = #) (error code #).

Cause: A software error has occurred.

Action: Carefully record the entire text of this error message. On a floppy diskette, save a copy of the sample file being analyzed. Contact your Micromeritics Service Representative.

6450- Warning. The requested gas flow is not possible because all inlet valves for this MFC are closed.

Cause: A Manual control operation to set a gas flow in a Mass Flow Controller was requested, but all associated valves are closed.

Action: Use Manual Control to open the desired gas valve, then repeat the Set Flow Rate operation.

6451- Error. Operation suspended. Communications with the furnace controller were unsuccessful due to a time-out.

Cause: Internal communications between the instrument software and the furnace controller were unsuccessful; several retries were attempted.

Action: Make a note of the code number included in the error message. Contact your Micromeritics Service Representative.

6452- Error. Operation suspended. Sample temperature ramping/control has failed because the actual sample temperature differs from the temperature control element by more than 100 degrees C.

Cause: The sample temperature has differed significantly from that of the furnace for at least 10 minutes.

Action: Ensure that the sample thermocouple is properly installed and that the furnace is closed.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6453- Error. Operation suspended. An operation was requested that is not currently valid.

Cause: A software error has occurred.

Action: Carefully record the entire text of this error message and the name of the sample file. Contact your Micromeritics Service Representative.

6454- Error. Operation suspended. The flow control interface board (channel <number>) was not ready within the allowed time.

6455- Error. Operation suspended. The heat zones interface board (channel <number>) was not ready within the allowed time.

Cause: A hardware error has occurred.

Action: Carefully record the entire text of this error message and the name of the sample file. Contact your Micromeritics Service Representative.

6456- Error. Operation suspended. A heat zone <code number> did not reach its target of <number> (current temperature = <number>) within the allowed time of <number> minutes.

6457- Error. Operation suspended. The furnace did not reach a temperature between <number> and <number> in the allowed time of <number> minutes.

6458- Error. Operation suspended. Sample temperature equilibration is not allowed because sample temperature control is not active (code).

Cause: A hardware error has occurred.

Action: Carefully record the entire text of this error message and the name of the sample file. Contact your Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

6463- Warning. The Cold Trap rotary valve did not operate correctly.

Cause: An attempt to manually control the Cold Trap valve resulted in incorrect operation, possibly due to a mechanical problem.

Action: Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

6464- Warning. The Preparation rotary valve did not operate correctly.

Cause: An attempt to manually control the Preparation (also known as the Analysis) valve resulted in incorrect operation, possibly due to a mechanical problem.

Action: Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and try the operation again. If this is unsuccessful, exit the program and turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and restart the program. The valve should be reinitialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

6465- Warning. The Inject rotary valve did not operate correctly.

Cause: An attempt to manually control the Inject (also known as the loop) valve resulted in an incorrect operation, possibly due to a mechanical problem.

Action: Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and try the operation again. If this is unsuccessful, exit the program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and restart the program. The valve should be reinitialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

6466- Warning. The Vapor Generator rotary valve did not operate correctly.

Cause: An attempt to manually control the Vapor Generator valve resulted in an incorrect operation, possibly due to a mechanical problem.

Action: Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is unsuccessful, exit the program and turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and restart the program. The valve should be reinitialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

6467- Warning. The KwikCool option is not installed.

Cause: An attempt to control manually the KwikCool option failed because the KwikCool option was not detected at the time the control was attempted. If analysis is resumed before the problem is corrected, the current step will NOT be completed.

Action: Confirm that the KwikCool option cable is properly connected to the analyzer. It is recommended that the power to the analyzer be turned off when you attach the KwikCool connector to the analyzer (or detach it). Once again, use Manual Control to attempt the KwikCool operation. If the problem persists, you may choose to continue operations without using the KwikCool. Or, you may try exiting the software program and then turning the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. If the problem cannot be corrected, contact a Micromeritics Service Representative.

6468- Error. Operation suspended. The Cold Trap rotary valve did not operate correctly.

Cause: An attempt to control the Cold Trap valve during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If analysis is resumed before the problem is corrected, the current step will NOT be completed.

Action: Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact a Micromeritics Service Representative.

6469- Error. Operation suspended. The Preparation rotary valve did not operate correctly.

Cause: An attempt to control the Preparation (also known as the Analysis) valve during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If the analysis is resumed before the problem is corrected, the current step will NOT be completed.

Action: Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact a Micromeritics Service Representative.

6470- Error. Operation suspended. The Inject rotary valve did not operate correctly.

Cause:	An attempt to control the Inject (also known as the loop valve) during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If the analysis is resumed before the problem is corrected, the current step will NOT be completed.
Action:	Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer’s power off. After a few seconds, turn the analyzer’s power back on and then re-start the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact a Micromeritics Service Representative.

6471- Error. Operation suspended. The Vapor Generator rotary valve did not operate correctly.

Cause:	An attempt to control the Vapor Generator valve during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If the analysis is resumed before the problem is corrected, the current step will NOT be completed.
Action:	Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer’s power off. After a few seconds, turn the analyzer’s power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact your Micromeritics Service Representative.

6472- Error. Operation suspended. The KwikCool option is not installed.

Cause: An attempt to operate the KwikCool option during an analysis failed because the KwikCool option was not detected at the time the operation was attempted. If the analysis is resumed before the problem is corrected, the analysis will continue without the KwikCool.

Action A: Confirm that the KwikCool option cable is properly connected to the analyzer. It is recommended that the power to the analyzer be turned off when you attach the KwikCool to the analyzer (or when you detach it).

Action B: Use Manual Control to attempt the KwikCool operation that was requested during the analysis. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. Restart the analysis. If the problem persists, contact your Micromeritics Service Representative.

6473- Warning. The CryoCooler option is not installed.

Cause: You attempted to change the CryoCooler's settings but the CryoCooler was not detected.

Action A: Verify that the CryoCooler is physically connected to the instrument and that the retaining screws on the connector are securely tightened.

Action B: Exit the 2920 application, turn off the instrument, then restart the application. If the CryoCooler still does not respond, contact your Micromeritics service representative.

6474- Warning. The CryoCooler is almost empty. Temperature control may not be stable.

Cause: The liquid level in the CryoCooler is low. It may be impossible to achieve subambient temperatures unless cryogen is added.

Action: If the current analysis requires subambient temperatures, suspend the analysis, refill the CryoCooler with liquid nitrogen, then resume the analysis.

6475- Warning. Manual control of the CryoCooler is not supported when the Sample Temperature system is active.

Cause: You attempted to change the CryoCooler's settings while a temperature ramp was in progress.

Action: Wait until the temperature ramp is finished before adjusting the CryoCooler's settings.

6476- Warning. Calibration of the detector heat zone failed. The previous calibration will be used.

6477- Warning. Calibration of the loop heat zone failed. The previous calibration will be used.

6478- Warning. Calibration of the analysis heat zone failed. The previous calibration will be used.

6479- Warning. Calibration of the cold trap heat zone failed. The previous calibration will be used.

6480- Warning. Calibration of the vapor valve heat zone failed. The previous calibration will be used.

6481- Warning. Calibration of the vapor flask heat zone failed. The previous calibration will be used.

6482- Warning. Calibration of the vapor reflux heat zone failed. The previous calibration will be used.

Cause A: The measured temperature when the high calibration value was entered was less than the measured temperature when the low calibration value was entered.

Action A: Repeat the calibration, ensuring that the entered temperatures are accurate. If you are calibrating the vapor flask zone, ensure that the heating mantle is attached to the instrument properly.

Cause B: The temperature is being measured improperly by the instrument.

Action B: Contact your Micromeritics service representative if you continue to receive this message.

6483- Error. analysis cannot proceed. System pressure has been above the allowed limit for more than 10 seconds.

Cause: Gas flow has been restricted at some point in the system.

Action: Examine the instrument schematic display to determine the current flow path from the carrier Mass Flow Controller, then examine the instrument's components that correspond to the flow path. Use Manual Control to identify the location of the restriction. A typical source of blockage is a sample that is too tightly packed in the tube. Clear the restriction, if possible. If necessary, contact a Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

J. TUTORIALS

This appendix contains tutorials for performing a:

- typical analysis, beginning on this page
- TPR analysis, page **J-9**
- TPD analysis, page **J-14**
- TPO analysis, page **J-19**
- BET analysis, page **J-23**
- Pulse chemisorption analysis, page **J-32**
- Loop calibration, page **J-38**
- Gas concentration calibration, page **J-42**
- Vapor generator calibration, page **J-45**

Typical Analysis

The instructions in this section are provided to familiarize you with the software and analyzer. More detailed instructions for performing each analysis type are found later in this appendix.

Analysis conditions sets are included with the software for TPR, TPO, TPD, Pulse Chemisorption, and BET experiments. For this tutorial, you may perform any one of these analyses, using any sample you choose. Using a reference sample is recommended.

As always, the operator is responsible for making sure that safe combinations of samples and gases are used.

In this example, we use Cupric Oxide (15-20 mg) to perform a TPR experiment. This analysis uses a mixture of hydrogen with an inert gas as the carrier gas. No loop gas is used.

1. Prepare and determine the mass of the sample, then load it on the analyzer. Make other preparations for the type of analysis you plan to perform (such as preparing the cold trap and dewar, and positioning the thermocouple) as directed in Chapter 4.

2. Make sure that the correct gases are connected. Also check to be sure that the Gas Selections table (on the Unit menu) accurately reflects the current gas connections.

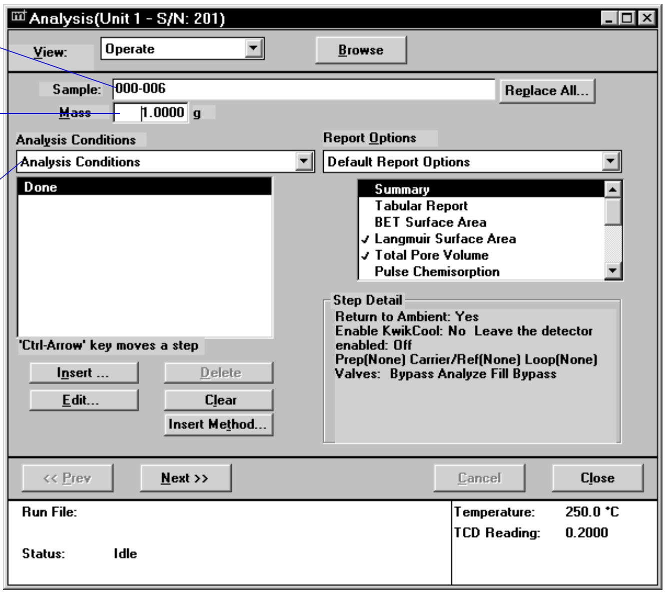
3. Select **Unit > Start Analysis**; the Analysis dialog sequence opens. You are first presented a dialog from which to choose a sample file. In this example, you are creating a new sample file; click **OK** to accept the default.

