

# ***Console Acceptance Tests & Specifications***

*MERCURYplus NMR Spectrometer Systems*

*Pub. No. 01-999186-00, Rev. B0902*



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*MERCURYplus* NMR spectrometer systems

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# SAFETY PRECAUTIONS

The following warning and caution notices illustrate the style used in Varian manuals for safety precaution notices and explain when each type is used:

**WARNING:** *Warnings are used when failure to observe instructions or precautions could result in injury or death to humans or animals, or significant property damage.*

**CAUTION:** *Cautions are used when failure to observe instructions could result in serious damage to equipment or loss of data.*

## Warning Notices

*Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these warnings, or with specific warnings elsewhere in Varian manuals, violates safety standards of design, manufacturing, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.*

**WARNING:** **Persons with implanted or attached medical devices such as pacemakers and prosthetic parts must remain outside the 5-gauss perimeter from the centerline of the magnet.**

The superconducting magnet system generates strong magnetic fields that can affect operation of some cardiac pacemakers or harm implanted or attached devices such as prosthetic parts and metal blood vessel clips and clamps.

Pacemaker wearers should consult the user manual provided by the pacemaker manufacturer or contact the pacemaker manufacturer to determine the effect on a specific pacemaker. Pacemaker wearers should also always notify their physician and discuss the health risks of being in proximity to magnetic fields. Wearers of metal prosthetics and implants should contact their physician to determine if a danger exists.

Refer to the manuals supplied with the magnet for the size of a typical 5-gauss stray field. This gauss level should be checked after the magnet is installed.

**WARNING:** **Keep metal objects outside the 10-gauss perimeter from the centerline of the magnet.**

The strong magnetic field surrounding the magnet attracts objects containing steel, iron, or other ferromagnetic materials, which includes most ordinary tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Unless restrained, such objects can suddenly fly towards the magnet, causing possible personal injury and extensive damage to the probe, dewar, and superconducting solenoid. The greater the mass of the object, the more the magnet attracts the object.

Only nonferromagnetic materials—plastics, aluminum, wood, nonmagnetic stainless steel, etc.—should be used in the area around the magnet. If an object is stuck to the magnet surface and cannot easily be removed by hand, contact Varian service for assistance.



## Warning Notices (*continued*)

Refer to the manuals supplied with the magnet for the size of a typical 10-gauss stray field. This gauss level should be checked after the magnet is installed.

**WARNING:** Only qualified maintenance personnel shall remove equipment covers or make internal adjustments.

Dangerous high voltages that can kill or injure exist inside the instrument. Before working inside a cabinet, turn off the main system power switch located on the back of the console, then disconnect the ac power cord.

**WARNING:** Do not substitute parts or modify the instrument.

Any unauthorized modification could injure personnel or damage equipment and potentially terminate the warranty agreements and/or service contract. Written authorization approved by a Varian, Inc. product manager is required to implement any changes to the hardware of a Varian NMR spectrometer. Maintain safety features by referring system service to a Varian service office.

**WARNING:** Do not operate in the presence of flammable gases or fumes.

Operation with flammable gases or fumes present creates the risk of injury or death from toxic fumes, explosion, or fire.

**WARNING:** Leave area immediately in the event of a magnet quench.

If the magnet dewar should quench (sudden appearance of gasses from the top of the dewar), leave the area immediately. Sudden release of helium or nitrogen gases can rapidly displace oxygen in an enclosed space creating a possibility of asphyxiation. Do not return until the oxygen level returns to normal.

**WARNING:** Avoid liquid helium or nitrogen contact with any part of the body.

In contact with the body, liquid helium and nitrogen can cause an injury similar to a burn. Never place your head over the helium and nitrogen exit tubes on top of the magnet. If liquid helium or nitrogen contacts the body, seek immediate medical attention, especially if the skin is blistered or the eyes are affected.

**WARNING:** Do not look down the upper barrel.

Unless the probe is removed from the magnet, never look down the upper barrel. You could be injured by the sample tube as it ejects pneumatically from the probe.

**WARNING:** Do not exceed the boiling or freezing point of a sample during variable temperature experiments.

A sample tube subjected to a change in temperature can build up excessive pressure, which can break the sample tube glass and cause injury by flying glass and toxic materials. To avoid this hazard, establish the freezing and boiling point of a sample before doing a variable temperature experiment.

## Warning Notices (*continued*)

**WARNING:** Support the magnet and prevent it from tipping over.

The magnet dewar has a high center of gravity and could tip over in an earthquake or after being struck by a large object, injuring personnel and causing sudden, dangerous release of nitrogen and helium gasses from the dewar. Therefore, the magnet must be supported by at least one of two methods: with ropes suspended from the ceiling or with the antivibration legs bolted to the floor. Refer to the *Installation Planning Manual* for details.

**WARNING:** Do not remove the relief valves on the vent tubes.

The relief valves prevent air from entering the nitrogen and helium vent tubes. Air that enters the magnet contains moisture that can freeze, causing blockage of the vent tubes and possibly extensive damage to the magnet. It could also cause a sudden dangerous release of nitrogen and helium gases from the dewar. Except when transferring nitrogen or helium, be certain that the relief valves are secured on the vent tubes.

**WARNING:** On magnets with removable quench tubes, keep the tubes in place except during helium servicing.

On Varian 200- and 300-MHz 54-mm magnets only, the dewar includes removable helium vent tubes. If the magnet dewar should quench (sudden appearance of gases from the top of the dewar) and the vent tubes are not in place, the helium gas would be partially vented sideways, possibly injuring the skin and eyes of personnel beside the magnet. During helium servicing, when the tubes must be removed, carefully follow the instructions and safety precautions given in the manual supplied with the magnet.

## Caution Notices

*Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these cautions, or with specific cautions elsewhere in Varian manuals, violates safety standards of design, manufacturing, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.*

**CAUTION:** Keep magnetic media, ATM and credit cards, and watches outside the 5-gauss perimeter from the centerline of the magnet.

The strong magnetic field surrounding a superconducting magnet can erase magnetic media such as floppy disks and tapes. The field can also damage the strip of magnetic media found on credit cards, automatic teller machine (ATM) cards, and similar plastic cards. Many wrist and pocket watches are also susceptible to damage from intense magnetism.

Refer to the manuals supplied with the magnet for the size of a typical 5-gauss stray field. This gauss level should be checked after the magnet is installed.

## Caution Notices (*continued*)

**CAUTION:** Keep the PCs, (including the LC STAR workstation) beyond the 5-gauss perimeter of the magnet.

Avoid equipment damage or data loss by keeping PCs (including the LC workstation PC) well away from the magnet. Generally, keep the PC beyond the 5-gauss perimeter of the magnet. Refer to the *Installation Planning Guide* for magnet field plots.

**CAUTION:** Check helium and nitrogen gas flowmeters daily.

Record the readings to establish the operating level. The readings will vary somewhat because of changes in barometric pressure from weather fronts. If the readings for either gas should change abruptly, contact qualified maintenance personnel. Failure to correct the cause of abnormal readings could result in extensive equipment damage.

**CAUTION:** Never operate solids high-power amplifiers with liquids probes.

On systems with solids high-power amplifiers, never operate the amplifiers with a liquids probe. The high power available from these amplifiers will destroy liquids probes. Use the appropriate high-power probe with the high-power amplifier.

**CAUTION:** Take electrostatic discharge (ESD) precautions to avoid damage to sensitive electronic components.

Wear a grounded antistatic wristband or equivalent before touching any parts inside the doors and covers of the spectrometer system. Also, take ESD precautions when working near the exposed cable connectors on the back of the console.

## Radio-Frequency Emission Regulations

The covers on the instrument form a barrier to radio-frequency (rf) energy. Removing any of the covers or modifying the instrument may lead to increased susceptibility to rf interference within the instrument and may increase the rf energy transmitted by the instrument in violation of regulations covering rf emissions. It is the operator's responsibility to maintain the instrument in a condition that does not violate rf emission requirements.

## Chapter 1. Introduction

Sections in this chapter:

- 1.1 “Overview of the Acceptance Testing Process” this page
- 1.2 “General Acceptance Testing Requirements” page 14
- 1.3 “Samples Required for Acceptance Tests” page 14
- 1.4 “General Testing and Specification Requirements” page 14
- 1.5 “Varian Sales Offices” page 17
- 1.6 “Posting Requirements for Magnetic Field Warning Signs” page 18

Following each installation of a Varian, Inc. *MERCURYplus* NMR spectrometer system, an installation engineer tests and demonstrates the instrument’s operation using the procedures in this manual.

This manual contains the acceptance test procedures and specifications for *MERCURYplus* NMR spectrometers. The following is an overview of the chapters in this manual:

- Chapter 2, “Console and Magnet Test Procedures,” provides the acceptance test procedures.
- Chapter 3, “Consoles and Magnets Specifications,” provides the acceptance test specifications.
- Chapter 4, “Customer Training,” provides basic spectrometer maintenance and operation training.
- Chapter 5, “Acceptance Test Results,” contains forms for recording test results.

The acceptance test procedures and specifications for each probe are contained in a separate probe family manual, for example procedures and specifications for AutoSwitchable probes are contained in the *AutoSwitchable NMR Probes* manual.

Only the lineshape and signal to noise tests are preformed manually. All other probe calibrations are performed by the instrument during the auto calibration procedures. The manual tests are provided as a reference.

### 1.1 Overview of the Acceptance Testing Process

The objectives of the acceptance tests procedures are threefold:

- To identify the tests to be performed during system installation.
- To identify the precise methods by which these tests are performed.
- To leave the instrument in a calibrated, ready to use, state.

## Acceptance Tests

Acceptance test procedures are arranged by the type of specification. These procedures cover the basic specifications of the instrument—signal-to-noise, resolution, and lineshape—and are not intended to reflect the full range of operating capabilities or features of a *MERCURYplus* NMR spectrometer. The order in which the tests are performed is determined by the installation engineer, although the order does not matter except that some procedures use results from other procedures

Performance of additional tests beyond those described in this manual must be agreed upon in writing as part of the customer contract.

## Acceptance Specifications

All specifications are subject to change without notice. The specifications published in this manual shall prevail unless negotiation or customer contract determines otherwise. Refer to the text in each chapter for other conditions.

