Thermo
ELECTRON CORPORATION

TRACE GC

Gas Chromatograph Setup

User's Manual

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Chapter 1
TRACE GC Ultra Configuration

This chapter contains the instruction to configure your TRACE GC Ultra.

This chapter contains these topics:

1.1 Introduction

This dialog box is available from the Instrument Configuration window.
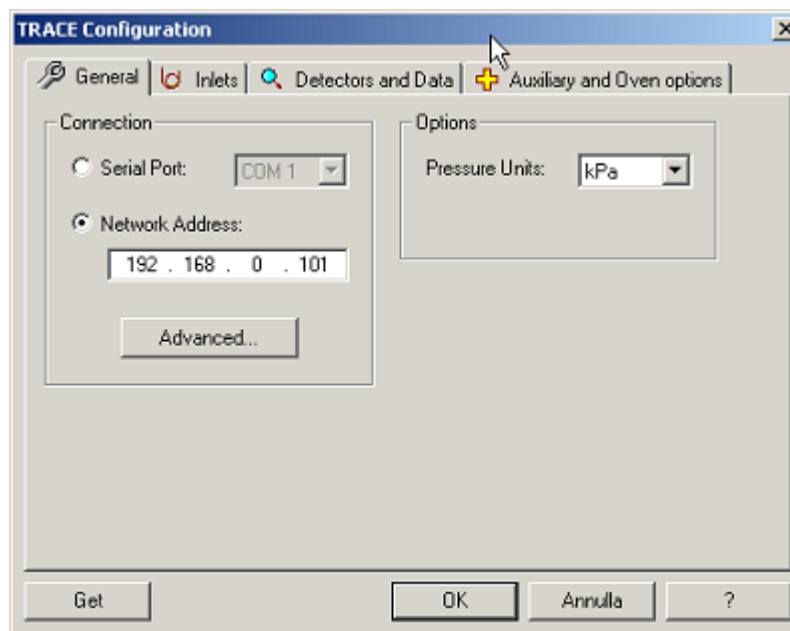
Figure 1-1. TRACE GC Ultra Configuration Window

Select the TRACE GC Ultra and then select the configure button. The following TRACE GC Ultra detector pages display:

• *General Page*

• *Inlets Page*

• *Detectors and Data Page*

• *Auxiliary Page*

GET   Use this button any time you want to automatically enter the configurations already entered into your TRACE GC Ultra. This program automatically enters the exact configuration parameters that were programmed into the TRACE GC Ultra key panel (embedded system). However, you will still have to enter the channel parameters located on the Detectors and Data page.

To open this view:

1.   Choose Start | Programs | Xcalibur | Instrument Configuration

2. Click the TRACE GC Ultra button located in the Available Devices column.

3. Click Add, then Configure.

1.2 General Page
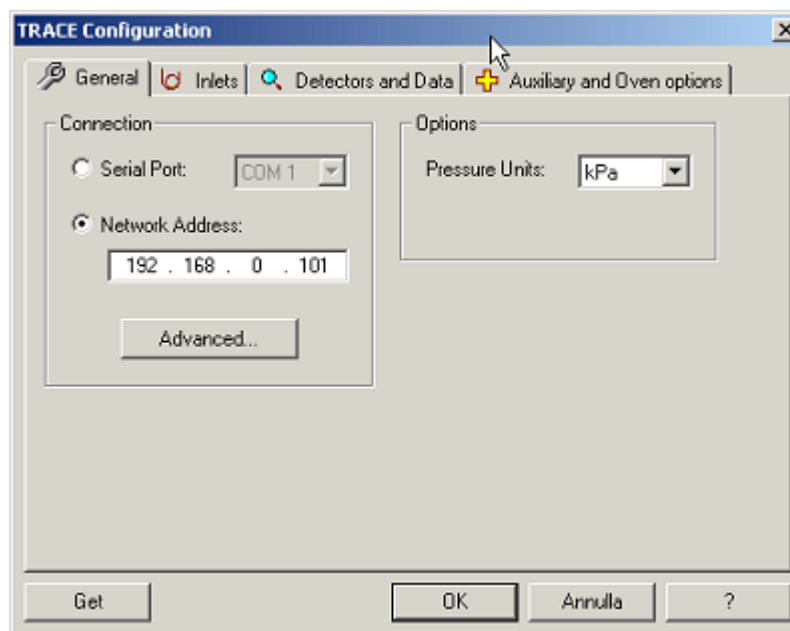
Figure 1-2. General Page

Connection Group Box

Serial Port   Select the COM port in which your TRACE is connected. A COM port is a 9-pin cable connection located on the back of your computer.

Network Address   Enter the I.P. address to allow the LAN control of the GC through the Thermo data systems.

ADVANCED   Select this button to set the LAN communication port used by the TCP-IP ptotocol and timeout.

Options Group Box

Pressure Units   Specify the pressure unit measurement to use in your work sessions. Pressure measurement units to select include kPa, bar, and psi.

1.3 Inlets Page

Use this page to select the inlets installed on the top of your TRACE GC Ultra. The inlet you select determines which options are available on this page and the instrument setup (method editor) pages.
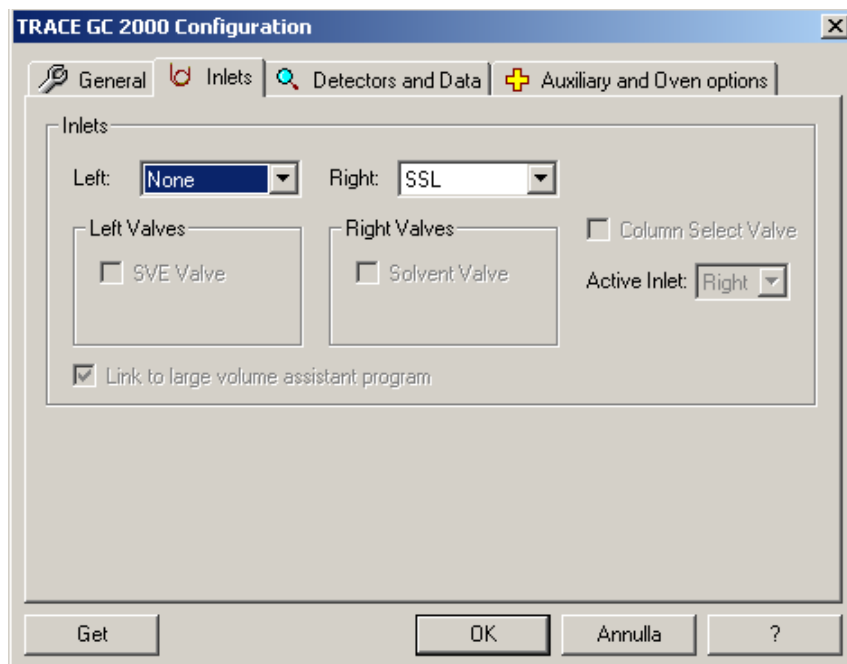
Figure 1-3. Inlet Page.

Inlets Group Box

Choose the inlet installed on the left or right side of your TRACE GC Ultra.

Left Inlets   Choose one: None, SSL, OCI, HOTOC, LVOCI, PKD, PPKD

Right Inlets   Choose one: None, SSL, PTV, PKD, PPKD

Left and Right Valves Group Box

Select the following to further control your selected inlet.

SVE Valve   Check this box to display **Sub Ambient** controls in the selected inlet's instrument setup pages. This allows you to enter cryogenic programmable entries below zero if you have installed cryogenic hardware (optional purchase). Additionally, SVE Valve allows volatile vapor to bypass the

analytical column. Typically used with a large volume injector. If you have the cryogenic hardware installed be sure that configurations have been set manually into the TRACE GC Ultra using the TRACE GC Ultra keypad.

Solvent Valve   Check this box to use a split valve that is attached to the GC top. This selection prevents condensation from reaching the split valve in a large volume injection when using a PTV.

Column Select Valve   Check this box when using a column select valve with a single DPFC and two inlets. The carrier gas diverts to the selected inlet that you activate.

Active Inlet

When you select Column Select Valve, specify which inlet you want to exclusively use. When you activate an exclusive side, the other inlet side defaults to no settings. Carrier gas diverts to the specified inlet.

Link to large volume assistant program

Checking this box allows you to use the Run Large Volume Program found on the autosampler menu. However, it does disable other functions found on other method editor pages (see the note below). This box is available only after selecting LVOCI left inlet. Check this box only if you plan to use a LVCI to run the Large Volume Program.

Checking this box disables the initial oven temperature controls in instrument stup. This is because Large Volume Assistant Program sets the temperature automatically. If you want to activate the initial oven temperature control on te Oven page located in instrument setup, then uncheck this box.

1.4 Detectors and Data Page

Use this dialog tab to configure the detectors you will use with your TRACE GC Ultra. If you disconnect your detector from your GC, remember to reconfigure your TRACE here and from the TRACE GC Ultra front panel. Detectors chosen here display instrument setup pages and list in the Detector Events located in the Run Table page.
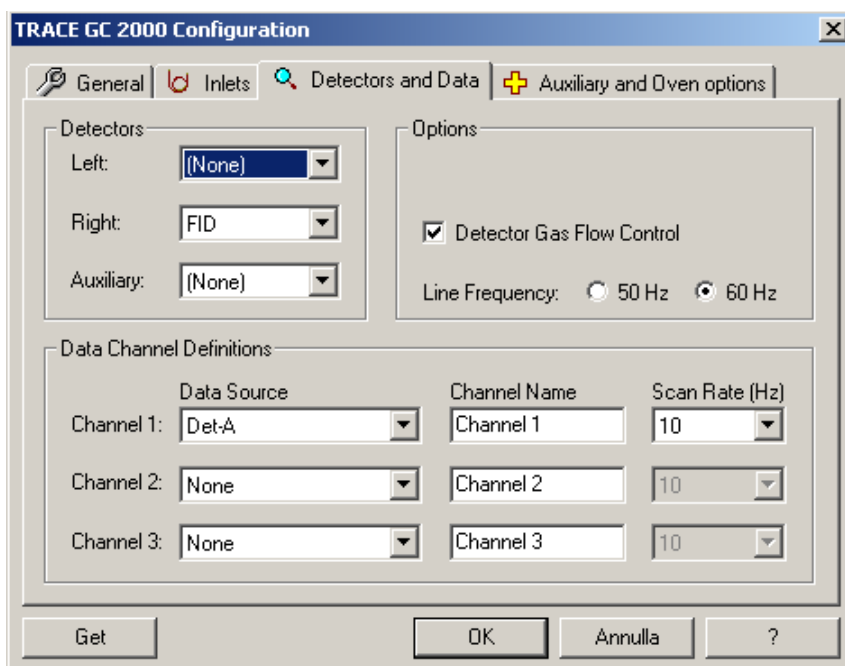
Figure 1-4. Detector and Data Page

Configuration changes are stored with a method's configuration file. A method uses the stored configurations so that a change in configuration results in making the method incompatible. You will not be able to load a method if it was created with a different configuration. Remember that you can upload a method from the GC to save with the new method.

Detectors group box

Right   Select the detector type (None, FID, ECD, NPD, TCD, FPD, PID) installed on your GC's right inlet connection. The right side is as you are facing the front.

Left   Select the detector type (None, FID, ECD, NPD, TCD, FPD, PID) installed on the GC's left inlet connection. The left side is as you are facing the front.

Auxiliary   Select the detector type (None, FID, NPD, FPD) installed on the GC's AUX position.

Option Group Boxes

Detector Gas Flow Control   Configuration option for the Detector method editors. The default for this check box is checked (on), in which case you get the detector method editor controls with Flow text-edit controls. If you unselect this checkbox, then all detector gas flow text-edit boxes in the detector method editors are removed then you may check (on) or uncheck (off) the gas flow check boxes. In other words, if this configuration option is on then you can turn detector gases on and off and specify their flow rates in the text boxes. If this option is off then you can only turn the gas flows on & off.

Line Frequency   Select the AC power frequency (50 Hz or 60 Hz) that your GC is plugged into. The line frequency control indicates allowable scan rate values.

Data Channel Definition Group Boxes

Channel 1 -3   Channels 1 through 3 are the data source connected to your TRACE GC Ultra. Select the detector card position located nearest to the front of the GC as Det-A. The detector card just behind Det-A is Det-B, and the detector card closest to the rear of the GC is Det-C. Other data source connections include Oven Actual, Oven % Power, Oven Exhaust % Open, Chassis Temp, and Line Voltage.

Channel Name   Type a name for the channel's data source displaying the chromatogram during runtime. For example if you have an FID installed on the right position of your TRACE GC Ultra type Right FID.

Scan Rate (Hz)   elect the number of data points to take per second during the acquisition.

1.5 Auxiliary Page

These controls allow you to configure and use an auxiliary interface. Your selections on this page directly influence the controls you will use on the Aux Zones page located in the Instrument Setup window.Whatever configurations you manually enter into your TRACE GC Ultra must *exactly* match what controls you enter into this Auxiliary configuration dialog.
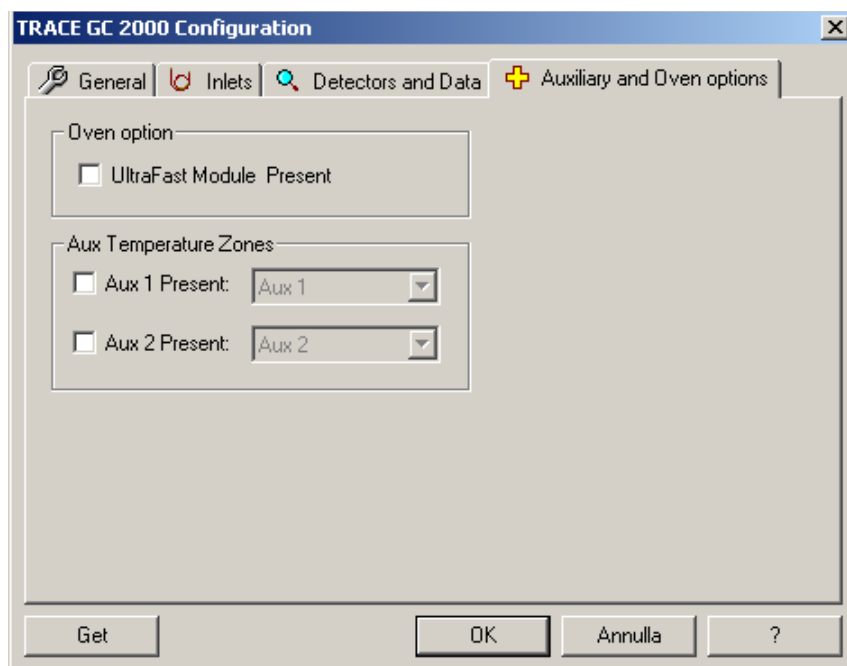
Figure 1-5. Auxiliary Page.

Aux Temperature Zones Group Box

• Check the Aux 1 Present control if you have one of these devices attached: Aux1, Valve Oven , or MS Transfer Line

• Check the Aux 2 Present if you have one of these devices attached: Aux 2 , Jet Separator, and Open Split

Aux Pressure Zones Group Box

• Check Aux 1 –3 Present controls if you will use auxiliary pressure zones during your data acquisition.

Oven Option Group Box

Check the Ultra Fast Module Present box if the **UltraFast Module (UFM)** device is installed inside the GC.
See also *UltraFast Module*

UltraFast Module   The UFM is a GC device allows the direct heating of the analytical capillary column properly installed inside the GC oven. The UFM device integrates a GC capillary column with the components for its temperature control. Compared to conventional air circulating oven, this device features faster temperature programming.

Auxiliary Interface   An optional piece of equipment that must be used as the interface between the GC and MS if carrier gas flow rates in excess of 4 mL/min through the GC column are required for the chromatographic separation. This is true when GC columns with inner diameters of 0.53 mm or larger are used. There are two Auxiliary Interfaces available for use with the TRACE GC Ultra; a jet separator and an open split .

Valve Oven   This is an independent heat zone, other than the analytical oven, where valves are kept at a temperature to prevent analyte from condensing inside them. Valves are often used in GC analysis for introducing sample into the analytical column (sampling valve) or for enhancing the analytes separation from other compounds in the sample matrix (column switching valve). Some gas analyses require the use of two or more columns. The first column would be used to hold some compounds while allowing others to pass on to a second column for back-flushing to vent or for subsequent analysis. Valves are mainly used in gas samples analysis; although liquid pressurized gas samples may be analyzed using a liquid sampling valve. It is critical for the valve or valves to be heated and to be in a purged in a closed environment such as an oven, which prevents atmospheric gases from leaking into them. This would apply to the gas sample analysis for the components that are in air, such as oxygen or nitrogen.

transfer line   A heated tube that the GC column effluent passes through to enter the ion source of the MS detector.

jet separator   An optional Auxiliary Interface available for use with the GCQ data system. The jet separator is sometimes referred to as a momentum separator or an enrichment device. A jet separator is used to reduce the flow of the column effluent eluting from the GC column to a lower one that is compatible with the vacuum system of the GCQ and TRACE.

A jet separator consists of two quartz jets that are separated by a small gap, which is held under vacuum. The first jet is connected to the end of the GC column where it receives the GC column effluent as it elutes from the column. The second jet is connected to a piece of tubing (1/16 inch i.d., glass-lined stainless steel or 0.53 mm i.d. deactivated fused silica) that is

connected to or passes through the transfer line of the GCQ. As the column effluent exits the first jet it must jump across the gap into the second jet to be transferred to the MS. Because the carrier gas has a low molecular weight, it tends to diffuse (and its stream spreads out in space) as it exits the first jet; thus, only a small percentage of the carrier gas makes it into the second jet. Analyte compounds are of higher molecular weight than the carrier gas, and do not spread out as much in space as they cross the gap. Thus, a higher percentage of the analytes (relative to carrier gas) are able to cross the gap into the second jet and move into the MS. This acts as a discriminatory split, such that the flow volume is reduced but the analyte concentration is enriched in the exiting gas stream.

See also: *open split*.

open split   An optional Auxiliary Interface . An open split interface is used to reduce the flow of the column effluent eluting from the GC column to a lower one that is compatible with the vacuum system of the TRACE GC Ultra. An open split interface typically consists of a column transfer line with a mass spectrometer transfer line placed near, or inserted into, that column transfer line. The system may also use an additional makeup or purge gas for finer flow control. The column transfer line is typically of a wider bore than the mass spectrometer transfer line and as such, only a specific percentage of the column effluent enters the mass spectrometer. This split is non-discriminatory, that is, an approximately equal percentage of both sample and carrier gas is split away from the mass spectrometer.

See also:*jet separator*.

1.6 Road Map Home Page Status Tabs

Status pages for your TRACE GC Ultra are located on your Xcalibur Roadmap-Home page. Just highlight TRACE GC Ultra from the Roadmap Status tab scroll list and see the following pages (status, temperatures, flows, and pressures) display the current GC status.

Status Tabs
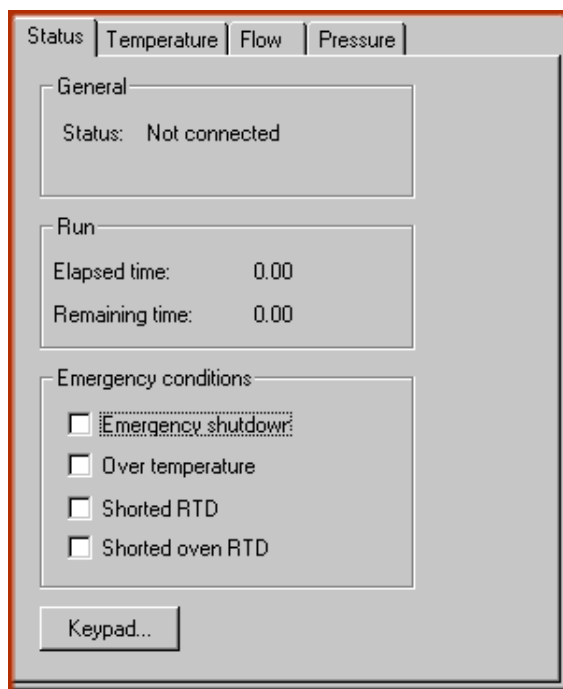
Figure 1-6. Status Tabs

- *Status*

- Temperatures

- Flows

- Pressures

Status

General Group Box

Status: Indicates if instrument is communicating to Xcalibur.

Run Group Box

Elapsed time The amount of time that has elapsed since the run started.

Remaining time The amount of time that remains before the run finishes.

Emergency Conditions Group Box

Emergency shutdown Your Xcalibur software automatically checks this box if an error condition like a leaking hydrogen sensor or something is malfunctioning internally in your TRACE GC Ultra. The software automatically shuts the TRACE GC Ultra off. Check your TRACE GC Ultra Maintenance and Troubleshooting Manual for possible solutions.

Over temperature This box is checked only when Xcalibur software detects the temperature is over the recommended level.

Shorted RTD This detected problem requires certified technical support. Contact your local TMQ Tech Support office for service.

Shorted oven RTD This detected problem requires certified technical support. Contact your local TMQ Tech Support office for service.

Temperatures page

These values show the actual and setpoint Temperature parameters for your TRACE GC Ultra.

Flows page

These values show the actual and setpoint Flow parameters for your TRACE GC Ultra.

Pressure page

These values show the actual and setpoint Pressure parameters for your TRACE GC Ultra.

Keypad

Located in your Instrument Setup TRACE menu. Your Xcalibur virtual TRACE GC Ultra Keypad operates just like if you were using it from the TRACE GC Ultra front panel. The only difference is where you are pressing the keys; from the instrument panel or your Xcalibur software. You might find it helpful to learn the TRACE Keypad according to the following groups:

Action Keys: Stop, Prep Run, and Start.

Zone and Device Information Keys: Oven, Left Inlet, Right Inlet, Left Detect, Right Detect, Aux, Left Carrier, Right Carrier, Left Signal, Right Signal.

Instrument Setup Keys: Leak Check, Column Eval, Config

Shortcut Keys: Temp, Press, Flow, Time, Ramp #

Data Entry Keys: On/Yes, No/Off, Mode/Type, ¥, Clear, Enter, st, 0-9

Information Keys: Status, Info/Diag, Run Log

Method Storage and Automation Keys: Load, Store, Method, Seq, Edit/Active, Run Table, Clock Table, Auto Sampler, Valves, Seq Control.

Look for specific instructions for operating each key in the TRACE GC Ultra Operating Manual, Chapter 2.

Chapter 2
TRACE Menu

This chapter describes the Instrument Setup menu.

This chapter contains these topics:

2.1 Menu Description

The TRACE menu is located on the Instrument Setup-menu bar.
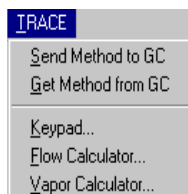
Figure 2-1. Menu

The pull-down mwnu contains the following options:

Send Method to GC command

Downloads the GC portion of the current method from the Instrument Setup window to the gas chromatograph.

Get Method from GC command

Uploads the current method from the gas chromatograph to the Instrument Setup Window.

Keypad

Located in your Instrument Setup TRACE menu. Your Xcalibur virtual TRACE GC Ultra Keypad operates just like if you were using it from the TRACE GC Ultra front panel. The only difference is where you are pressing the keys; from the instrument panel or your Xcalibur software. You might find it helpful to learn the TRACE Keypad according to the following groups:

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Shortcut Keys: Temp, Press, Flow, Time, Ramp #

Data Entry Keys: On/Yes, No/Off, Mode/Type, ∞, Clear, Enter, ▲▼, 0-9

Information Keys: Status, Info/Diag, Run Log

Method Storage and Automation Keys: Load, Store, Method, Seq, Edit/Active, Run Table, Clock Table, Auto Sampler, Valves, Seq Control.

Look for specific instructions for operating each key in the *TRACE GC Ultra Operating Manual*, Chapter 2.

Flow Calculator

Column Flow Calculator allows you to make changes in non-DPFC pneumatics, which affect carrier gas flow and velocity.

Additionally you may:

- Change the **column length, id (inner diameter), temperature, or inlet pressure** by moving any of the **"Column Parameters** slider bars will change the calculated values for flow, velocity, and hold-up time. (Changing **outlet pressure** or **carrier gas type** will also result in recalculation of the carrier flow and velocity, but they are set on different parts of the screen because they are less likely to be varied during method development than the other column parameters.)

- Change the **"Carrier Gas Parameters** slider bars for either flow or linear velocity***** will calculate the inlet pressure needed to give that flow rate or velocity, keeping the other column parameters constant.

- See the **Flow, linear velocity**, and **hold-up time** simultaneously when a column parameter changes.

While all calculations are solved directly, calculating inlet pressure from velocity (at greater than zero outlet pressure) is done using an approximation technique.

For more information, refer to:

- *Using Flow Calculator*

Vapor Calculator

Calculates the vapor volume for the PTV and SSL inlets from the injection volume in proportion to the installed liner column. Assuming, if the vapor behaves like an ideal gas and only solvent vapor needs to be considered for dilute samples: vapor volume (V) for n solvent moles at temperature T and pressure p is then approximated by the ideal gas law: $V = nRT / p$.