4. Click **Yes** to create the file. The Analysis dialog opens containing the sample file you are creating; all fields contain default values. Complete the dialog as shown below. (Basic sample file presentation format is shown; Advanced format may also be used.)

If desired, change or add to the default identifier.

Enter the sample mass.

From the drop-down list, select the analysis conditions set that is appropriate for the type of analysis you wish to perform.



Analysis(Unit 1 - S/N: 201)

View: Operate

Sample: 000-006

Mass: 1.0000 g

Analysis Conditions: Analysis Conditions

Report Options: Default Report Options

Done

'Ctrl-Arrow' key moves a step

Summary
Tabular Report
BET Surface Area
✓ Langmuir Surface Area
✓ Total Pore Volume
Pulse Chemisorption

Step Detail
Return to Ambient: Yes
Enable KwikCool: No Leave the detector enabled: Off
Prep(None) Carrier/Ref(None) Loop(None)
Valves: Bypass Analyze Fill Bypass

Run File:

Status: Idle

Temperature: 250.0 °C

TCD Reading: 0.2000

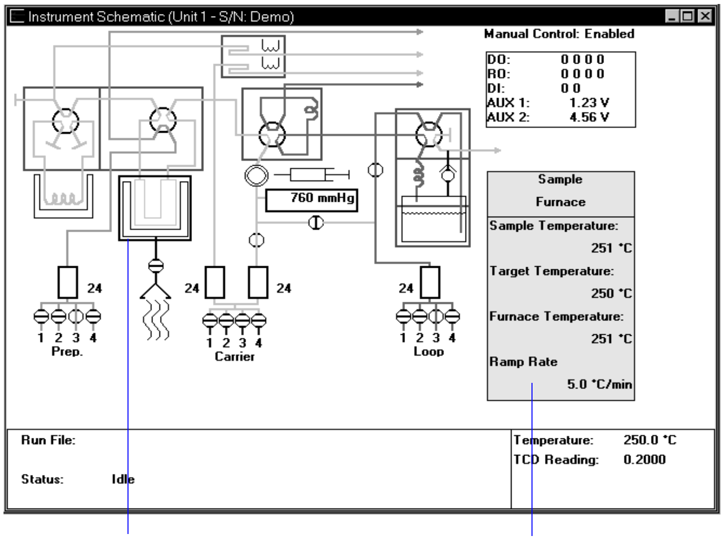
5. Click **Next** three times to accept the calibration and reports defaults (although these can be changed when you are performing your own analyses) and to verify the caution information.

6. Click **Start** and the analyzer begins the experiment.

Observe the Analysis

The Analysis window contains four different views in which to observe the progress of your analysis. You select the view from the View drop-down list. The Instrument Schematic view shows the current status of the internal components of the instrument. The Results view displays the results, periodically updating the view as data are collected. The Sample Log view lists the completed steps contained in the current experiment, as well as any error messages that may have occurred during the analysis. During the time your analysis is running, open each view and explore the system’s features.

- Select the **Instrument Schematic** view and click on the sample tube illustration. The box at the lower right corner of the schematic shows updated details about the temperatures related to the sample furnace heat zone. Clicking on other parts of the schematic causes their details to be displayed in the detail box.



Manual Control: Enabled

DO: 0 0 0 0
RO: 0 0 0 0
DI: 0 0
AUX 1: 1.23 V
AUX 2: 4.56 V

Sample Furnace
Sample Temperature: 251 °C
Target Temperature: 250 °C
Furnace Temperature: 251 °C
Ramp Rate 5.0 °C/min

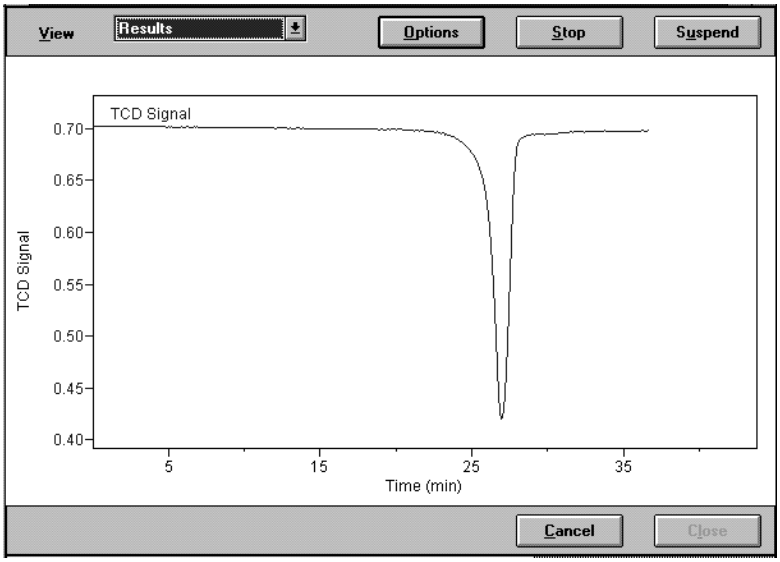
Run File:
Status: Idle

Temperature: 250.0 °C
TCD Reading: 0.2000

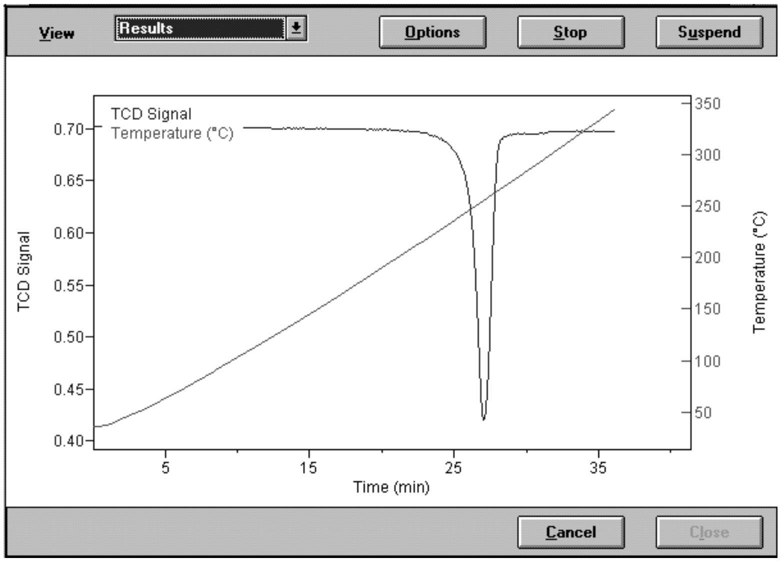
Sample Tube Illustration

Detail Box

- Select the **Results** view to observe the peaks **after** the baseline stabilizes and recording begins. This illustration shows the Results view of a TPR analysis on CuO in progress.



In the following illustration, Y-axis Temperature readings have been added to the display. (Instructions for making such changes to the display are given below.)

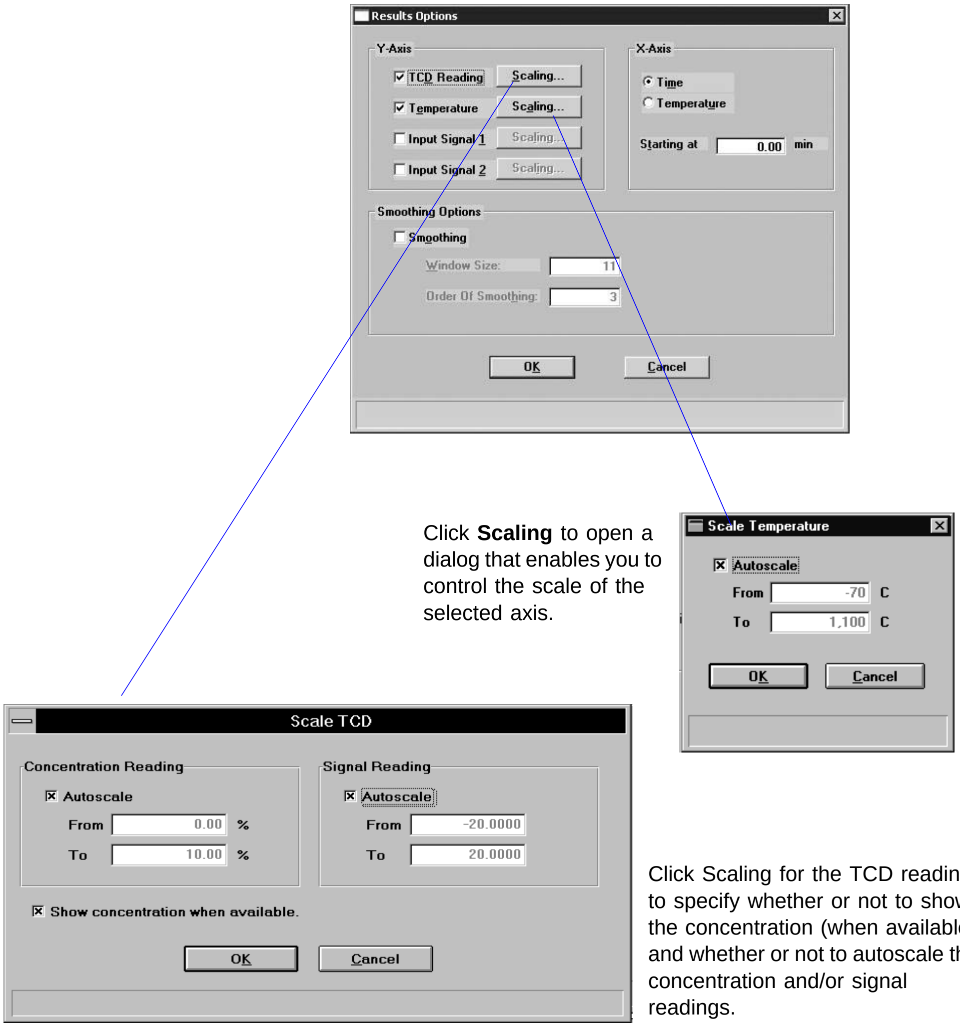


View Results

Note that the display is rescaled automatically as the experiment progresses.

- While the **Results** view is displayed, click **Options**. A dialog opens that allows you to change the presentation of the results.

Y-axis	Mark the check box(es) for the results you wish to display.
X-axis	Mark a radio button to choose Time or Temperature for the X-axis. You can also specify the starting value for the X-axis.



Click **Scaling** to open a dialog that enables you to control the scale of the selected axis.

Click Scaling for the TCD reading to specify whether or not to show the concentration (when available) and whether or not to autoscale the concentration and/or signal readings.

- Select the **Sample Log** view to review a list of each step as the analyzer begins it. The Sample Log also lists any error messages that occur during the analysis.