Request for additional specifications beyond those listed in this manual must be agreed upon in writing as part of the customer contract. The following policies are in effect at installation:

- **Specifications Policy for Probes Used in Systems other than *MERCURYplus*** – No guarantee is given that probes purchased for use in systems other than *MERCURYplus* will meet current specifications.
- **Testing Policy for Indirect Detection Probes used for Direct Observe Broadband Performance** – Probes designed for indirect detection applications are tested for indirect detection performance only. Indirect detection acceptance tests are performed only if an indirect detection probe was purchased for use with the *MERCURYplus*.
- **Sample Tubes Policy** – Tests are performed in 5-mm sample tubes with 0.38 mm wall thickness (Wilmad 528-PP, or equivalent) and 10-mm sample tubes with 0.46 mm wall thickness (Wilmad 513-7PP, or equivalent). Using sample tubes with thinner walls (Wilmad 5-mm 545-PPT, or equivalent; Wilmad 10-mm 513-7PPT, or equivalent) increases signal-to-noise.

## Computer Audit

A computer audit form is included in “**Computer Audit,**” page 61. The information from this form will help Varian assist you better in distributing future software upgrades and avoiding hardware compatibility problems. You are asked for information about all computers directly connected to the spectrometer or else used to process NMR data.

## Installation Checklist

An installation checklist form is included in “**System Installation Checklist,**” page 65.

## System Documentation Review

Following the completion of the acceptance tests and computer audit, the installation engineer will review the following system documentation with the customer:

- Software Object Code License Agreement.
- Varian and OEM manuals.
- Warranty coverage and where to telephone for information.

## Basic System Demonstration

The installation engineer will also demonstrate the basic operation of the system to the laboratory staff. The objective of the demonstration is to familiarize the customer with system features and safety requirements, as well as to assure that all mechanical and electrical functions are operating properly.

The system demonstration includes the following items:

### *Magnet Demonstration*

The following are demonstrated:

- Posting requirements for magnetic field warning signs.
- Cryogenics handling procedures and safety precautions.
- Magnet refilling.
- Flowmeters.
- Homogeneity disturbances.

### *Console and Probe Demonstration*

The following are demonstrated:

- Loading programs (VNMR, Optional VNMR packages, Solaris).
- Experiment setup, including installing the probe in the magnet.
- Basic instrument operation to obtain typical spectra, including probe tuning, magnet homogeneity shimming, and printer/plotter operation. (Note that Varian installation engineers are not responsible for, or trained to, run any spectra not described in this manual.)
- Auto calibration, using the *GLIDE* or Tc1-dg interface, of key probe parameters such as  $^1\text{H}$  pw90,  $^{13}\text{C}$  pw90, decoupler field, gradient strength (if gradients are present), and other probe specific parameters.
- Demonstration of automated data acquisition via *GLIDE* interface. Using the 2-Ethyl-1-indanone sample provided with the console the following experiments will be run:
  - 1D Experiments:  $^1\text{H}$ ,  $^{13}\text{C}\{^1\text{H}\}$ , APT and DEPT.
  - 2D Non gradient experiments: NOESY and TOCSY (for non-gradient system or probe COSY is also run).
  - 2D Gradient experiments: gCOSY, gHSQC, gHMBC will be demonstrated if gradients are present.
- Walk through the demonstration spectra and the “Data Acquisition – Calibration and Indanone Spectra,” page 44.
- Demonstration of gradient shimming using PFG gradients, if present, or homospoil.
- Review how to use the manuals (online and hard copy) and where to find information.
- Review the post installation 30 day check list.

Detailed specifications and circuit descriptions are not covered.

Formal training in the operation and maintenance of the spectrometer is conducted by Varian at periodically scheduled training seminars held in most Varian Application Laboratories. Installation engineers are responsible for guiding you through the acquisition of the spectra as provided in the manual. The installation engineer is not responsible for interpretation of the results beyond what is provided in this manual. On-site training is

available in some geographic locations. Contact your sales representative for further information on availability and pricing for these courses.

To make the system demonstration most beneficial, the customer should review Varian and OEM manuals before viewing the demonstration.

## 1.2 General Acceptance Testing Requirements

Each *MERCURYplus* spectrometer is designed to provide high-resolution performance when operated in an environment as specified in the *Installation Planning Guide*. Unless both the specific requirements of this manual and the general requirements specified in the *MERCURYplus Installation Planning Guide* are met, Varian cannot warrant that the NMR spectrometer system will meet the published specifications.

## 1.3 Samples Required for Acceptance Tests

The *MERCURYplus* spectrometer is supplied with the samples listed in [Table 1](#)

**Table 1.** Samples Required for Console Acceptance Tests

<i>Test Sample</i>	<i>Sample Tube (mm)</i>	<i>Nucleus</i>	<i>Sample Part Number</i>
<sup>13</sup> C enriched 1% methyl iodide, 1% Trimethyl phosphite, and 0.2% Cr(AcAc) in Chloroform-d	5	<sup>13</sup> C	00-968120-96
Doped 2-Hz H <sub>2</sub> O/D <sub>2</sub> O (0.1 mg/ml GdCl <sub>3</sub> in 1% H <sub>2</sub> O in D <sub>2</sub> O)	5	<sup>1</sup> H	01-901855-01
2% 2-ethyl-1-indanone in Chloroform-d	5	<sup>1</sup> H and <sup>13</sup> C	01-901855-03
0.1% ethylbenzene, 0.01% TMS, 99.89% deuteriochloroform (CDCl <sub>3</sub> )	5		00-968120-70
0.1% ethylbenzene, 0.01% TMS, 99.89% deuteriochloroform (CDCl <sub>3</sub> )	10		00-968123-70
chloroform in acetone-d <sub>6</sub> lineshape	5		00-968120-xx
100% methanol (reagent grade) -50 to +25 (Low)	5		00-968120-80
100% ethylene glycol (reagent grade) +25 to +100 (High)	5		00-968120-79

The samples required for acceptance testing of any individual probe are supplied with the probe. Typical samples required for probe acceptance tests are: chloroform in acetone-d<sub>6</sub>, ethyl benzene in chloroform-d, and ASTM (40% p-dioxane in 60% benzene-d<sub>6</sub>). Other samples might be required depending upon the type of probe. The specific sample requirements and test procedures are specified in the manual for each probe.

## 1.4 General Testing and Specification Requirements

- The *MERCURYplus* performance specifications in effect at the time of your order are used to evaluate the system.
- The appropriate quarter-wavelength cable must be used for each nucleus except autoswitchable probes operated in 4-nucleus mode. The <sup>13</sup>C quarter-wavelength cable is used in this case.

- Homogeneity settings must be optimized for each sample (manual shimming may be required in any or all cases). The shim parameters for resolution tests on each probe should be recorded in a log book and in a separate file name (in the directory `/vnmr/shims`) for each probe. For example, for a 5-mm switchable probe, the shim parameters can be saved with the command `svs('sw5res')`. These values can then be used as a starting point when adjusting the homogeneity on unknown samples, by the command `rts('sw5res')`.
- The probe must be tuned to the appropriate frequency.
- Spinning speed must be set to the following:

Sample (mm)	Nuclei	Speed (Hz)
5	all	20–26
10	all	15

Spinning 10-mm tubes faster than 15 Hz can cause vortexing in samples, severely degrading the resolution.

- Some test parameters are stored in the disk library `/vnmr/tests` and can be recalled by entering `rtp('/vnmr/tests/xxx')`, where `xxx` is the name of the file that contains the parameters to be retrieved—e.g., `rtp('/vnmr/tests/H1sn')`. To see the parameter sets available for the standard tests, enter `ls('/vnmr/tests')`. Other sets come from `/vnmr/stdpar`.
- Many of the probe parameters and performance specifications will be determined automatically and the corresponding manual tests will NOT be run by the installer. Certain tests, such as signal to noise and lineshape will be run manually. Tests corresponding to the automatic performance tests are provided should you want to run them at a later time.
- For all sensitivity tests, the value of `pw` must be changed to the value of the 90° pulse found in the pulse width test on the same probe and nucleus.
- During calibration, *GLIDE* creates an appropriate `pw` array to determine the 90° pulse width. For manually run observe pulse width tests, an appropriate array of `pw` values must be entered to determine the 360° pulse. The 360° pulse is the first non-zero pulse that gives minimum intensity of the spectrum. The 360° pulse is usually determined by interpolation between a value that gives a positive signal, and a value that gives a negative signal. The 90° pulse width is one quarter the 360° pulse (360/4).
- Signal-to-noise (S/N) is measured by the computer as follows:

$$S/N = \frac{\text{maximum amplitude of peak}}{2 \times \text{root mean square of noise region}}$$

- Lineshape should be measured digitally with the aid of the system software. The properly scaled spectra should also be plotted and the plot retained.
- Digital determination of lineshape:
  1. Display and expand the desired peak.
  2. Enter **nm**, then **dc** for drift correction to ensure a flat baseline. Set **vs=10000**. Click the menu button labeled **Th** to display the horizontal threshold cursor. Set **th=55** (the 0.55% level).
  3. Click the menu button labeled **Cursor** or **Box** until two vertical cursors are displayed, and align them on the intersections of the horizontal cursor and the peak. Enter **delta?** to see the difference in Hz between the cursors.



4. Set **th=11** (the 0.11% level) and repeat.
  5. You can also use the macro **res**. Place the cursor on the resonance of interest and type **res** on the command line.
- Determination of lineshape from a plot:
    1. Use a large enough plot width to allow accurate determination of the baseline. The baseline should be drawn through the center of the noise, in a region of the spectrum with no peaks.
    2. The 0.55% and 0.11% levels are then measured from the baseline and calculated from the height of the peak and the value of **vs**. For example, if a peak is 9.0 cm high with **vs=200**, then the 0.55% level on a 100-fold vertical expansion (**vs=20000**) is  $9 \times 0.55$ , or 4.95 cm from the baseline.

If the noise is significant at the 0.55% and 0.11% levels, the linewidth should be measured horizontally to the center of the noise.
  - Use the **dsnmax** macro to determine signal-to-noise (sensitivity). You can also choose a noise region free of any anomalous features with the cursors. Neither cursor should be placed any closer to an edge of the spectrum than 10 percent of the value of **sw**. This should produce the best possible signal-to-noise that is representative of the spectrum.
  - The results of all tests should be plotted to create a permanent record. Include a descriptive label and a list of parameters. These plots can then be saved as part of the acceptance tests documentation.