Absolute temperature and pressure are used for the calculations, and n is

determined from the solvent volume, density, and molecular weight. Since 1 mole of an ideal gas occupies 22.4 liters at 0° C and 1 atmosphere pressure, vapor volume in microliters can be calculated from the following equation:

V = [22.4 x] [r / MW] [(T + 273) / 273] [patm / (pi + pa)] [Vinj]

where:

V = vapor volume, microliters, at inlet temperature and pressure

Vinj = solvent (liquid) volume, microliters injected

r= solvent (liquid) density, g/ml

MW= solvent molecular weight, g/mole

22.4= liters occupied by 1 mole of ideal gas at 0 °C and 1 atm pressure

T= inlet temperature, °C (absolute temperature = °K = °C + 273)

pi = inlet pressure, gauge (absolute pressure = pi + pa)

patm = 1 atm pressure (14.7 psi, 101 kPa, or 1.01 bar)

pa = ambient pressure, usually taken as patm for this approximation

Solvent boiling points, molecular weights, and densities can be found in references such as the Handbook of Chemistry and Physics (CRC Press) and the Merck Index (Merck & Co.).

For more information refer to:

• *Using Vapor Calculator*

• *Comparing Vapor Volume to Liner Volume*

2.2 Using Flow Calculator

Use the Column Flow Calculator to determine column pressure settings and flow rates through a capillary GC column when operating with manual pneumatics. Column dimensions, temperature, inlet and outlet pressure, and carrier gas type can be varied when calculating an outlet flow rate, average linear velocity, and holdup time.

Column Flow Calculator Page

Figure 2-2. Column Flow Calculator Page

Column parameters group box

Each control listed in this box is affected by the choices made in the Carrier Gas Parameters group box.

Length   Slide the slider bar to the desired column length in meters.

Inside diameter   Slide the bar to the columns inside diameter in millimeters.

Temperature   Slide the bar to the oven temperature you want to use (ºC).

Inlet pressure   Slide the bar to the pressure amount to supply to the column head (k).

Column outlet pressure group box

1 Atm Atmospheric pressure.

Vacuum Select this if your TRACE is connected to a mass spectrometer.

Other Select this if you want to adjust column outlet pressure above or below atmospheric (0-43.5 psi)

Outlet Pressure Slide the bar to the desired pressure amount the column flow will push against at the column exit.

Carrier gas parameters group box

Gas type Select the type of gas you are using for column flow.

Flow Actual flow calculated based on items listed in Column Parameter box.

Velocity This is the speed in which the flow travels to the column.

Holdup time Time for non-retained peak to travel through the column. Hold-up time is measured to determine the average linear velocity.

Using slider bar and buttons

Slider Bars

Click here to display the main topic for this procedure.

You might hear the slider bar referred to as a scroll bar. But whereas scroll bars typically scroll vertically slider bar scroll horizontally. Use the slider bar to adjust the displayed values.

Quick tips
- Grab (click and hold) the slider and drag it along the length of the slider bar.

- Click on the arrow buttons on either end of the slider bars to increase or decrease the associated value. The amount of increase or decrease depends on the number of decimal places in the parameter, and will vary for different slider bars.

- Click on the area between the slider and the arrow buttons to increase

or decrease the slider bar's associated value at 10 times the rate of clicking on the arrows.

- Use your mouse or tab key to select a slider bar and then use the right and left arrow keys located on your PC's keyboard to adjust the values. This method produces the same results as using the arrows.

Radio Button

○ 1 Atm
○ Vacuum
◉ Other

Use radio buttons to select one or several choices, however, only one radio button from any group can be selected at the same time.

Reset Button

Click to reset all control parameters back to the factory settings. The only parameters for which default values can be changed and saved are pressure units (kPa, psi, or bar) and the controls listed in the Column Outlet Pressure group box (1 atm, vacuum, other, and outlet pressure).

Done Button

Press Done to exit the screen. The pressure units and outlet pressure currently selected save to disk and become the new defaults for the remaining work session. Current pressure units (kPa, psi, or bar) and outlet pressure (1 atm, vacuum, or outlet) settings save and load automatically when you open the screen again. Values for all of the other parameters return to the factory default settings.

Related Topics

For other details refer to

- *Pressure/Flow Calculations*

- *Split Ratio Calculations*

- *Average Linear Velocity*

- *Vacuum Outlet Conditions*

- *Other Pressures*

Pressure/Flow Calculations

The rate of carrier gas flow through an open tubular column can be calculated using the well-understood relationships between pressure and flow in capillary GC. These calculations are useful in your method development and for you to determine the inlet pressure for a particular column and carrier flow or the change in flow resulting from a temperature or pressure program. The TRACE GC Ultra performs a column evaluation where the actual column resistance is calculated. Thus, insuring and achieving accurate column flows for a specific column length, i.d., and the wall coating.

Split Ratio Calculations

The split ratio for a capillary inlet is determined by the relationship between the split vent flow and the column flow. Chromatographers calculate split ratio in several ways and refer to this subject as: split flow/column flow, column flow/split flow, or total flow/column flow. For these calculations, split ratio is defined as

Split Ratio= SR_1 = (split vent flow) / (column flow)

Split ratio is usually expressed as a ratio relative to one. For example, for a split vent flow of 100 ml/min and a column flow of 2 ml/min,

SR_1 = (split vent flow/column flow) : (column flow/column flow) = 50:1

On the pressure/flow calculator screen, the split ratio is always based on the current value for column flow. Entering a value for either split vent flow or split ratio and clicking on "Calc Flow/Ratio calculates the other.

Column Outlet Flow

The *Poiseuille equation* gives the gas flow carrier rate through an open tubular GC column. For a given set of experimental conditions - column length and id, carrier gas type, temperature, and outlet pressure - this equation can be used to calculate the flow expected with a known inlet pressure, or the pressure setting needed to give a desired flow rate.

F=outlet flow in ml/min, measured at Tref and pref(standard conditions)

= [60 p r / 16 h L] [(pi - po) / po] [po / pref] [Tref /T](Eq. 1)

where:

r = column inner radius, cm

L = column length, cm

pi = inlet pressure (absolute), dynes/cm

po = outlet pressure (absolute), dynes/cm

pref = reference pressure, typically 1 atm

T = column (oven) temperature, °K

Tref = reference temperature, typically 25 ° C (298 °K)

h= carrier gas viscosity at column temperature, poise

60 = conversion from seconds (cgs units) to minutes

Average Linear Velocity

The rate at which carrier gas moves through the GC column can also be expressed in terms of its linear velocity. Because pressure on the carrier gas changes at each point along the column, the gas will expand as it flows through the column, and linear velocity will increase from the inlet to the outlet. The retention time for a component reflects the average linear velocity, which is measured by determining the elution time for an unretained peak.

$\bar{u}$= average linear velocity at column temperature T, cm/sec=L / t_M(Eq. 2)

where:

L= column length, cm

t_M = hold-up time, elution time for an unretained component, sec

The *average linear velocity* is related to the outlet velocity by the compression correction factor.

$\bar{u} = u_o \times j$

where:

j= compression correction factor (compressibility correction factor)

$= 3 \, p_o \, (p_i^2 - p_o^2) / 2 \, (p_i^3 - p_o^3)$

As with equation 1 for flow under a given set of experimental conditions, the average linear velocity can be calculated from the column dimensions,

viscosity, and pressure.

$$\bar{u} = [\, 3\, r^2 \,/\, 32\, \eta\, L][\, (p_i{}^2 - p_o{}^2)^2 \,/(p_i{}^3 - p_o{}^3)] \quad \text{(Eq. 3)}$$

Linear velocity is calculated here at oven temperature, to correspond with retention time measurements. (Temperature does not appear explicitly in equation 3, but the viscosity value is a function of temperature.)

Vacuum Outlet Conditions

With most GC detectors, pressure at the column outlet (po) is about 1 atm. In GC/MS systems, however, the column outlet pressure is zero. This makes the flow rate at outlet conditions very high, but column flow under reference conditions can still be calculated from equation 1. (This corresponds to the flow that would be measured with a flow meter connected to the discharge of the vacuum pump, if such a measurement could be made). With $p_o{}^2 \ll p_i{}^2$, equation 1 becomes,

$$F = [\, 60\, \pi\, r^4 \,/\, 16\, \eta\, L]\, [p_i{}^2 \,/\, p_{ref}\,]\, [\, T_{ref}/T] \quad \text{(Eq. 4)}$$

Other Pressures

There are some cases where the column outlet is neither at atmospheric pressure nor under vacuum. One example is a column with a restrictor installed at the end. This could be a length of narrow-bore tubing connected to a mass spectrometer, as discussed in the topic Vacuum Outlet Conditions , or a splitter with connections to two different detectors.

Another example is in work with the atomic emission detector (AED), where the cavity has a positive pressure of about 1.5 psig. When the total pressure drop is low (that is., with short, wide-bore columns), this can cause a significant difference between the calculated and actual flow values. Selecting the "other setting for outlet pressure on the flow calculator allows the appropriate value (14.7 + 1.5 = 16.2 psia) to be entered and used in the calculations.

2.3 Using Vapor Calculator

A liquid sample injected into a heated GC inlet rapidly vaporizes and expands. Vapor Volume Calculator rapidly calculates the expansion volume using several factors (solvent, injected liquid volume, temperature, and inlet pressure). Included with this calculation is the resulting vapor expansion volume relative to the inlet liner's inner dimensions. Because, if the volume of vapor is greater than that of the liner, the excess can expand into other areas of the inlet and result in loss of sample or contamination of the gas lines.

Vapor Calculator Page

Figure 2-3. Vapor Calculator Page

Inlet parameters group box

Injection volume   Slide the bar to the volume amount to inject.

Temperature   Slide the bar to the temperature your method calls for.

Pressure gauge   Slide the bar to the pressure amount your method calls for.

Vapor volume group box

Vapor Volume   An injected solvent's calculated expansion volume. Compare this to a list of the most frequently used liners and their available internal volume.

If you are using an DPFC system, pressure programming can be used to help control vapor volume. With the Pressure Pulse technique, pressure is briefly increased at the time of injection, then returned to the setpoint best for column flow rate during the rest of the run. Advantages for this technique include:

Optimizing conditions for sample transfer to the column

- Facilitating larger injection volumes
- Reducing sensitive components residence time inside the inlet
- Solvent parameters group box

Solvent parameters group box

Type   Select the solvent type you wish to use. Choosing a specific type automatically sets preferred values in all the other fields. However, you still may make adjustments. If any of your adjustments exceed the recommended allowance for the liner, then Xcalibur displays an error message.

Values you see listed in Type are for the most common solvents that have densities at 20° C. Select Other to calculate expansion volumes for other solvents not listed here. Then you may enter the appropriate values for the rest of the Solvent Parameters (boiling point, molecular weight, and density.)

Boiling Point   This is shown on the screen for reference, although it is not used in the calculations. Because the approximation for vapor volume does not really apply near the solvent boiling point, calculations are not done if the inlet temperature is set within 20°C of this value. Instead, a message displays, indicating that a higher temperature should be specified.

Density   For dense solvents with low molecular weights give the largest vapor volume per microliter injected. The effects of inlet temperature and pressure on vapor volume can also be seen from the equation - increasing temperature increases volume, while increasing pressure decreases volume.

Mol. Weight   Molecular weight. The average mass of a molecule of a compound compared to 1/12 the mass of carbon 12 and calculated as the atomic weights sum of the constituent atoms. Or simply stated, the average mass of a molecule of a compound expressed in atomic mass units (AMU).

Liner volume group box

Type   Select a liner type from the list box. Vapor Volume Calculator automatically displays the Liner Volume

Related Topics

• *Comparing Vapor Volume to Liner Volume*

Comparing Vapor Volume to Liner Volume

Comparing vapor expansion volumes with liner volumes gives an estimate of the sample volume that can be injected under different conditions. In practice, mixing and diluting sample vapor with carrier gas during rapid evaporation means that the actual volume occupied by vapor may be greater than the calculated volume, and it depends on the liner design and how rapidly carrier gas is moving through the inlet. A good starting point in method development is to choose the liner, injection volume, temperature, and pressure so that the vapor volume does not exceed the liner volume. (For splitless injections, the liner volume should also be considered in choosing the purge hold time, so that the inlet will be swept at least once by carrier gas flowing onto the column.)

Typical volumes for capillary inlet liners have a range of 250 to 1000 microliters. Volume for an open liner can be calculated from the length and inner diameter, but it may be reduced by internal constrictions or packing.

Rate of carrier gas flow at the column outlet, usually given as volumetric flow in ml/minute. Because the volume for a given mass flow will depend on temperature and pressure ($pV=nRT$), the conditions under which flow is calculated or measured must be specified. See the section on "Column Outlet Flow for details of the calculations.

Velocity, at which carrier gas moves through the column at temperature T, is represented in cm/sec. Because pressure varies continuously along the column (from pi to po), linear velocity also changes at each point between inlet and outlet. The elution time for an unretained component provides a measure of the average linear velocity. This is related to outlet velocity (and flow) through the compression correction factor j.

See the topic on Average Linear Velocity for details of the calculations.

An unretained component's (at column temperature T) elution time is a measure of time that sample components spend in the mobile phase. Hold-up time is measured to determine the average linear velocity.

F = outlet flow in ml/min, measured at Tref and pref (standard conditions)

$F = [\, 60\, p\, r^4 / 16\, h\, L\,]\, [\, (pi^2 - po^2)/po]\, [\, po/pref]\, [\, Tref/T\,]$ (Eq. 1)

$\bar{u}$ = average linear velocity at column temperature T, cm/sec

$$= \quad L / tM \qquad\qquad\qquad (Eq.\ 2)$$

Under a given set of experimental conditions, the average linear velocity can be calculated from the column dimensions, viscosity, and pressure.

$$\bar{u} \quad = [\ 3\ r^2\ /\ 32\ h\ L]\ [\ (pi^2\ -\ po^2\ )^2\ /\ (\ pi^3\ -\ po^3\ )](Eq.\ 3)$$

Flow can then be determined indirectly from the average linear velocity.

$$F \quad = [\ 60\ p\ r^2\ ]\ [Tref\ /\ T]\ [\ 2(\ pi^3\ -\ po^3\ )\ /\ 3\ pref\ (pi^2\ -\ po^2\ )]\ \bar{u}\ (Eq.\ 4)$$

The equation used to calculate inlet pressure from velocity could be quite slow under certain column conditions. At outlet pressures greater than vacuum the equation cannot be solved directly. Instead, the equation is solved by a successive approximation method. You can speed this process up by using the flow slider bar to indirectly change the velocity value.

Electronic pressure control (EPC) provides an alternative to mechanical pressure control for regulating the gas flow in your TRACE GC Ultra. Additionally, EPC provides stability and precision in flow settings, which makes it possible to use pressure/flow programming in GC methods and to document pressures along with the other parameters.

Chapter 3
Instrument Setup

This chapter contains the instructions to edit the TRACE GC Ultra parameters according to the inlets and detector installed.
This chapter contains these topics:

3.1 Edit TRACE GC Ultra Parameters

Introduction

The parameters to set for the TRACE GC Ultra (oven, injectors and detector) should be set according to your GC configuration.

All parameters can be sent to or loaded from the instrument connected by using the functions available in *TRACE Menu*

To open this view:

1. Choose **Instrument Setup** from the Home Page window.

2. Click on the TRACE panel button located in the **Instrument Setup** window.
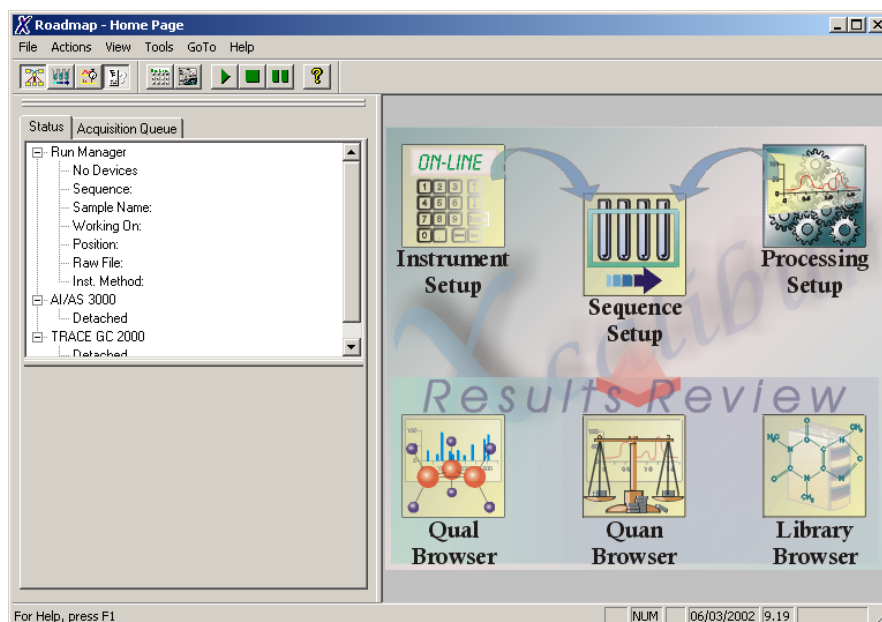
Figure 3-1. Roadmap - Home Page

Using the TRACE Tabs

After you have configured the TRACE and options, you are ready to set the controls to run your method.

Related Topics

How To...:

- *Installing the Desolvation Column and Connecting the Tee*

- *Characterizing Columns*

- *Post Column Splitting of the GCQ with the Flame Ionization Detector*

- *Operating an FID with Xcalibur Software*

- *Simultaneous Analysis on the Ion Trap and the FID*

3.2 Oven Page

This page is the method editor for setting up parameters in the GC run.

To display this page: Click on the Oven page from the Instrument Setup view.
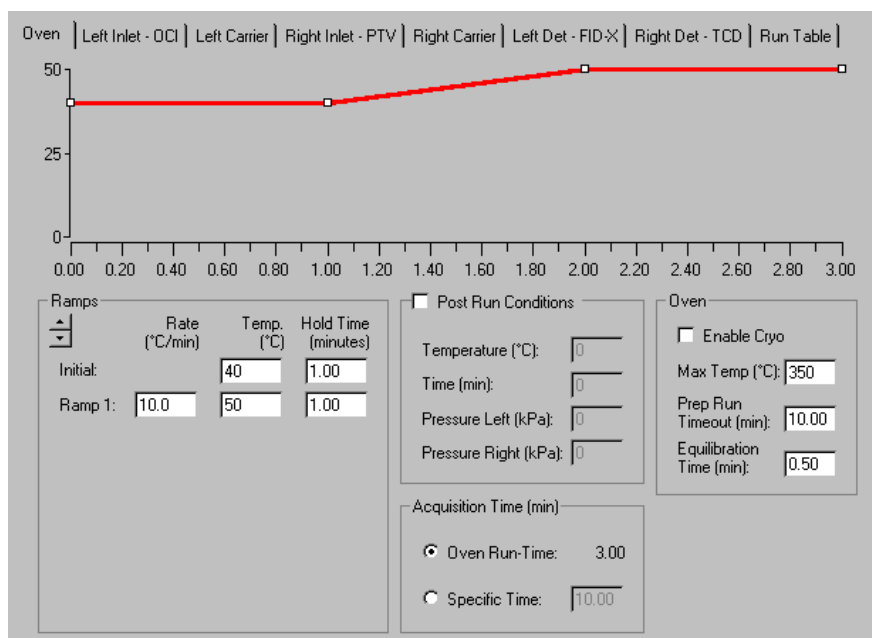
Figure 3-2. TRACE GC Ultra Oven Page View

Related topics:

•   *Setting Up Oven Ramp Parameters*

Oven Page Parameters

Oven Temperature Program graph

Graphical representations of the oven temperature program including any post run events. The axes are temperature in degrees centigrade and time in minutes. An isothermal run just gives a flat line

Oven Temperature Program Parameters

Ramps arrow buttons   Use the up-down control to add or subtract the number of ramps to use in the oven temperature program. This is the rate in degrees Centigrade per minute

the GC oven is ramped up or down from the initial temperature or the previous level's final ramp temperature. Clicking on the Down arrow automatically adds a ramp level after the last one listed. Clicking on the Up arrow deletes the highest level or the one being displayed on the bottom

Initial / Temp text box
The initial temperature the oven is set at the beginning of the temperature program. For isothermal runs, this is the oven temperature for the entire run. After Preready or Prep Run this is the temperature the oven will be at. Checking the Link to Large Volume Assistant program located on the Configuration»Inlet dialog disables this control. This is because you are able to set these controls in the Large Volume Assistant program, which you will find located in the autosampler menu. If you want to activate this control, then uncheck the Link to Large Volume Assistant program located on the Configuration»Inlet dialog box.

Initial / Hold Time text box
The amount of time to maintain the initial temperature.

Ramp / Rate text boxes
The rate of change of the temperature when increasing the temperature from one value to the next. Described in degrees Celsius per minute (°C/min). The ramp begins at the previous step's final temperature value and proceeds to the current step's final temperature at the specified rate.

Ramp / Temp text boxes
The temperature the oven is set at for the next ramp of the temperature program. For isothermal runs, this is the oven temperature for the run. After Preready or Prep Run this is the temperature the oven will be at.

Ramp / Hold Time text boxes
The time to maintain the temperature specified in the Final Value control box.

Oven Group Box

Oven Subambient check box
This refers to an installed subambient (additional purchase), which allows subambient oven cooling.

Max Temp text box
Specify the maximum temperature for this run. The GC has a maximum temperature of 450°. The maximum allowable temperature for the column being used in the method is automatically set.

PrepRun Timeout
Specify how long the GC should remain in the Ready to Inject mode before returning to Stand By. If the GC Start button is not pressed within the prep run timeout period, then the GC will return to Stand By mode. This feature is a protective measure to keep the GC from being in the Ready to Inject mode too long. For example, this will prevent accumulation of contaminants on the analytical column when doing splitless injections.

**Equilibration Time (min)
text box**

Specify how much time the GC should wait after the initial conditions are
met before going to standby.

Acquisition Time Group Box

**Use Oven Run Time
radio button**

Check this box for the Acquisition Time to be the same as the oven run time;
however this does not include the post-run time.

**Use Other Time button
and text box**

You can define the time for acquisition. Enter a value in the text box.

Post Run Conditions check box and group box

Use this option to specify post run conditions. When you select the Post Run
Conditions check box, the group box becomes active.

Temperature text box

The post-run temperature setpoint. Use as a bakeout to clean up the column
or use as a cool down phase after the run has finished.

Time text box

The amount of time the oven remains at the post-run temperature setpoint.

Pressure text box

The post-run pressure setpoint for the left and right EPC inlet. Setting Up
Oven Ramp Parameters

Setting Up Oven Ramp Parameters

Set oven ramp parameters

1. Open the Instrument Setup window.

2.   Display the Oven page by clicking on the TRACE panel button.

3. Type in the temperature (degrees Celsius) and time (in minutes) for the
    initial oven parameters.

4. To add ramps to the oven temperature program, click on the down arrow
    of the up-down control. If you add too many ramps, click on the up arrow
    to delete them.

Set post run conditions

Select the Post Run Conditions check box and enter the time, temperature
and pressure parameters.

1. To keep the default analysis time, check the Total Run Time box. If you
    want to set a different analysis time, leave the Total Run Time box
    unchecked and enter a value for the analysis time.

2. Select Oven Subambient (if your system has this option) for subambient cooling.

3. Enter the Max Temp and Equilibration Time values.

3.3 UFM Page

This page is the method editor for setting up parameters in the GC run.

To display this page: Click on the UFM page from the Instrument Setup view.
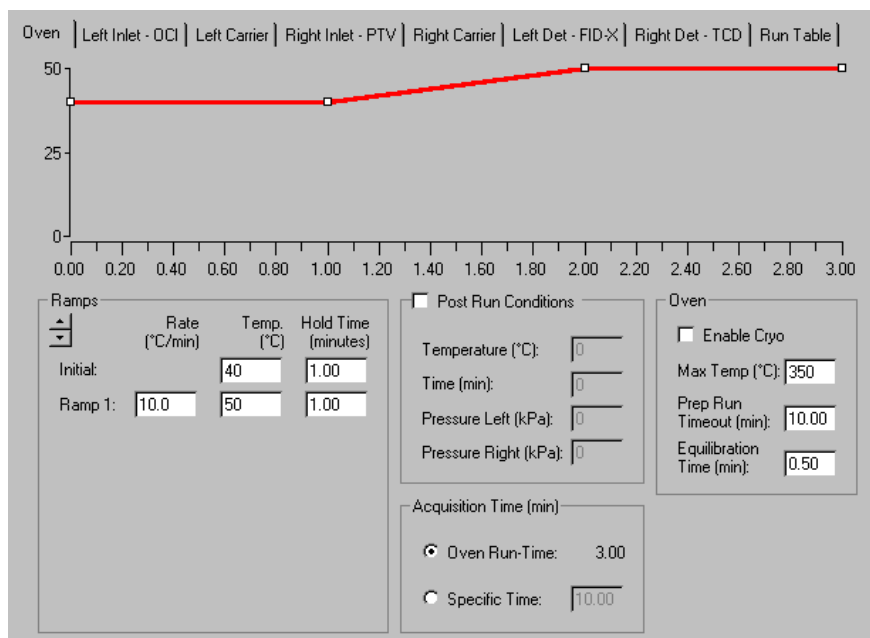
Figure 3-3. UFM Page View

Related topics:

- *Setting Up UFM Ramp Parameters*

UFM Page Parameters

UFM Temperature Program Graph

UFM Temperature Program graph Graphical representations of the column temperature program. The axes are temperature in degrees Centigrade and time in minutes. An isothermal run just gives a flat line.

Block Temperature Group Box

Temperature Enter here the temperature of the auxiliary heating block.