Ending the Analysis

During an analysis, the software executes each step in the analysis conditions set. The last step in every analysis is the Done step. The Done step lets you specify the ending state of the analyzer. The Done step is completed when the analyzer reaches the state specified.

Do not close the Analysis dialog until you have finished observing the data using its various views.

After the Done step is completed, you can continue to examine the data using the various views of the Analysis dialog, or you can close the Analysis dialog by clicking **Next**.

After you close the Analysis dialog, you can no longer use its views to assess this sample's data. However, you can view this sample's data in reports, by using the Peak Editor, and (if you use Advanced format for your sample files) by opening the sample information file to view the collected data.

Printing Reports

Desired report options are selected (and edited if required) from the sample information file. These reports can be generated automatically to a choice of destinations by selecting the report option on the third dialog of the Start Analysis series of dialogs (Chapter 6).

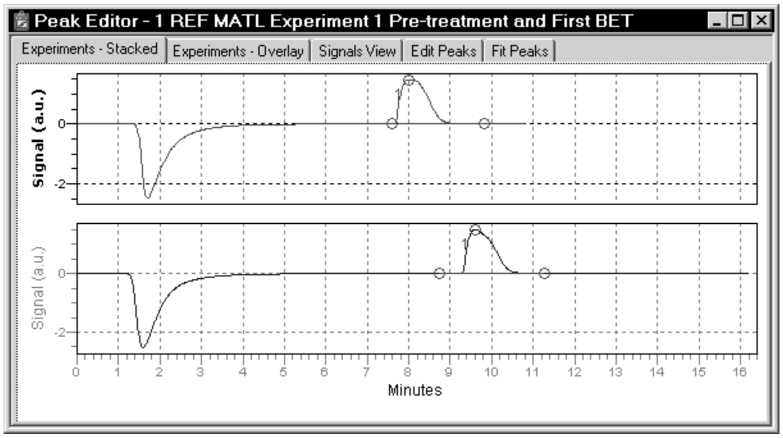
If desired, you can generate partial reports at any time during the analysis or additional reports after the analysis. To do so, select **Start Report** from the Report menu. Select the sample file you wish to report on, then click **OK**. In the reports list box, deselect and/or select the reports you do not wish to print, then click **OK**.

Refer to Chapter 7 for additional information on reports.

Editing the Peaks

A set of peak-picking parameters is contained in the sample information file. The software automatically detects peaks that meet these criteria, and all such peaks are included in the results. In some cases, the automatically detected peaks accurately represent the results. However, it is often helpful to edit the peaks using the Peak Editor. The Peak Editor can be used at any time during or after an analysis.

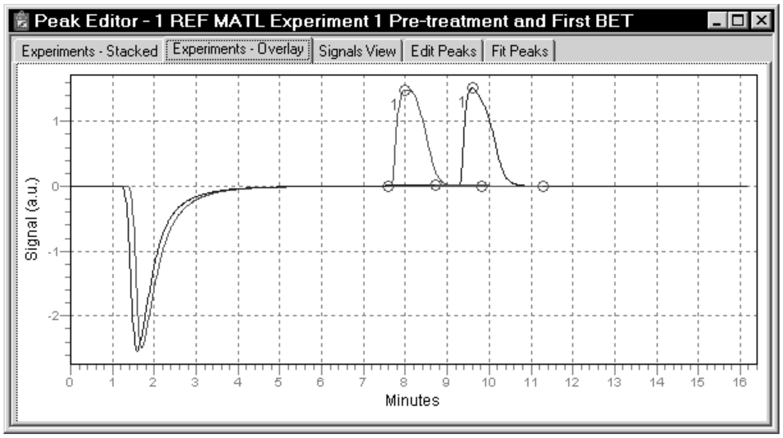
1. Select **Report > Peak Editor**; a files list box is displayed. Select the desired sample file, then click **OK**.



The Peak Editor defaults to the Experiments - Stacked view. This view shows the signals for all experiments contained in the file in a stacked fashion, one after the other. This example shows two experiments.

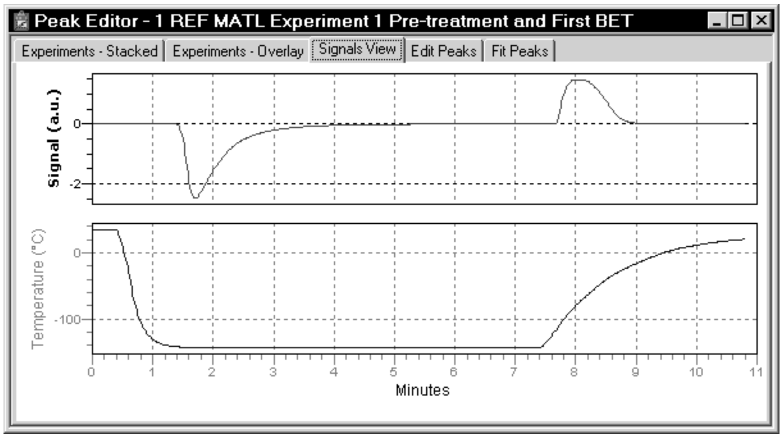
2. Right-click in the graph area to display a menu of the functions available while the Peak Editor is in this view. Refer to Chapter 7 for a description of these functions.

3. Click **Experiments - Overlay**; this view displays the results of all experiments (in this example, the same two shown previously) in an overlaid manner.



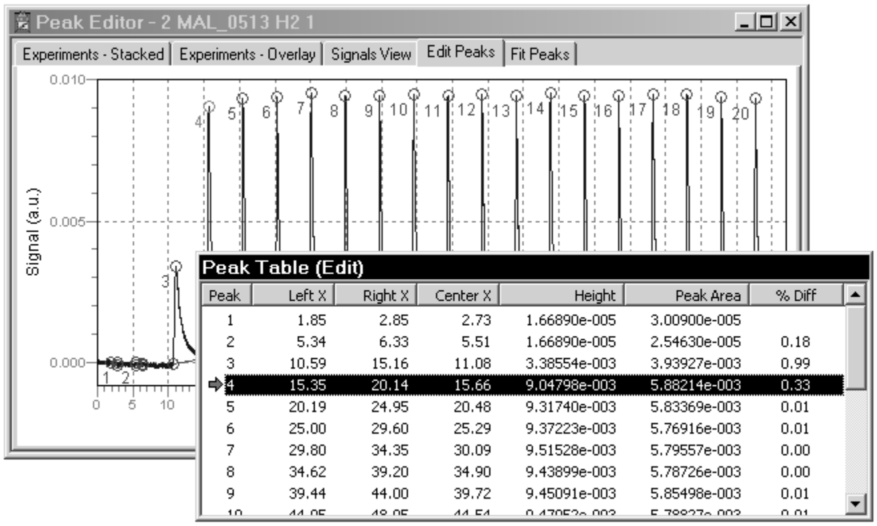
4. Right-click in the graph area to view the functions available for this view.

5. Click **View Signals**; this view shows the currently selected experiment in context with the temperature conditions that occurred during the analysis (as well as auxiliary signals, if they were recorded).



6. Right-click in the graph area to view the functions available in this view.

7. Click **Edit Peaks**; the selected experiment is shown with a peak table detailing peak information positioned on top.



Peak Table (Edit)

Peak	Left X	Right X	Center X	Height	Peak Area	% Diff
1	1.85	2.85	2.73	1.66890e-005	3.00900e-005	
2	5.34	6.33	5.51	1.66890e-005	2.54630e-005	0.18
3	10.59	15.16	11.08	3.38554e-003	3.93927e-003	0.99
➨4	15.35	20.14	15.66	9.04798e-003	5.88214e-003	0.33
5	20.19	24.95	20.48	9.31740e-003	5.83369e-003	0.01
6	25.00	29.60	25.29	9.37223e-003	5.76916e-003	0.01
7	29.80	34.35	30.09	9.51528e-003	5.79557e-003	0.00
8	34.62	39.20	34.90	9.43899e-003	5.78726e-003	0.00
9	39.44	44.00	39.72	9.45091e-003	5.85498e-003	0.01
10	44.05	48.05	44.54	9.47052e-003	5.78827e-003	0.01

When multiple peaks are displayed, the peak markings for the selected peak are always green.

8. Right-click in the graph area to view the functions available for editing the peaks. Experiment with some of the options if you like, then click **No** when asked if you wish to save the peak data.

9. Click **Fit Peaks**; this view shows the selected experiment with a peak table positioned on top.

   This view allows you to define a set of peaks; for example you may wish to create a table containing only the peaks of interest.

10. Right-click in the graph area to view the functions available for creating peak sets. Experiment with some of the options; you can delete the peak sets when finished.

Example Analyses

This section contains a description and instructions for performing each type of analysis. The descriptions provide a very basic overview of each type of analysis. Recommendations for gases, ramp rates, etc. are generalizations which are valid for many types of samples, but not all. The examples provided are the same as the standard analysis conditions sets that are included in the software.

The operator is responsible for understanding the sample's properties, in order to make gas and temperature selections that are safe and that yield useful data.

Analysis summaries include a basic explanation of the scientific theory upon which the analysis is based. If more extensive theory information is needed, you may wish to obtain a copy of *Analytical Methods in Fine Particle Technology*. Refer to Chapter 10 for ordering information.

Remember that other chapters in this manual describe each menu, window, and field in detail. Refer to these chapters if you have a question about a step described in these procedures.

TPR Analysis

Description

Temperature-Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperature at which reduction occurs. An important aspect of TPR analyses is that the sample need not have any special characteristics other than containing reducible metals.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) over the sample, usually starting at ambient temperature. While the gas is flowing, the temperature of the sample is increased linearly with time and the consumption of hydrogen by adsorption/reaction is monitored. Changes in the concentration of the gas mixture are determined. This information yields the hydrogen uptake volume.

Preparation

Pretreatment

Oxidize by flowing O_2 over the sample.

Analysis

Flow 5-10% hydrogen/argon OR 5-10% hydrogen/ nitrogen, while ramping the temperature. The analyzer records hydrogen consumption as a function of temperature. Nitrogen is sometimes used because it is more economical than argon. However, argon is recommended over nitrogen because the resultant peak(s) show no reaction between sample and gas.

Cold Trap

A cold trap is not required if you use argon and only traces of water are formed as a product of the reduction, and if the internal analyzer temperature zones are maintained at 100 °C or higher during the entire experiment. This method is preferred for longer experiments, because it eliminates the need to replenish the dewar.

When you use argon, the need for a cold trap may vary. In some cases a small amount of water may be found, so the cold trap may be needed. If only a small amount of water is produced, no cold trap is needed, because the thermal conductivity of argon and water vapor are very similar, so the water vapor's presence does not affect the signal reading. Best baseline stability and lowest noise always result from using a cold trap.