## 1.5 Varian Sales Offices

For product sales and service information, contact one of the Varian sales offices:

- Argentina, Buenos Aires, (114) 783-5306
- Australia, Mulgrave, Victoria, (3) 9566-1138
- Austria, Vösendorf, (1) 699 96 69
- Belgium, Brussels, (02) 721 51 51
- Brazil, Sao Paulo, (11) 829-5444
- Canada, Ottawa, Ontario, (613) 260-0331
- China, Beijing, (10) 6846-3640
- Denmark, Herlev, (42) 84 6166
- France, Orsay, (1) 69 86 38 38
- Germany, Darmstadt, (6151) 70 30
- Italy, Milan, (2) 921351
- Japan, Tokyo, (3) 5232 1211
- Korea, Seoul, (2) 3452-2452
- Mexico, Mexico City, (5) 523-9465
- Netherlands, Houten, (0118) 61 71 56
- Norway, Oslo, (9) 86 74 70
- Russian Federation, Moscow, (95) 241-7014
- Spain, Madrid, (91) 472-7612
- Sweden, Solna, (8) 445 1601
- Switzerland, Zug, (41) 749 88 44
- Taiwan, Taipei, (2) 2698-9555
- United Kingdom, Walton-on-Thames, England (1932) 898 000
- United States, Palo Alto, California,  
 Varian, Inc., NMR Systems  
 Customer Sales Support, (650) 424-5434  
 Service Support, Palo Alto, California, **1 (800) 356-4437**  
 E- mail: [custserv@varianinc.com](mailto:custserv@varianinc.com)  
 North American Service Manager  
 9017 Mendenhall Ct., Ste D, Columbia, MD 21045  
 (410) 381-7229
- Venezuela, Valencia (41) 257608

## 1.6 Posting Requirements for Magnetic Field Warning Signs

The strong magnetic fields that surround a superconducting magnet are capable of causing death or serious injury to individuals with implanted or attached medical devices such as pacemakers or prosthetic parts. Such fields can also suddenly pull nearby magnetic tools, equipment, and dewars into the magnet body with considerable force, which could cause personal injury or serious damage. Moreover, strong magnetic fields can erase magnetic media such as tapes and floppy disks, disable the information stored on the magnetic strip of automated teller machine (ATM) and credit cards, and damage some watches.

To warn of the presence and hazard of strong magnetic fields, the customer is responsible for posting clearly visible signs warning of magnetic field hazards. This responsibility includes measuring stray fields with a gaussmeter.

Radio-frequency emissions may also pose a danger to some individuals. The rf emission levels from Varian NMR equipment have been measured and compared to the IEEE/ANSI C95.1-1991 standard. For further information, refer to the *RF Environment* section of the *Installation Planning Guide*.

### Warning Signs

Varian provides signs to help customers meet this posting responsibility. These signs *must* be posted according to the following requirements *before the magnet is energized*:

1. *10-gauss warning signs (Figure 1)* – Post along the 10-gauss perimeter of the magnet so that a sign can be easily seen by any person about to enter the 10-gauss field from any direction. Refer to the manuals supplied with the magnet for the size of a typical 10-gauss stray field. Check this gauss level after the magnet is installed.

Note that the stray field may extend vertically to adjacent floors, and additional signs may be needed there. A sign is not required if the 10-gauss field extends less than 30 cm (12 in.) beyond a permanent wall or less than 61 cm (24 in.) beyond the floor above the magnet.

2. *5-gauss warning signs (Figure 2)* – Post along the 5-gauss perimeter of the magnet so that a sign can be easily seen by any person about to enter the 5-gauss field from any direction. Refer to the manuals supplied with the magnet for the size of a typical 5-gauss stray field. Check this gauss level after the magnet is installed. Note that the stray field may extend vertically to adjacent floors, and additional signs may be needed there.

3. *Magnet area danger signs (Figure 3)* – Post at each entrance to the magnet area. Be sure each sign is outside the 5-gauss perimeter.



Figure 1. 10-Gauss Warning Sign

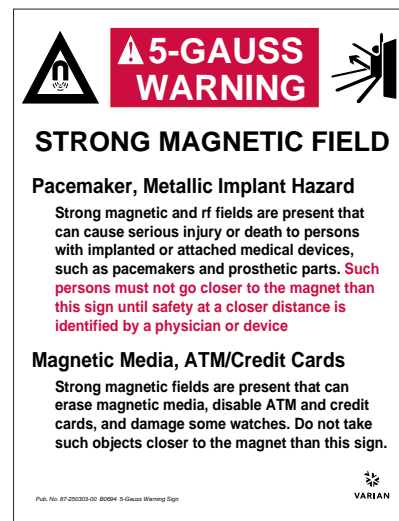


Figure 2. 5-Gauss Warning Sign

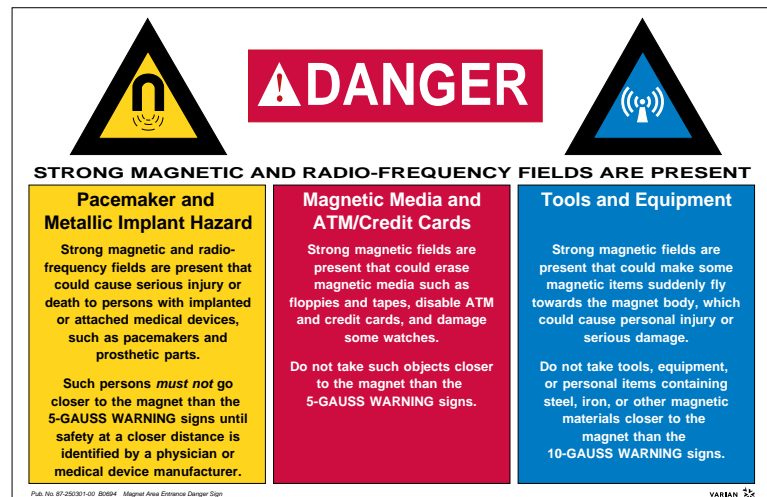


Figure 3. Magnet Area Danger Sign

Stray magnetic fields can reach beyond the published distances when two or more magnetic fields intersect or when the field extends over large ferromagnetic masses or structures (steel doors, steel construction beams, etc.). In this case, the customer *must* measure the stray field using a gaussmeter to determine how the 5- and 10-gauss fields are altered (contact a scientific instrumentation supplier for information on acquiring a gaussmeter).

You can request additional signs from Varian by telephoning 1-800-356-4437 in the United States or by contacting your local Varian office in other countries.



## Chapter 2. Console and Magnet Test Procedures

Sections in this chapter:

- 2.1 “AutoCalibration and GLIDE Operation Demonstration” page 21
- 2.2 “Automated Data Acquisition” page 26
- 2.3 “Homonuclear Decoupling” page 31
- 2.4 “Magnet Drift Test” page 32
- 2.5 “Variable Temperature Operation (Optional Hardware)” page 33
- 2.6 “Temperature Accuracy for VT Systems (Optional Test)” page 34
- 2.7 “Stability Calibration for High-Stability VT (Optional Test)” page 36

This chapter contains the procedures required to demonstrate the specifications for *MERCURYplus* consoles and magnets. Chapter 5, “Acceptance Test Results,” contains forms for writing the results.

Lineshape and resolution tests described in the probe manual shipped with your probe must be run before these console tests are run. During the console tests probe calibration files are created that are used during some of the console tests. The probe calibrations written to these probe files will meet or exceed the specifications for the probe. Probe performance tests and calibrations that are executed during AutoCalibration will not be repeated manually. These probe calibration files are required for some of the console tests.

### 2.1 AutoCalibration and *GLIDE* Operation Demonstration

The AutoCalibration procedures calibrates the probe and demonstrates the performance of the probe. During the AutoCalibration, a probe calibration file containing the  $^1\text{H}$  and  $^{13}\text{C}$  90° pulse widths, decoupler calibration, gradient calibration (if present) is set up as described in the probe installation manual that shipped with your probe.

Table 2 lists the samples used for the AutoCalibration.

**Table 2.** Samples for System Calibration

<i>Sample</i>	<i>Nucleus</i>	<i>Sample Tube (mm)</i>	<i>Part Number</i>
$^{13}\text{C}$ enriched 1% methyl iodide, 1% trimethyl phosphite, and 0.2% Cr(AcAc) in Chloroform-d	$^{13}\text{C}$	5	00-968120-96
Doped 2-Hz $\text{H}_2\text{O}/\text{D}_2\text{O}$ (0.1 mg/ml $\text{GdCl}_3$ in 1% $\text{H}_2\text{O}$ in $\text{D}_2\text{O}$ )	$^1\text{H}$	5	01-901855-01

The total time for the tests and calibrations should be about 1 hour. Run the tests and calibrations in the following general order:

- Run the lineshape tests described in the probe manual before running AutoCalibration.
- Run the AutoCalibration as described in this section.
- Run the signal-to-noise test described in the probe manual. Use the pw90 determined by the AutoCalibration routine.

## Setting Up Probe Calibration Files

Before you calibrate a probe for the first time, you must set up the probe calibration file with the `addprobe` command as described below.

1. Log in as **vnmr1**.
2. Enter one of the following command:  
`addprobe(probe_name)`  
The probe calibrations are written to the probe file in `~/vnmr1/vnmrsys/probe/probe_name` and are available only to the user `vnmr1`.  
– OR –  
`addprobe(probe_name, 'system')`  
The calibrations are written to `/vnmr/probe/probe_name` and are available to all users.

Some probes, like the Autoswitchable and 4 nucleus probes, require additional calibrations not covered in this manual. For information on using *GLIDE* to complete the calibration of these probes, see the *Walkup NMR* manual and installation, testing, and specifications manual the Probe.

## Calibrating Z0 and Make LOCK gmap

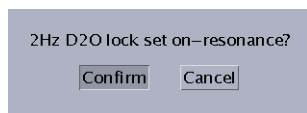
Before preceding any further, the lock and gradients must be calibrated for the autoshim and autolock procedures to function efficiently. This procedure calibrates Z0 and makes a gradient map for gradient shimming.

1. Insert the doped 2-Hz D<sub>2</sub>O sample (see [Table 2](#)).
2. Open the `acqi` window by pressing the `acqi` button on the VNMR menu. Lock onto the D<sub>2</sub>O resonance. The lock must be set on-resonance. Adjust Z0 as necessary.
3. Adjust Lock Gain and Lock Power and set the lock level at 80%.
4. Exit `acqi`.
5. Open *GLIDE* by clicking on the **GLIDE** button in the VNMR menu and click the **Setup** icon in *GLIDE*.