UFM Temperature Program Parameters

Ramps arrow buttons   Use the up-down control to add or subtract the number of ramps to use in the column temperature program. This is the rate in degrees Centigrade per minute (°C/min) the column is ramped up or down from the initial temperature or the previous level's final ramp temperature.

- Clicking on the Down arrow automatically adds a ramp level after the last one listed.

- Clicking on the Up arrow deletes the highest level or the one being displayed on the bottom.

Initial / Temp text box   The initial temperature the column is set at the beginning of the temperature program.

Initial / Hold Time text box   The amount of time to maintain the initial temperature.

Ramp / Rate text boxes   The rate of change of the temperature when increasing the temperature from one value to the next. Described in degrees Centigrade per minute (°C/min). The ramp begins at the previous step's final temperature value and proceeds to the current step's final temperature at the specified rate.

Ramp / Temp text boxes   The temperature the column is set at for the next ramp of the temperature program.

Ramp / Hold Time text boxes   The time to maintain the temperature specified in the Final Value control box.

Oven Group Box

Prep Run Timeout   Specify how long the GC should remain in the Ready to Inject mode before returning to Stand By. If the GC Start button is not pressed within the prep run timeout period, then the GC will return to Stand By mode. This feature is a protective measure to keep the GC from being in the Ready to Inject mode too long. For example, this will prevent accumulation of contaminants on the analytical column when doing splitless injections.

Max Temp text box   Specify the maximum temperature for this run. The UFM has a maximum temperature of 350 °C.

Equilibrium Time (min) text box   Specify how much time the GC should wait after the initial conditions are met before going to standby.

Acquisition Time (min) Group Box

Use Oven Run Time radio button

Check this box for the Acquisition Time to be the same as the UFM device.

Use Other Time button and text box

You can define the time for acquisition. Enter a value in the text box.

Setting Up UFM Ramp Parameters

Set UFM ramp parameters

1. Open the Instrument Setup window

2. Display the UFM page by clicking on the TRACE panel button.

3. Type in the temperature (degrees Centigrade) and time (in minutes) for the initial column parameters.
   To add ramps to the column temperature program, click on the down arrow of the up-down control. If you add too many ramps, click on the up arrow to delete them.

Set block temperature parameter

1. Set the temperature of the column auxiliary heating

3.4 SSL Page

This page is the method editor for the Split/Splitless inlet.

To display this page: Click on the SSL page from the Instrument Setup view.
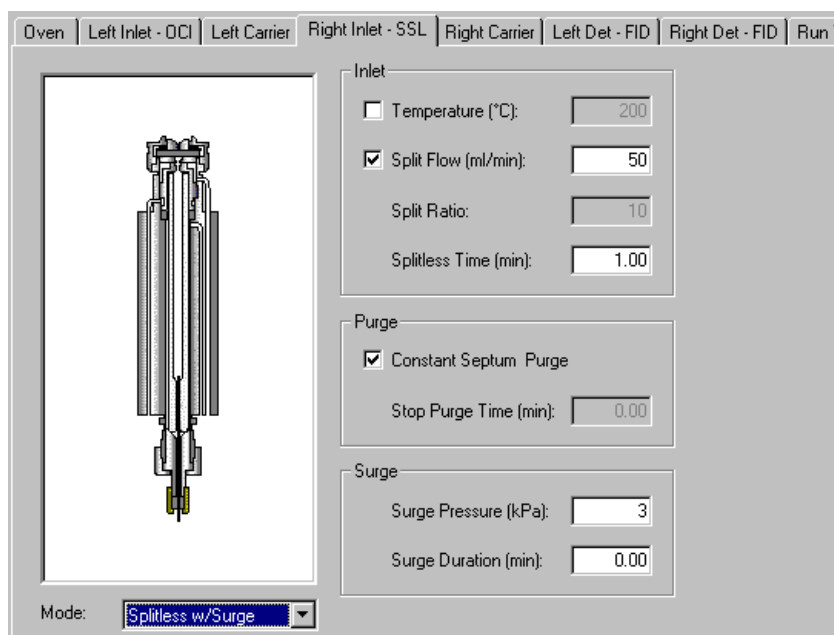
Figure 3-4. S/SL Page View

Related topics:

- *Setting Up Parameters for Split Mode*

- *Setting Up Parameters for Splitless Mode*

- *Setting Up Parameters for Splitless with Surge Mode*

SSL Page Parameters

SSL Modes list box

Use this spin box to select one of the following:

Split   The carrier flow is split in the injection port with the bulk going out the split vent. Use this injection type when analyzing high concentration or neat samples, or in instances where sensitivity is less important. The split vent remains open all the time. This method yields the sharpest peaks, if the split

gas is properly mixed. Additionally, this technique can improve peak shape and resolution.

Splitless The split vent is closed during the injection to drive most of the sample into the column. Solvent effect is required to refocus the analytes , especially more volatile components. You can achieve solvent effect by keeping the analytical column or guard column near the sample's solvent boiling point. This technique also requires the spilt valve be opened after the injection to prevent band broadening due to solvent flooding. Splitless times of ~ 1 minute are typical.

Splitless w/surge A surge is applied during the splitless time to reduce residence time of the analytes in the inlet. This will reduce band broadening and may sharpen peaks, especially for more volatile components, which are not improved by cold trapping effects on the column. Usually this technique involves an oven temperature ~ 50C° below BP of analyte.

Inlet Group Box

Temperature check box and text box The setpoint for the inlet's temperature. Depending on the mode of injection, it should be set at a temperature appropriate to vaporize the sample and solvent.

Split Flow check box and text box Specify the total split flow coming out of the inlet. This is the total flow out of the inlet when the split valve is open.

Split Ratio text box The ratio of split vent flow to the column flow. Calculate the split ratio as shown: Split Ratio = (column flow + split vent flow) / (column flow).

Splitless Time text box The amount of time after the injection at which the split vent opens.

Purge group box

This group controls the septum purge for the inlet. Septum purge is used to sweep the bottom of the septum to reduce contamination from sample analytes. This prevents carryover from run to run Septum purge also prevents contamination of the inlet from septum bleed.

Constant Septum Purge check box The septum purge may be turned off during a splitless injection. Checking this box will keep the septum purge valve open continuously. By leaving this box unchecked, the analyst may close this valve during the injection splitless time.

Stop Purge Time text box Enter the time (in minutes) for the septum purge valve to close after beginning the injection. The Constant Septum Purge checkbox has to be unchecked for this option to be used. This is usually set to the same time as

the splitless time.

Surge Pressure   The pressure applied during the splitless time to produce a surge of flow in the inlet to speed transfer of the sample. It may be used, depending upon the analysis, to sharpen peaks closer to the solvent's boiling point where cold trapping is ineffective and solvent effect is the main refocusing mechanism. Try adding 8 psi or 55.157 kPa to the inlet and observe early peaks after the solvent to see if peak tailing is reduced.

Surge Duration   The amount of time a pressure surge is administered after the injection. Typically, set to coincide with the splitless time.> GC Setup View - Split Mode

Setting Up SSL Parameters

Setting Up Parameters for Split Mode

Use the following procedure to specify the carrier gas flow rate and pressure for constant flow.

1. Open the Instrument Setup window.

2. Click on the TRACE GC Ultra panel button.

3. Select the SSL tab.

4. Choose Split from the Mode list box.

If you would like to set a temperature for the injector, click in the temperature check box and enter a temperature value (in degrees Celsius) in the Temperature text box.

Setting Up Parameters for Splitless Mode

Use the following procedure to specify parameters for a splitless GC run.

1. Open the Instrument Setup window.

2. Click on the TRACE GC Ultra panel button.

3. Select the SSL tab.

4. Choose Splitless from the Mode spin box.

If you would like to enter a temperature for the injector, click in the temperature check box, then enter the desired temperature in the temperature text box.

If you would like to enter a split flow, click in the Split Flow check box and

enter a value (mL/min) in the Split Flow text box.

5.   Enter a value in the Splitless time text box.

If you want to use constant septum purge, click in the Constant Septum Purge check box. Otherwise, enter the time in minutes in the Stop Purge Time textbox.

Setting Up Parameters for Splitless with Surge Mode

Use the following procedure to specify the carrier gas flow rate and pressure for ramped pressure.

1.   Open the Instrument Setup window.

2.   Click on the TRACE GC Ultra panel button.

3.   Select the SSL tab.

4.   Choose Splitless w/ surge from the Mode list box.

If you would like to enter a temperature for the injector, click in the temperature check box, and enter the desired temperature in the temperature text box.

If you would like to enter a split flow, click in the Split Flow check box and enter a value (mL/min) in the Split Flow text box.

5.   Enter a value in the Splitless time text box.

If you want to use constant septum purge, click in the Constant Septum Purge check box. Otherwise, enter the time in minutes in the Stop Purge Time text box.

6.   Enter values in the Surge Pressure and Surge Duration text boxes.:

3.5 OCI Page

This is the method editor for the on-column inlet.

To display this page: Click on the OCI page from the Instrument Setup view.

Figure 3-5. On Column Page View

Related topics:

*   *Setting Up Parameters for an OCI*

OCI Page Parameters

Sec. Cooling Time text box

The amount of time the secondary cooling stays on after the start of injection. This begins when the TRACE GC Ultra display-panel displays READY. The value you enter in this text box is the time duration for secondary cooling, after selecting Run Start. For example, if you enter a one in this text box, then secondary cooling and the secondary valve turn on when the GC begins READY mode and remains on for one minute after the GC run has started.

Setting Up Parameters for an OCI

1. Open the Instrument Setup window.

2. Click on the TRACE GC Ultra panel button.

3. Select the OCI tab.

4. For secondary cooling enter a value equivalent to the sample injection time.

5. If you have auxiliary purge controls available and the optional hardware is installed, then select column purge to continuously purge the inlet during injection.

6. If constant purge is not selected, then enter a Stop Purge Time value that is greater than the sample injection time.

3.6 HOTOC Page

This page is the method editor for the high oven cold on-column inlet.

To display this page: Click on the HOTOC page from the Instrument Setup view.
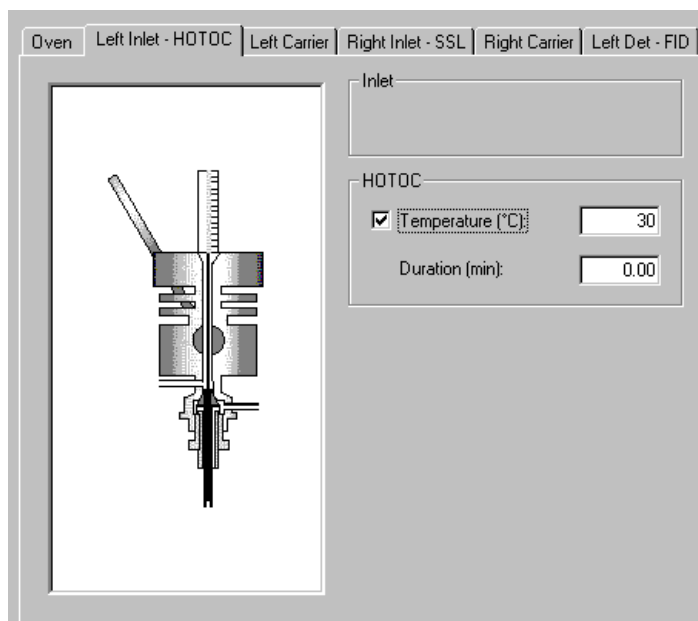
Figure 3-6. HOTOC Page View

Related topics:

• *Setting Up Parameters for an HOTOC*

HOTOC Page Parameters

Inlet Group Box

This group box is intentionally left blank for future developments.

HOTOC Group Box

HOTOC Temperature check box and text box

Activates the secondary cooling valve and prevents the GC from going into READY mode until the entered temperature value you enter in this box is achieved.

Duration text box   Enter the amount of time the secondary cooling remains on once the GC advances to READY.

SVE Valve Group Box

These controls display ONLY if you selected SVE Valve during Inlet configuration.

SVE Temperature check box and text box   The solvent vapor exit valve is normally heated to a temperature above the boiling point of the solvent being injected, in order to prevent solvent condensation in the valve, which may cause a flow change during injection. Allowable values are 0-200 ºC or On/Off.

SVE Duration text box   This is the time that the SVE valve is open after injection to vent most of the solvent's large volume. The LVI program usually calculates this parameter since the exact time to cut off is critical for the LVI injection to properly operate. This time determines the volume of residual solvent that is allowed to enter the analytical column.

Setting Up Parameters for an HOTOC

Click here to display the main topic for this procedure

1. Cool the area around the column where sample is introduced, since the GC column oven generally heats to a temperature higher than the injected solvent' boiling point. Generally this temperature is set to a value below a solvents boiling point.

2. Set the HOTOC Time Duration to the same amount of time as it takes for a sample injection.

3. If the SVE Valve is installed, heat it to approximately 150 ºC. Turn on the SVE Valve for the necessary time needed to vent the solvent. Solvent vent valve turns on when GC READY begins. After RUN START the SVE duration time turns off. However, this duration time stays on until the required amount of solvent is vented. It is necessary that the GC run be started at the beginning of sample injection.

3.7 LVOCI Page

This is the method editor for the large volume on-column inlet.

To display this page: Click on the LVOCI page from the Instrument Setup view.
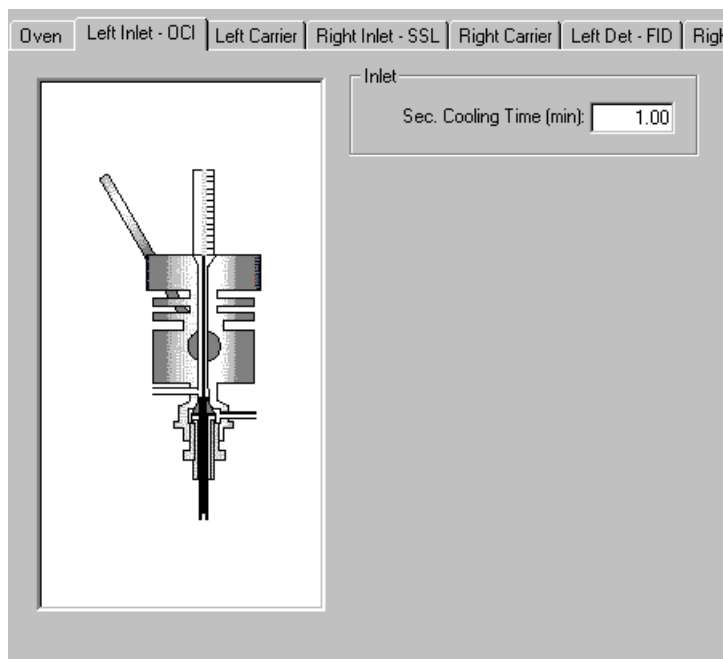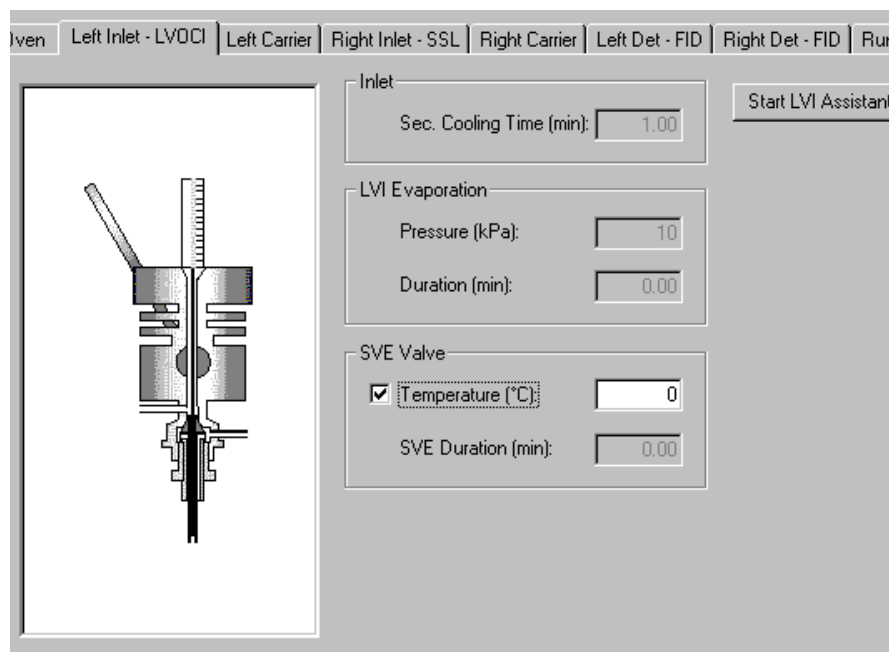

Figure 3-7. LVOCI Page View

Related topics:

- *Setting Up Parameters for an LVOCI*

- *Operating Precautions for the LVOCI*

- *Tips for Performing Large Volume Injections*

LVOCI Page Parameters

Inlet Group Box

Sec. Cooling Time text box
This is the time after injection that the secondary cooling air (or other cooling fluid, for example N_2 or CO_2 s on. Allow the cooling to be on long enough to allow the liquid injection to move away from the syringe needle tip before the syringe is withdrawn. After the syringe is withdrawn, this cooling is no longer necessary.

LVI Evaporation Group Box

Pressure text box

This is the inlet pressure used during the evaporation stage of the large volume injection. This parameter is usually generated by the LVI software program, and not entered by the user. This can be the same as inlet pressure, if a single pressure program is being used, and this entry will be grayed out. Allowable values are 0-250 or 0-700 kPa, depending on the carrier flow module.

Duration text box

Enter a duration time for the injection's LVI evaporation stage. This parameter is usually calculated by the LVI software and transferred into this page.

SVE Valve Group Box

Temperature check box and text box

The solvent vapor exit valve is normally heated to a temperature above the boiling point of the solvent being injected, in order to prevent solvent condensation in the valve, which may cause a flow change during injection. Allowable values are 0-200 ºC or On/Off.

SVE Duration text box

This is the time that the SVE valve is open after injection to vent most of the solvent's large volume. The LVI program usually calculates this parametersince the exact time to cut off is critical for the LVI injection to properly operate. This time determines the volume of residual solvent that is allowedto enter the analytical column.

Setting Up Parameters for an LVOCI

Setting up parameters for an LVOCI method is a three-step process: *configuring* instruments for LVI, *calculating* optimum parameters and then *downloading* parameters to the autosampler and GC. Mass spectrometer parameters download with the GC.

Configuring for LVI

1. Configure the autosampler, TRACE or TOP GC, and MS for LVOCI.

2. Open the autosampler method editor and create a new method for the LVI method.

3. Configure your autosampler to use the Large Volume Program. Select Left for the Autosampler Placement and Large Volume and enter the maximum volume for the syringe that is installed. Select OK. The system configures for EPC gas control for the left LVI injector.

Calculating Optimum Parameters

When using the TRACE GC Ultra:

1. Select the left carrier and set the flow mode to constant flow or programmed pressure with vacuum compensation.

2. Next, select LVOCI and select Temperature On. Enter 200°C for the SVE valve temperature to prevent residue accumulating from the solvent during desolvation.

3. Enter the required ramped pressure setting for downloading the two pressures that the LVI software calculates for the two step process of desolvation and then analyte analysis. The first pressure is high for solvent evaporation andthe second pressure setting is lower for analytes enhanced resolution throughthe capillary column.

Note: Do not enter the oven ramps for the analysis at this time; they are entered after loading the parameters for the LVI. You may also setup parameters for isobaric operations.

When using the GCQ Plus Mass spectrometer:

1. Press the mass spectrometer icon button. Select the first line for the first segment and double click the left mouse button. The dialog box for the MS opens. Enter the appropriate values and set the start time to 5.0 minutes or longer. You will have a residual amount of solvent going through the detector and want to delay the filament ignition until after this solvent has passed through. Select a time that provides detection of the first analyte but not the solvent

2. ook at the fore-pressure (located on the homepage Status page) during the first run to verify the time for the solvent to pass out of the mass spectrometer. The fore pressure may be monitored during filament delay.

3. Save the method.

4. Set the mass spectrometer to a high-pressure operation mode by selecting the CI mode from the GCQ Plus front panel.

Downloading LVI Parameters into the GC Method

1. From Instrument Setup, click on the autosampler icon. The autosampler menu displays on the menu bar, which contains two selections necessary to run a large volume injection select Run Large Volume Program and the Load Large Volume File.

2. Select **Run Large Volume Program** to begin creating a LVI method file. For step-by-step instructions on how to create LVI method files, just click the Help button listed in the LVI dialog box. Be sure to save the file

with a specific name and close the LV program.

Run Large Volume Program   Launches the large volume program to automatically make a Large Volume File which calculates the appropriate oven temperature, injection speed and pressure setting required for desolation and analysis.

3. Select **Load Large Volume File** to see a list of available methods. Select the appropriate method. The instrument parameters for the large volume injection insert into the GC and autosampler methods accordingly.

Load Large Volume File   Actually downloads the file made during the Run Large Volume Program.

4. Enter the desired oven program, analytical column flow required, other autosampler and mass spectrometer parameters.

5. Enter a long filament delay for the first injection for 5-8 minutes. The actual fore pressure to the mass spectrometer may be monitored during the filament delay. Just look at the GCQ Plus Status page located on the home page for desolation step optimization and filament delay time.

Operating Precautions for the LVOCI

To conserve air consumption, install a step-down single stage regulator to use secondary coolant set at 10 psi or 70 kPa. Stop all sequence runs not using secondary coolant and do not have a vial located in the tray. This is because the GC begins using initial conditions when not using secondary coolant andwill stop the run when it does not detect a vial in the autosampler tray.

Set the GCQ Plus to the CI mode (press the GCQ Plus EI/CI button locatedon the GCQ front panel) to operate in high pressure mode.

Tips for Performing Large Volume Injections

Read the LVI Manual.

•   Select the smallest retained volume when optimizing your large volume injection method. You can choose larger retained volumes if the smallest amount is not adequate.

•   During the filament delay process, the GCQ extends the filament delay until adequate vacuum has been detected (the blue blinking light on the front panel of the GCQ-MS will turn off and the green light will be on). This is completely normal. You can increase your filament delay time in the MS method to match the actual time it takes for the vacuum system to recover from the large volume injection.

•   Get the best pressure value results by using the LVI Assistant program to select the Injected Volume option. Then use the LVI calculated pressure values to download to the instruments.

- Before loading an LVI method from the AS method editor, make sure the GC is configured for pressure program mode. This will download the pressure values from the LVI optimization program and ensure that the correct times and flows are present to evaporate the solvent from the pre-column. Using the constant-flow or constant-pressure modes results in too much flow into the mass spectrometer during the run analysis. Using programmed flow will cause too little flow to be present during the desolvation period, overloading the vacuum system of the mass spectrometer.

- Some parameters will be grayed out in the GC and autosampler method parameter after downloading the LVI method. These values are calculated and automatically set by the LVI software.

- If you are using a TRACE GC Ultra, check to see that the initial pressure time is equal to the inject time plus the SVE delay time. You may need to change this manually.

- If you are using a CE 8000 Top, and you note residual solvent in the background spectrum throughout the run, shorten the fused silica line on the SVE valve. For injection of 100 uL of Hexane, remove 15 cm.

3.8 PKD Page

This page is the method editor for the packed column inlet.

To display this page: Click on the PKD page from the Instrument Setup view.
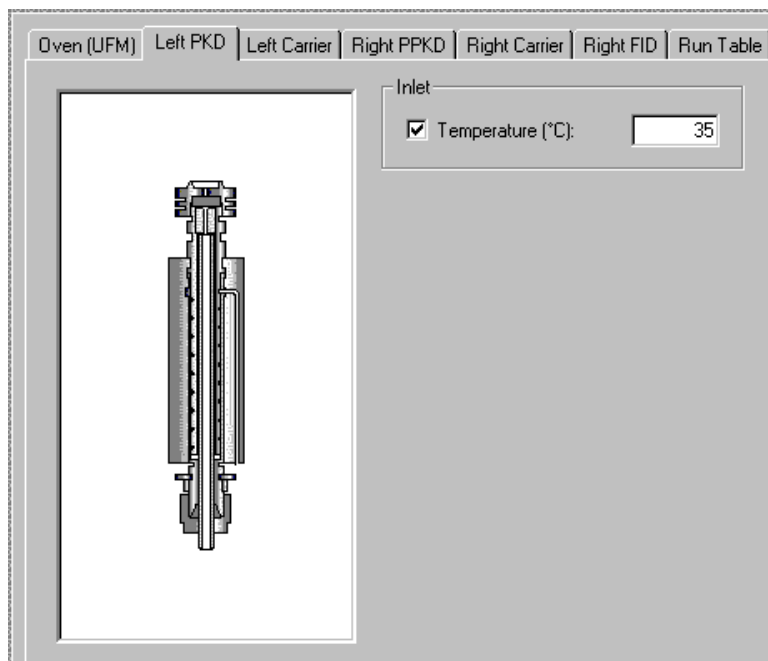
Figure 3-8. PKD Page View

Related topics:

• *Setting Up Parameters for PKD*

PKD Page Parameters

Inlet Group Box

Temperature check box and text box
This is the temperature setting for the packed column (PKD) inlet. The allowable entry range is 30-400 ℃. This temperature is usually selected to be 20° above the evaporation temperature for all components of interest in the analysis sample. Optimum temperature for an analytical method varies with he method and sample requirements. The entry also has a check box for on/off values, so that the temperature setting may be turned off without affectingthe setpoint (usually used for troubleshooting purposes.)