A cold trap is needed for the entire analysis if you are using nitrogen, because water vapor and nitrogen have different thermal conductivities.

Before performing an analysis, make sure you have adequately prepared the sample and analyzer using the instructions in Chapter 4.

Procedure

The following steps are provided as an example of how to perform a TPR analysis of copper oxide. Make the appropriate modifications to these steps for the material you wish to analyze.

Some reactions begin at temperatures below ambient. In such cases, a dewar containing an appropriate coolant should be used instead of the furnace at the beginning of the experiment. For example, reduction of PtO should begin at approximately -50 °C, because the reaction begins at about -30 °C. Alternatively, the optional CryoCooler can be used.

1. Obtain the sample weight, then install the loaded sample tube on the analyzer. If the analysis begins below ambient, either place a dewar of coolant around the sample tube, or close the furnace around the sample tube and install the CryoCooler. If the analysis begins at ambient, close the furnace around the sample tube.

2. Install the cold trap (if you are using one), then place a dewar filled with coolant around it. Make sure that the dewar contains enough coolant to cover the cold trap loops.

   A mixture of isopropyl alcohol (IPA) and liquid nitrogen (LN_2) is the recommended coolant for this experiment. Place the isopropyl alcohol in a dewar and slowly pour LN_2 into the dewar as you stir the mixture. Continue to add and stir the mixture until it becomes a slush. The mixture must be capable of achieving a temperature of about -90 ºC.

Extreme caution should be used when mixing the IPA/LN_2. If the dewar is damaged during this process, the mixture could be expelled from the dewar, causing injury.

3. Create a sample information file for this analysis. (Descriptions of the File menu items are found in Chapter 5.)

   a. Select **File** > **Open** > **Sample Information**. Enter a file name or accept the default; click **OK**, then **Yes** to confirm.

   b. Complete the Sample Information dialog. Enter the correct weight and, if desired, complete the optional descriptive fields (comments, operator, etc.).

   c. Click the Analysis Conditions tab and insert the experiment steps listed below:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select **Temperature Programmed Reduction** from the drop-down list.
	Zones	Block Filament Cold Trap Analysis Loop Vapor generator Reflux Flask	100 175 110 110 110 40 40 40
	Gas Flows	Prep	Helium
		Carrier/Reference	10H2-Ar OR 10H2-N2
		Loop	None
		Valve states	Trap, Prepare, Fill, Bypass
		Flow rate (all gases)	50 cm^3/min
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: **Wait for operator**	Type: *Add sample and set up cold trap.*
Change Gas Flows	Gas Flows	Prep	None
		Carrier	10H2-Ar
		Loop	None
		Valve states	Trap, Analyze, Fill, Bypass
Wait	Wait	Select: **Wait until baseline is stable**.	
Recording	Start Recording	One measurement every:	1.0 seconds
Temperature Ramp	Temperature Ramp	Ramp Type	Sample Ramp
		End Temperature	400
		Rate	10.0
		Hold Time	0.00
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop.			

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Done	Done (Click **Edit**, if necessary)	Return to ambient	Yes
		Detector	Off
		Gas Flows	Not applicable
		Zones	Not applicable

d. Click on the Report Options tab and set the values to those you wish to use.

e. Click **Save** then **Close**.

4. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6.)

a. From the Unit menu, select **Start Analysis**. From the Files list box, select the sample file you just created.

b. You can edit the sample file, if needed. For this example, no editing is required; click **Next** to continue.

c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.

Calibration files can also be associated with a sample file after analysis through the Peak Editor using Set Calibration.

d. Click **Next**; the next Analysis dialog appears: the reports dialog, no changes are needed.

e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.

As the temperature increases, the copper oxide is reduced, the water produced by the reaction is collected in the cold trap (if used), and the amount of hydrogen consumed is detected and transmitted to the software. Use the Results view to display a chromatogram of the hydrogen consumed from the detector signal as a function of the ramping temperature.

A hydrogen consumption peak which corresponds to the reduction capacity of copper oxide is displayed. The maximum peak should occur at approximately 280 ºC. This temperature varies highly, depending on the CuO particle size. Larger particle size shifts the temperature upward to 330 °C or more. Under certain combinations of sample, hydrogen concentration, and flow rate, two peaks may appear due to the transition state of Cu^{2+} to Cu^{+} to Cu.

5. Allow the TCD signal to return to the initial baseline after the peak has been recorded. When the analysis concludes, remove the sample tube.

Use the *cotton* gloves provided in the accessory kit if you wish to remove the sample tube before it has cooled. You may use rubber gloves to handle the sample tube when it has cooled.

TPD Analysis

Description

Temperature-Programmed Desorption (TPD) analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows over the sample and reacts with the active sites. (Alternatively, a step-like stream of gas is used when Pulse Chemisorption is used to react with active sites.) Programmed desorption begins when the temperature is ramped linearly over time while a constant stream of inert carrier gas passes over the sample.

At a certain temperature, the heat will overcome the activation energy, breaking the bond between the adsorbate and adsorbent. The adsorbed species will then desorb. If different active metals are present, they usually will desorb the reacted species at different temperatures. The desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which measures the gas concentrations. The volume of desorbed species combined with the stoichiometry factor, and the temperature at which pre-adsorbed species desorb, yield the number and strength of active sites.

Preparation

Pretreatment	Degas by flowing inert gas (such as helium, argon, or nitrogen) over the sample while ramping the temperature.
Analysis	Choose a gas mixture from the following table based on the results desired. Flow the gas over the sample, then ramp the temperature beginning at ambient.

Results Desired	Carrier Gas	Analysis Gas
Active Metal Surface Area H_2 Chemisorption	argon	hydrogen
Active Metal Surface Area CO Chemisorption	helium	carbon monoxide
Acidity	helium	ammonia (15% maximum NH_3 blended in helium)

Cold Trap Not required.

Before performing an analysis, make sure you have adequately prepared the sample and analyzer using the instructions in Chapter 4.

Procedure

Instructions given here are specific for analyzing zeolite with ammonia (use 15% maximum NH_3 blended in helium) at 3 to 20 ºC/min. You should adjust the values given in the example to accommodate the analysis you are performing.

1. Obtain the sample weight, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.

2. Create a sample file for this analysis.

   a. Select **File > Open > Sample Information**. Type a file name or accept the default; click **OK**, then **Yes** to confirm.

   b. Complete the Sample Information dialog. Enter the correct weight and, if desired, complete the optional descriptive fields (comments, operator, etc.).

   c. Click the Analysis Conditions tab and insert the experiment steps listed below:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select **Other** from the drop-down list.
	Zones	Block Filament Cold trap Analysis Loop Vapor generator Reflux Flask	100 175 110 110 110 20 20 20
	Gas Flows	Prep	Helium
		Carrier/Reference	Helium
		Loop	None
		Flow rate	50 cm^3/min
		Valve states	Bypass, Prepare, Fill, Bypass
	Outputs	Use defaults	
	Peaks	Use defaults	
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	350
		Ramp Rate	50.0
		Hold Time	10.00
Experiment	New Experiment	Experiment description	Type an appropriate description.
		Type of analysis	Select **Temperature Programmed Desorption** from the drop-down list.
	Zones	Block Filament Cold trap Analysis Loop Vapor generator Reflux Flask	100 175 110 110 110 50 40 50
	Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment *(continued)*	Gas Flows	Flow rate	50 cm^3/min
		Valve states	Bypass, Prepare, Fill, Bypass
	Outputs	Use defaults	
	Peaks	Use defaults	
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Change Gas Flows	Gas Flows	Prep	Ammonia (15% maximum NH_3 blended in helium)
		Carrier	Helium
		Loop	Ammonia (15% maximum NH_3 blended in helium)
		Flow rate	50 cm^3/min
		Valve states	Bypass, Prepare, Fill, Bypass
Wait	Wait	Select: **Wait**	Type: **60.0 minutes**
Change Gas Flows	Gas Flows	Prep	None
		Carrier	Helium
		Loop	Ammonia (15% maximum NH_3 blended in helium)
		Valve states	Bypass, Analyze, Fill, Bypass
Wait	Wait	Select: **Wait**	Type: **30.00 minutes**
Wait	Wait	Select: **Wait until baseline is stable.**	
Recording	Start Recording	One measurement every	1.0 seconds
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	500
		Rate	3.0
		Hold Time	0.0
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop.			
Wait	Wait	Select: **Wait**	Type: **30.00 minutes**
Temperature Ramp	Temperature Ramp	Type	Return to Ambient

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Repeatedly insert the last several steps from the step that is double-outlined above (*Wait until baseline is stable* step). Use a different ramp rate for each repetition; after ramping at 3°/min. and 5°/min., use 10, 15, and 20°/min. (This shifts the peak as a function of the Heat of Desorption.)			
Done	Done (Click **Edit**, if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable

d. Click the Report Options tab and set the values to those you wish to use. Click **Save**, then **Close**.

3. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6.)

   a. Select **Unit > Start Analysis**. From the Files list box, select the sample file you just created.

   b. You can edit the sample file, if needed. For our example, no editing is required; click **Next** to continue.

   c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.

   d. Click **Next**; the next Analysis dialog appears: the reports dialog. For this example, no changes are needed.

   e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.

When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, you may wish to use the optional CryoCooler or KwikCool. Remove the sample tube when the analysis is complete.

Use the *cotton* gloves provided in the accessory kit if you wish to remove the sample tube before it has cooled. You may use rubber gloves to handle the sample tube when it has cooled.

TPO Analysis

Description

Temperature-Programmed Oxidation (TPO) examines the extent to which a catalyst can be re-oxidized. Generally, TPO analyses are used to measure the degree of reduction of certain oxides.

Usually the sample is pretreated and the metal oxides are reduced to the base metal. Then the reactant gas is applied to the sample in pulses or (alternatively) as a steady stream. The AutoChem 2920 measures the uptake of the reactant gas.

TPO is always performed after TPR is performed. When the TPR experiment concludes, the sample is returned to room temperature. (This process can be accelerated using the optional CryoCooler or KwikCool.) Then, the analysis gas is changed to 2-5% O_2 + He. This gas mixture is flowed through the sample at ambient temperature, then the temperature is ramped up to the same maximum temperature used for the preceding TPR analysis. The portion of the sample that had been reduced is re-oxidized, and the degree of reduction can be calculated.

If the TPR and TPO results are different, there are two possible causes: the sample material sintered or it reacted with the gas.

Preparation

Pretreatment	TPR
Analysis	2 to 5% oxygen/helium is flowed through the sample while temperature is ramped, beginning at ambient temperature.
Cold Trap	None needed

Before performing an analysis, make sure you have adequately prepared the sample and analyzer by following the steps described earlier in this chapter.