## Running AutoCalibration

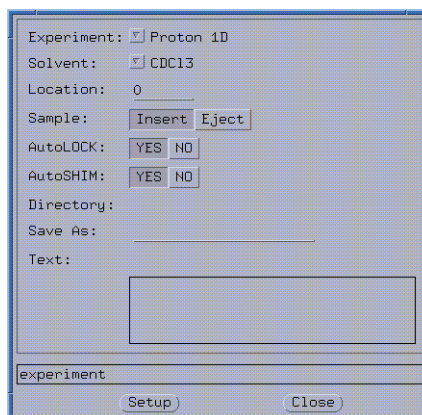
1. Click on the **GLIDE Setup** button in the **GLIDE** interface. The Experiment and Calibration Setup window (Figure 4A) is displayed.
2. Right mouse click the button next to **Experiment** to display the experiment and calibration menu.
3. Right mouse click on “**Generate 1k gmap & calibrate z0 “(D2O)”**” selection from the menu (Figure 4B).
4. Right mouse click the button next to **Solvent** to display the solvent menu (Figure 4C).
5. Right mouse click on **D2O**.
6. Set **Autoshim** and **Autolock** to **NO**
7. Click on **Setup**.

Standard proton parameters are recalled and the sample confirmation window appears.

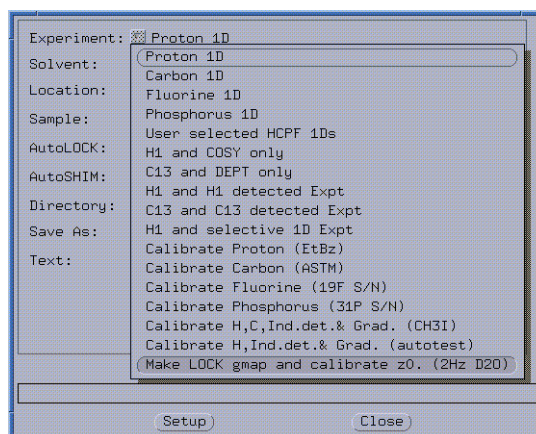


8. The message “**Set z0 exactly on-resonance before starting acquisition**” displays in the VNMR window. Open the lock display and set the lock as directed
9. Click on the **GO** button in GLIDE to run the calibration.

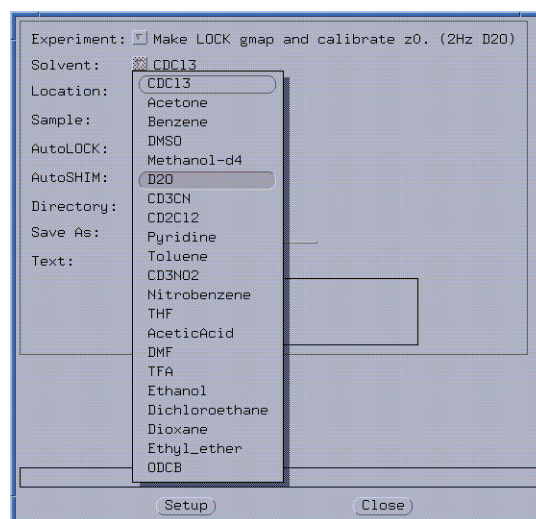
When the calibration is completed, the probe calibration file is updated.



(A) Experiment Setup Window



(B) Select Calibration from Experiment Menu



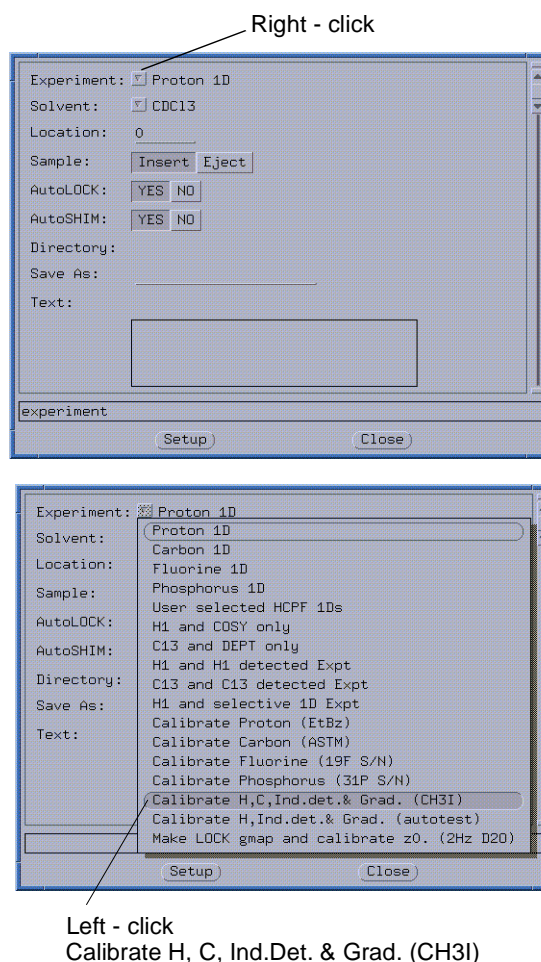
(C) Solvent Menu

**Figure 4.** GLIDE Calibrate LOCK



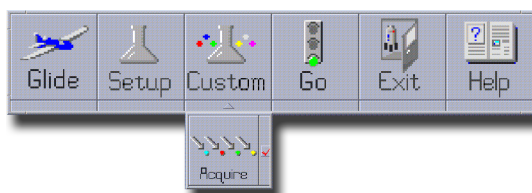
## Calibrating Probe and System Files

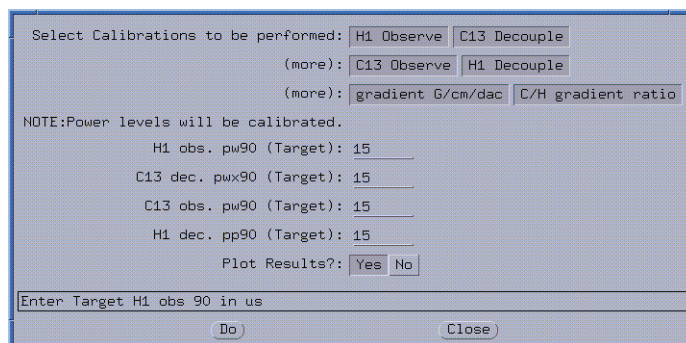
1. Open *GLIDE* by clicking on the **GLIDE** button in the **VNMR** menu, and click the **Setup** icon in *GLIDE*.
2. Use the **Sample: Eject / Insert** buttons (Figure 5) to eject the 2-Hz D<sub>2</sub>O sample and insert the chloroform sample (Table 2). Tune the probe if needed.
3. **Right - click** on the **Experiment select** button using the right mouse button.  
**Left - click** on **Calibrate H,C,Ind.Det.&Grad. (CH3I)** in the drop down experiment menu, (see Figure 5) (place the mouse pointer on the experiment selection **Calibrate H,C,Ind.Det.&Grad. (CH3I)** and click with the left mouse button). Make this selection for pfg and non pfg systems.
4. From the **Solvent** menu, select **CDCl<sub>3</sub>**.
5. Set **Autoshim** and **Autolock**.
  - Click the **No** button if your sample is already locked and shimmed.
  - Click the **Yes** button to lock and shim automatically.



**Figure 5.** *GLIDE* Setup Window and Drop Down Experiment Menu

6. Enter a relevant text in the **Text field** (e.g., calibration of ASWprobe) and click the **Setup** button. If the **text box** is not visible, place the mouse pointer on the bottom of the **Setup** Window boarder. The mouse pointer will change to an arrow pointing down to a short bar. Press and hold the left mouse button and drag the bottom boarder down until the text entry box is full visible.
7. Click the **Confirm** button.  
At the end of the setup operation, the Custom and Go buttons are no longer shaded and the **GLIDE Acquire** button appears.
8. Click the **Acquire** button to open the Acquisition Setup window, see Figure 6.
9. For the purposes of the ATP/ATS, select all the calibration routines appropriate for the current probe.





**Figure 6.** Acquisition Setup Window

No matter what order you select, the calibration order is always the following:

- H1 observe
- C13 decouple, gradient
- H1-C13 gradient ratio, C13 observe
- H1 decouple

If you do not select one of these calibrations, it is skipped. If you do not have gradients, the gradient related calibrations are skipped.

10. Enter the H1 and C13 **pulse specifications** of your probe's for H1 and C13 observe pw90 and decoupler pulses. If you do not enter a value, the calibration routine defaults to 15 usec for all pulses.
11. **Plot Results** - select **Yes**.
12. Click the **Do** button to start the calibration routine.

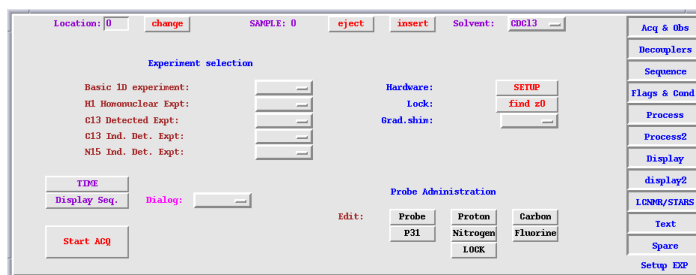
At the end of the calibration routine, the power and pulse width values are automatically incorporated into the probe file and the calibration spectra are plotted.

## Reviewing the Probe Calibration

Probe calibrations executed by vnmr1 are written to probe files one of two places.

- If the probe *was created as a system probe* and the probe name is unique (it does not exist in /export/home/vnmr1/vnmrsys/probe) it is written to /vnmr/probes/probe\_name.
- If the probe *was not created as a system probe* or the probe name is not unique (it does exist in /export/home/vnmr1/vnmrsys/probe) it is written to /export/home/vnmr1/vnmrsys/probes/probe\_name.

You can read or edit the file with a text editor (e.g., vi) or you can use the **setup EXP** pane.

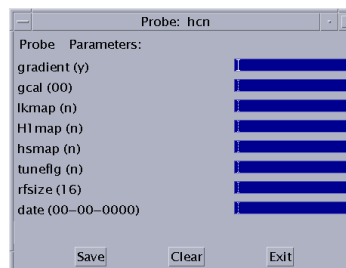


**Figure 7.** Probe Administration from Setup EXP

1. Log in as `vnmr1` and start VNMR.
2. Click on the **Setup EXP** tab.
3. In the **Probe Administration** part of the **Setup EXP** pane, select the probe calibration you want to edit (see [Figure 7](#)).
4. Click on a field to review data.

The current values are shown in parentheses next to the probe parameter. To change a parameter value, enter the new value in the blank

If you are a user other than `vnmr1`, the system probe calibrations are displayed in the **TEXT** pane. The calibrations cannot be changed.



## 2.2 Automated Data Acquisition

The automated data acquisition consists of several 1D and 2D experiments using the indanone sample listed in [Table 3](#).