Setting Up Parameters for PKD

1. Set up parameters for the temperature.

Typically, you set the inlet temperature to approximately 20 ℃ above the temperature necessary to volatilize a sample's components of interest. ThePKD operates in a closed loop flow control manner. This means that the carrier-gas-flow-module measures the flow of gas to the inlet for direct flowcontrol. Typical gas flow rates for 1/8 PKDs are 15-30 cc/min.

Note: Operating this inlet with micro packed or wide-bore capillary columns make it necessary to optimize the carrier gas flow to match the installed column.

3.9 PPKD Page

This is method editor for the purge packed column inlet.

To display this page: Click on the PPKD page from the Instrument Setup view.
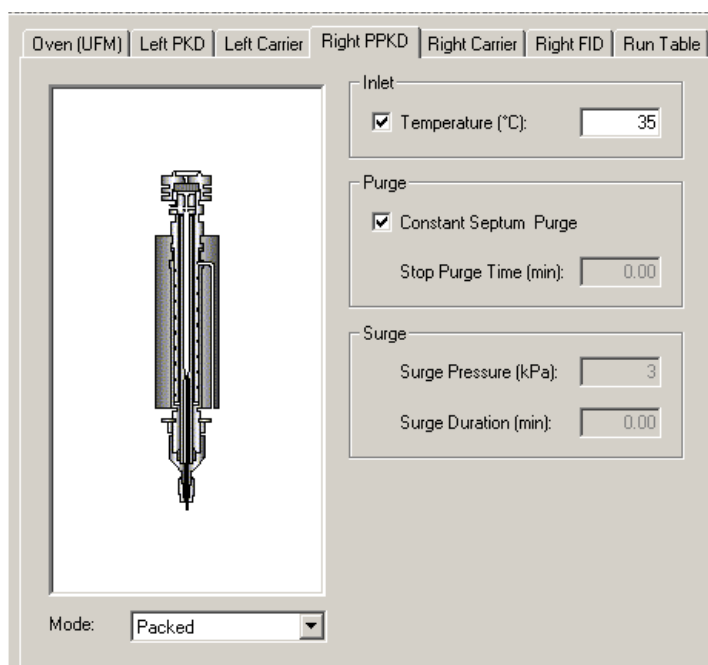
Figure 3-9. PPKD Page View

Related topics:

• *Setting Up Parameters for PPKD*

PPKD Page Parameters

Inlet Group Box

Temperature check box and text box

This is the temperature setting for the purged-packed column (PPKD) inlet. The allowable entry range is 30-400 °C. This temperature is usually selected to be 20° above the evaporation temperature for all components of interest in the analysis sample. Optimum temperature for an analytical method varies with the method and sample requirements. The entry also has a check box for on/off values, so that the temperature setting may be turned off without affecting the setpoint (usually used for troubleshooting purposes.)

Purge Group Box

Constant Septum Purge check box

This entry allows the septum purge flow to be turned off for a specified amount of time after injection. Allowable entries is On or Off. If you select On using a needle to penetrate to a normal injection depth then you will see little sample loss.

Stop Purge Time text box

If the constant septum purge box is unchecked, specify the time after injection that the septum purge flow is turned off. Keep the flow off for the minimum time amounts so those septum bleed products are not transferred onto the analytical column. Typical entries are 0.2-1.0 minutes, with allowable entries from 0-99.99 minutes.

Surge Group Box

Surge Pressure text box

This control is enabled when you select wide bore/w surge or packed/w surge as the injection mode. This injection mode allows a higher inlet pressure to be applied during injection. This serves to reduce the volume of the vapor cloud created when injected sample is vaporized, and may improve separated analytes resolution. Allowable entries are 0-250 or 0-700 kPa, depending on the carrier-flow-module you have installed.

Surge Duration text box

Enter a time-duration for the inlet pressure to be set at the surge pressure setting. This entry should be only the time necessary to ensure the injected sample transfers to the analytical column; so that the column flow is slightly affected.

Mode

Wide Bore mode

The PPKD inlet operates in both packed mode and wide bore (capillary) column mode. If you select wide bore, the carrier flow module operates like conventional capillary modes, and uses the column evaluation K-factor to calculate flow from pressure applied to the inlet. This mode is appropriate for capillary column's length equal or greater than 30 m and operating at column flow rates less than 5 mL/min. When a column's length is less than 30 m, the flow resistance is so small that the packed mode, with its closed loop flow control is more accurate.

Packed mode

If packed mode is selected for the PPKD inlet to operate in then the column flow control is in a closed loop. This means that the carrier module is directly measuring the column flow. This mode is useful for a short (< 30m) wide bore capillary column (0.53 mm or >) as well as packed columns. These column usually operate at flow rates > 5 cc/min, so that direct column measurement is feasible. Column evaluation is not performed for this mode.

Wide Bore w/Surge mode   This selection allows you to program a higher inlet pressure during the sample injection. This reduces the vaporized sample volume and enhances the injection vapor cloud transfer onto the column in a narrow time window. Thus, yielding a better component resolution by the column.

Packed w/Surge mode   This selection allows you to program a higher inlet pressure during the sample injection. This reduces the vaporized sample volume and enhances the injection vapor cloud transfer onto the column in a narrow time window. Thus, yielding a better component resolution by the column. Setting Up Parameters for PPKD mode

Setting Up Parameters for PPKD

1. Select the desired operation mode: Wide Bore Packed mode , Wide Bore w/Surge , or Packed w/Surge mode .

2. Go to the Inlet group box and select Temperature.

3. Go to the Purge group box and select Constant Septum Purge or Stop Purge Time .

If you selected any mode w/surge then go to the Surge group box. Enter the desired parameters into the Surge Pressure control and the Surge Duration control.

The PPKD inlet may be operated in two primary modes: wide bore or packed. In wide-bore mode, the inlet operates like a direct capillary inlet. Column flow is not measured directly, but is calculated from inlet pressure, oven temperature, and a measured k-factor. The k-factor is automatically measured by the TRACE GC Ultra in the column evaluation process and represents a summation of selected factors (such as the carrier gas type, outlet pressure, column resistance versus pressure, and others).

In packed mode, the carrier flow module measures the column flow. This mode is appropriate for packed column and short wide bore (< 30 m and > .53 mm i.d.) columns. These short columns do not provide enough resistance to flow to allow measurement of k-factors for typical capillary column control.

As a general rule, the inlet must provide column carrier gas flow control, andvaporize the injected samples for transfer to the analytical column. Because the proper temperatures and flow are sample and method dependent, noguidelines are available.

3.10 PTV Page

This is the method editor for the programmable temperature vaporizing inlet.This is a right inlet only. Several controls require purchasing additional TRACE GC Ultra options.

To display this page: Click on the PTV page from the Instrument Setup view.
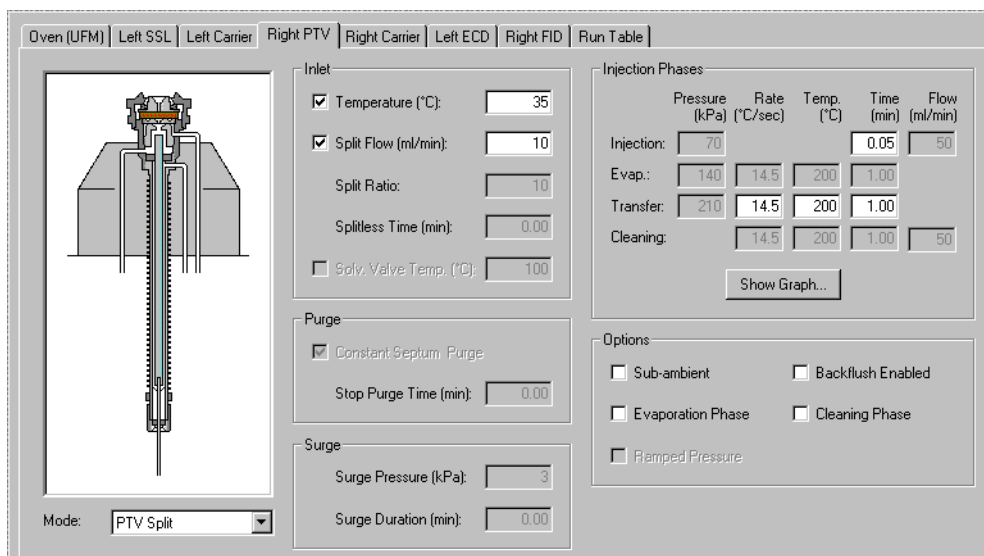
Figure 3-10. PTV Page View

Related topics:

- *Setting Up Parameters for PTV*

PTV Page Parameters

Mode list box

Select a PTV mode from the Mode list box .

PTV Modes

All PTV injection modes are programmable ramped temperatures you operate at varying temperatures with specified times and temperature increments. Selecting PTV modes activates various controls listed the Injections Phases group box.

PTV Split The carrier flow is split in the injection port with the bulk going out the split

vent. Use this injection mode when analyzing high concentration or neat samples, or in instances where sensitivity is less important. The split vent remains open all the time. This method yields the sharpest peaks if the split gas is properly mixed.

PTV Splitless The split vent is closed during the injection to drive most of the sample into the column. The solvent effect is required to refocus the analytes, especially more volatile compounds. You can achieve the solvent effect by keeping the analytical column or guard column slightly below the solvent's boiling point. Splitless times of ~ 1 minute are typical. Activates controls listed in the Purge group box.

PTV Solvent Split Injection technique used when it is necessary to eliminate the solvent before the sample enters the column. This is mainly for large volume injections when components have a lower volatility than the solvent. This item does not list if the Solvent Valve control was selected in for PTV in the Inlet Configuration dialog.

PTV Large Volume Displays when the Solvent Valve control is selected or the PTV in the Inlet Configuration dialog. Activates Purge controls.

CT Modes

All **CT injection modes** are constant temperature (isothermal) modes you operate at set temperatures and time increments. Selecting CT modes disables all controls in the Injections Phases group box.

CT Split Constant temperature (isothermal) split valve. Isothermal temperatures operate at a single temperature during the entire analysis.

CT Splitless Constant temperature (isothermal) splitless valve. The split vent is closed during the injection to drive most of the sample into the column. The solvent effect is required to refocus the analytes, especially more volatile compounds. You can achieve the solvent effect by keeping the analytical column or guard column slightly below the solvent's boiling point. Splitless times of ~ 1 minute are typical. Activates controls listed in the Purge group box.

CT Splitless w/Surge Constant temperature (isothermal) activates controls listed in the Surge group box. Same as CT Splitless but can also program a surge during an injection. Surge starts at Prep Run and continues until the surge duration time is finished. Surge is further defined in the Surge Group Box**.**

Inlet Group Box

Temperature checkbox and text box Allowable entry ranges are 30-400 ℃. However, if you selected the SVE Valve in the Inlet Configuration dialog then you can enter cryogenic temperature values (below 0 ℃) in the range –50º to 400º.

For non-cryogenic use, this temperature is usually set for 20º above the evaporation temperature for the sample's components of interest. Optimum temperature for an analytical method varies with the method and sample requirements. The entry also has a check box for on/off values, so that the temperature setting may be turned off without affecting the setpoint (usually used for troubleshooting purposes.)

Split Flow checkbox and text box Specify the total split flow coming out of the inlet. This is the total flow out of the inlet when the split valve is open.

Split Ratio The ratio of split vent flow to the column flow. Calculate the split ratio as shown: Split Ratio = (column flow + split vent flow) / (column flow). You must select the PTV Split or CT Split mode to use the Split Ratio control. Then select the Carrier page and select Constant or Programmable Pressure mode to activate the Split Ratio control.

Splitless Time text box The amount of time after the injection at which the split vent opens.

Solv. Valve Temp This control activates only after selecting SVE Valve from the **Inlet Configuration** dialog (see *Inlets Page*). Check this box to enter a temperature value for a PTV large volume injection instead of the PTV solvent. With this control you can regulate the temperature for the PTV large-volume heating valve.

Purge Group Box

Constant Septum Purge checkbox This entry allows the septum purge flow to be turned off for a specified amount of time after injection. Allowable entries is On or Off. If you select On using a needle to penetrate to a normal injection depth then you will see little sample loss.

Stop Purge Time text box If the constant septum purge box is unchecked, specify the time after injection that the septum purge flow is turned off. Keep the flow off for the minimum time amounts so those septum bleed products are not transferred onto the analytical column. Typical entries are 0.2-1.0 minutes, with allowable entries from 0-99.99 minutes.

Surge Group Box

Surge Pressure text box This control is enabled when you select wide bore/w surge or packed/w surge as the injection mode. This injection mode allows a higher inlet pressure to be applied during injection. This serves to reduce the volume of the vapor cloud created when injected sample is vaporized, and may improve separated analytes resolution. Allowable entries are 0-250 or 0-700 kPa, depending on the carrier-flow-module you have installed.

Surge Duration text box   Enter a time-duration for the inlet pressure to be set at the surge pressure setting. This entry should be only the time necessary to ensure the injected sample transfers to the analytical column; so that the column flow is slightly affected.

Injection Phase group box

Injection   Use this control for the injection's ramped pressure. Available only with PTV Splitless or PTV Large Volume mode. The injector temperature is automatically set to be lower than the solvent's boiling point all you have to do is specify the time the injector temperature must be maintained.

Evaporation   This group of controls allows you to specify solvent evaporation controls: pressure (PTV Splitless and PTV Large Volume only), rate, temperature, and time in minutes. Set the solvent evaporation temperature, set the rate to reach the solvent evaporation temperature.

Transfer   This group of controls allows you to specify controls for the sample transfer into the column: Pressure (PTV Splitless and PTV Large Volume only), rate, temperature, and time in minutes. Specify the *pressure* if available. Set the *rate* in °C/s to reach the sample transfer temperature. Set the *temperature* for sample transfer into the column. Set the *time* in minutes for the transfer temperature to be maintained.

Cleaning   Set the *rate* necessary to reach the cleaning temperature. Set the injector cleaning *temperature*. Set the *time* in minutes the cleaning temperature must be maintained.

Options Group Box

Start here. Selections made in this group box determine which controls to activate in other group boxes.

Sub-ambient   Select this option to enter sub-ambient temperatures (–50 °C to 400 °C) in the Temperature text box.

Evaporation Phase   Check this box to activate the Evap controls listed in the Injection Phases group box.

Ramped Pressure   If you selected the PTV Splitless or PTV Large Volume mode then this control is available for you to activate pressure controls for solvent injection, evaporation, and transfer.

Cleaning Phase   Check this box to activate the Cleaning controls listed in the Injection Phases group box.

Setting Up Parameters for PTV

1. In the Options group box, select an injection mode and then check or uncheck the available controls listed in this group box.

2. Set the available controls listed in the Injection Phases group box.

3. Set the available controls listed in the Inlet group box.

4. Set the available controls listed in the Purge group box.

5. Set the available controls listed in the Surge group box.

6. For PTV mode or SSL inlets and all isothermal modes select *Vapor Calculator* from the TRACE menu bar to automatically calculate the expansion volumes.

7. Select *Keypad* from the TRACE menu bar for quick access to your TRACE GC Ultra keypad functions.

3.11 Carrier Page

This page is the method editor for setting the carrier gas for the TRACE GC Ultra inlet. Carrier is gas used as the mobile phase in gas chromatography. The carrier gas carries the analyte mixture through the GC column, where it is separated into its individual components. The carrier gas flows through the GC column at a specific rate, measured either as a linear velocity (cm/sec) or as a flow rate (mL/min). Common carrier gases are helium, hydrogen, and nitrogen. Hydrogen offers the best chromatographic properties (optimum resolution at the highest flow rates). However, because hydrogen is flammable, helium is often used as an alternative for a carrier gas.

To display this page: Click on the Carrier page from the Instrument Setup view.
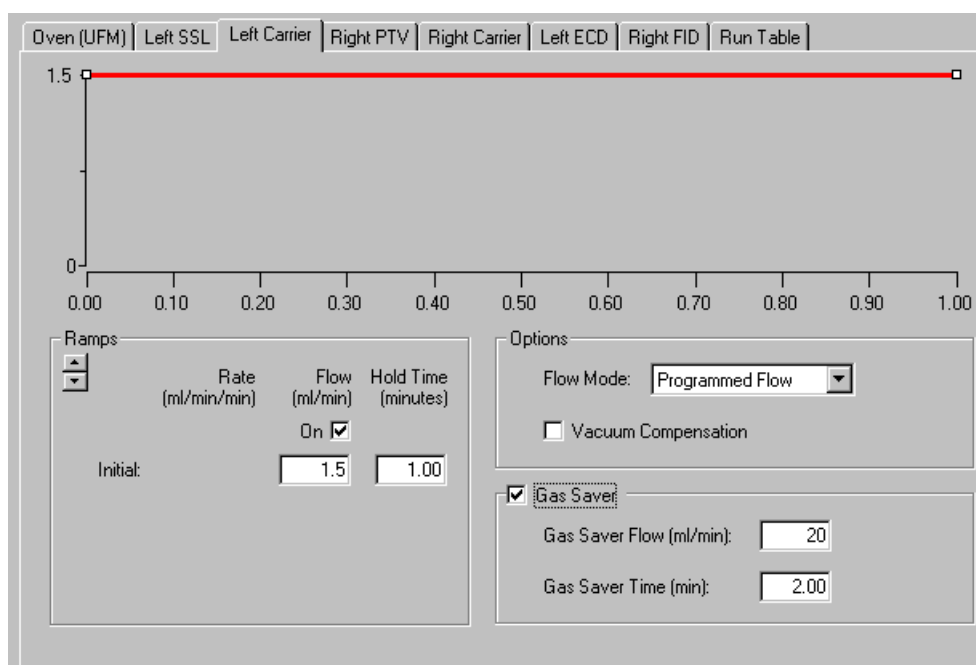

Figure 3-11. Carrier Page View

Related topics:

- *Setting Up Parameters for Constant Flow Mode*

- *Setting Up Parameters for Constant Pressure Mode*

- *Setting Up Parameters for Programmed Flow Mode*

- *Setting Up Parameters for Programmed Pressure Mode*

Flow Mode list box

The controls you see on this page are dependent on the Flow Mode selection. Please select one of the following modes and jump to that topic for detailed information.

- *Constant Flow Mode*

- *Constant Pressure Mode*

- *Programmed Flow Mode*

- *Programmed Pressure Mode*

Four flow modes are available and affect the controls listed in the Ramps group box:

Constant Flow — controls in the Ramp group box display Flow (cc/min). Enter a column flow for the analytical column. When using an oven temperature program, run the GC to automatically increase the pressure constant flow on the column. This compensates a gas's increased viscosity due to an increase in temperature. In this mode, the inlet pressure is varied during an analytical run as needed to maintain the column flow at a specified setpoint. This means that when a column is heated and its restriction to flow increases the inlet pressure is increased.

Constant Pressure — controls in the Ramp group box display Pressure. Enter inlet pressure for the analytical run. In this mode, a specified pressure is applied to the column and remains unchanged throughout the analysis.

Programmed Flow — controls in the left group box display Ramps, Rate, Flow, and Hold Time. Enter program to control column flow for analytical run.

Programmed Pressure — controls in the left group box display Ramps, Rate, Pressure, and Hold Time. Enter inlet pressure program for analytical run.

In both the programmed flow and programmed pressure modes, the flow or pressure changes according to a timetable similar to temperature programming. The analyst may specify an initial pressure, initial time up to three program ramps with rates, targets, and hold times for each needed ramp. The programmed modes are typically used to shorten analysis times by increasing column flow to drive higher boiling components off the column faser at the end of analysis.

Constant Flow Mode

Click on the Carrier tab of the GC Setup view, and then click on **Constant Flow** in the Flow Mode list box

Use the Carrier page, constant flow option to select a constant flow without ramping.
The parameter available are:

Flow Program graph　　A graphical representation of a flow program for the carrier gas. It is a flat line if a constant flow program is selected. The axes represent time for the x-axis and flow in mL/min for the y-axis.

Flow text box　　A flow for the analytical column. For an oven temperature programmed run the GC automatically increases pressure to keep a constant flow on the column compensating for increased viscosity of gas due to increase in temperature.

Vacuum Compensation check box

Check this box to compensate for a vacuum at the end of the analytical column. This is usually the case where the detector is a mass spectrometer. When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

Gas Saver check box and group box

This group box contains gas saver controls, which reduce carrier gas consumption, especially when a large split flow is used. The gas saver options are set to come on at some point well after the injection to conserve gas. Checking the box will activate the other two features of gas saver.

Gas Saver Flow text box　　The desired flow after the injection or in standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

Gas Saver Time text box　　The time after the injection when gas saver flow will be activated.

Constant Pressure Mode

Click on the Carrier tab of the GC Setup view, and then click on **Constant Pressure** in the Flow Mode list box.

Use the Carrier page with constant pressure option to select a constant pressure for the analysis. The parameters are:

Pressure Program graph

The graph represents a pressure program for the carrier gas. It is a flat line if a constant pressure program is selected. The axes represent time for the x-axis and either flow in mls/min or pressure in kPa for the y-axis.

Pressure text box

Enter the inlet pressure for the analytical run in this text box.

Vacuum Compensation check box

Check this box to compensate for a vacuum at the end of the analytical column. This is usually the case where the detector is a mass spectrometer. When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

Gas Saver check box and group box

This group box contains gas saver controls, which reduce carrier gas consumption, especially when a large split flow is used. The gas saver options are set to come on at some point well after the injection to conserve gas. Checking the box will activate the other two features of gas saver.

Gas Saver Flow text box

The desired flow after the injection or in standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

Gas Saver Time text box

The time after the injection when gas saver flow will be activated.

Programmed Flow Mode

Use the Carrier page, programmed flow option to set up a programmed flow for the GC. The available parameters are:

Use the Carrier page, programmed flow option to set up a programmed flow for the GC. The available parameters are:

Ramps arrow buttons

The controls in the Ramps group box depend on the Flow Mode you choose. Use the up-down control to add or subtract the number of ramps to use in the pressure or flow program. This is the rate of change in mL/min that the column flow is increased or decreased. Click the down arrow to add another ramp level. Click the up arrow to delete the last ramp displayed.

Initial / Flow text box

The initial flow for the inlet and column at the start of the analytical run.

Initial / Hold Time text box

The amount of time the flow is held at the beginning of the run before the first ramp.

Ramp / Rate text boxes

The rate of change in mL/minute that the columns flow is increased or decreased.

Ramp / Flow text boxes   The amount of flow to be used for each ramp.

Ramp / Hold Time text boxes   The amount of time the flow is held at the end of the ramp. Setting Up Parameters for Programmed Flow Mode

Vacuum Compensation check box

Check this box to compensate for a vacuum at the end of the analytical column. This is usually the case where the detector is a mass spectrometer. When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

Gas Saver check box and group box

This group box contains gas saver controls, which reduce carrier gas consumption, especially when a large split flow is used. The gas saver options are set to come on at some point well after the injection to conserve gas. Checking the box will activate the other two features of gas saver.

Gas Saver Flow text box   The desired flow after the injection or in standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

Gas Saver Time text box   The time after the injection when gas saver flow will be activated.

Programmed Pressure Mode

Click on the Carrier tab of the GC Setup view, and then click on **Programmed Pressure** in the Flow Mode list box

Use the Carrier page, programmed pressure option to program the pressures to the carrier. The parameters are:

Ramps arrow buttons   The controls in the Ramps group box depend on the Flow Mode you choose. Use the up-down control to add or subtract the number of ramps to use in the pressure or flow program. For pressure programs, this is the rate of change in kPa per minute that the inlet pressure increased or decreased. Click the down arrow to add another ramp level. Click the up arrow to delete the last ramp displayed.

Initial / Pressure text box   The initial pressure for the inlet and column at the start of the analytical run.

Initial / Hold Time text box   The amount of time the pressure is held at the beginning of the run before the first ramp.

Ramp / Rate text boxes   The rate of change in pressure that the ramp is intended to reach.

Ramp / Pressure text boxes The pressure to be applied during injection and at the beginning of the ramp.

Ramp / Hold Time text boxes The time to maintain the value in the ramp/pressure field.

Vacuum Compensation check box

Check this box to compensate for a vacuum at the end of the analytical column. This is usually the case where the detector is a mass spectrometer. When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

Gas Saver check box and group box

This group box contains gas saver controls, which reduce carrier gas consumption, especially when a large split flow is used. The gas saver options are set to come on at some point well after the injection to conserve gas. Checking the box will activate the other two features of gas saver.

Gas Saver Flow text box The desired flow after the injection or in standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

Gas Saver Time text box The time after the injection when gas saver flow will be activated.

Setting Up Parameters for Constant Flow Mode

Use the following procedure to set up parameters for the constant flow option.

1. Open the Instrument Setup window.

2. Select the Carrier tab.

3. Check the On box in the Flow group box to turn on the flow.

4. Type in a value for the flow for the analytical column.

5. Check the Vacuum Compensation box to compensate for a vacuum at the end of the analytical column.

    When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

6. Check the Gas Saver box to activate gas saver controls. Checking the box will activate the other two features of gas saver:

    Gas Saver Flow (mL/min): The desired flow after the injection or in

standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

Gas Saver Time (minutes): The time after the injection when gas saver flow will be activated.

Setting Up Parameters for Constant Pressure Mode

Use the following procedure to set up parameters for constant pressure.

1. Open the Instrument Setup window:

2. Select the Carrier tab.

3. Check the pressure box to enable flow to the inlet. When this box is not checked gas flow goes to the inlet and through any installed column. If the oven is taken to a high temperature, the column may be damaged as well as any detectors or components attached to it.