For the following example, a TPO is performed on WO3. Because TPR is used as the preparation for TPO, the TPR process was included in this example.

Procedure

1. Obtain the sample weight, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.

2. Create a sample file for this analysis.

    a. Select **File** > **Open, Sample Information**. Type a file name or accept the default; click **OK**, then **Yes** to confirm.

    b. Complete the Sample Information dialog. Enter the correct weight and, if desired, complete the optional descriptive fields (comments, operator, etc.)

    c. Click on the Analysis Conditions tab and insert the experiment steps in the following table:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select **Temperature Programmed Reduction** from the drop-down list.
	Zones	Block Filament Cold trap Analysis Loop Vapor generator Reflux Flask	100 175 110 110 110 20 20 20
	Gas Flows	Prep	Helium
		Carrier	10 H2-Ar
		Loop	None
		Flow rate	50 cm^3/min
		Valve states	Trap, Prepare, Fill, Bypass
	Outputs	Use defaults	
	Peaks	Use defaults	
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Change Gas Flows	Gas Flows	Prep	None
		Carrier/Reference	10 H2-Ar
		Loop	None
		Valve states	Bypass, Analyzer, Fill, Bypass

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Wait	Wait	Select: **Wait for operator**	Type: **Position cold trap dewar and close furnace.**
Wait	Wait	Select: **Wait until baseline is stable**.	
Recording	Start Recording	One measurement every	1.0 seconds
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End temperature	980
		Rate	10.0
		Hold time	0.00
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop.			
Change Gas Flows	Gas Flows	Prep	Helium
		Carrier/Reference	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Bypass
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select **Temperature Programmed Oxidation** from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
		Vapor generator	20
		Reflux	20
		Flask	20
	Gas Flows	Prep	Helium
		Carrier/Reference	2 02-He
		Loop	None
		Flow rate	50 cm^3/min
		Valve states	Bypass, Prepare, Fill, Bypass
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: **Wait until baseline is stable.**	

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Recording	Start Recording	One measurement every:	1.0 seconds
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	980
		Rate	20.0
		Hold Time	0.00
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop			
Change Gas Flows	Change Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Bypass
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Done	Done (Click **Edit**, if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable

d. Click the Report Options tab and set the values to those you wish to use.

e. Click **Save**, then **Close**.

3. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6.)

   a. Select **Unit > Start Analysis**. From the Files list box, select the sample file you just created.

   b. You can edit the sample file if needed. For this example, no editing is needed; click **Next** to continue.

   c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.

   d. Click **Next**; the next Analysis dialog appears: the reports dialog. For our example, no changes are needed.

   e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.

When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, you may wish to use the optional CryoCooler or KwikCool.

4. As the temperature increases, the sample is oxidized, and the software calculates the volume of oxygen taken up.

5. Allow the TCD signal to return to the initial baseline after the peak has been displayed.

Use the *cotton* gloves provided in the accessory kit if you wish to remove the sample tube before it has cooled. You may use rubber gloves to handle the sample tube when it has cooled.

6. When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

BET Surface Area Analysis

Description

The BET Surface Area analysis lets you evaluate total surface area of the catalyst before and after chemisorption. Disactivation, which may occur due to the blocking of pores during the chemisorption reaction, as well as the occurrence of sintering of the support, can be studied.

After outgassing the sample, a mixture of 30% nitrogen and 70% helium is applied to the sample which is immersed in a liquid nitrogen (LN_2) bath. The amount of nitrogen adsorbed (usually measured on desorption) at LN_2 temperatures is used to calculate total surface area.

Because nitrogen uptake is a function of pore size, sample sintering can cause results to show reduced surface area. Therefore, it may be helpful to perform BET as the first and last experiments in your analysis, in order to check for sintering of the support.

Preparation

Pretreatment	Flow helium over the sample to remove impurities, usually at the maximum temperature tolerated by the sample (for example, the temperature at which the sample was calcined).
Analysis	Flow 30% N_2/He, using a dewar of liquid nitrogen, to measure the uptake of nitrogen. Then, remove the LN_2 dewar and replace it immediately with a dewar of water at ambient temperature. The amount of N_2 desorbed is measured and the BET equation is used to calculate the active surface area.
Cold Trap	Install the external long delay path. Do not use a cold trap dewar.
Pressure regulator	For BET Analysis, gas cylinders should be set to a level between 10 and 15 psig (69 and 103.5 kPag).
Furnace temperature	During pretreatment, select a temperature high enough to remove any contaminants or moisture, but not so high as to cause sintering or fusing of the sample. Hold the temperature long enough to remove any contaminants or moisture. Maintain the target temperature for at least 30 minutes to ensure adequate degassing. Make sure the Done step is set to return the sample temperature to 0 ºC.

Calibration

BET experiments require a separate calibration consisting of three manual injections of nitrogen through the septum. You can perform this calibration either before or after the BET analysis is performed. As with all TCD calibrations, you will use the Peak Editor to associate the calibration file with the analysis file. For this reason, it is necessary that the data on the manual injections are collected in a separate experiment from the BET experiment. The injections may be included in the same analysis as the BET experiment, OR they may be performed as a completely separate analysis. You should use the same recording options (zero, invert) for the Calibration experiment and the BET experiment.

The method below includes the calibration injections as a separate experiment which follows the BET experiment in the analysis.

Injection Size

You should inject three volumes of gas with the goal of approximating the volume of gas taken up by the sample. Use one injection slightly larger than the volume you expect, one injection slightly smaller than the volume you expect, and one injection between these two volumes. The largest injection should be two to three times greater than the smallest injection.

Because you must use the same syringe for all three injections, be sure to select a syringe that can accommodate all three injection volumes. You can estimate the volume of gas uptake if you know the approximate surface area (SA) of your sample. Use the following formula to determine the approximate volume (V_m) to inject.

$$V_m(cm^3\ STP) = \frac{SA(\ m^2)}{(6.023 \times 10^{23}\ molecule/mole)(16.21 \times 10^{-20}\ m^2/molecule)} \times 22414\ cm^3/mole$$

So:

$$V_m cm^3\ STP = SA(\ m^2) \times 0.229$$

For example, a sample of 0.1 g of 50 m^2/g material has a surface area of 5 m^2. Therefore, the volume of the gas which will be adsorbed is approximately 1.145 cm^3 STP.

Procedure

Before performing an analysis, make sure you have adequately prepared the sample and analyzer using the instructions in Chapter 4.

1. Obtain the sample weight, then install the loaded sample tube and thermocouple on the analyzer.

2. Make sure the Delay Path is installed in the Cold Trap port.

3. Create a sample file containing the appropriate analysis conditions and report options. (Descriptions of the options on each dialog are found in Chapter 5.)

    a. Select **File > Open > Sample Information**. Type a file name or accept the default; click **OK**, then **Yes** to confirm.

    b. Complete the Sample Information dialog using appropriate values.

    c. Click on the Analysis Conditions tab and insert the experiment steps listed below:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select **BET Surface Area** from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
		Vapor	20
		Reflux	20
		Flask	20
	Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None
		Flow rate	50 cm^3/min
		Valve states	Trap, Prepare, Fill, Bypass
	Outputs	Use defaults	
	Peaks	Use defaults	
	Recording options	Select: **Invert the TCD Signal**	
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	350
		Rate	50.0
		Hold Time	0.00
		Enable KwikCool	No
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
		Enable KwikCool	Yes
Change Gas Flows	Gas Flows	Prep	None
		Carrier	30 N2-He
		Loop	None
		Valve states	Trap, Analyze, Fill, Bypass
Wait	Wait	Select: **Wait until baseline is stable.**	
Wait	Wait	Select: **Wait for operator**	Type: **Click OK to proceed with BET measurement.**
Recording	Start Recording	One measurement every:	0.1 second

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Dose	Dose	Select: **Get BET Point**	
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Dose step and the Temperature Ramp step within the Start/Stop Record loop.			
Done	Done (Click **Edit**, if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable

To include the calibration data collection (three manual injections through the septum) within this analysis but as a separate Experiment, also insert the following steps (**before the Done step**):

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select **Other** from the drop-down list.
	Zones	Block Filament Cold trap Analysis Loop Vapor generator Reflux Flask	100 175 110 110 110 20 20 20
	Gas Flows	Prep	None
		Carrier	30 N2-He
		Loop	None
		Flow rate	50cm^3/min
		Valve states	Trap, Analyze, Inject, Bypass
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: **Wait until baseline is stable.**	
Recording	Start Recording	One measurement every:	0.1 second

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Repeat	Start Repeat Sequence	Select: **Repeat**	Type: **3 times**
Wait	Wait	Select: **Wait for operator.**	**Type: Prepare manual N_2 injection. Click OK.**
Dose	Dose	Select: **Manual Injection**	
The software automatically inserts a Stop Repeat step when a Start Repeat step is inserted. Make sure that you insert the Wait step within the Start/Stop Repeat loop.			
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Dose step and the Temperature Ramp step within the Start/Stop Record loop.			

d. Click the Report Options tab and set the values to those you wish to use.

e. Click **Save**, then **Close**.

4. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6.)

   a. Select **Unit** > **Start analysis**. From the Files list box, select the sample file you just created.

   b. You can edit the sample file if needed, but for our example, editing is not required; click **Next** to continue.

   c. The second dialog box appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select None.

   d. Click **Next**; the next Analysis dialog appears: the reports dialog. For our example, no changes are needed.

   e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis. During the analysis, you are prompted to change the dewar.

Change the dewar rapidly. It is recommended that you hold the water dewar in one hand and remove the LN_2 dewar with the other to minimize the time required.

5. When the analysis ends, remove the sample tube from the analyzer, place the caps on the tube, and dry off the outside of the tube with a paper towel.

Use the *cotton* gloves provided in the accessory kit if you wish to remove the sample tube before it has cooled. You may use rubber gloves to handle the sample tube when it has cooled.

6. Weigh the sample tube, sample, and caps. Subtract this weight from the weight you obtained for the sample tube and caps. The resulting weight is the dry sample weight (the “after analysis weight”). This weight will be used when you calculate the BET surface area, as described in the following sections.

Generating the BET Report

The following tasks must be performed before you can generate the BET report:

- Edit the sample file and the calibration file to ensure that peaks are properly marked
- Create a TCD calibration file
- Associate the TCD calibration file with the sample file

After completion of these tasks, select **Start Report** from the Reports menu to generate the report. Be sure that **BET Surface Area** is selected (preceded with a check mark).

Edit the peaks in the sample file

1. Select **Reports > Peak Editor**; the Edit Sample Peaks dialog is displayed.

2. Select the BET sample file you used with the analysis, then click **OK**.

3. Click on the desired experiment, then click **Edit Peaks**.

4. Right-click in the graph area and choose **Mark All Peaks**; all peaks that meet the current peak-picking parameters are marked.