**Table 3.** Sample for Automated Data Acquisition

<i>Sample</i>	<i>Sample Size (mm)</i>	<i>Sample Part Number</i>
2% 2-ethyl-1-indanone in chloroform- <i>d</i>	5	01-901855-03

The system (console and probe) configuration determines the experiment selection as follows:

Tests	Gradient Systems	Nongradient Systems
Four 1 D experiments:		
Acquisition of a proton spectrum	✓	✓
Acquisition of a proton decoupled carbon observe spectrum	✓	✓
DEPT (distortionless enhancement by polarization transfer)	✓	✓
APT (attached proton test)	✓	✓
Nongradient 2D experiments:		
TOCSY (total correlation spectroscopY)	✓	✓
NOESY (nuclear overhauser spectroscopY)	✓	✓
Gradient 2D experiments (requires PFG option and gradient probe)		
gCOSY (gradient correlation spectroscopY)	✓	
gHSQC (gradient heteronuclear single quantum correlation)	✓	
gHMBC (gradient heteronuclear multiple bond correlation)	✓	
Nongradient		
COSY (correlation spectroscopY)		✓

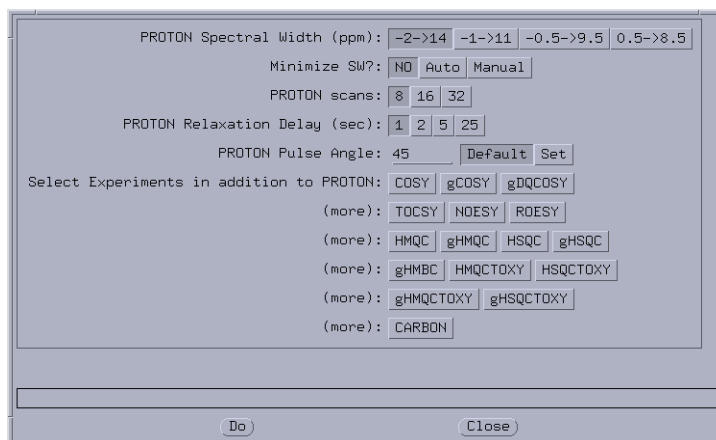
These experiments demonstrate the capabilities of the *MERCURYplus* spectrometer, the correct calibration of the instrument, and validate the correct functioning of the instrument.

You will be setting up two sets of experiments. You will be clicking on the Setup icon on the GLIDE interface once for H1 and H1 detected experiments and once of the C13 and C13 detected experiments.

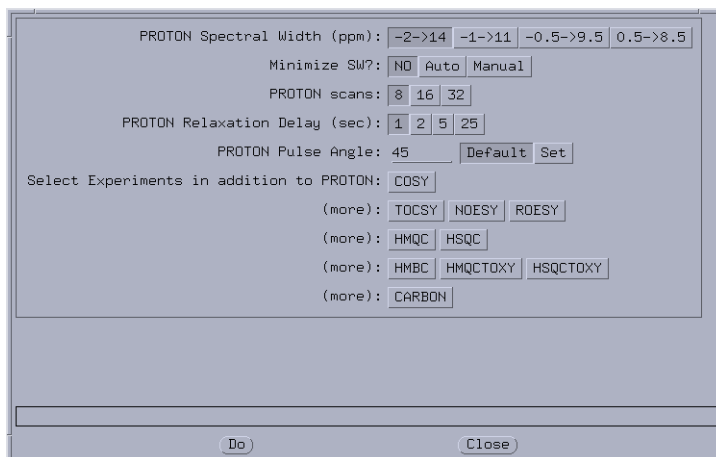
## H1 and H1 Detected Experiments

1. Open *GLIDE* by clicking on the *GLIDE* button in the VNMR menu. Click the **Setup** icon.
2. Insert the indanone sample (Table 3); use the Eject and Insert buttons (see Figure 5).
3. From the **Experiment** menu, select **H1 and H1 detected Expt.**  
Right-click the Experiment menu and left click H1 and H1 detected Expt.
4. From the **Solvent** menu, select **CDC13**.
5. Set **Autoshim** and **Autolock**.
  - Click the **No** button if your sample is already locked and shimmed.
  - Click the **YES** button to lock and shim automatically.
6. Enter the sample name **2-ethyl-1-indanone** in the **text box**.  
If the Text box is not visible, resize the Setup window until the text entry box is fully visible. The Setup window in Figure 5 has been expanded to show the text box.
7. Enter an appropriate directory name (e.g., 2-ethyl-1-indanone\_H1spectra) on the **Save As:** line.  
A directory is created using the name you entered on the **Save As** line with the current date and time to the name. As each experiment finishes, the experiment is saved in this directory. The individual FIDs have the experiment names, e.g. cosy.fid, tocsy.fid, etc.
8. **Click the Setup** button.  
The Setup window closes, the Custom icons become active, and the Acquire icon drops down.
9. **Click the Acquire** icon to select the experiments.  
After you click on the **Acquire** icon, one of two possible Experiment Selection windows appears. The window that appears is determined by the value of the gradient field in the probe definition file.
  - If you have a gradient probe (and the gradient field in the probe definition file is set to *Y*) the Experiment Selection window shown in Figure 8 is displayed.
  - If you have a non-gradient probe (or the gradient field in the probe definition file is set to *N*) the Experiment Selection window shown in Figure 9 is displayed.

If gradients are installed on your system and you do not see gradient experiments listed, the gradient field in the probe file is incorrect or an entry has not been made. The correct entry in the gradient field of the probe file is either *y* (yes there are gradients) or *n* (there are no gradients).
10. Use the proton 1D default acquisition parameters displayed in the Experiment Selection window.
11. Select the following experiments in the order listed. Experiments are executed, following the proton 1D experiment, in the order that they are selected.
  - Gradient-equipped systems with gradient probe select:



**Figure 8.** Experiment Selection Window for Gradient Experiments



**Figure 9.** Experiment Selection Window for Nongradient Experiments

**gCOSY** – click **OK** to accept the default parameters.

**gHMBC** – click **OK** to accept the default parameters.

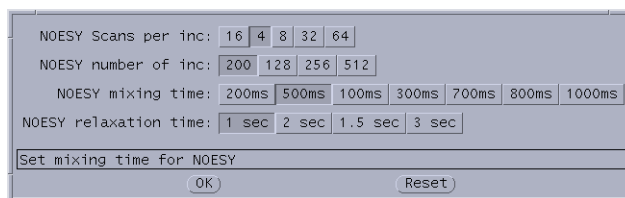
**gHSQC** – click **OK** to accept the default parameters.

- Non Gradient systems or non-Gradient Probe select:

**COSY**– click **OK** to accept the default parameters.

- Both Gradient and Non-Gradient systems or non-Gradient Probe select:

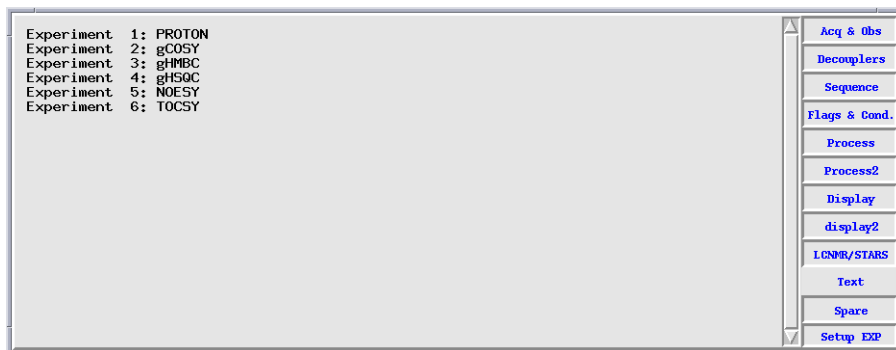
**NOESY** – Change the following acquisition parameters: **4 Scans per increment**, **mixing time = 1 sec**; then, click **OK** (see **Figure 10**).



**Figure 10.** NOESY Acquisition Parameter Window

**TOCSY** – click **OK** to accept the default parameters.

Each time the **OK** button is clicked an experiment is added to the list of experiments displayed in the VNMR TEXT pane in the order they are run, as shown in **Figure 11**.



**Figure 11.** 1H and 1H Detected Experiment List

12. Click **Do** to start data acquisition.

### C13 and C13 Detected Experiments

All systems and broadband-capable probes can run these experiments.

1. Open *GLIDE* and click the **Setup** icon.  
The indanone sample (**Table 3**) should already be inserted. If not, insert it now.
2. From the **Experiment** menu, select **C13 and C13 detected Expt.**
3. From the **Solvent** menu, select **CDC13**.
4. Set **Autoshim** and **Autolock**.
  - Click the **No** button if your sample is already locked and shimmed.
  - Click the **YES** button if you would prefer to lock and shim automatically.

5. In the Text box, enter the name of the sample, in this case **2-ethyl-1-indanone**.

If the Text box is not visible, resize the Setup window until the text entry box is fully visible. The Setup window in **Figure 5** has been expanded to show the text box.

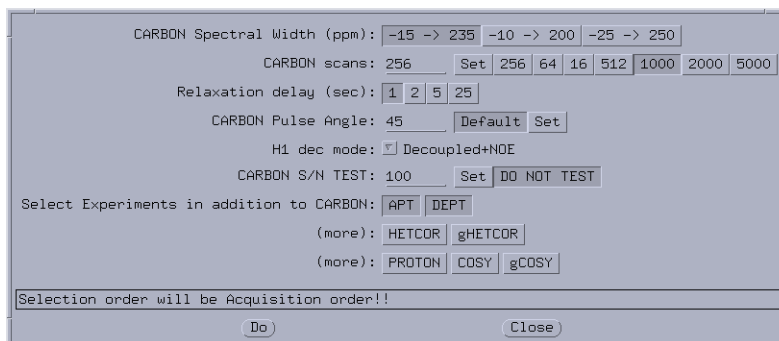
6. Enter an appropriate directory name (e.g., 2-ethyl-1-indanone\_C13spectra) on the **Save As:** line.

A directory is created using the name you entered on the Save As line with the current date and time to the name. As each experiment finishes, the experiment is saved in this directory. The individual FIDs have the experiment names, e.g. APT data named APT.fid and the DEPT data named DEPT.fid etc.

7. Click on the **Setup** button.

The Setup window closes, the CUSTOM icon becomes active, and the Acquire icon drops down.