4. Enter a value for the pressure for the analytical column.

5. Check the Vacuum Compensation box to compensate for a vacuum at the end of the analytical column.

6. When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

7. Check the Gas Saver box to activate gas saver controls. Checking the box will activate the other two features of gas saver:

    Gas Saver Flow (mL/min): The desired flow after the injection or in standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

    Gas Saver Time (minutes): The time after the injection when gas saver flow will be activated in the Instrument Setup window.

Setting Up Parameters for Programmed Flow Mode

Use the following procedure to set up parameters for the Programmed Flow option.

1. Open the Instrument Setup window.

2. Select the Carrier tab.

3. Select Programmed Flow from the Flow Mode spin box.

4. Check the Flow box to enable flow to the inlet.

   When this box is not checked gas flow goes to the inlet and through any installed column. If the oven is taken to a high temperature, the column may be damaged as well as any detectors or components attached to it.

5. Type in values for the initial flow and hold time for the analytical column.

6. Use the Ramp control to add extra ramps to the program.

7. Enter rate, flow, and hold time values for each additional ramp in the program.

8. Check the Vacuum Compensation box to compensate for a vacuum at the end of the analytical column.

   When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

9. Check the Gas Saver box to activate gas saver controls. Checking the box will activate the other two features of gas saver:

   Gas Saver Flow (mL/min): The desired flow after the injection or in standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

   Gas Saver Time (minutes): The time after the injection when gas saver flow will be activated.Related topics:

Setting Up Parameters for Programmed Pressure Mode

Use the following procedure to set up parameters for the Programmed Pressure option.

1. Open the Instrument Setup window.

2. Select the Carrier tab.

3. Select Programmed Pressure from the Flow Mode spin box.

4. Check the Pressure box to enable flow to the inlet.

   When this box is not checked gas flow goes to the inlet and through any

installed column. If the oven is taken to a high temperature, the column may be damaged as well as any detectors or components attached to it.

5. Type in values for the initial pressure and hold time for the analytical column.

6. Use the Ramp control to add extra ramps to the program.

7. Enter rate, pressure, and hold time values for each additional ramp in the program.

8. Check the Vacuum Compensation box to compensate for a vacuum at the end of the analytical column.
    When the box is not checked calculations are made for a normal GC detector, which is usually at atmospheric or slightly higher pressure.

9. Check the Gas Saver box to activate gas saver controls. Checking the box will activate the other two features of gas saver:

    Gas Saver Flow (mL/minute): The desired flow after the injection or in standby. Gas saver flow displays the carrier gas in milliseconds and minutes until the end of the run.

    Gas Saver Time (minutes): The time after the injection when gas saver flow will be activated.

3.12 ECD Page

This page is the method editor for the electron capture detector.

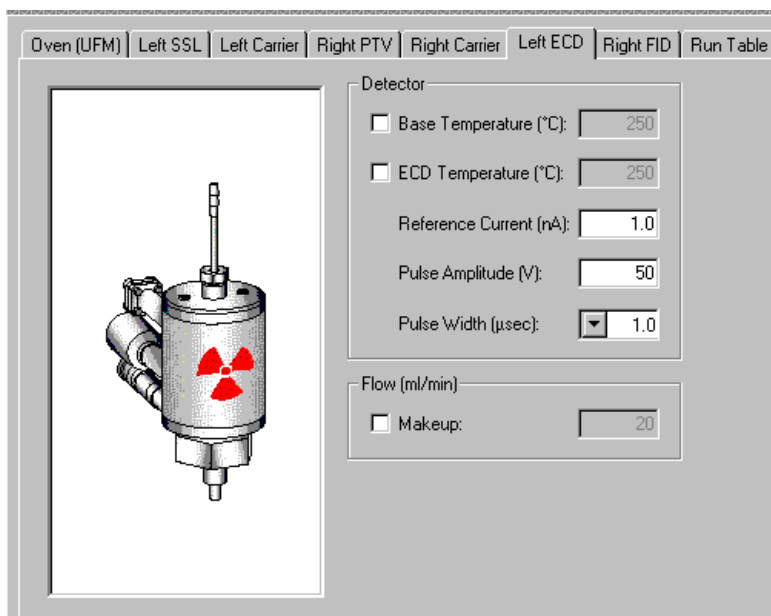To display this page: Click on the ECD page from the Instrument Setup view.

Figure 3-12. ECD Page View

Related topics:

•   *Setting Up Parameters for ECD*

ECD Parameters

Detector group box

Base Temperature   This is the ECD detectors base body temperature in degrees Celsius (300º to 350 ºC).

ECD Temperature   The temperature of the Electron Capture Detector in degrees Celsius. Select a temperature above the final oven temperature and high enough to prevent sample condensation inside the detector. The ECD temperature heats separately from the base temperature. Typical operating temperatures for the ECD are 300º to 350 ºC.

Reference Current   The amount of current in nano Amps applied as reference current to the

detector. Sometimes referred to as standing current. The value entered here is the current for the detector to maintain at a constant level. The settings for this current depends on the type of detector used with the ECD. Values from 0-3 nano Amps may be entered. Changes in this value may effect linearity. See the TRACE GC Ultra Operators Manual for more detailed information.

Pulse Amplitude   The voltage applied to the detector. Consult your ECD Detector manual for more detailed information. Values from 5 V to 50 V may be entered. Typically the pulse amplitude is set to 50 V. However, very clean systems may operate equally as well at 25 V or 15 V. This voltage is used to aid in achieving the selected reference current or standing current. Detectors exhibiting line out frequencies below 1-2 kHz should be operated at 25 V or 15 V.

Pulse Width   Select from 0.1, 0.5, or 1 microseconds to control the pulse going to the detector. The use of Nitrogen as make-up requires 1 ms pulse. Use of Argon/Methane requires a 0.1 ms pulse to produce the highest linear range. The value selected depends on the type of detector gas being used with the ECD. Nitrogen requires a pulse width value of 0.5 or 1.0. Usually 1.0 is the desired value.

Flow (mL/min) Group Box

Makeup   Specify how much make-up gas (0-90 cc/min) to activate. Nitrogen or Argon/Methane is suggested as the make-up gas to the ECD detector. Checking this box enables the use of make-up gas into the detector. To reduce line out time, use higher flow rates around 60 mL/min when the detector is first turned on. Reduce the makeup to 30 mL/min for optimumresults.

Setting Up Parameters for ECD

1. .Set the detector base temperature to 250 ºC

2. Set the ECD temperature to 300 ºC.

3. Set the Reference Current to 1 nA

4. Set the Pulse Width to 1 μ sec.

5. Set nitrogen makeup gas at 30-40 mL min.

Allow several hours for the system to line out. The detector frequency should stabilize below 5 kHz.

3.13 FID Page

Note: For auxiliary, right, and left usage only.
This page is the method editor for the flame ionization detector.
If you are using a stacked detector and using an ECD, then the makeup gas is usually plumbed separately for the ECD, and is specified as the Electron Capture medium.

This page is the method editor for the electron capture detector.

To display this page: Click on the FID page from the Instrument Setup view.
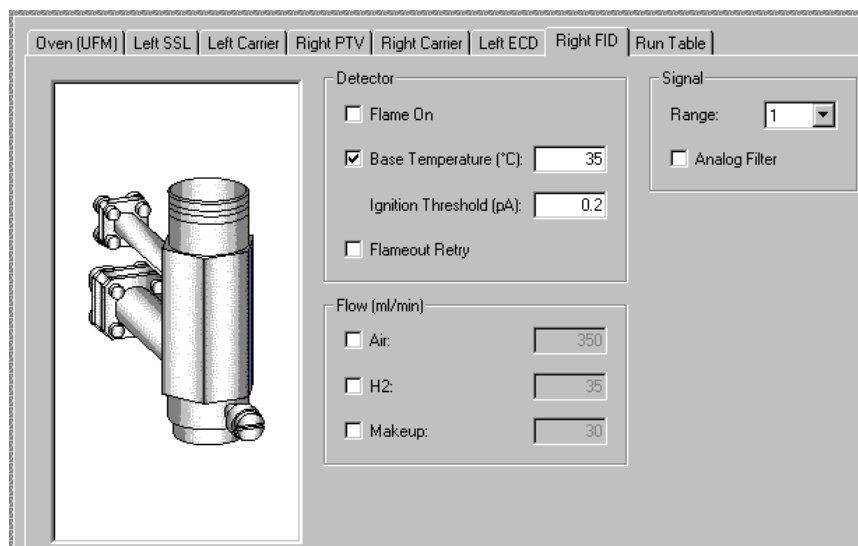

Figure 3-13. FID Page View

Related topics:

• *Setting Up Parameters for FID*

FID Parameters

Detector Group Box

Flame On Checking this box turns on the detector gases if not previously on, and turns on the ignition coil to light the flame. Unchecking this box shuts off the detector gases, extinguishing the flame.

Performs several functions: turns on detector gases, increases hydrogen flow above the setpoint to 40 mL/min, decreases air below the setpoint to approximately 200 mL/min, turns the makeup gas off, supplies current to the ignition coil.

Base Temperature This is the temperature in degrees Celsius of the detector base body. Enter values 250º to 350º. To prevent stationary phase condensation on the analytical column, operate the detector at a higher temperature than the oven program. Pay special attention to the analytical column's upper limit.

Flameout Retry Use this to monitor the signal. If the signal should drop below the Ignition Threshold , it will turn on the ignition coil and attempt to light the FID. Flameout Retry repeats up to three times. Flameout Retry compares the ignition threshold value to the signal pA.

Ignition Threshold The point at which the FID attempts to re-ignite. The signal in picoamps for the GC to use to compare the current signal to determine if the FID flame is still lit. If the signal pA is lower than the threshold the FID flame function indicates igniting When the flame ignites the signal pA increases above the ignition threshold and the ignition process stops. If the signal pA does not increase above the ignition threshold value, the flame function attempt to ignites the FID three times and then stops. The flame function indicates out.

Flow (mL/min) Group Box

Checking any of the following controls enables gas flow from the DGFC to the detector. Checking any of these boxes and downloading the method will turn on the particular gas even if the Flame On box is not checked.

Air Specify how much airflow (0-600 cc/min) to activate. Acceptable values are On/Off. When the air is turned on, enter the value of the air supplied to the detector to optimize the flame. Typical flow is 350 mL/min.

H2 Specify how much Hydrogen flow (0-200 cc/min) to activate. Acceptable values are On/Off. When the Hz is turned on, enter the value of Hydrogen to supply to the detector to optimize the flame. Typical flow is 25 mL/min.

Makeup Specify how much makeup gas (0-90 cc/minute) to activate. Nitrogen is recommended as the makeup gas to the FID detector. Acceptable values are On/Off. Makeup gas enhances FID sensitivity. Typical flow is 30 mL/min.

Signal Group Box

Range (nA) The range for the detector signal to be acquired. Selections are 1, 10, 100, 100 however, the lowest setting, 0, gives the most sensitivity. A higher range may be needed for high concentration samples. Range represents the amount of current required in nano amps from the detector to offer a full-scale output at the selected range.

Analog Filter Electronic filtering used to reduce baseline noise. Acceptable values are On/Off. The analog filter applies a time constant to the analog output reducing

the response times to high frequency baseline noise thus filtering the analog output.

Setting Up Parameters for FID

1. Check the FID base temperature check box.

2. Check the base temperature box and set the temperature to 250 ºC.

3. Turn on the detector gas settings to: H_2 = 30 mL/min, Air=350 mL/min, Makup=30 mL/min.

4. Turn the flame on.

3.14 NPD Page

Note: Use in Auxiliary, right, and left detector positions.

This page is the method editor for the nitrogen phosphorus detector.

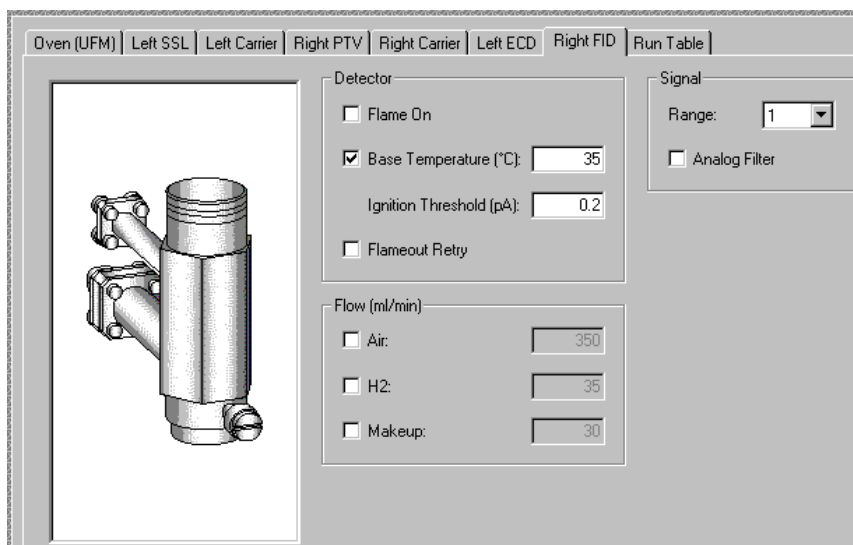To display this page: Click on the NPD page from the Instrument Setup view.

Figure 3-14. NPD Page View

Related topics:

• *Setting Up Parameters for NPD*

The Nitrogen-Phosphorus detector detects Nitrogen and or Phosphorus containing organic compounds.

The NPD source is a ceramic bead impregnated with alkali metal. The source is mounted just above the jet with air, makeup and a dilute concentration of hydrogen combined with the column effluent. The source is heated to high temperature producing plasma around the bead, which decomposes the sample. Interaction with the thermionic source is thought to produce the ions attracted and detected by the collector. A low hydrogen flow (< 4mls/min) is used which will not sustain a flame. An NPD (Nitrogen/ Phosphorus Detector), is an alkali-sensitized thermionic detector used for the specific detection of phosphorus and organic nitrogen. The NPD is very sensitive and is instrumental in pesticide and herbicide residue analysis in various EPA methods. This detector is also useful for the clinical analysis of caffeine, nicotine, and other nitrogen-containing drugs.

NPD Parameters

Detector Group Box

Source Current　Specify the current amperes necessary to generate a background current.

Base Temperature　This is the temperature in degrees Celsius of the detector's base body. Maximum range is 450 ºC. Typically detectors that use the detector base temperature as the sole source of heat are set to 300 ºC. Of course the detector base temperature should always be at least 10 ºC higher than the maximum column oven operating temperature. Detectors that have a secondary heat source require a base temperature of typically 250 ºC.

Polarization Voltage (v)　Select a polarization voltage (1.0-9.9) to apply to the collector. Polarizing voltage is a DC voltage that is superimposed on to the NP source assembly. Adjust the polarizing voltage from 1.0 to 9.9 in .1 V increments.

Flow (mL/min) Group Box

Checking any of these boxes activates the gas flow from the DGFC module to the detector and downloads the method to turn on the particular gas.

Air　Specify how much airflow (60-100 µL/min) to activate. When the air is turned on, enter the value of the air supplied to the detector to optimize the flame. Typical flow is 70 mL/min.

NPD H2　Specify how much hydrogen air flow (2-4 cc/min) to activate. Acceptable values are On/Off. When the H_2 is turned on, enter the hydrogen range (0-10 mL/min) to supply to the detector, which optimizes the flame. Typical flow is 3 mL/min.

H2 Delay　Specify how many minutes to delay the H_2 flow within the detector.

Makeup　Specify how much makeup gas (10-30 µL/min) to activate. Nitrogen is recommended as the makeup gas to the NPD detector. Makeup gas has less thermal capacity and tends to cool the plasma and bead less. Makeup gas serves to sweep the column effluent into the detector energy field on plasma. Makeup should be nitrogen since Helium tends to cool the source. Typical makeup flow is 15 mL/min.

Signal Group Box

Range (nA)　The range for the detector signal to be acquired. Selections are 1, 10, 100, 100 however, the lowest setting, 0, gives the most sensitivity. A higher range

may be needed for high concentration samples. Range represents the amount of current required in nano amps from the detector to offer a full-scale output at the selected range.

Analog Filter   Check this box to enable electronic filtering, which reduces the response to high frequency baseline noise and signal.

Setting Up Parameters for NPD

1. In the Detector group box, set the Source Current to 2.720 or as required to generate a background current. Set the Base Temperature to 300 ºC. Set the Polarization Voltage to 3.5.

2. In the Flow group box, set the Air to 7.0. Set the H_2 to 3.0. Set the H_2 delay to how many minutes you want to wait before starting the hydrogen flow. Set the Makeup gas to 15.

3. In the Signal group box, set the Range to the desired sensitivity. Check the Analog Filter box if you want to reduce the response to high frequency noise and signal.

3.15 TCD Page

This page is the method editor for the thermal conductivity detector.

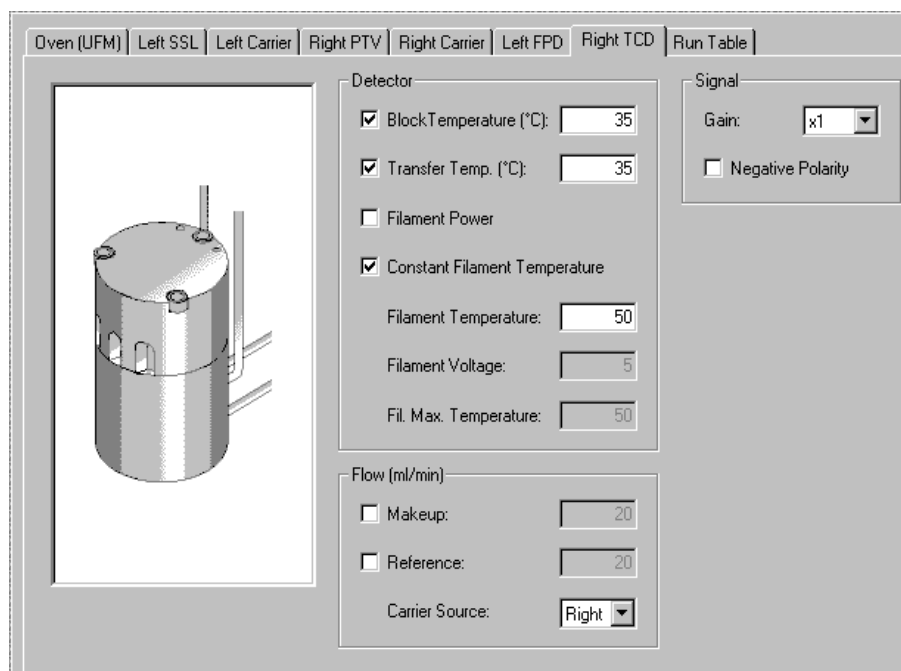To display this page: Click on the TCD page from the Instrument Setup view.

Figure 3-15. TCD Page View

Related topics:

• *Setting Up Parameters for TCD*

TCD Parameters

Detector Group Box

Block Temperature Sets the TCD cell temperature. Enter an integer between 30 and 450 ºC. Typical temperature is 200 ºC depending on the application you are using. A general rule to apply is to set the temperature to a higher value than the maximum temperature reached by the GC column oven during the analysis.

See Recommended Filament Temperature and Voltages .

Transfer Temp. Enter an integer between 20 and 400 ºC. This temperature heats the transfer line between the GC oven and the TCD cell. We recommend setting a value

higher than or equal to the set oven temperature.

Filament Power   Check this box to turn on power to the TCD filaments.

Constant Filament Temperature   Check this box to activate the Filament Temperature control only and disables Filament Voltage and Fil. Max Temperature controls. This operating mode keeps a constant filament temperature based on the relationship between the block temperature and the filament temperature and carrier gas. When using low thermal conductivity gases the filament temperature is high for low voltage supply. See Recommended Filament Temperature and Voltages .

Filament Temperature   Enter an integer between 50 and 450. Adjust in 10 ° increments. Filament temperatures should be kept at least 20 ºC higher than the block temperature . The greater the difference the better the sensitivity. However, the useable difference between the block temperature and the filament temperature depends on the carrier gas you are using. See the TRACE GC Ultra Operators Manual for suggested carrier gas values and the TCD. See also *TCD Recommended Filament Temperature and Voltages*

Filament Voltage   Enter an integer between 5 and 15 in 1 V increments. Filament voltages are closely related to filament temperature values. Typical values are 5 to 7 V. See *TCD Recommended Filament Temperature and Voltages*

TCD Recommended Filament Temperature and Voltages

Table 3-1.

Detector Temperature 100 ºC

Table 3-2.

Voltage (v)	5	6	7	8	9	10

Table 3-3.

Filament Temperature	235	275	315	355	395	435

The detector's block temperature and filament temperatures, voltages, and carrier gas influence the analysis sensitivity and filament's average life-time.

Fil. Max. Temperature   Limits the filament temperature when the detector operates in the constant voltage mode. Enter an integer between 50 to 450 ºC. See Recommended Filament Temperature and Voltages.

Flow (mL/min) Group Box

Makeup   Makeup indicates the make-up gas flow rate. Enter an integer between 0 and 100. If the value is less than or equal to 10 mL/min, the filament power is

disabled.

Reference   Set the appropriate reference gas flow. Enter an integer from 1 to 100. If the value is less than or equal to 10 mL/min, the filament power is disabled. Reference gas is used in addition to the sample's carrier gas to measure the gas conductivity when sample moves across both filaments. Reference gas must be same type of carrier gas and use the same flow rate.

Signal Group Box

Gain   Set the gain to x1 or x10. When a x10 gain is selected the GCs sensitivity increases the detector output signal and electrical and mechanical noise amplification.

Negative Polarity   Select this control to reverse the polarity output signal as a TC carrier gas function versus the sample and the analytical column (channel right or hannel left) used.

Setting Up Parameters for TCD

These steps are typical parameters for analyzing samples using Helium as a carrier gas.

1. In the Detector group box, select Block Temperature and enter 150. Select Transfer Temp and enter 150. Check the Constant Filament Temperature box to turn the filament power on. Enter 250 in the Filament Temperature text box.

2. In the Flow group box select Makeup if you are using makeup gas. Select Reference if you need to set a reference gas flow.

3. In the Signal group box, set the Gain ifyou need amplified sensitivity. Select Negative Polarity to reverse the TC gaspolarity.

3.16 FPD Page

Auxiliary usage only.

This page is the method editor for the flame photometric detector.

To display this page: Click on the FPD page from the Instrument Setup view.
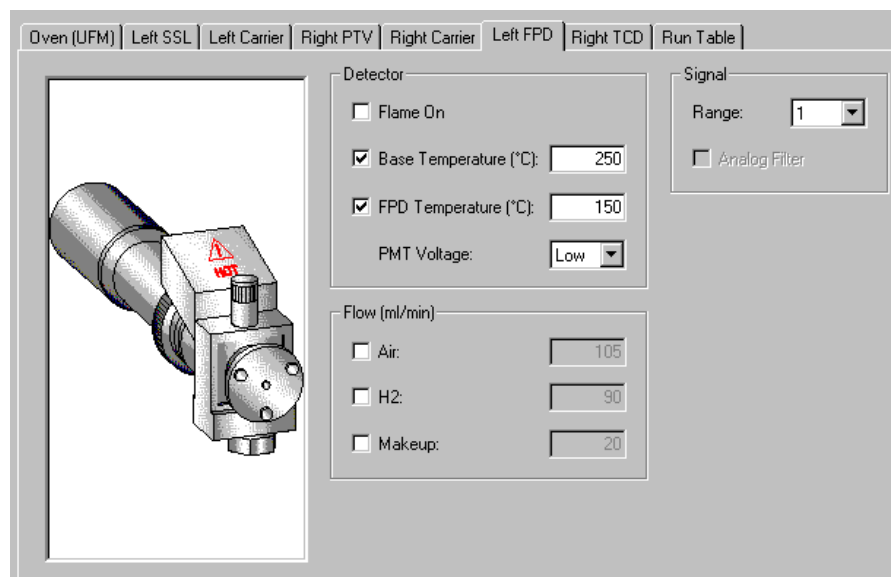

Figure 3-16. FPD Page View

Related topics:

•   *Setting Up Parameters for FPD*

FPD Parameters

Detector Group Box

Flame On   Select this control to turn the detector ignition and detector gases on.

Base Temperature   Select to set the detector base body temperature. For an FPD set at 250 ºC.

FPD Temperature   Sets the detector cell temperature. A typical operating temperature is 180 ºC.

PMT Voltage   Sets the value for the dynode voltage to apply to the photo-multiplier table. Values are high and low. Low set the voltage to –800 V and high sets the voltage to –900 V.

Flow (mL/min) Group Box

Air   Adjusts the air supply to the detector. Typical values are 200 mL/min.

H2   Adjusts the hydrogen gas supply to the detector. Typical value is 150 mL/min.

Makeup   Adjusts the makeup gas to the detector. Preferred gas type is Nitrogen and a typical setting is 30 mL/min.

Signal Group Box

Range   Changes the electrometer gain. Valid selections are 1, 10, and 100. With 1 yielding the most sensitivity.

Analog Filter   Electronic filtering used to reduce baseline noise. Acceptable values are On/Off. The analog filter applies a time constant to the analog output reducing the response times to high frequency baseline noise thus filtering the analog output.

Setting Up Parameters for FPD

1. In the Detector group box, set the Base Temperature to 250 ºC. Set the FPD Temperature to 180 ºC. Set the PMT Voltage to low. Optional: Flame On.