5. If required, edit the edges of the desorption peak as follows:

The desorption peak is most often used to determine the surface area because the desorption process starts with the adsorbate equilibrated on the surface. Also, it is simple and convenient to develop a calibration for desorption.

   a. Drag the cursor over the entire baseline of the peak to enlarge the editing area.

   b. Click on the baseline of the left side of the peak, right-click and choose **Mark left edge**.

   c. Repeat step b for the right side of the peak.

6. As you adjust the peak edges, the values associated with the peak (listed in the peak table) are also adjusted. When you are satisfied with the appearance of the peak, right-click and choose **Save**.

Edit the peaks in the calibration file

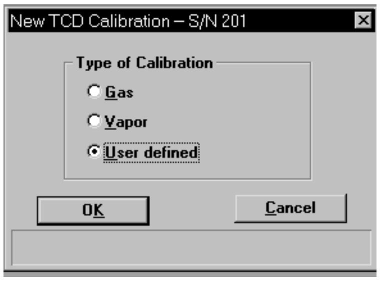
1. Select **Peak Editor** from the Reports menu; the Edit Sample Peak dialog is displayed.

2. Select the file containing the calibration data you used with the analysis, then click **OK.**

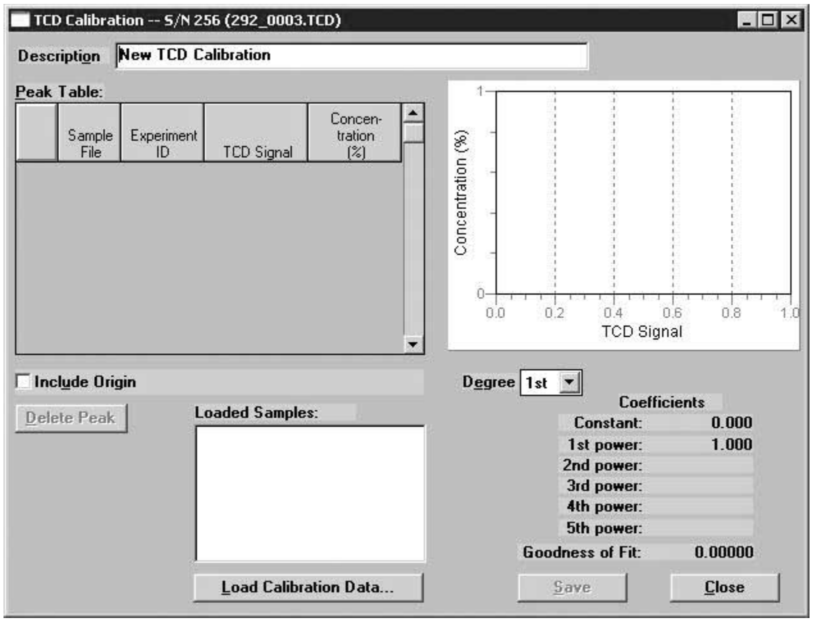
3. Edit the peaks in the same manner as described for the sample file.

Create a TCD Calibration file

1. Select **Unit > TCD Calibration > New**; the New TCD Calibration dialog box is displayed.



2. Choose **User defined**, then click **OK**; the TCD Calibration dialog box is displayed.



Description: New TCD Calibration

☐ Include Origin

Degree: 1st

Coefficients

Constant:	0.000
1st power:	1.000
2nd power:	
3rd power:	
4th power:	
5th power:	
Goodness of Fit:	0.00000

3. Click **Load Calibration Data;** the Load Calibration Sample File dialog is displayed.

4. Select the sample file containing the calibration data, then click **OK**. The data in the file are inserted into the appropriate fields of the TCD Calibration window.

5. The values in the Peak Volume column are defaults. The correct values for the volume of each injection at STP must be determined using the following formula:

   $$V_{STP} = V(\,injected\,)\left(\frac{273.15}{273.15 + ambient\ temp}\right) \times \left(\frac{ambient\ pressure}{760\ mmHg}\right)$$

   Determine the volumes for each calibration peak listed in the table.

6. Enter the converted values (cm^3 STP) into the table.

   Assess the Peak Area plot, Goodness of Fit, and Coefficients to decide if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, however, you should use a calibration file with a very low value for Goodness of Fit (less than 0.1), when 1st or 2nd Degree is specified.

   You may wish to delete any peaks that are outliers (highlight the peak data in the table and click **Delete Peak**). You may also wish to evaluate the results when you change the degree (select a different degree from the drop-down list).

7. Enter a new description into the **Description** field.

8. When you are satisfied with all values in the file, click **Save**, then **Close**

Associate the Calibration file with the Sample file

1. Select **Reports > Peak Editor**; the Edit Sample Peak dialog is displayed.

2. Select the BET sample file you used with the analysis, then click **OK**.

3. Click on the experiment, then right-click in the graph area and choose **Set Calibration**; the Set TCD Calibration dialog box is displayed.

4. Click the down arrow to the right of the Calibration field and choose the Calibration file you just created; click **OK**.

5. Repeat steps 3 and 4 for each experiment.

6. Click **Save**, then **Close.**

Pulse Chemisorption

Description

A Pulse Chemisorption analysis determines active surface area, percent metal dispersion, and active particle size by applying measured pulses of reactant gas to the sample, depending upon what technique is used. Pulse Chemisorption is performed in the same manner as TPD, except that the sample is dosed with the analysis gas using the injection loop until each active site has reacted. After the active sites have completely reacted, the discretely injected gas volumes emerge unchanged. If TPD is performed after Pulse Chemisorption, additional information about the distribution of active sites and the strength of active sites is collected.

The amount chemisorbed is the difference between the total amount of reactant gas injected and the amount that did not react with the active sites of the sample. The size of each pulse is determined by the loop, which is located on an electrically operated valve.

Loop Volume

In general, it is desirable for the sample to require at least two doses of gas, but no more than ten doses, before the reaction ends (although this may vary from lab to lab). Some factors that influence the number of doses required are sample size, the active surface area per unit of sample, and the size of the loop. If your pulse chemisorption analysis requires more doses or fewer doses than you prefer, you can change either the sample size or the loop size.

Three loops of different sizes are provided with the analyzer; each loop must be calibrated before its use. To replace or install a loop, refer to **Changing the Injection Loop** on page **9-7**. As an alternative to physically replacing the loop, more experienced operators may wish to adjust the loop volume by adjusting its temperature. If you change the loop volume by changing its temperature, results will be more accurate if you recalibrate the loop volume at the new temperature.

Loop Calibration

Before data from an analysis that uses an injection loop can be reduced, a loop calibration experiment must be performed. You can perform an independent loop calibration before or after the analysis.

The following example assumes that the loop calibration will be performed after the analysis. See the instructions later in this chapter for performing a loop calibration. You can also include a Loop Calibration step in your pulse chemisorption analysis.

Preparation

Pretreatment	Degas by flowing inert gas (such as helium, argon, or nitrogen) over the sample while ramping the temperature.
Analysis	Pulse the loop (analysis) gas over the sample until the peak area remains constant.
Cold Trap	No cold trap is needed during pretreatment During the analysis, use the cold trap fixture with a dewar of water in place as a short delay path. This delay compensates for the flow disturbance caused by each loop injection.
Pressure regulator	For Pulse Chemisorption, gas cylinders should be set to a level between 10 and 15 psig (69 and 103.5 kPag).
Furnace temperature	Select a temperature high enough to remove any contaminants or moisture, but not so high as to cause sintering or fusing of the sample. Make sure the Done step is set to return the sample temperature to ambient.

In the following example, Pulse Chemisorption is performed on a sample of Pt/Al2O3 with CO. You should alter the analysis conditions to accommodate the analysis you are conducting.

Procedure

Before performing an analysis, make sure you have adequately prepared the analyzer using the instructions in Chapter 4.

1. Obtain the sample weight, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.

2. Create a sample file for this analysis.

   a. Select **File** > **Open** > **Sample Information**. Enter a file name or accept the default; click **OK**, then **Yes** to confirm.

   b. Complete the Sample Information dialog using appropriate values.

   c. Click the Analysis Conditions tab and insert the experiment steps listed below:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select **Pulse Chemisorption** from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
		Vapor generator	20
		Reflux	20
		Flask	20
	Gas Flows	Prep	Helium
		Carrier/Reference	Helium
		Loop	None
		Flow rate	50 cm^3/min
		Valve states	Bypass, Prepare, Fill, Bypass
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: **Wait**	Type: 5 minutes

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Change Gas Flows	Gas Flows	Prep	Hydrogen
		Carrier	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Bypass
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	120
		Rate	10.0
		Hold Time	30
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	220
		Rate	10.0
		Hold Time	30
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	350
		Rate	10.0
		Hold Time	120.0
Change Gas Flows	Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Bypass
Wait	Wait	Select: **Wait**	Type: **90 minutes**
Temperature Ramp	Temperature Ramp	Type:	Return to Ambient
Wait	Wait	Select: **Wait**	Type: **30 minutes**
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	35
		Rate	5.0
		Hold Time	10.0
Change Gas Flows	Change Gas Flows	Prep	None
		Carrier	Helium
		Loop	Carbon Monoxide
		Valve states	Trap, Analyze, Fill, Bypass
Wait	Wait	Select: **Wait until baseline is stable.**	

Step to Insert	**Dialog or subdialog(s)**	**For this parameter:**	**Use this value:**
Recording	Start Recording	One measurement every:	0.2 second
Wait	Wait	Select: **Wait**	Type: **3.0 minutes**
Repeat	Start Repeat Sequence	Select: **Repeat until peaks are equal or 20 times.**	
Dose	Dose	Select: **Inject Loop Gas**	
		Select: **Wait for**	Type: **1.5 minutes**
The software automatically inserts a Stop Repeat step when a Start Repeat step is inserted. Make sure that you insert the Dose step within the Start/Stop Repeat loop.			
The software automatically inserts a Stop Record step when a Start Record step is inserted. Make sure that you insert the Start/Stop Repeat step and the Dose step within the Start/Stop Record loop.			
Done	Done (Click **Edit**, if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable

d. Click the Report Options tab and set the values to those you wish to use.

e. Click **Save**, then **Close.**

3. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6.)

a. Select **Unit** > **Start analysis**. From the Files list box, select the sample file you just created.

b. You can edit the sample file if needed. For our example, no editing is needed; click **Next** to continue.

c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.

d. Click **Next**; the next Analysis dialog appears: the reports dialog. For our example, no changes are needed.

e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.

When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, you may wish to use the optional CryoCooler or KwikCool.

Use the *cotton* gloves provided in the accessory kit if you wish to remove the sample tube before it has cooled. You may use rubber gloves to handle the sample tube when it has cooled.

When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

Generate the Report

Before proceeding with the steps below, you may wish to open the sample file using the Peak Editor on the Reports menu to ensure that the peaks are properly marked.