8. Click on the **Acquire** icon and set up the proton decoupled carbon observe experiment by selecting **1000 scans**, the **DO NOT TEST** option for **CARBON S/N TEST**, and accept the defaults for the remaining options (see **Figure 12**).
9. Click on the following experiments in the order listed:

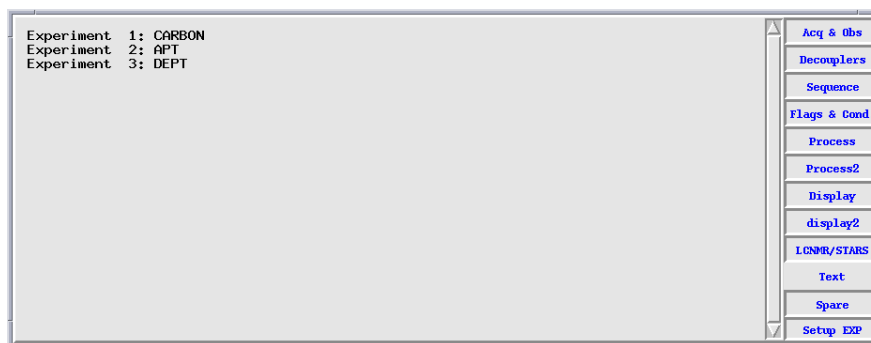


**Figure 12.** Experiment Window for <sup>13</sup>C and <sup>13</sup>C Detected Experiments

**APT** – click **OK** to accept the default parameters.

**DEPT** – click **OK** to accept the default parameters.

Each time the OK button is clicked, an experiment is added to the list of experiments, similar to **Figure 13** displays in the VNMR Text pane (see **Figure 13**) in the order they will be run.



**Figure 13.** <sup>13</sup>C and <sup>13</sup>C Detected Experiment List

10. Click the **Do** button to begin acquisition.

## 2.3 Homonuclear Decoupling

Use this procedure to calibrate homonuclear decoupling power values.

### Samples

**Table 4.** Samples for Homonuclear Decoupling Test

<i>Test Sample</i>	<i>Sample Tube (mm)</i>	<i>Sample Part Number</i>
0.1% ethylbenzene, 0.01% TMS, 99.89% deuteriochloroform (CDCl <sub>3</sub> )	5	00-968120-70
0.1% ethylbenzene, 0.01% TMS, 99.89% deuteriochloroform (CDCl <sub>3</sub> )	10	00-968123-70

### Hardware

A 5-mm probe capable of <sup>1</sup>H direct observe should be used for this test.

### Procedure

1. Enter `rtp ('/vnmr/tests/H1sn')`.
2. Tune the probe. Set `nt=1`. Run a normal spectrum without decoupling.
3. Set `dm='nny'`. Use the cursor and `sd` to set the decoupler on the central line of the triplet, and then run a decoupled spectrum.  
Possible `dpwr` values are 0 to 49 (49 is maximum power), in steps of 1.0 dB. The best values of `dpwr` must be found by experiment. Too much power might show increased noise; too little might not decouple the quartet. Setting `dpwr=25` is a good starting point.
4. Observe that the quartet collapses to a single peak with no remaining evidence of splitting.
5. Write the results on the form in [Chapter 5.5, "Console and Magnet Test Results,"](#).



## 2.4 Magnet Drift Test

The magnet drift test is an overnight test.

### Samples

Depending upon the probe, the concentration of H<sub>2</sub>O. The H<sub>2</sub>O concentrations are 1% for the -01 and autotest samples and 2% for the -02 sample. Use the sample that provides a signal with good a good signal to noise ratio, in most cases the 1% H<sub>2</sub>O / 99% D<sub>2</sub>O samples will a good signal.

**Table 5.** Sample for Magnet Drift Test

<i>Sample</i>	<i>Sample Tube (mm)</i>	<i>Sample Part Number</i>
Doped 2-Hz H <sub>2</sub> O/D <sub>2</sub> O (0.1 mg/ml GdCl <sub>3</sub> in 1% H <sub>2</sub> O in D <sub>2</sub> O)	5	01-901855-01
Doped 2-Hz H <sub>2</sub> O/D <sub>2</sub> O (0.1 mg/ml GdCl <sub>3</sub> in 2% H <sub>2</sub> O in D <sub>2</sub> O)	5	01-901855-02
autotest sample; 0.1% <sup>13</sup> C enriched methanol in 1% H <sub>2</sub> O/99% D <sub>2</sub> O	5	01-96812068-xx

### Probe and Hardware Requirements

A 5-mm probe capable of <sup>1</sup>H direct observe is recommended.

### Test Procedure

1. Enter **rtp ('/vnmr/tests/shmd2o')** to retrieve the test parameter set to the current experiment.
2. Tune the probe.
3. Acquire a normal spectrum and shim the HDO signal to 2 to 3 Hz linewidth at 50%.
4. Connect to the **acqi** window, turn the lock off, turn the spinner off, and set the spinner speed to 0. Make sure the lock signal is on-resonance (the lock signal display should be flat). Disconnect the **acqi** window. Then disconnect the lock cable from the probe.
5. Enter **in='n' spin='n' nt=1 array('d1',11,3600,0) d1[1]=60.**  
This sets up an array of d1 values, with the first spectrum to be collected after 1 minute and subsequent spectra to be collected at 60 minute intervals.
6. Enter **ga** to acquire the spectra. The test takes approximately 10 to 11 hours to finish.
7. Phase the first spectrum by entering **ds (1)** to display the first spectrum of the array and by entering **lp=0 aph0** to apply a first-order phase correction to the spectrum.
8. Enter **ai** to scale all of the spectra to the same vertical scale, and enter **dssa** to display the arrayed spectra stacked vertically.
9. Compare the frequency shift of the HDO peak of the arrayed spectra to the frequency of the first spectrum in the array.
10. Write the results on the form in **Chapter 5.5, "Console and Magnet Test Results,"**.

## 2.5 Variable Temperature Operation (Optional Hardware)

This demonstration shows that the basic variable temperature (VT) unit and probe changes to the desired temperature and displays on the VT controller. If the system is equipped with a VT unit, read through the VT operation instructions before this demonstration.

Dry nitrogen is required as the VT gas if the requested temperature is over 100<sup>o</sup> C or below 10<sup>o</sup> C. Otherwise, air can be used. Dry nitrogen gas is recommended for cooling the bearing, spinner, and decoupler. This prevents moisture condensation in the probe and spinner housing.

**CAUTION:** The use of air as the VT gas for temperatures above 100<sup>o</sup> C is not recommended. Such use destructively oxidizes the heater element and the thermocouple.

### *Demonstration Limitations*

If dry nitrogen gas and liquid nitrogen are unavailable at the time of installation, the range of VT demonstration is limited to temperatures between 30<sup>o</sup>C and 100<sup>o</sup>C.

### *Sample*

No sample is used.

### *Probe and Hardware Requirements*

Any VT probe is used.

### *Procedure*

1. In the `config` window, make sure VT Controller is set to Present. Alternatively, enter `vttype?` to check that `vttype` is set to `2`.
2. Set N2 gas flow to 9.5 to 10.0 LPM (for temperatures below –100<sup>o</sup> C, increase N2 flow to 12 LPM).
3. Enter a value for `temp`, then enter `su`. For values below room temperature, the heat exchanger must be in place. Maintain the temperature for 5 minutes.
4. Operate the VT unit within the specifications of the probe. Test the temperature at set points that correspond to the following:
  - Maximum, minimum, and midpoint of the allowed temperature: 95, 80, 60 if air is used; 120, 30, 20 if dry nitrogen is used; or 120, –100, 40 if a heat exchanger is used.
  - Ambient temperature.

The software limits the ramp rate to 12<sup>o</sup>C per minute up or down. Wait for the temperature to equilibrate.

## 2.6 Temperature Accuracy for VT Systems (Optional Test)

The optional tests in this section check temperature accuracy calibrations for high and low temperatures using ethylene glycol and or methanol, respectively.

Table 6 lists the samples for low-temperature and high-temperature tests.

**Table 6.** Samples for Optional VT Accuracy Test

<i>Sample</i>	<i>Temperature Range (°C)</i>	<i>Sample Tube (mm)</i>	<i>Sample Part Number</i>
100% methanol (reagent grade)	–50 to +25 (Low)	5	00-968120-80
100% ethylene glycol (reagent grade)	+25 to +100 (High)	5	00-968120-79

### Probe and Hardware Requirements

The variable temperature accessory and a VT probe are required. Run VT tests with a 5-mm probe capable of <sup>1</sup>H direct observe from –150°C to +200°C. For probes that have a more limited temperature range (particularly PFG probes), run the test at two or three temperatures that fall within the VT range of the probe. These tests can also be run using the <sup>1</sup>H decoupling coil of the 5-mm broadband probe as <sup>1</sup>H direct observe.

### High-Temperature Calibrations Test

1. Tune the probe. Use a 99.8% D<sub>2</sub>O sample (not supplied by Varian) for shimming.
2. Enter `rtp ('/vnmr/tests/shmd2o')` to retrieve the test parameter set to the current experiment. Acquire a normal spectrum and shim the water signal to about 3 to 4 Hz linewidth at 50%.
3. Replace the 99.8% D<sub>2</sub>O sample with the 100% ethylene glycol sample (Part No. 00-968120-79). Set the following parameters: `pw=2 gain=5` (or some value that doesn't overload the receiver) `sw=10000 at=2 nt=1 in='n'`.

In the **acqi** window, set the **lock to Off**. Disconnect the lock cable and set `alock='n'`. The test is run unlocked, because the sample has no deuterated solvent to lock on. Enter `su` and check the probe tuning for the ethylene glycol sample. Enter `ga` to acquire the spectrum. Place the cursor between the two peaks and enter `movetof` to move the transmitter offset.

4. Click the mouse on the Box menu button to call up right and left cursors. Position the right and left cursors on the right and left peaks. Enter `tempcal ('glycol')`.
5. Record the temperature reading from the VT controller (displayed face of the VT controller) and the computer-calculated temperature based on the chemical shift frequencies of the two peaks.

**CAUTION:** Extreme temperatures can damage the probe. The high and low temperatures must be within the specified range of the probe.

6. Enter `temp=50 su` to change the temperature to 50°C. Allow the sample to stabilize at 50°C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter `ga` to acquire a spectrum. Repeat steps 4 and 5.
7. Make sure that the VT gas flow and cooling air flow levels are between 9.5 to 10 LPM and gas flow to the probe is not restricted in any way. Enter `temp=100 su` to change the temperature to 100°C.

8. Allow the sample to stabilize at 100°C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter **ga** to acquire a spectrum. Repeat steps 4 and 5.

### Low-Temperature Calibrations Test

**CAUTION:** For low-temperature calibrations, fill the VT dewar with liquid nitrogen. If a chemical mixture is used instead of liquid nitrogen for low-temperature calibrations, choose the chemical slurry carefully. A mixture of crushed dry ice and acetone is not recommended, because it will dissolve the polystyrene VT dewar.