2. In the Flow group box, set the Air to 200 ºC. Set the H2 to 150 ºC. Set the Makeup gas to 30 ºC.

3. Optional: In the Signal group box, set the Range and Analog Filter.

3.17 PID Page

This page is the method editor for the photoionization detector.

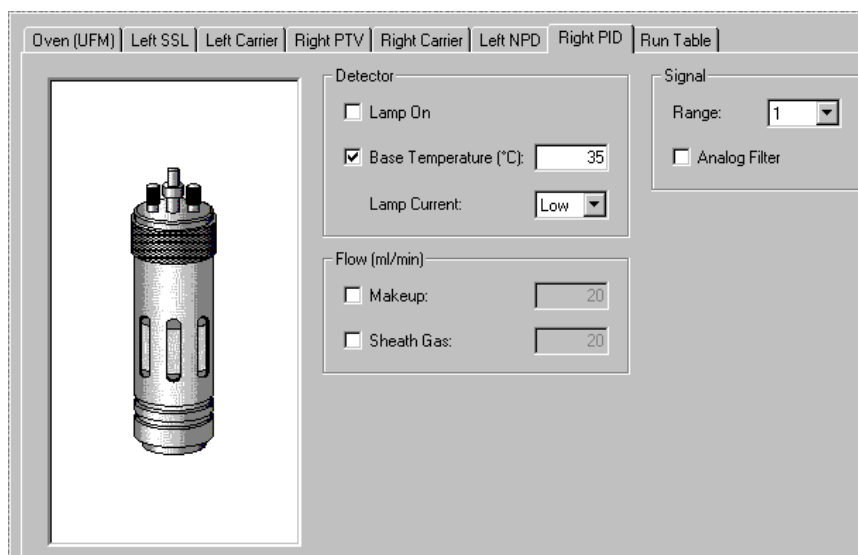To display this page: Click on the PID page from the Instrument Setup view.

Figure 3-17. PID Page View

Related topics:

•   *Setting Up Parameters for PID*

PID Parameters

Detector Group Box

Lamp On   Turns the UV lamp power on, which strikes the lamp and thus creates a lamp current.

Base Temperature   Turns the detector base body temperature on.

Lamp Current   Selects the PID lamp current. Valid selections are low and high. The higher the lamp current the more intense the transmitted lamp transmission, which enhances sensitivity, but reduces lamp life.

Flow (mL/min) Group Box

Makeup   Turns on the makeup gas. Typical value is 15 mL/min.

Sheath Gas   Flows around the outside of the cell to reduce temperature. Typical flow value is 30 mL/min.

Signal Group Box

Range   Changes the electrometer gain. Valid selections are 1, 10, 100, 1000. With one yielding the most sensitivity.

Analog Filter   Electronic filtering used to reduce baseline noise. Select On or Off. The analog filter applies a time constant to the analog output reducing the response times to high frequency baseline noise thus filtering the analog output.

Setting Up Parameters for PID

1. In the Detector group box, set the Base Temperature to 180 ℃. Set the Lamp Current to low. Optional selections: Lamp On.

2. In the Flow group box, set the Makeup gas to 15-20 mL/min. Set the Sheath Gas to 20-30 mL/min.

3. In the Signal group box, set the Range to X1. Optional selections: Analog Filter.

3.18 Aux Zones Page

Ths page is the method editor for the setting auxiliary connections.

All selections on this page must exactly match the settings you have manually entered into your TRACE GC Ultra and the TRACE GC Ultra detector configuration window. See *Detectors and Data Page*.

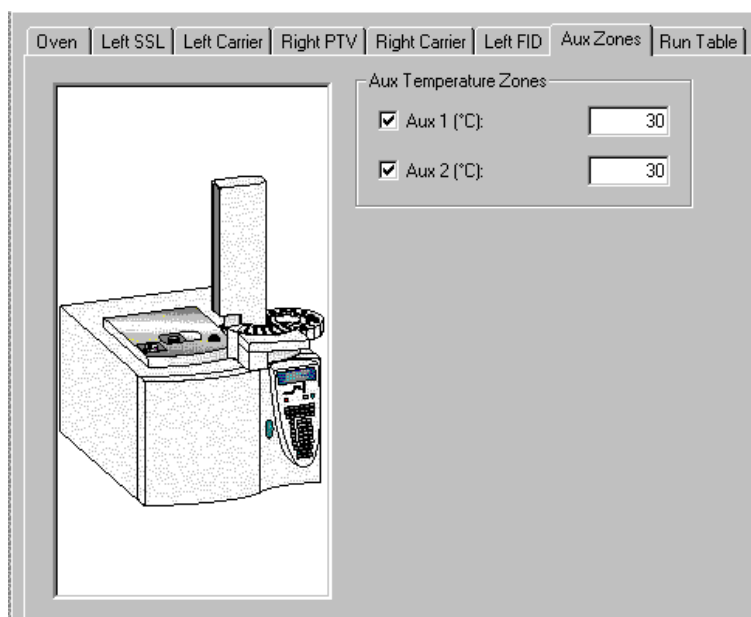To display this page: Click on the Aux Zones page from the Instrument Setup view.

Figure 3-18. Aux Zones Page View

Related topics:

•   *Setting Up Parameters for Aux Zones*

Aux Zones Parameters

Aux Pressure Zones Group Box

When you check one of these boxes (Aux 1 or Aux 2) you let the GC know you have an auxiliary heater.
Set a temperature from 0-450. If any of these pressure channels (Aux 1-3) are connected to your TRACE GC Ultra, then check the box and enter a value from 0-700 kPa.

Setting Up Parameters for Aux Zones

1. Make sure that auxiliary configurations are selected in the *TRACE GC Ultra Configuration* program.

2. Check either Aux 1 or Aux 2 in the Aux Temperature Zones box and then enter a temperature value of 0-450.

3. Check if any of these pressure channels are present (Aux 1, Aux 2, or Aux 3) controls located in the Aux PressureZones box and then enter a temperature value of 0-700kPa.

3.19 Run Table Page

This page is the method editor to program events to happen before or during a run. For example, a valve could open two minutes into a run. Listed in the run table page are settings entered prior to the run in the Run Time Event dialog. Time, item, and settings display in this run table. Controls listed in the Initial Values group box are for switching external devices On/Off.

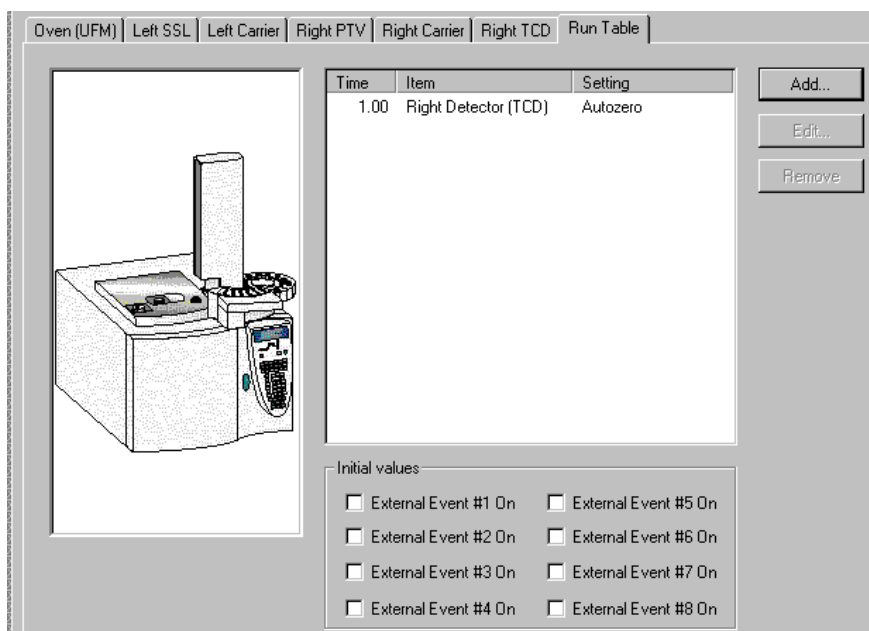To display this page: Click on the Run Table page from the Instrument Setup view.

Figure 3-19. Run Table Page View

Related topics:

•   *Setting Up Parameters for Run Table*

•   *Using the Add/Event Run-Time Event Dialog*

Run Table Parameters

Time, Item, and Settings

Time   Displays which Event Time (Prep run or Run Time) was selected in the Run Time Event dialog.

Item   Displays which event (Detector, Valve, or External) was configured in the

Run Time Event dialog.

Setting   Displays the Detector Events settings or On/Off states for Valve and External Events.

Click [Add...] to view the Add/Event Run-Time Event dialog box. Run items you select from this dialog are added to the time, item, and setting table.

Click [Edit] to edit a selected run item listed in the Time, Item, and Setting table.

Select an item listed in the time, item, and setting table. Click [Remove] to remove the selected run item from the run table.

Initial Values Group Box

External Event   Located on the back of your TRACE GC Ultra is a port with eight contacts that switches external devices On/Off. During a specific run times you can use these controls to set the status of these external switches prior to the run. Factory defaults for the beginning of each run is off (not checked).

Setting Up Parameters for Run Table

1. Select a control from the Event Time Group Box. When do you want to start the run at Prep Run or at Run Time?

2. Select a Detector Event, Valve Event, or External Event.

3. Select the controls available under the evnt type you selected

4. Click Ok.

Using the Add/Event Run-Time Event Dialog

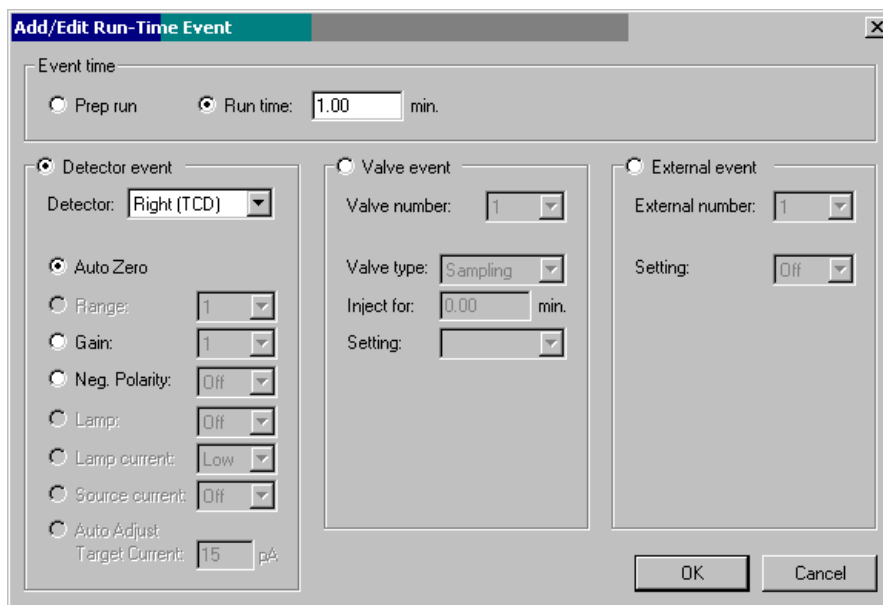If you selected Add or Edit from the Run Table Page this dialog box displays.

Figure 3-20. Add/Run-Time Event Page View

Event Time group box

Select one of the following two options

Prep Run This option makes the selected (Detector, Valve, or External) happen *before the run* begins.

Run Time This option makes the selected (Detector, Valve, or External) happen at the time specified *during the run*.

Detector Event Group Box

Click the detector event button to activate a selected detector group of events:

Detector Only the detectors you have selected from your TRACE GC Ultra Instrument Configuration program display in the Instrument Setup window. Select the one you wish to add a run time event for during this run. Each detector has some programmable events, and the ones you have available depend on your detector selection.

Autozero   Select this option to cause the detector offset to automatically adjust and force the output signal to zero (1000 signal counts).

Range   Select this option to change the electrometer sensitivity for ionization detectors. This function is useful for known sample matrices, which exhibit wide and varying analyte concentrations.

Gain   Select this option to change the TCD detector sensitivity. This is useful for known sample matrices, which exhibit wide and varying analyte concentrations.

Neg. Polarity   Select this option to invert the TCD detector response. This is useful for quantifying a negative going peak such as Hydrogen when a carrier such as Helium is used as carrier and reference gas.

Source current   Select this option to allow for source current changes for the NPD detector during runtime.

Lamp   PID specific. Turns the UV lamp power on, which strikes the lamp and thus creates a lamp current. This event is useful to enable the PID lamp only during specific analysis times, such as after solvent elution.

Lamp current   PID specific. Selects the PID lamp current. Valid selections are low and high. The higher the lamp current the more intense the transmitted lamp transmission, which enhances sensitivity, but reduces lamp life.

Valve Event Group Box

If you have either a valve oven or sample valves then click the valve event button to activate valve events:

Valve number-   Select from 1 to 8 for each external event to turn on from the Run Table page. If you use all 8 then none are available for other events.

Valve type-   Select one: Switching, sampling, or Multiposition. Switching is On or Off. Sampling is a valve used to inject a sample volume into the carrier stream. Multiposition allows you to use 1 to however many positions your valve has.

Setting   Select Load or Inject for the sample valve settings. Whichever valve type you select determines the available setting selections in this box.

External Event Group Box

External events are external valves, switches, and options.Click the external event button to activate external events:

External number   Select which of the eight (8) external events you wish to program. There are 8 available open collector outputs on the GC rear panel, which can be configured to switch external valves, switches, and other options.

Setting   Select On or Off to set the external event at the specified programmed run time.

Chapter 4
How To....

This Chapter contains these topics:

4.1 Installing the Desolvation Column and Connecting the Tee

1. Turn the carrier gas off at the keypad on the GC. Open the butterfly valve to the OC injector, and manually insert an extra 250 uL into the inlet. The syringe needle will extend into the GC oven. Install the desolvation column by sliding it over the needle, using the needle as a guide. Tighten the male nut with the tapered end of the ferrule pointing up while pressing the column upward as far as it will go. Tighten the nut with a wrench ¼ turn beyond finger-tight. Be sure to install the uncoated or front end of the column into the injector.

2. Use the GCQ software to open a new method in the method editor and save it to a new name. This is your LVI method. Press the GC icon and then select Inlet and change the inlet selection to left. Save the method and then send it to the GC. This will switch the DFPC or carrier gas control to divert the carrier gas from the right inlet to the left. Turn the carrier gas on at the keypad on the GC.

3. Place the effluent end of the desolvation column into a vial of solvent and note bubbles indicating flow.

4. Position the end of the desolvation column with a ferrule and nut loosely into the tee. If the tee has not been installed, remove it from the bag and insert it into the solvent vent valve bulkhead from inside of the oven with a ferrule and 1/16 nut. Slide the analytical column into the tee and inside the desolvation column with the ferrule and nut in place. The analytical column should protrude through the desolvation column about 4 cm. Tighten the fittings ¼ turn beyond finger tight. Note flow through the end of the analytical column by placing the end in a vial of solvent.

5. Install the column into the mass spectrometer as described in the *GCQ Plus Hardware Manual*. If the analytical column was previously installed in the right inlet, simply move it to the tee. You do not have to shut down the mass spec, since the restriction of the narrow bore capillary will prevent venting the detector to atmosphere.

6. Proceed to *Characterizing Columns*.

4.2 Characterizing Columns

Columns must be characterized after they are installed in order to deliver the correct flow rate set in the GC Method Editor. The procedure is performed at the keyboard of the GC. A constant for the algorithm for regulation of column flow is determined. This constant, K, is the restriction of the column.

Column characterization may be invoked at the keyboard of the GC. Press the column characterization button. The display will show the electronic gas control change the pressure and record the actual flow rate for each setting. These values will be inserted into a formula to calculate the K value or restriction of the column.

To test the column for leaks, raise the oven 50°C higher than the temperature that the column characterization was performed and press the LEAK CHECK button on the GC.

4.3 Post Column Splitting of the GCQ with the Flame Ionization Detector

Introduction:

Through Xcalibur a simultaneous analysis may be made on a GC detector and the Mass Spec from of a single injection. A post column or effluent splitter may be installed to "split the analytical column flow between the Mass Spec and the Flame Ionization Detector (FID). An SGE outlet splitter union with fused silica transfer lines, at a combined i.d. of 70-80% if the i.d. of the analytical column, is used to make the split. The kit includes a single hole ferrule for the analytical column side of the union and a two hole ferrule for the split side for attachment of the two transfer lines to the Mass Spec and the GC detector. The actual split is initially calculated to balance the flows between the two outlets, with one operating under vacuum (Mass Spec) and the GC Detector, at atmospheric pressure. The response may be enhanced on the Mass Spec by shortening the transfer line, diverting more of the split to it than the GC detector. Since the sample is being split between two outputs, the injection volume must be doubled.

Installing the System to Meet Instrument Specifications:

The GCQ should be installed to manufacturing specs with the standard test column connected to the splitless injector and the Mass Spec. After specs have been met, the Mass Spec is shut down and the splitter installed. Two microliters of the 3 component GCQ test mix is injected at a concentration of 1 ng/uL in hexane. The response, retention time, and peak shapes are checked. The retention times on the Mass Spec and the FID should be within 0.02 minutes of each other and the peaks should not show any significant tailing. The response for the ion trap will be quite a bit higher than on the Flame Ionization Detector (FID).

Connecting a Post Column SGE Splitter:

Using an SGE Capillary Outlet Splitter, two pieces of 0.1 mm i.d. fused silica transfer line were cut: 60 cm in length for the Mass Spec and 20 cm in length for the FID. Connect the analytical column to the inlet side of the splitter union and connect the transfer lines to the outlet side with a SGE two hole ferrule. Connect the outlet end of the transfer line to the FID with the standard detector nut and the CE Instruments graphite ferrule Part # 29013489.

Materials Required:

- SGE phone # (800) 945-6154

- SGE Outlet Splitter # 123630

- Fused Silica 0.1mm x 10 meter # 062469

- Two hole ferrules 0.3mm i.d. #072660

- Single hole ferrule 0.4 mm i.d # 072663

Calculating Transfer Line Dimensions for the Mass Spec and the GC Detector:

The flow calculator in Xcalibur was used to determine the length and i.d. of the transfer lines for the split. The flow calculator is accessed by selecting the TRACE GC Ultra icon button under the Instrument Setup. Press the word Trace, and Flow Calculator will be listed in the menu. After selecting the Flow Calculator, a dialog box will be displayed. Select helium for the carrier gas type. First determine the pressure above atmospheric pressure (14.69 psi) required providing a flow rate of 1.5 ml/min, using the dimensions of the test column at 50°C. The outlet is set to 1 Atm and the inside diameter to 0.25 mm with the length at 30 meters. The inlet pressure slide bar is increased to achieve a flow rate of 1.5 ml/min. This value is 16.37 psi or about 2-3 psi above atmospheric pressure.
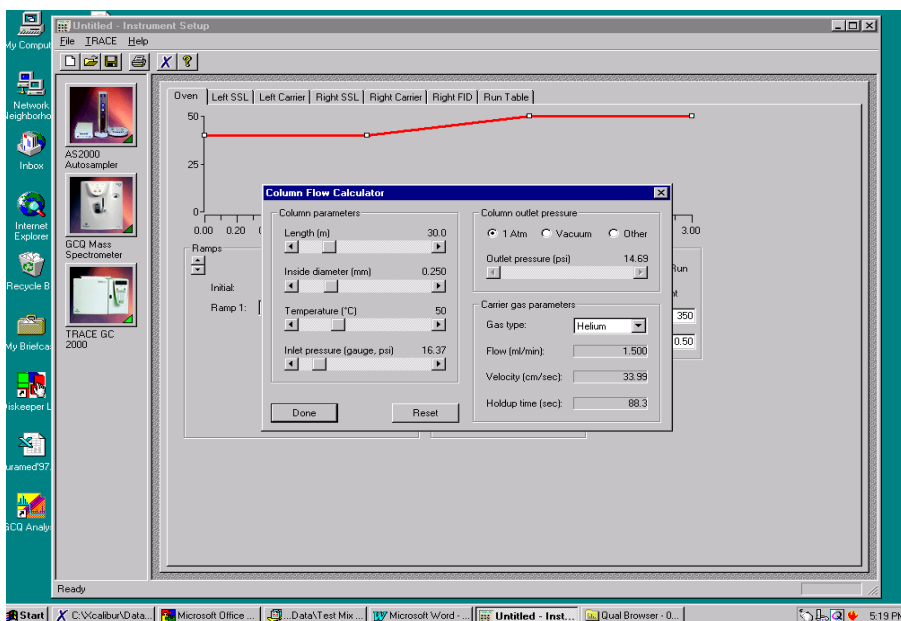
Now you can determine the length of the transfer line for the FID. Set the column outlet for the splitter to 1 Atm and the inlet pressure to 3 psi. The oven temperature remains at 50°C. The i.d. was set to 0.1 mm. Reduce the splitter length to a value such that a flow rate of ½ of 1.5 ml/min is shown or

0.747 ml/min. The length should be 0.2 meters or 20 cm. The dimensions for the FID splitter transfer line are 0.1 mm x 20 cm. Now calculate the length for the transfer line to the Mass Spec.

Leaving the oven temperature at 50°C and the i.d. at 0.1 mm set the outlet of the splitter to vacuum. With 3 psi at the inlet, increase the length until a flow of 0.8 ml/min is shown. The dimensions for the Mass Spec splitter transfer line should be 0.1 mm x 0.6 meters (60cm). At these conditions the flow should be split evenly between the Mass Spec and the FID.

Installing the Post Column Splitter:

Insert the effluent end of the analytical column into the union with a ferrule and insert the two transfer line sections into the other end of the union with the two-hole ferrule. Note that there is flow of carrier out each transfer line

section by placing them in a vial of solvent. A steady stream of bubbles will be seen. The FID transfer line will be inserted into the FID beyond the jet and then pulled back 2-3 mm below the jet to prevent burning the end of the fused silica in the hydrogen flame, which would result in loss of response. The loss would be severe; only a small peak for the solvent would be observed. The make up flow for the FID is set to 30 ml/min of nitrogen or helium, with the air at 350 ml/min and hydrogen to 30 ml/min. The detector temperature is set to 250°C. The transfer line to the Mass Spec is inserted 2mm beyond the vespel tip of the heated transfer line into the trap. The Mass Spec will indicate a vacuum of 20 mTorr at the lower flow rate of 0.8 ml/min.

Setting up the Carrier Flow:

The carrier gas flow exiting the analytical column was set to 1.5 ml/min helium in the constant flow mode. The flow may be entered at the keypad on the GC during set up and then in Xcalibur in the method under Instrument Setup. With the restrictor attached to the Mass Spec, the carrier gas control is set to vacuum compensation "off.

Leak Checking:

It is critical to tighten the splitter union so that no leaks are observed by checking with a gas leak detector. It is not recommended to use any soap leak test solutions like SNOOP on any fittings on the GCQ. Also check the bulkhead fittings on the back of the GC for leaks.

Evaluating the DFPC Modules for the Detector and Carrier Gases:

The DPFC module for the carrier gas should give a K value of about 1.879 with the outlet splitter installed to the FID and the Mass Spec when the standard test column is installed (Rtx 5 0.25 mm x 30 meter, 0.25 micron film). Close the split and septum purge vent valves on the keypad of the GC and test by pressing the column evaluation button and selecting the right inlet. If the value is lower, then you have a leak. If the value is higher like 14 or 22, you have a defective DPFC module.

The FID has a gas flow test for validation of the gas module calibration. The FID module is a type AC. This module provides control for the hydrogen, air and make up flows. A special adapter is enclosed to attach to the top of the FID for measuring the flows for this test. Turn the heater off to the FID and allow it to cool. Remove the chimney to the detector and install the adapter. Using an electronic flow meter verify the following flows:

Test	Low Flow Test	High Flow Test
Hydrogen	20 +/- 2	100 +/-5
Air	60 +/-6	300 +/-15
Make up	10 +/-1.5	50 +/-3

If the flows are not within the specifications, order a new module.

4.4 Operating an FID with Xcalibur Software

Introduction:

Xcalibur software allows the user to acquire from the Mass Spec and the Flame Ionization Detector (FID) simultaneously or independently.

Configuring Xcalibur and the TRACE GC Ultra:

First configure Xcalibur for all hardware that is installed on the TRACE GC Ultra. In order to acquire from the FID, a Channel will have to be selected for acquisition of the analog signal in the TRACE GC Ultra Configuration under the Detectors and Data Tab.
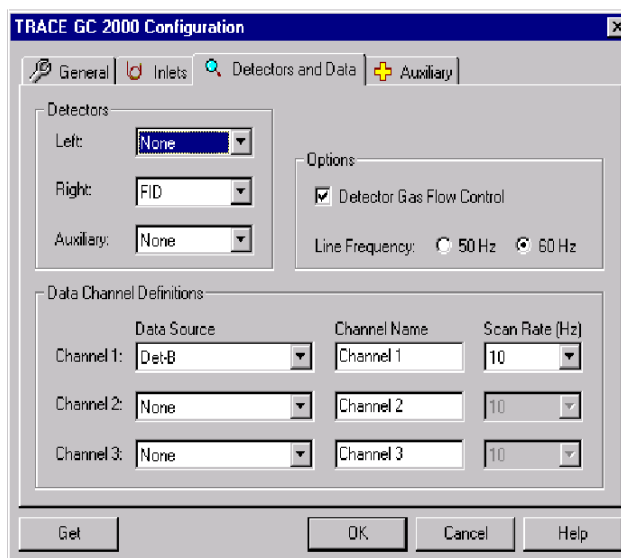
Verify that the FID is configured on the keypad of the TRACE GC Ultra. Unplug the Handshake (HS) cable from the back of the TRACE GC Ultra that connects to the Mass Spec. Review the menu for the Right Detector and be sure that the gases are all configured correctly. Standing in front of the GC, the right detector position is on your right side.