There are three possibilities for each dose of gas injected during Pulse Chemisorption: all of the gas is taken up by the sample, some of the gas is taken up by the sample, or none of the gas is taken up by the sample. When Pulse Chemisorption is properly performed, there will be some injections of each type. When you view the data using the Peak Editor, however, only those injections in which some or none of the gas is taken up will appear as peaks. When all of the gas is taken up by the sample, none of it reaches the detector and, therefore, the peak area is zero. These types of peaks are detected automatically by the software and do not require marking.

After verifying that the peaks are properly marked, perform the following steps to generate the report:

1. Select **File > Open > Sample Information**.
2. Select the sample file used with this analysis and click **OK**.
3. Click the Collected Data tab to display its associated dialog box.
4. Enter the percent of active gas in the **Active concentration** field.
5. Click **Active Metals**; the Active Metals Table is displayed. Verify that the **Stoichiometry Factor** and the **% of Sample Weight** values are correct.
6. Click **OK** to close the Active Metals dialog box.
7. Verify that the values for the physical injection volume, ambient temperature, and ambient pressure are accurate.
8. Select **Reports > Start Report** to generate the report.

Loop Calibration

You must calibrate each injection loop before its first use to determine its precise volume under local conditions. Calibration consists of:

- Determining the average area of a series of peaks generated by injections of a known volume of gas through the analyzer septum using a syringe.
- Determining the average area of a series of peaks generated by injections of the same gas using the analyzer's internal loop.
- Calculating the volume of the loop by comparing the average peak area generated by the loop injections with that generated by the syringe injections.

Update Ambient Pressure and Temperature

Ambient pressure and temperature at the time of the analysis affect the results of a loop calibration. In calculations, the analyzer uses the ambient pressure and temperature recorded in the Environmental Defaults dialog box (accessed from the Options menu). You may wish to check and update your environmental defaults before beginning a loop calibration. If necessary, you can also update the ambient temperature and pressure on the Collected Data dialog of the sample information file after the analysis. (The sample information file must be opened in Advanced presentation in order to view or edit collected data.)

Create a Sample File

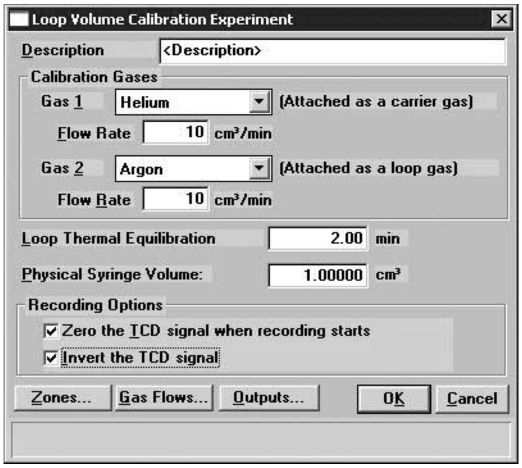
First, you must create a sample file:

1. Select **Open**, **Sample information** from the File menu; the Sample Information File selection dialog appears.
2. Enter the name you wish to use for this calibration in the **File name** field. Click **OK**, then **Yes** to confirm.

If the file is in Basic presentation format, click Advanced to switch to the Advanced format.

3. Complete the fields of the Sample Information dialog as needed.
4. Click the **Analysis Conditions** tab. Click **Insert**, then select **Loop Calibration** from the list of available steps.

5. Complete the Loop Calibration Experiment Step dialog using Helium for Gas 1 and Argon for Gas 2.



Loop Volume Calibration Experiment

Description: <Description>

Calibration Gases

Gas 1: Helium (Attached as a carrier gas)

Flow Rate: 10 cm³/min

Gas 2: Argon (Attached as a loop gas)

Flow Rate: 10 cm³/min

Loop Thermal Equilibration: 2.00 min

Physical Syringe Volume: 1.00000 cm³

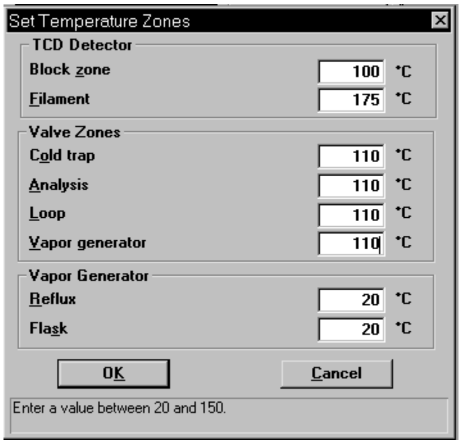
Recording Options

☑ Zero the TCD signal when recording starts

☑ Invert the TCD signal

6. Click **Zones** to set the temperature for the analyzer’s heated zones. Complete the fields of the Temperature Zones dialog box as shown below, using 175 °C for the filament and 100 °C for the block zone. Use the defaults for all other zones. Click **OK** to close the dialog.

You must create a Loop Calibration for each loop you use, using the temperatures used in the analyses that will be associated with the calibration file. If you use different temperatures for different analyses, you must create a separate calibration file for each temperature configuration.



Set Temperature Zones

TCD Detector

Block zone: 100 °C

Filament: 175 °C

Valve Zones

Cold trap: 110 °C

Analysis: 110 °C

Loop: 110 °C

Vapor generator: 110 °C

Vapor Generator

Reflux: 20 °C

Flask: 20 °C

Enter a value between 20 and 150.

7. Click **Save**, then **Close**.

Perform the Analysis

1. Make sure the injection loop you wish to calibrate is installed on the analyzer. Refer to **“Changing the Injection Loop” on page 9-7** for the steps to install or replace an injection loop.

2. Select **Start Analysis** from the Unit menu; select the sample file you just created.

3. Click **Next** three times to accept the values contained in this sample information file. (Although you can edit these values, no changes are needed for this example). Read the cautionary statement, then click **Start** to start the analysis.

4. As the test progresses, you are prompted to make three manual injections.

   For this example, use argon for your injections (because argon is specified as the loop gas in the sample information file). Use a volume that is close to the volume of the loop you are calibrating. For example, if you are calibrating the 1-cc loop, use a 1-cc syringe and inject as close to 1 cc of gas as possible.

Pay close attention to the instructions given in prompts that appear on the screen. Perform the prompted steps in the order given. For example, if you are prompted “Click OK, then perform manual injection,” you should perform these actions in the order given. Otherwise, data may not be recorded properly

Most accurate data results from keeping injection size as consistent as possible. Injection errors may be evident in the data, and may make it necessary to repeat the experiment.

After the third manual injection, the analyzer automatically makes three injections using the loop.

Generate the Report

Each manual and automatic injection results in a peak (if it has been properly performed). When the Results view of the Start Analysis window is selected, each peak can be viewed as it is collected. The area under the peak corresponds to the amount of argon injected.

Before proceeding with the steps below, you may wish to open the sample file using the peak Editor on the Reports menu to ensure that the peaks are properly marked. Instructions for using the Peak Editor are located on page **7-14**.

1. Select **Start Report** from the Reports menu.

2. Select the sample file you created for this calibration; click **OK**.

3. Deselect all the reports in the reports list box except the Loop Calibration Report; click **OK**.

4. Select **Unit > Unit Configuration** and enter the value printed for the **Computed Loop Volume @ 0 ºC** in the **Loop Volume** field.

Reports use the loop volume from the System Configuration dialog (Unit menu). Environmental conditions are taken from the Environmental Defaults dialog on the Options menu. If these values were incorrect during analysis, you can change them for this sample file by clicking the sample file's Collected Data tab and updating the appropriate fields.

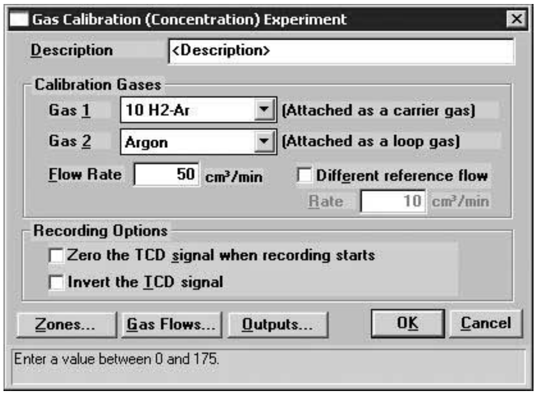
Gas Concentration Calibration

After you perform an analysis, you may wish to convert the peak area data it yielded to volume data. The analyzer can do this automatically, but it must first have some additional information.

In some cases (such as in TPR analyses), the analyzer needs to know how to correlate the signal readings collected in the analysis with the volume of gas uptake at any given point in the analysis. If we provide the analyzer with a series of known gas concentrations, and it records the signal associated with each known concentration, then it can use this information to calculate the concentrations associated with the signals it recorded during the analysis. From that point, it can also calculate the volume of gas associated with each peak in the data.

1. Prepare a clean, empty sample tube and install it on the analyzer.
2. Make sure the correct gases are connected. Also check to be sure that the Gas Selections table (Unit menu) accurately reflects the current gas connections.
3. Create the sample file:
    a. Select **File > Open > Sample information**; the sample information file selection dialog is displayed.
    b. Enter a name (or accept the default). Click **OK**, then **Yes** to confirm,
    c. Complete the Sample Information dialog as needed.
4. Click the **Analysis Conditions** tab. Click **Insert** and select **Gas Calibration**; the Gas Calibration (Concentration) Experiment dialog is displayed.

The values shown and given in this example are for a typical TPR calibration.


Gas Calibration (Concentration) Experiment

Description <Description>

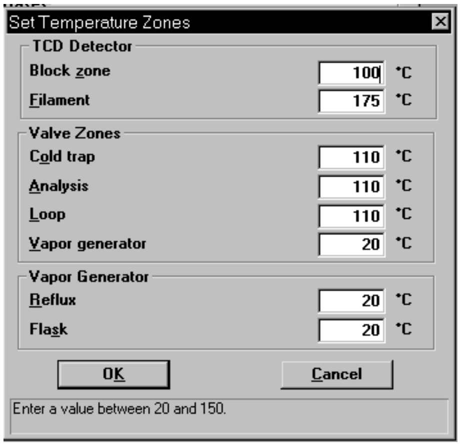
Calibration Gases
Gas 1: 10 H2-Ar (Attached as a carrier gas)
Gas 2: Argon (Attached as a loop gas)
Flow Rate: 50 cm³/min
☐ Different reference flow
Rate: 10 cm³/min

Recording Options
☐ Zero the TCD signal when recording starts
☐ Invert the TCD signal

Enter a value between 0 and 175.

5. Choose **10 H2-Ar** for **Gas 1** and **Argon** for **Gas 2**, then enter **50** as the **flow rate**.

6. Click **Zones** to set the temperature for the analyzer's heated zones. Complete the fields in the Set Temperature Zones dialog box as shown below.