1. Tune the probe. Use a 99.8% D<sub>2</sub>O sample for shimming the probe.
2. Enter **rtp('/vnmr/tests/shmd2o')** to retrieve the test parameter set to the current experiment. Acquire a normal spectrum and shim the water signal to about 3 to 4 Hz linewidth at 50%.
3. Replace the 99.8% D<sub>2</sub>O sample with the 100% methanol sample (Part No. 00-968120-80). Set the following parameters: **pw=2 gain=5** (or some value that doesn't overload the receiver) **sw=10000 at=2 nt=1 in='n'**.
4. In the **acqi** window, set the **lock to Off**. Disconnect the lock cable and set **alock='n'**. The test is run unlocked (the sample lacks deuterated solvent).
5. Enter **su** and check the probe tuning for the methanol sample. Enter **ga** to acquire the spectrum. Place the cursor between the two peaks and enter **movetof** to move the transmitter offset.
6. Click on the Box menu button to call up right and left cursors. Position the right and left cursors on the right and left peaks, and enter **tempcal('methanol')**.
7. Record the temperature reading from the VT controller (displayed on the front face of the VT controller). Record also the computer-calculated temperature based on the chemical shift frequencies of the two peaks. If low-temperature calibrations are performed immediately following high-temperature calibrations, allow the probe to cool to room temperature before continuing with the rest of the procedure.
8. Enter **temp=-20 su** to change the temperature to -20° C. Allow the sample to stabilize at -20° C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter **ga** to acquire a spectrum. Repeat steps 4 and 5.
9. Enter **temp=-80 su** to change the temperature to -80° C. Allow the sample to stabilize at -80° C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter **ga** to acquire a spectrum. Repeat steps 4 and 5.
10. After finishing the low-temperature test, enter **temp='n' su** to turn off the temperature regulation. While keeping the dry nitrogen gas flowing to the probe and upper barrel, remove the polystyrene VT dewar containing liquid nitrogen. The flow of dry nitrogen gas to the probe will prevent condensation inside the probe. Allow the dry nitrogen gas to flow through the probe and upper barrel for at least 15 minutes while the probe warms up to room temperature.
11. Plot a graph of the VT controller reading (horizontal axis) as compared with the calculated VT reading from the chemical-shift differences between the two peaks (vertical axis). Draw a straight line through the points.

## 2.7 Stability Calibration for High-Stability VT (Optional Test)

This optional test is for high-stability VT units only (Part No. 00-992953-00). The test demonstrates that the VT unit can hold the temperature with  $\pm 0.1^{\circ}\text{C}$ . The test requires preconditioning of the laboratory air and restricts on the room temperature fluctuations.

**Table 7.** Samples for Optional High-Stability VT Test

<i>Test Sample</i>	<i>Nucleus</i>	<i>Sample Tube (mm)</i>	<i>Part Number</i>
Doped 2-Hz H <sub>2</sub> O/D <sub>2</sub> O (0.1 mg/ml GdCl <sub>3</sub> in 1% H <sub>2</sub> O in D <sub>2</sub> O)	<sup>1</sup> H	5	01-901855-01

Alternatively, the customer can request using a 10-mM DSS in D<sub>2</sub>O (sample volume of 0.6 ml in a 5-mm NMR tube) DSS= 3-(trimethylsilyl)-1-propane sulfonic acid. The customer must make this sample using DSS and deuterium oxide (99.8 or 99.9 atom% D). Upon request, Varian can make this sample if DSS is not available at the customer site.

### Probe and Hardware Requirements

High-stability variable temperature accessory and a 5-mm probe capable of <sup>1</sup>H direct observe are required.

### Test Procedure

1. Enter **rtp ('/vnmr/tests/shmd2o')** to retrieve the test parameter set to the current experiment.
2. Enter **temp=40 at=10 sw=10000**. Set pw to the <sup>1</sup>H 90° pulse width for the probe, and then enter **spin='n' su**.
3. Allow the VT controller to regulate to 40° C, which should be about 10° higher than the room temperature. Check that the probe is tuned.
4. Acquire a normal spectrum by entering **ga**. Move the cursor to the DSS signal (right-most peak). Enter **movetof sw=1000 at=10**.
5. Acquire a normal spectrum and shim the DSS signal to about 0.6 Hz or less linewidth at 50%. The sample of DSS in D<sub>2</sub>O should equilibrate at 40° C for at least 2 hours before the next step.
6. Enter **in='n' spin='n' nt=1 and array('d1',73,600,0) d1[1]=0**.
7. This sets up an array of d1 values with the first spectrum to be collected at time 0 minutes, and subsequent spectra to be collected at 10 minute intervals for up to 12 hours. Enter **ga** to acquire the spectra. The test takes about 12 hours to complete.
8. After the spectra are acquired, phase the first spectrum by entering **ds(1)** to display the first spectrum of the array, and by entering **lp=0 aph0** to apply a first-order phase correction to the spectrum.
9. Enter **ai** to scale all of the spectra to the same vertical scale, and enter **dssa** to display the arrayed spectra stacked vertically.
10. Measure the difference between the left-most peak and the right-most peak in Hz.

## Chapter 3. Consoles and Magnets Specifications

This section contains the following specifications:

- 3.1 “Homonuclear Decoupling” [this page](#)
- 3.2 “Variable Temperature Operation” [this page](#)
- 3.3 “Magnet Drift” [page 38](#)
- 3.4 “Temperature Accuracy for VT Accessories” [page 38](#)
- 3.5 “Stability Calibration for High-Stability VT Accessory” [page 38](#)

### 3.1 Homonuclear Decoupling

The quartet shows a single peak with no remaining evidence of splitting.

### 3.2 Variable Temperature Operation

For basic variable temperature (VT) accessories (Varian Part No. 00-992957-00), demonstrate that the VT unit and probe go to the desired temperature as registered on the window of the VT controller. If the system is equipped with a VT unit, the system user should read through the VT operation instructions before the demonstration.

Dry nitrogen is required as the VT gas if the requested temperature is over 100°C or below 10°C. Otherwise, air can be used. For temperatures below –40°C, dry nitrogen gas is recommended for cooling the bearing, spinner, and decoupler. This prevents moisture condensation in the probe and spinner housing.

**CAUTION:** The use of air as the VT gas for temperatures above 100°C is not recommended. Such use destructively oxidizes the heater element and the thermocouple.

**CAUTION:** Extreme temperatures can damage the probe. The high and low temperature must be within the specified range of the probe.

#### *Demonstration Limitations*

If dry nitrogen gas and liquid nitrogen are not available at the time of installation, the range of VT demonstration is limited to temperatures between 30°C and 100°C.

#### *Basic Specifications*

The specifications for variable temperature ranges are listed with each probe.

### 3.3 Magnet Drift

**Table 8** lists the drift specifications for magnets. Specifications for nominal field decay rate are less than or equal to the values listed in the table.

**Table 8.** Magnet Drift Specifications

<i>System (MHz/mm)</i>	<i>Field Strength (T)</i>	<i>Nominal Field Decay Rate (Hz/hr)</i>
200/54	4.70	2
300/54	7.05	3
400/54	9.40	8

### 3.4 Temperature Accuracy for VT Accessories

The temperature reading displayed on the VT unit display panel should be within  $\pm 1^{\circ}\text{C}$  of the actual temperature reading, as measured from the chemical shift.

### 3.5 Stability Calibration for High-Stability VT Accessory

The high-stability VT accessory holds the set temperature to within  $\pm 0.1^{\circ}\text{C}$ . ( $\pm 0.1^{\circ}\text{C} = 0.001$  ppm or in field dependent terms:  $\pm 0.2$  Hz at 200 MHz,  $\pm 0.3$  Hz at 300 MHz, and  $\pm 0.4$  Hz at 400 MHz)

## Chapter 4. Customer Training

Sections in this chapter:

- 4.1 “Where to Look for Answers” this page
- 4.2 “Host Computer Setup and Software Installation” page 41
- 4.3 “VNMR Directory Structure” page 41
- 4.4 “Managing Disk Space” page 43
- 4.5 “Tuning Probes” page 44
- 4.6 “Data Acquisition – Calibration and Indanone Spectra” page 44
- 4.7 “Magnet Maintenance” page 56
- 4.9 “Warranty and Who to Call for Assistance” page 58
- 4.8 “30-Day System Maintenance” page 57

This chapter describes training provided by the installer. This training is intended as a general overview of the instrument, basic maintenance requirements, software features, data acquisition and storage, file maintenance, and other routine tasks. Comprehensive training classes are offered at various Varian, Inc. Applications Laboratories around the world. Call your sales representative or contact the Varian, Inc. NMR systems office nearest you for class offerings, schedules and cost.

### 4.1 Where to Look for Answers

Knowing where to look is the first step to answering a question. All manuals are available in hard copy and can also be installed online. Figure 14 shows the menu for the *MERCURYplus* online manuals.

The online manuals are accessed from the Workspace menu.

- To access the Workspace menu in Solaris 2.5 and newer, place the mouse pointer on the background and click with the right mouse button.
- To access the online manuals in Solaris 2.6, click on Online manuals on the Workspace menu.
- To access the online manuals in Solaris 2.7 and later, select VNMR from the CDE menu, then select Online Manuals.

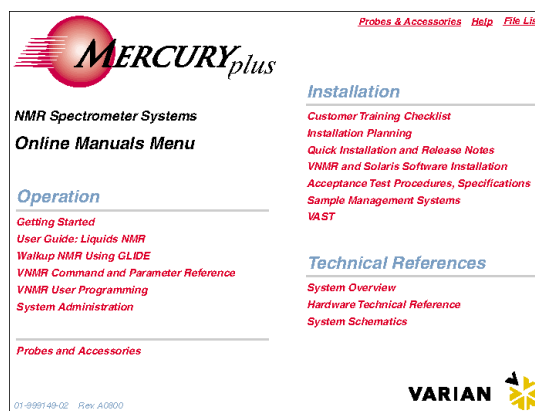


Figure 14. *MERCURYplus* Online Manual Menu



## Operation Manuals

After the installation has been finished you will find many of your routine questions answered in the following manuals.