Connecting the Gases:

Connect hydrogen, air, and a make up of helium or nitrogen to the bulkhead fittings on the back of the GC. Set the pressure on the regulators at each tank to about 50 psi. The DPFC must be checked by placing a plug in the column end of the detector and installing the brass flow adapter for measuring flows on the detector base of the FID. You must remove the outside chimney to the detector first by unscrewing the thumbscrew and lifting the entire detector

body off. Verify the following flows and tolerances:

- H2   20 ml/min +/-2   60 ml/min +/- 5

- Air   60 ml/min +/-6   300 +/-15

- Makeup   10 +/-1.5   50 +/-3

If the flows do not match the tolerances, then change the DPFC module. Remove the flow adapter and reinstall the detector body.

Installing the Test Column:

The test column is installed into the split/splitless injector.   The column is inserted beyond the FID jet and then pulled back 2-3 mm. This prevents burning the end of the column in the flame. If the column is burned at the tip, the response will be low and only a small peak for the solvent will be seen.

Column Characterization:

Once the column has been connected, set the base temperature to 250°C and perform the column characterization. The Right carrier is set to constant pressure at 30 kpa with vacuum compensation off. Check to see that the split/splitless septum purge and split vents are off, by pressing VALVE on the keypad and ENTER. Next perform the column characterization. A "K value of 0.148 is typical for the Rtx 5 0.32mm x 7 meter, 0.25 micron film GC test column.

Igniting the Flame:

Once the column has been characterized, the detector gases may be turned on at the key pad of the GC and the flame ignited. Press ON at the first display for the Right Detector. The flame will ignite and the signal value displayed will go up. You will hear a soft pop. A shiny wrench may be held next to one of the exhaust holes at the top of the detector body and a condensation of water will be seen from the flame.

Setting up the Method:

In Xcalibur, under Instrument Setup, enter the values shown below for the method:
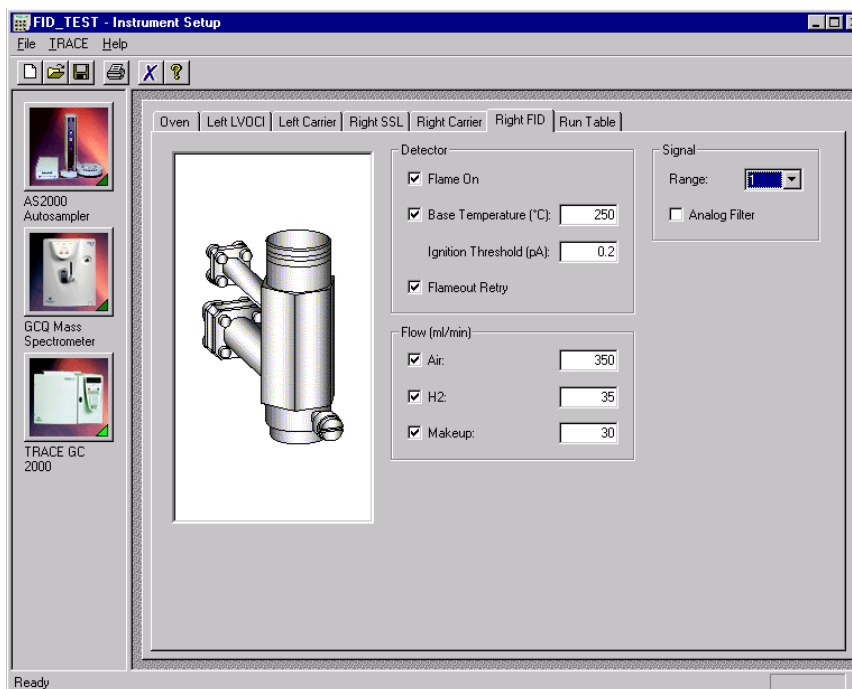
The Mass Spec will not be run, but you should set the temperatures to appropriate values for the ion source and the transfer line. Also put in a 5 minute filament delay, as a precautionary measure.

The oven program is set to 50°C, 1.0 min; 20°C/min; 200°C, 1.0 min. The split/splitless injector is set with a split flow of 60 ml/min in the splitless mode with a 1 minute inject time at 230°C.

## Setting up a Sequence in Xcalibur for Acquisition of the FID Only:

Write a sequence for injection of the test mix. When activating the sequence to run open the dialog box, "Change Instruments. Remove the Mass Spec from the list by a right mouse click on the word YES by Mass Spec. Press OK.
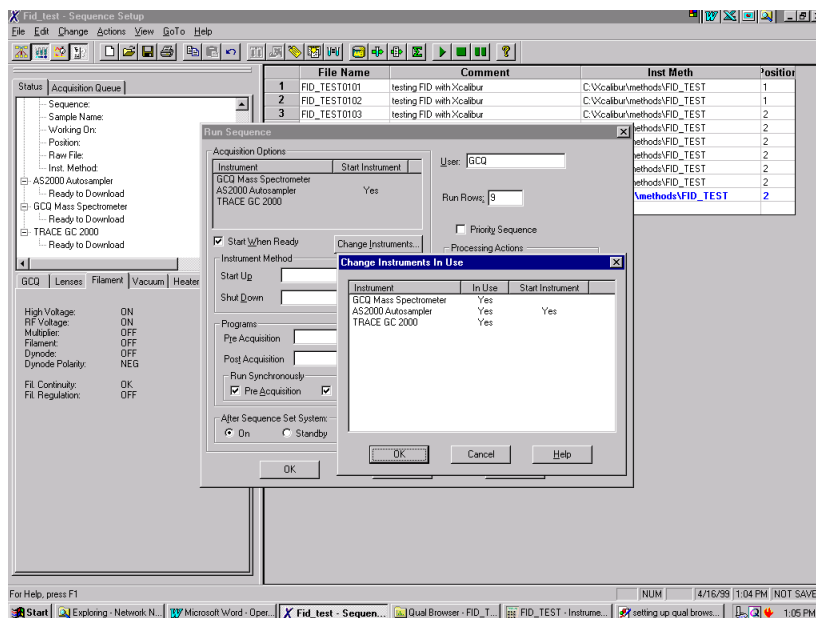
Now you are set up to run only the FID.
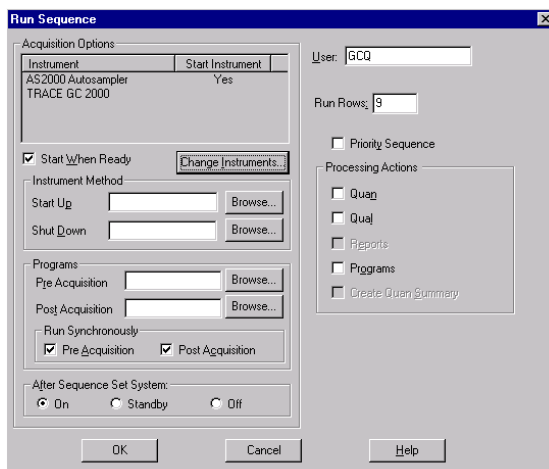
The real time plot will appear on the home page for the FID
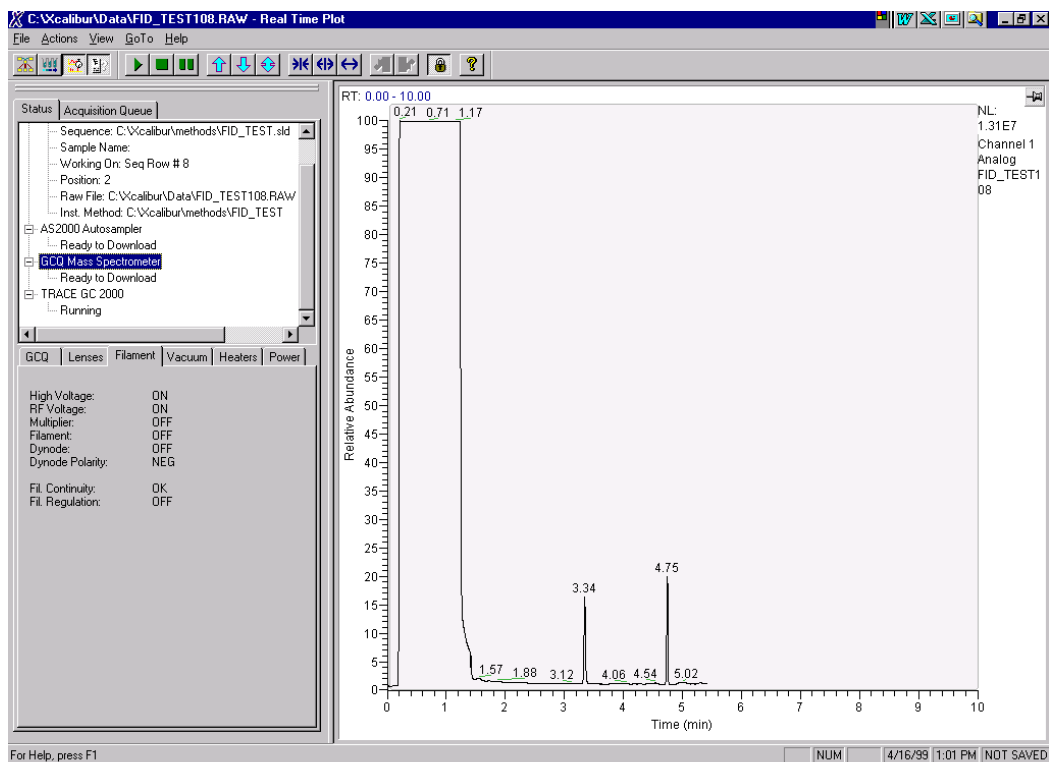
## Setting Up Qual Browser for the FID Analog Chromatogram:

Open the file. If this were a dual Mass Spec and FID run, you would have to select Channel 1 and set the Detector type to analog.
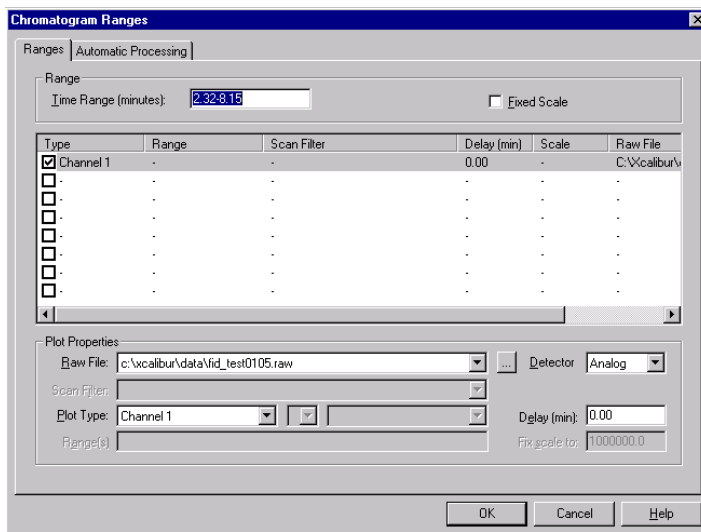
Meeting FID Specifications:

One uL of the test solution is injected at 20 ug/ml in hexane of C12H26, C14H30, and C16H34. A real time plot will be visible under the home page without the Mass Spec TIC. A representative chromatogram is shown below in Qual Browser.
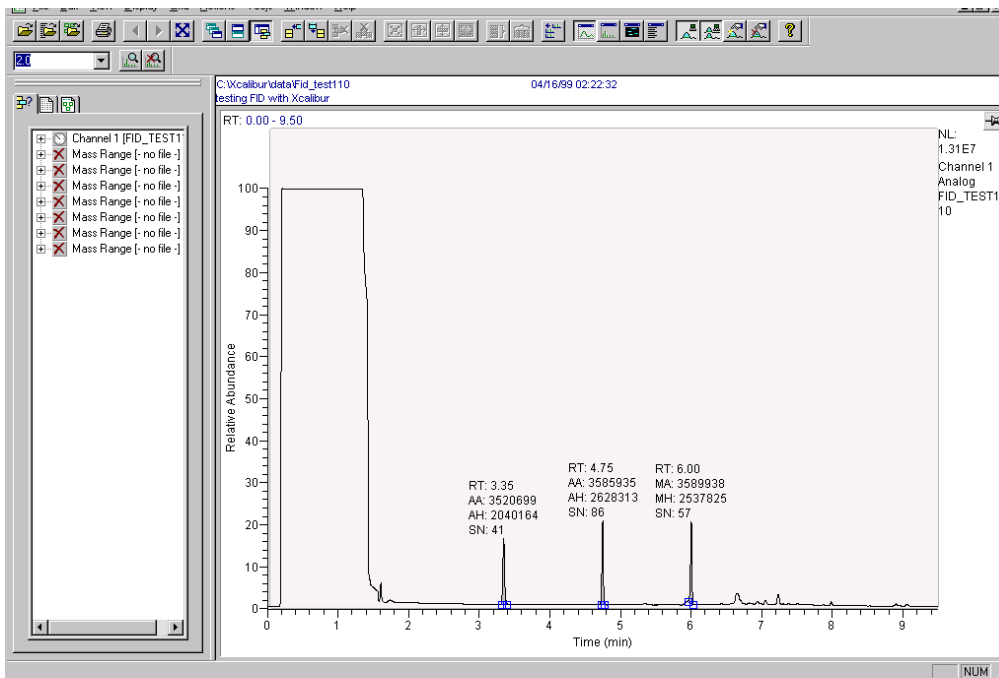

1 uL of the FID test solution is injected in the split/splitless inlet with an injection time of 1.0 minute. The FID gas flows were set to: hydrogen at 30 ml/min, air at 350 ml/min, and makeup at 30 ml/min nitrogen. The FID detector base temperature was set to 250°C and the inlet to 230°C. The range was put on 1. The spec calls for area counts   > 3,500,000 for each component. The ratio of C12H16/C16H34 must be 1.0 +/- 0.1. If the column was installed correctly and the injection time is set to 1.0 minute or less, a sharp front and back for the solvent peak should be seen. The peak shapes should be symmetrical.

# 4.5 Simultaneous Analysis on the Ion Trap and the FID

Configuration:

Close Xcalibur and open the Configuration icon. Select the TRACE GC Ultra and then configure. The configuration may be uploaded directly from the GC or entered. Go to the Detector and Data Tab and select Channel 1 for the Right FID with a scan rate of 10 Hz. Select Done and close Configuration. Open Xcalibur and proceed to Instrument Set up. By specifying a Channel for the detector, you invoke acquisition in every run.
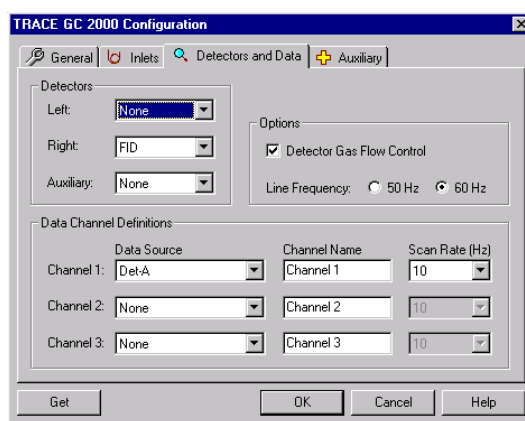
When you select "NONE for the Detector channel, no acquisition of a GC detector will occur. Press OK to save the changes. Then open Xcalibur.
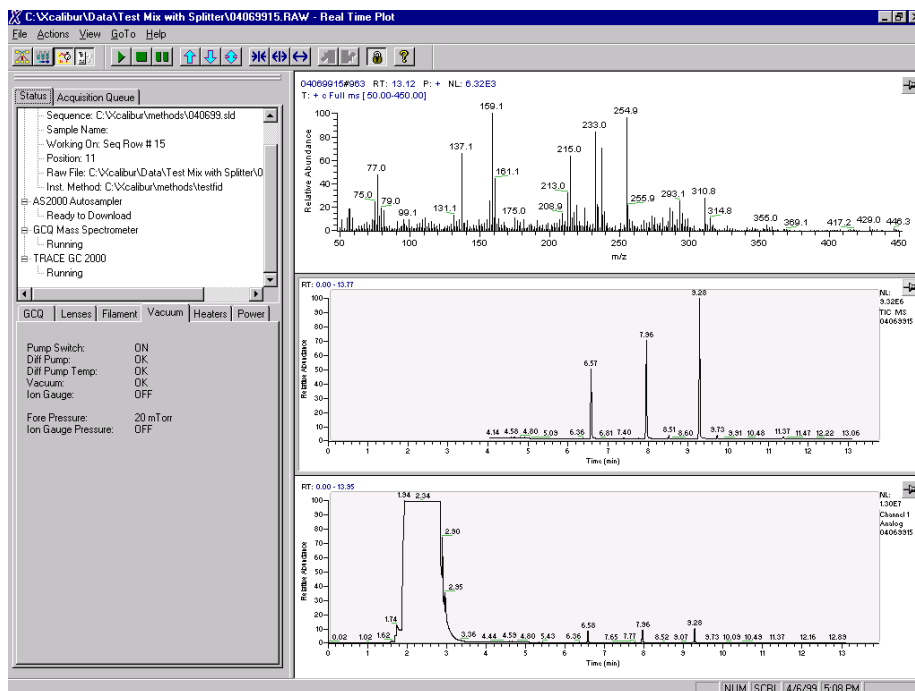
Instrument Method Set Up:

Go to Instrument Set Up from the Home Page and select new method. Enter the FID parameters listed below:
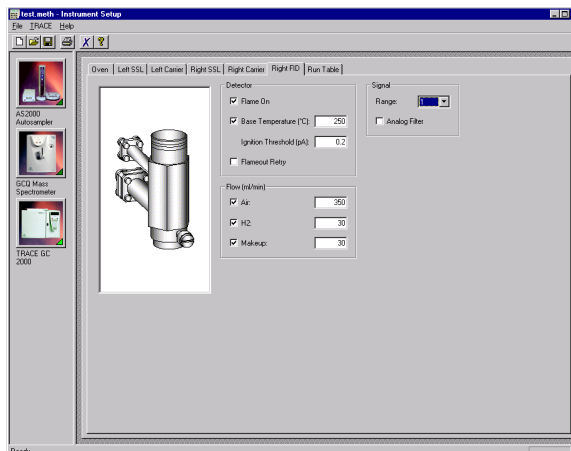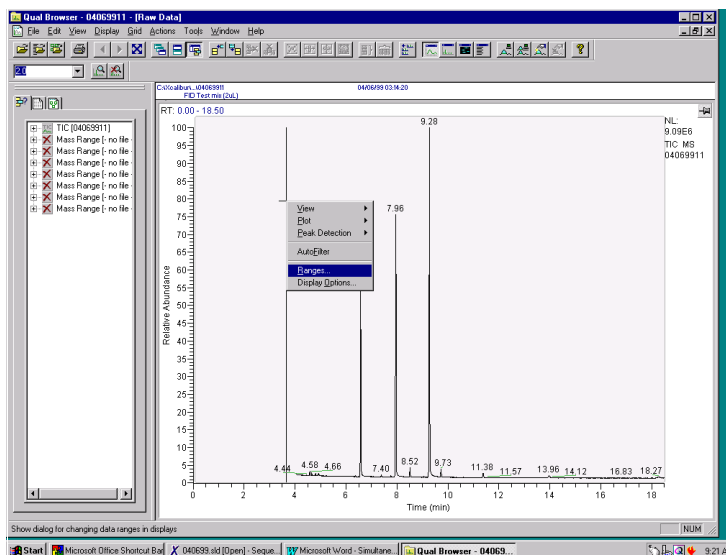
By entering a temperature for the FID, the detector will be turned on during acquisition and appear as an analog plot in real time at the bottom of the page with the real time spectrum at the top and TIC in the center.
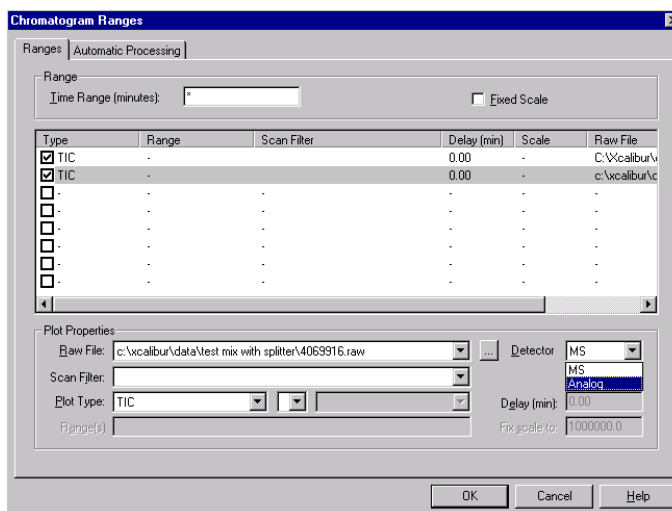

Opening a Data File in Qual Browser:

To open a data file in Qual Browser, select the file. The TIC will appear. Press the right mouse button and select ranges.
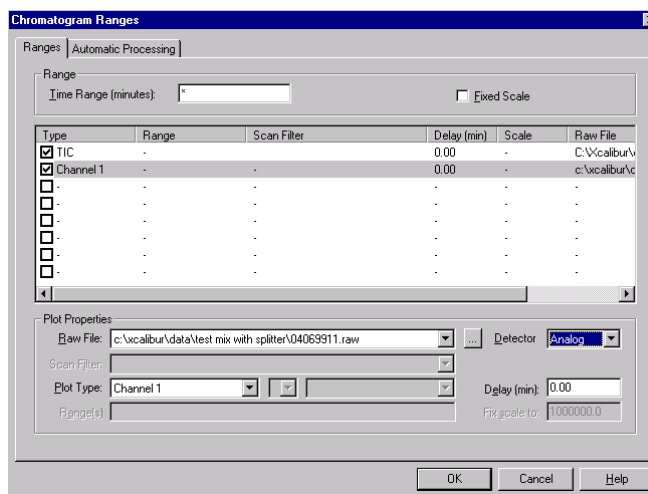
A dialog box will appear, select another TIC and then change the detector from Mass Spec to Analog.

Channel 1 will appear as selected type of file. Press OK.