Set Temperature Zones

TCD Detector
Block zone 100 °C
Filament 175 °C

Valve Zones
Cold trap 110 °C
Analysis 110 °C
Loop 110 °C
Vapor generator 20 °C

Vapor Generator
Reflux 20 °C
Flask 20 °C

Enter a value between 20 and 150.

7. Click **OK** to close the Zones dialog, then again to close the Calibration dialog.

8. Click **Save**, then **Close**.

9. Select **Unit > Start Analysis**. From the Files list box, select the sample information file you just created; the Analysis dialog box is displayed.

10. Click **Next** three times to accept the values contained in this sample information file. (Although you can edit these values, no changes are needed for this example.)

11. Click **Start** to start the analysis.

    At this time, you can use the other views of the Analysis dialog to observe the progress of the analysis.

    During the automatic analysis, the analyzer decreases the proportion of the analysis gas in 10% increments, beginning with 100% and ending with 0%. The resultant data should appear as a series of ten stepwise changes in the TCD signal.

12. When the analysis ends, close the Analysis dialog.

13. When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

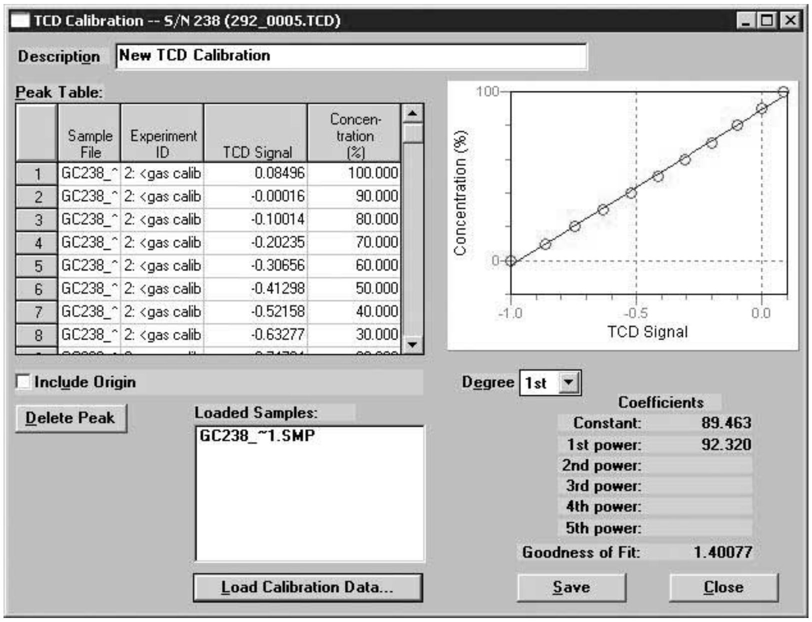
Use the *cotton* gloves provided in the accessory kit if you wish to remove the sample tube before it has cooled. You may use rubber gloves to handle the sample tube when it has cooled.

14. Select **Unit > TCD Calibration.> New**. Select **Gas**, then click **OK**; the TCD Calibration window opens.

15. Click **Load Calibration Data**; select the sample information file for this calibration and click **OK**. The TCD Signal vs. Concentration dialog box is displayed.

16. Verify that the active gas concentration for the carrier gas is correct; edit if necessary.

17. Click **OK**; the data contained in the sample file are automatically inserted into the appropriate fields of the TCD Calibration dialog box.



TCD Calibration -- S/N 238 (292_0005.TCD)

Description: New TCD Calibration

Peak Table:

	Sample File	Experiment ID	TCD Signal	Concentration (%)
1	GC238_^	2: <gas calib	0.08496	100.000
2	GC238_^	2: <gas calib	-0.00016	90.000
3	GC238_^	2: <gas calib	-0.10014	80.000
4	GC238_^	2: <gas calib	-0.20235	70.000
5	GC238_^	2: <gas calib	-0.30656	60.000
6	GC238_^	2: <gas calib	-0.41298	50.000
7	GC238_^	2: <gas calib	-0.52158	40.000
8	GC238_^	2: <gas calib	-0.63277	30.000

☐ Include Origin

Loaded Samples: GC238_~1.SMP

Degree: 1st

Coefficients

Constant:	89.463
1st power:	92.320
2nd power:	
3rd power:	
4th power:	
5th power:	
Goodness of Fit:	1.40077

18. Assess the data listed in the table. In the **Concentrations** column, enter the starting and ending gas mixture percentages. The analyzer calculates the percentages for the rest of the table.

19. Assess the gas concentration **plot**, **Goodness of Fit**, and **Coefficients** to determine if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, however, you should use a calibration file with a very low value for Goodness of Fit (less than 1/2% of the maximum concentration), when 1st or 2nd Degree is specified.

    You may wish to delete any data point that deviates significantly from linear. You may also wish to evaluate the results when you change the degree (select a different degree from the drop-down list).

20. When you are satisfied with the calibration file, enter an appropriate name in the Description field.

21. Click **Save** then **Close**.

22. If you wish to generate a report, select **Unit** > **TCD Calibration** > **Report** and select the desired file.

Calibrating the Vapor Generator

When you use the optional Vapor Generator, you must calibrate it for the type of vapor you are using. Associating your analysis data with a Vapor Generator calibration file enables you to convert your peak area data to volume data.

You can perform the Vapor Generator calibration either before or after performing your analysis. A single calibration file can be used with multiple analysis files.

1. Select **File > Open > Sample information**; the Sample Information File selection dialog appears.

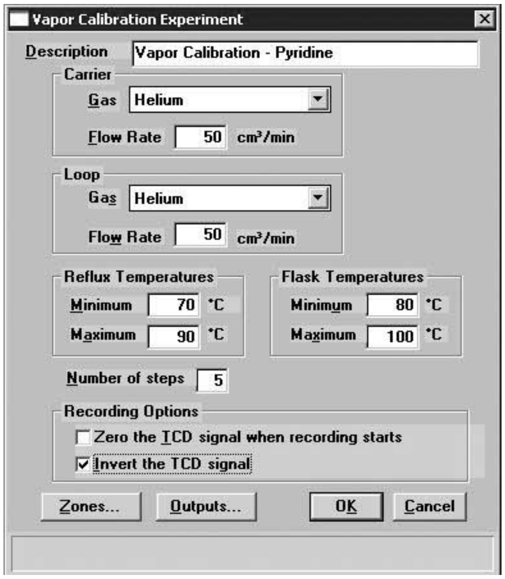
2. Enter the name you wish to use for this calibration in the **File name** field. Click **OK**, then **Yes** to confirm.

If the file is in Basic presentation format, click Advanced to change to the Advanced format.

3. Complete the fields of the Sample Information dialog as needed.

4. Click the **Analysis Conditions** tab. Click **Insert**, then select **Vapor Calibration**; the Vapor Calibration Experiment dialog is displayed.

The values shown and given in this example are for a typical Vapor calibration for pyridine.



Description: Vapor Calibration - Pyridine

Carrier
Gas: Helium
Flow Rate: 50 cm³/min

Loop
Gas: Helium
Flow Rate: 50 cm³/min

Reflux Temperatures
Minimum: 70 °C
Maximum: 90 °C

Flask Temperatures
Minimum: 80 °C
Maximum: 100 °C

Number of steps: 5

Recording Options
☐ Zero the TCD signal when recording starts
☑ Invert the TCD signal

5. Complete the dialog as follows:

a. Choose **Helium** for the Carrier and Loop gases, then enter a flow rate of **50** for each.

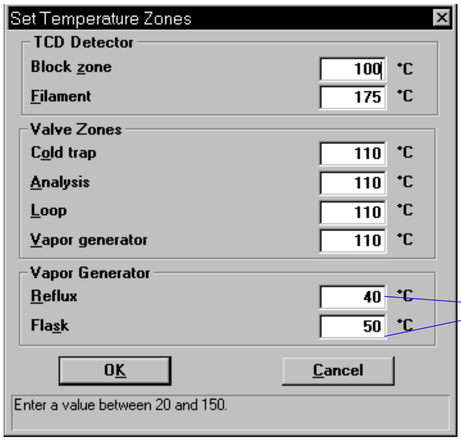
b. For the **Reflux** temperature, enter a minimum of **70** and a maximum of **90**.

c. For the **Flask** temperature, enter a minimum of **80** and a maximum of **100**.

d. Enter **5** for the number of steps.

e. Select **Invert the TCD Signal**. It is a good idea to select this option when using Helium for the carrier gas; it improves the performance of automatic peak picking.

f. Click **Zones**; specify temperatures as shown here:



Set Temperature Zones

TCD Detector

Block zone: 100 °C

Filament: 175 °C

Valve Zones

Cold trap: 110 °C

Analysis: 110 °C

Loop: 110 °C

Vapor generator: 110 °C

Vapor Generator

Reflux: 40 °C

Flask: 50 °C

Enter a value between 20 and 150.

You do not have to edit these fields; the Reflux and Flask temperatures entered on the Vapor Calibration Experiment dialog are used.

6. Click **OK** to close the Set Temperature Zones dialog, then again to close the Vapor Calibration Experiment dialog box.
7. Click **Save**, then **Close** to close the sample information file.
8. Select **Unit** > **Start Analysis**. From the Files list box, select the sample information file you just created; the Analysis series of dialogs is displayed.
9. Click **Next** three times to accept the values contained in this sample information file. (Although you can edit these values, no changes are needed for this example). Click **Start** to start the analysis.
10. At this time, you can use the other views of the Analysis dialog box to observe the progress of the analysis. Complete descriptions of these views are located in Chapter 6.
11. When the analysis ends, close the Analysis dialog.
12. When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

Use the *cotton* gloves provided in the accessory kit if you wish to remove the sample tube before it has cooled. You may use rubber gloves to handle the sample tube when it has cooled.

13. Edit the calibration file.

    a. Select **Unit > TCD Calibration > New**.

    b. Select **Load Calibration Data** and select the sample information file for this calibration; the data contained in the file are automatically loaded.

    d. Refer to **Vapor Generator** in Appendix B, page **B-9** for calculating the volume of each vapor injection, then enter the values for each peak.

    e. Assess the plot, Goodness of Fit, and Coefficients to decide if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, however, you should use a calibration file with a very low value for Goodness of Fit (less than 1 to 2% of the signal), when 1st or 2nd Degree is specified.

    f. You may wish to delete any peaks that are outliers (highlight the peak data in the table and click **Delete Peak**). You may also wish to evaluate the results when you change the degree (select a different degree from the drop-down list).

    g. When you are satisfied with the calibration file, click **Save** then **Close**.

Reports on calibration files can be printed by selecting TCD Calibration, Reports from the Unit menu.

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