The TIC and FID chromatogram will be displayed. Press the integration button on the tool bar and integrate the peaks in the TIC and FID chromatogram. Also set the display labels to area. Press the right mouse button with the mouse over the chromatogram for the FID and select Peak Detection settings to allow integration of the peaks. The default integration is set for 10% of the highest peak. With a FID the solvent is the highest peak, so the Peak Identification has to be set to 0.1%
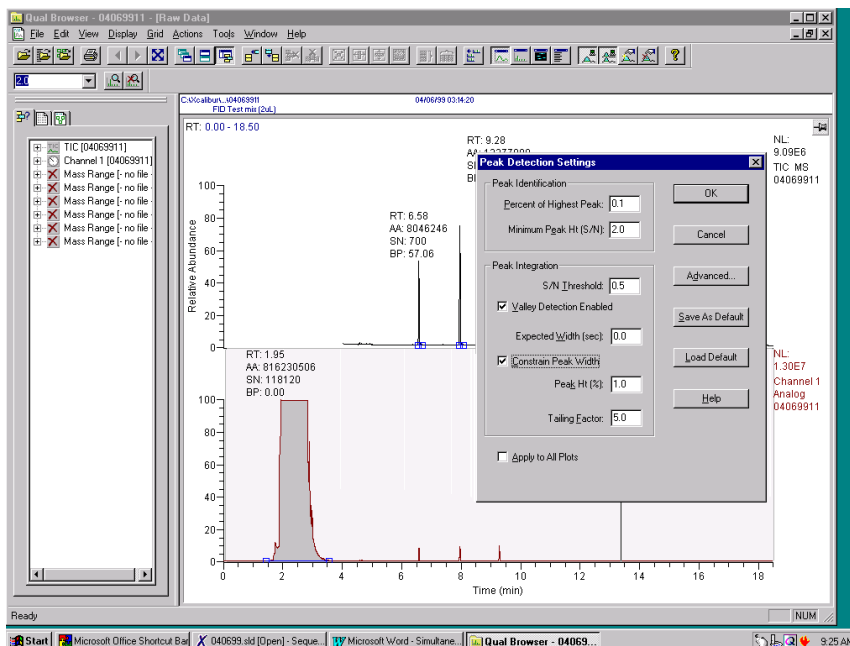
The spectrum may be viewed as usual by adding another cell and setting it to view spectrum. Then select a peak in the TIC and view the spectrum.
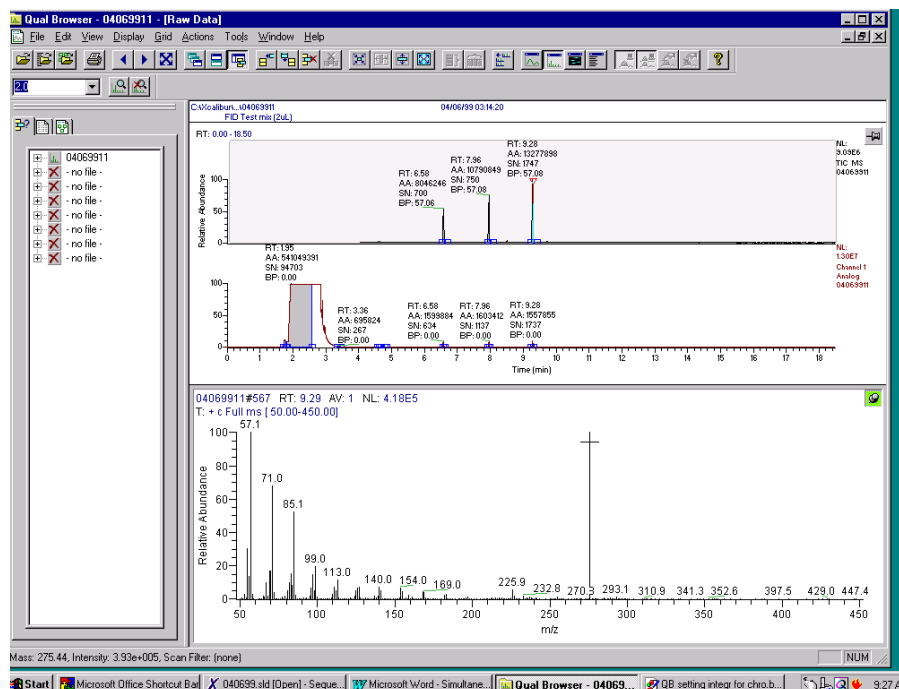
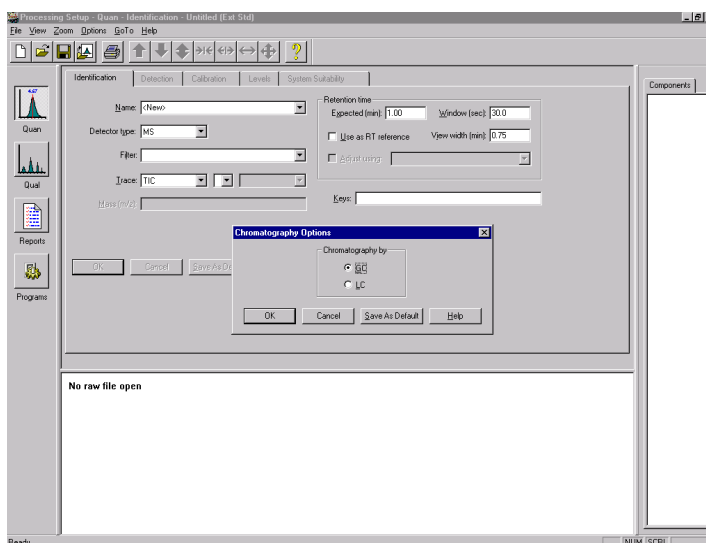## *Developing a Processing Method:*

Separate processing methods are created for the Mass Spec and the GCdetector. The following order must be followed to open a GC detectorchromatogram:

1. Open Processing Set Up and select new method file. Under Options verify that the chromatography is by GC.

2. Set the Detector Type to Analog.
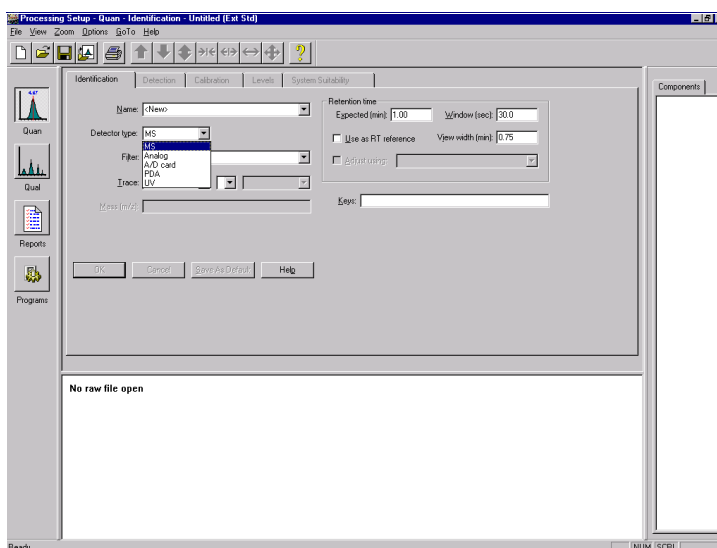
3. Open a raw data file and enter a name for the first analyte.

Press OK. Be sure that the Detector Type is still Analog. The FID chromatogram will appear.
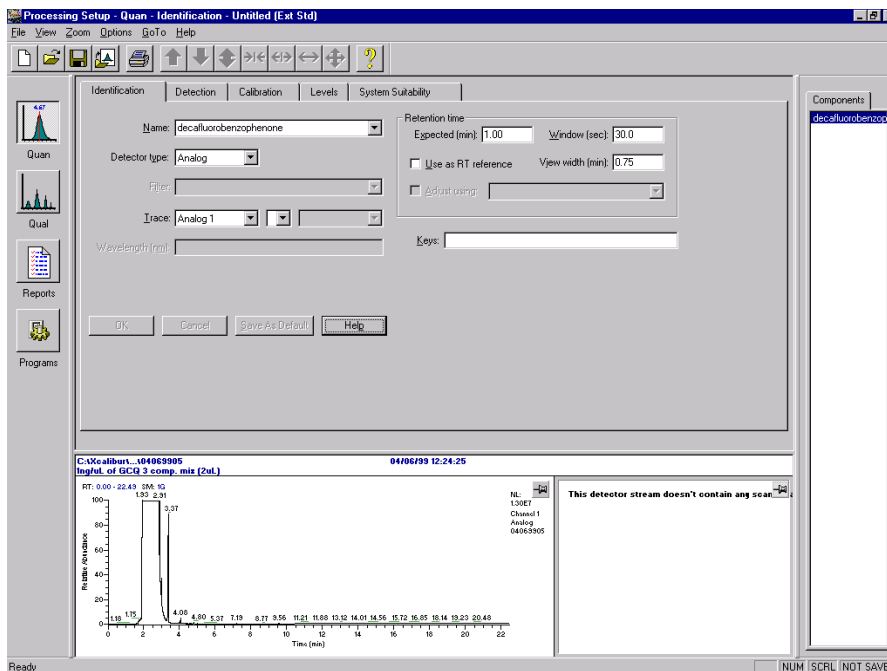
4. Zoom in on the analyte peaks by pinning the cell with the chromatogramand dragging the mouse across the area to be enlarged.
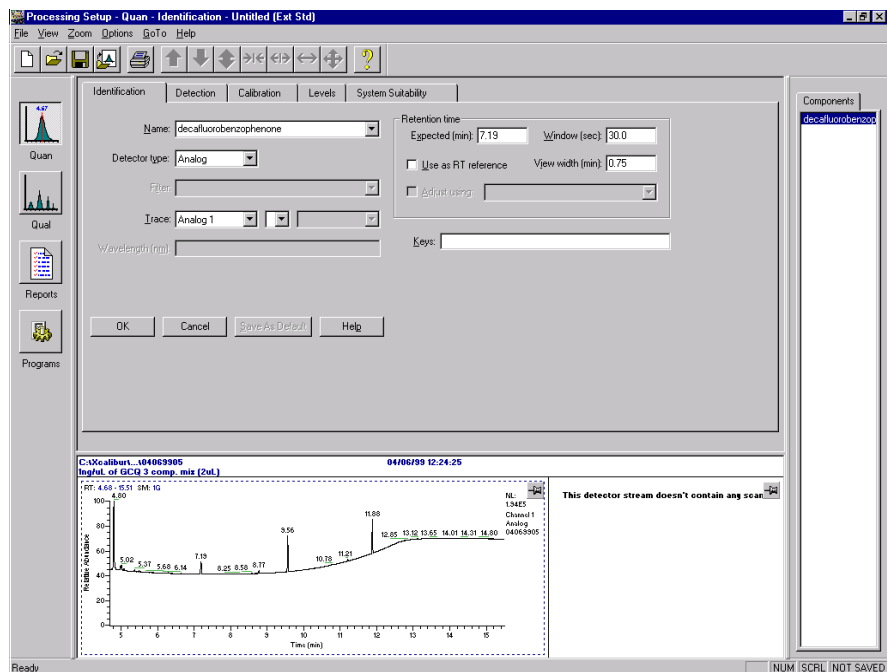
5. Enter the retention time for the first analyte and press OK. The peak of

interest will be integrated. Continue building your method as instructed in the on line Help.
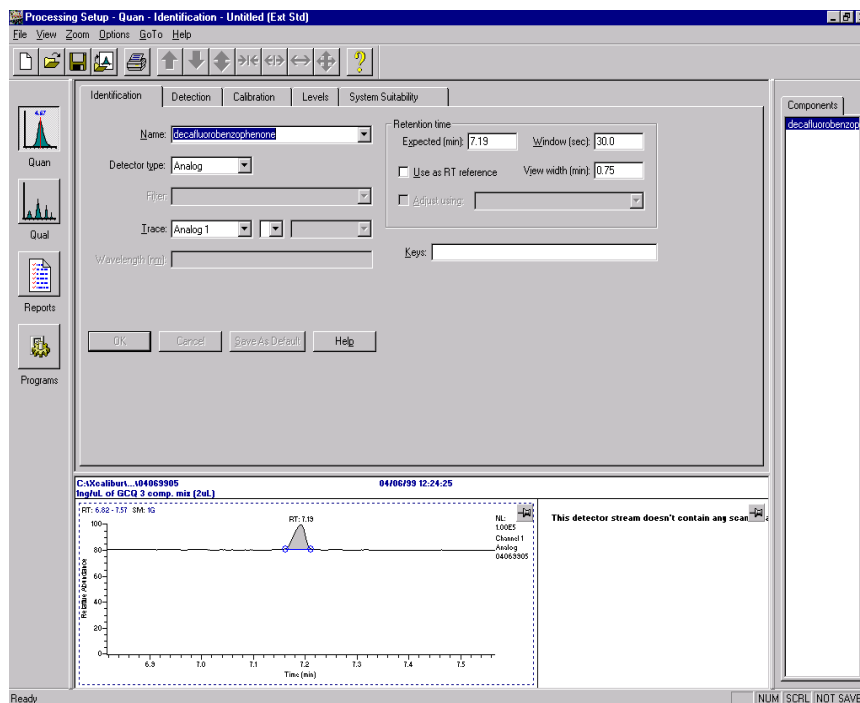
6. Select the remaining analytes by retention time and press OK with their appropriate name.
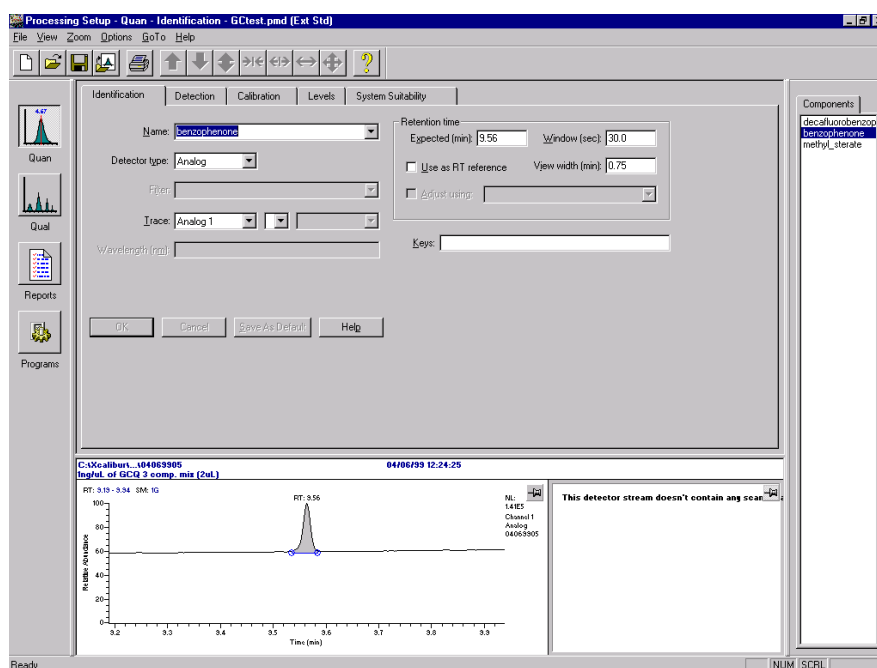
Report Writing:

After establishing a report template in Merlin, the raw data files may be reprocessed in a batch and reports printed out. A calibration curve may be viewed in Quan Browser.

analyte   Any chemical component to be analyzed. Internal standards, external standards, target components, quality control samples, knowns, and unknowns can all be considered analytes

carrier gas   The mobile phase in gas chromatography. The carrier gas carries the analyte mixture through the GC column, where it is separated into its individual components. The carrier gas flows through the GC column at a specific rate, measured either as a linear velocity (cm/sec) or as a flow rate (mL/min). Common carrier gases are helium, hydrogen, and nitrogen. Hydrogen offers the best chromatographic properties (optimum resolution at the highest flow rates). However, because hydrogen is flammable, helium is often used as a safer alternative for a carrier gas.

See also: *chromatography*, *constant flow*, *linear velocity*, *flow rate*, and *resolution*

chromatography   A process in which a chemical mixture carried by mobile phase (gas or liquid) is separated into its individual components as a result of differential distribution of the analytes as they flow around or over a stationary phase (liquid or solid).

See also: *liquid chromatography* and *gas chromatography*.

column flow   The flow of mobile phase of carrier gas through the chromatographic column. Column flow is usually reported as a linear velocity (cm/sec) or as a flow rate (mL/min).

See also: *linear velocity* and *flow rate*.

constant flow   The type of flow control available on gas chromatographs to regulate the flow of carrier gas through the GC column.
Flow rates are specified in units of mL/min.
In the Constant Flow mode, the flow rate of the carrier gas is maintained at a designated setting for the duration of the GC run. The head pressure of the carrier gas in the injector is electronically adjusted during the oven temperature program to compensate for changes in the gas viscosity and thereby maintain the desired flow rate.

data system   The computer hardware and software used to control the instrument and to record, analyze, and interpret the data collected by the instrument.

electronic pressure control	A system on the Finnigan GC, CE 8000 TOP GC, and the HP 5890 that controls the head pressure on a GC column at the injector. The pressure can be maintained or modulated during an analysis to affect the separation efficiency and speed of an analysis
final value	For the first step, this box sets the initial temperature for the program. For all other steps, this final value is the value the ramp is intended to reach. Temperatures can range from -80°C to the maximum oven temperature value.
flow rate	The rate of flow of the carrier gas through the GC column. The unit of measurement for flow rate is mL/min. Gas chromatography is defined by the separation of analytes by partitioning between a mobile phase (the carrier gas) and a stationary phase. This mobile phase flow rate must be regulated to provide reproducible results. See also: *flow control*
flow control	Method for controlling the flow of carrier gas through the GC column. Several methods are available depending on the type of GC
gas chromatograph	An instrument that separates a sample mixture into its chemical components by *gas chromatography*.A GC typically consists of an inlet for introduction of the sample, a column for separation of the analytes , and a detection system. The characteristic *retention time* of each component of the mixture determines when each component enters the detector. On the GCQ, the GC can be operated from the either the data system or the GC front panel. A serial I/O connection (COM1 port) between the GC and the data system allows the GC parameters to be set up and controlled directly by the data system through the Analysis window and the Instrument Setup window.
gas chromatography	One of the most common instrumental analysis techniques in use. When properly used, it provides both qualitative (what is it?) and quantitative (how much?) information about individual components in a sample. Gas chromatography involves separating the different components in a sample mixture from each other. The separation is produced by injecting the sample into a *carrier gas* stream (usually through an injector) that passes through a GC column that is packed with a solid packing or coated with a liquid stationary phase. The GC column is housed within a temperature-programmable oven. Varying the oven temperature during a GC run affects the *resolution* of components as well as their *retention time*. Under reproducible temperature and carrier gas flow conditions, the retention time of a component is descriptive of it. The components in a mixture can then be identified by comparing their retention times with those of known standard components. With a mass spectrometric detector, the mass spectra of the components are also descriptive of the components, offering another method of identification. In gas chromatography, the components are primarily separated by differences in volatility and structure. Some components and samples are not

suitable for gas chromatographic analysis due to their physical and chemical properties.

GC/MS Analysizing organic compounds by gas chromatography with a mass spectometer. The mass spectrometer detects analyte concentration found and the analytes identity by comparing the analytes to a library of known spectra.

head pressure The pressure of carrier gas in the injector of the GC. The head pressure creates a specific flow of the carrier gas through the GC column.

See also: *flow rate* , and *linear velocity*.

hold time The time in minutes to maintain the temperature specified in the Final Value control box. Referred to as *Holdup time* in the *Flow Calculator* . This refers to an unretained component's (at column termperature T) elution time. This is a measure of time that the sample components spend in the mobile phase. Hold up time is measured to determie the average linear velocity.

initial This is the initial temperature of the GC during injection and at the beginning of the run.

injection mode Type of injection used to introduce a sample mixture onto the GC column. Types of injection modes include *Split* , *Splitless*, *Large Volume Injection*, and *on-column injection*

injector The inlet system for a *gas chromatograph*. The injector is a gas-tight connection at the entrance of the GC column that delivers carrier gas to the column and provides a path for introduction of the sample into the GC.

See also: *Large Volume Injection*, *on column injector*, *split/splitless injector*, and *temperature programmable injector*.

ion polarity The charge on an ion (positive or negative).

ion source The part of a mass spectrometer where ions are formed (molecular ions and fragment ions, or in an MS/MS experiment, precursor ions).

Large Volume Injection Injection mode possible using a Finnigan TPI and the CE On-Column Injector (OCI). In this mode, up to 500 microliters of sample can be injected into the GC. With the Finnigan TPI, during the injection, the temperature of the injector is held slightly above the boiling point of the solvent, but below the boiling points of the analytes, with the split vent open. After most of the solvent evaporates, the split vent is closed, and the temperature of the injector is ramped quickly to a high enough temperature to cause the analytes to vaporize and be swept onto the analytical column. LVI is accomplished with the CE On-Column Injector by incorporation of a desolvation pre-column connected through a tee union to the analytical column and the solvent vent valve. During the injection, the oven temperature is held below the boiling

point of the solvent during desolvation while the vent valve is open. A high carrier gas flow rate sweeps the more volatile solvent out the vent valve while the analytes remain coated on the pre-column. The vent valve is then closed and the oven temperature programmed up to vaporize the analytes for transfer into the analytical column.

See also: *injection mode*, *temperature programmable injector* and *on column injector*.

linear velocity   The rate of movement of the carrier gas through the GC column measured in cm/sec.

See also: *flow rate* and *flow control*.

liquid chromatography   A form of elution chromatography in which a sample partitions between a stationary phase of large surface area and a liquid mobile phase that percolates over the stationary phase. As components elute from the chromatographic column they flow into a detector and generate a signal. The characteristic time each component elutes from the liquid chromatograph and enters the detector is called the component's retention time

negative ion   An atom, radical, molecule, or molecular moiety that has gained one or more electrons, acquiring an electrically negative charge. The charge state becomes -1, -2, -3, etc., depending upon the number or electrons acquired.

See also: *positive ion* and *ion polarity*.

on-column injection   Sample injection directly inside the capillary column rather than in a liner located in a flash vaporization off column mode.

See also: *negative ion* and *ion polarity*.

on column injector   The On-Column Injector (OCI) allows to inject a sample directly into a 0.25 or 0.32 mm capillary column or 0.53 mm wide-bore column. Primary and secondary cooling system keep the injection block at ambient temperature and the injection zone cool to prevent sample vaporizazion and ensure complete sample transfer from the syringe to the column.

A special versions of this injector allows:

- to introduce large volumes of liquid sample (LVOCI).

- to operate at high oven temperature by using an option HOT device (HOTOC).

See also: *Large Volume Injection*.

positive ion   An atom, radical, molecule, or molecular moiety that has lost one or more electrons or has been protonated, thus acquiring a positive charge. The charge state becomes +1, +2, +3, etc., depending upon the number of

electrons lost or protons gained.

See also: *negative ion* and *ion polarity*.

pressure program   Type of *flow control* used to regulate the flow of carrier gas through the GC column. *head pressure*at the injector is specified in units of psi, and the ramps are in units of psi/min. This produces a certain flow rate of the carrier gas through the GC column. The head pressure is controlled by an EPC . The pressure is programmed as a method development or optimization tool.

purge vent   On a Split/Splitless (SSL) Injector, the septum purge gas flows out this vent into the atmosphere. The purge vent may have a purge valve on it, which allows the purge flow to be regulated.

ramp   Use the ramps to increase the oven temperature at a specific rate in °C/minutes to a final value in °C. Using multiple ramps allows for finer control of the component separation. A graph is generated during GC temperature method development to display the temperature versus time plot that is being created or edited.

rate   This is the rate of change of the temperature when increasing the temperature from one value to the next. This rate is described in degrees Celsius per minute (°C/min). The ramp begins at the previous step's final temperature value and proceeds to the current step's final temperature at the specified rate.

resolution   A measure of the ability of a chromatographic column to separate components during a chromatographic experiment. Chromatographic resolution between two adjacent peaks is defined as the distance between the centers of the peaks divided by their average peak width (measured at 10% of the peak height). A resolution of 1.00 indicates baseline separation.

retention time   The total time that a component is in the chromatographic column. This is measured from the time of injection until elution. If the maximum signal from an analyte is detected 5 minutes and 14 seconds after injection, then the analyte has a retention time (on the column) of 5:14

secondary cooling   Cools the On-Column Injector and the first few centimeters of the GC column. This is an external gas supply (typically bottled air or CO_2) that is supplied to the secondary coloing valve located on the rear of the GC. Use this option to cool the column head and the first few centimeters of column where sample is introduced. Flow rate is dependcant upon the pressure supplied from the gas source. Secondary cooloing gas consumption is usually very large so you should minimze the time between the GC Ready mode and Run Start mode.

split injection   One type of injection mode available on a *split/splitless injector*. As the injection is made (and at all times during the GC run), the split vent is opened

to allow a specific portion of the sample to be swept into the atmosphere (that is, split away), while the rest of the sample is swept onto the GC column. Only a certain percentage of the sample makes it onto the GC column depending on the ratio of the split flow to the column flow (the split ratio). Split injection is used for the analysis of high concentration samples that would overload the GC column if all of the sample were placed onto the column. Some types of samples can not be diluted with a solvent because of the presence of volatile analytes that would co-elute with most commonly used solvents, making split injection necessary.

splitless injection
One type of injection mode available on a Split/Splitless (SSL) Injector. In this mode, the split vent is closed during the injection, and a large amount of sample is transferred to the GC column from the injection port. This type of injection is used for trace residue analysis.

See also: *split/splitless injector* and *split vent*.

split/splitless injector
A type of injector available on a gas chromatograph. This is the standard injector offered on the GCQ. The SSL Injector is made up of a septum nut, a septum, a liner, a metal body, an influent line for the carrier gas, a septum purge vent, and a split vent that allows a portion of the carrier gas to be vented into the atmosphere.

See also: *split vent*, *purge vent*, *carrier gas*, *flow rate*, *split injection* , and *splitless injection*.

split vent
The exit port of the Split/Splitless (SSL) Injector. The split vent is opened and closed by the split valve and/or split solenoid. When the split vent is open, a portion of the carrier gas and sample mixture (if present) is allowed to pass through the split vent into the atmosphere.

subambient temperature
Below room temperature or ambient temperature. The oven of the gas *gas chromatograph*, T*temperature programmable injector* , and *on column injector* can be cooled to subambient temperatures if the appropriate valving and coolant supply is configured accordingly.

surge pressure
An option available on the with the *split/splitless injector*. If the Use Surge Pressure check box is selected in the GC Instrument Setup window Pressure tab, you can enter the pressure to surge to (in psi), the time (minutes into the GC run) for the surge to come on and the time for the surge to go off. During the GC run, the *head pressure* will be set to the value specified in the Pressure box and the On Time, and it will hold at that pressure until the Off Time. The selected head pressure will override the EPC value(s) while it is on. *surge pressure* is often coordinated to be on during the injection of a sample into the injection port. By surging the pressure during the injection, then the sample is pushed more quickly onto the GC column in a tighter band. This helps improve loading of the components. The surge pressure it also useful with analytes that are thermally degradable and tend to break down when exposed to the high temperatures of the injector. Using surge

pressure helps to sweep the sample out of the injection port and onto the GC column quickly.

See also: *electronic pressure control.*

temperature program   A temperature program where the oven temperature is changed at a specified rate.

temperature programmable injector   The TPI is an injector available as an option for the Finnigan GC. This injector provides the capability for temperature programming of the injector. The injector is a universal injector for capillary gas chromatography. The injector may be operated in the cool on-column mode, large volume mode, isothermal split or splitless mode, or temperature programmed mode.

See also: *on-column injection*, *Large Volume Injection*, *split injection*, and *splitless injection*.

total time   This is the GC run time. It is calculated by summing the ramp times and hold times in the temperature program. The total elapsed time for a run cannot exceed 650 minutes. At 650 minutes the run terminates and the oven temperature returns to the initial oven temperature. In an isothermal operation (no ramp steps, or RATE=0), the instrument internally sets the run time to a maximum of 650 minutes.

UltraFast Module   The Ultra Fast Module (UFM) is a GC device allows the direct heating of the analytical capillary column properly installed inside the GC oven. The UFM device integrates a GC capillary column with the components for its temperature control.Compared to conventional air circulating oven, this device features faster temperature programming.