- *VNMR Command and Parameter Reference* – Provides an alphabetical listing of the VNMR commands, parameters, and macros. Refer to this manual when you are working at the command line and you need to review a specific command.
- *Walkup NMR* – Provides information about the walkup NMR interfaces available in VNMR. Step-by-step instruction on setting up experiments using *GLIDE* and three Tcl/Tk interfaces. Detailed system calibration and administration instructions are provided in this manual.
- *Getting Started* – Provides an overview of VNMR and instrument operations. This manual covers the VNMR menu systems in detail, as well as command line operations, locking and shimming, data acquisition parameters, probe tuning, digital signal processing, and data processing, display, and plotting. Data management, storage, retrieval, and archival is also addressed in this manual.
- *User Guide: Liquids NMR* – Lists and explains how to set up all the standard experiments provided with VNMR. Not every experiment listed can be run on every system. The type and configuration of the instrument will determine which experiments can be run. This is the manual to go to when you have very specialized experimental requirements and will most likely be running the experiments from the command line. You will also want to refer to this manual for a general overview of the experiments that are automatically set up through the *GLIDE* and VNMR menu systems.
- *User Guide: Solids NMR* – Explains how to run CP/MAS experiments and describes the XPOLAR1 pulse sequences.
- *System Administration* – Provides detailed information on both the UNIX operating system and VNMR software for the system administrator is presented. Also included are magnet maintenance and other administrative items.

## Installations

- *MERCURYplus Installation Planning Guide* – Provides site planning information for *MERCURYplus* NMR spectrometer systems.
- *MERCURYplus Acceptance Test Procedures and Specifications* – Covers the console installation test procedures and specifications for the *MERCURYplus* spectrometer and magnet. Included in this manual is the information used by the installation engineer in the introductory training at the end of the installation. This manual does not cover the probe test procedures.
- *VNMR and Solaris Software Installation* – Details the installation of the Sun host computer hardware and the Solaris and VNMR software. Detailed instructions on system configuration are included here. Also included in this manual are instructions for setting up various plotters and printers, and other administration tasks.
- *MERCURYplus CP/MAS Accessory Installation* – Describes how to install and test the CP/MAS accessory.
- Probe installation, testing, and specifications manuals – Each type of probe has its own installation, test, and specifications manual. In these manuals are the detailed instructions for installing, tuning, testing the probe.
- Accessory manuals – Each accessory is covered in an independent manual, which contains installation, testing, and sometimes operation instructions.

## Technical References

- *System Schematics* – Provides schematic and technical drawings for *MERCURYplus* NMR spectrometer systems.
- *Technical Reference* – Provides technical details of the spectrometer systems and electronics.
- *System Description* – Provides an overview of the *MERCURYplus* spectrometer system and hardware.
- *User Programming* – Provides details about the VNMR macro programming language Magical II and pulse sequence statements. This manual contains the information necessary to write custom macros, edit existing macro, write new pulse sequences, and edit existing pulse sequences.

## 4.2 Host Computer Setup and Software Installation

Host computer setup and software installation is covered in detail in the *VNMR and Solaris Software Installation* manual. NMR data is processed and displayed using the Sun host computer, which is connected to the *MERCURYplus* NMR spectrometer with an Ethernet cable.

Setting up the host computer involves the following general steps:

- Unpacking the Sun computer
- Connecting peripheral devices
- Connecting the Sun computer to the *MERCURYplus* console
- Installing the Solaris operating system software (including UNIX)
- Installing the VNMR software
- Configuring system hardware
- Setting up user accounts
- Setting printers and plotters

During this process, the Sun hard disk is laid out specifically for the needs of the VNMR software and NMR operation. VNMR is installed in the directory `/export/home` and a link called `/vnmr` is placed at the root level. User accounts are also installed in `/export/home`.

Software is installed from CD-ROMs.

## 4.3 VNMR Directory Structure

An overview of the VNMR directory and file structure is shown in **Figure 15**. This file structure is discussed in detail in the *Getting Started* manual.

The `vnmr` directory and file structure is set up with the global or system files and directories that have read and execute permission for all users and a group of user files and directories that the user has read, write, and execute permissions. The global or system files and directories are administrated by the user `vnmr1`. These files and directories are located in `/export/home/vnmr` which is also accessed via the symbolic link in root, `/vnmr`.

Only the user `vnmr1` and root have read, write, and execute permissions while others have read and execute permission for files and directories in this area. If you must make changes to VNMR files and directories, make them as `vnmr1`, not as root. The global or system files

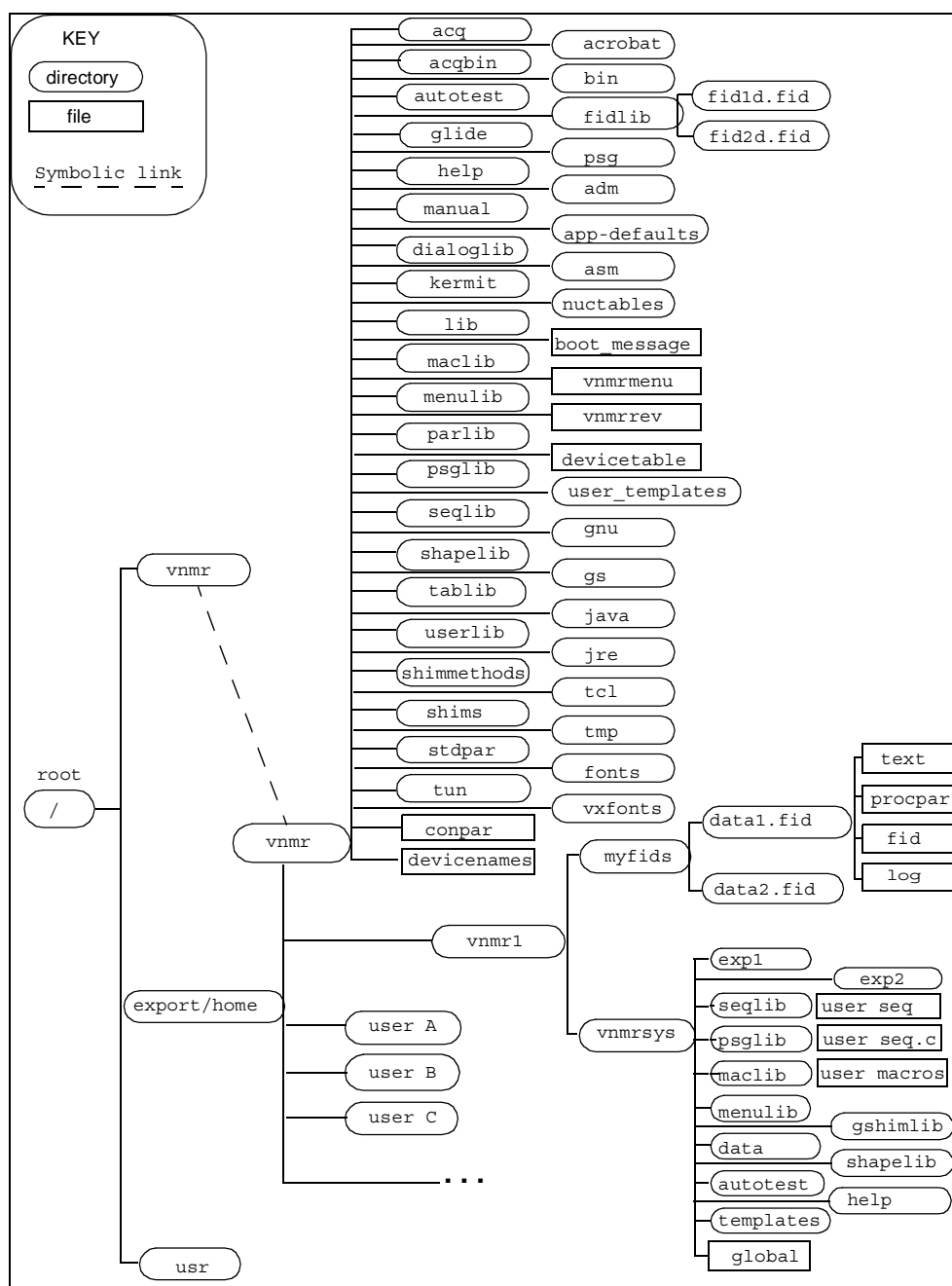


Figure 15. VNMR Directory Structure

and directories contain macros, pulse sequences, binary files, optional VNMR software and other files that have a common usage to all users. The users area contains files that are owned by that user only.

When VNMR is installed and user accounts are established, certain directories are created in the user's home directory. In some cases these directories have the same names in both the user work space in the /vnmr directory. When a command or instruction is executed in vnmr by a user, VNMR first searches the user's /vnmr/sys/ directory for the command, pulse sequence, etc. and then searches the global files in /vnmr. The *System Administration* manual provides more detailed explanation. When creating custom macros,

pulse sequences, menus, etc. it is good practice to give these files different names from the similar files in `/vnmr`. If you want the customized macros, pulse sequences, etc. to be available to all users, place them in the appropriate directory in `/vnmr`.

## 4.4 Managing Disk Space

The primary tool used by the system administrator to manage disk space is the UNIX command `df -k`, see the *System Administration* manual for more details. This command may be executed from any terminal window by any user. The output from this command may look like this:

In this example, user accounts are placed in `/export/home/<user>` in the `/` (root)

File system	kbytes	used	avail	capacity	Mounted on
<code>/dev/dsk/c0t0d0s0</code>	2467482	1818540	599593	76%	<code>/</code>
<code>/proc</code>	0	0	0	0%	<code>/proc</code>
<code>fd</code>	0	0	0	0%	<code>/dev/fd</code>
<code>swap</code>	243472	496	242976	1%	<code>/tmp</code>
<code>/dev/dsk/c0t0d0s1</code>	1372362	477485	839983	37%	<code>/space</code>

directory. In some installations `/export/home/<user>` maybe a separate slice or partition. There are two user accessible slices or partitions on this disk:

`/dev/dsk/c0t0d0s0` mounted on `/`

`/dev/dsk/c0t0d0s1` mounted on `/space`

When a partition or slice has reached 90% of its capacity it is time to do some maintenance. UNIX will, in some cases allow the capacity to exceed 100% but this is not a good condition. In the worst case, if the file system or slice containing the user's `vnmrsys` directory becomes full, the system may stop during a go or VNMR will not load or process a data set. This situation can lead to data being lost.

Each user account has a `vnmrsys` directory contain `maclib`, `seqlib` and other needed VNMR directories. Also within the user's `vnmrsys` directory is a directory for each experiment work space. Each work space or experiment provides temporary storage of the data. The data in the experiment work space remains intact until an acquisition is started in that experiment, a previously saved data set is loaded into the current experiment, or the current experiment is deleted (see the *Getting Started* manual for details on creating and deleting experiments in VNMR). The amount of disk space consumed by an experiment depends upon the type of NMR experiment. To manage this, users should save their data and either remove the experiment (delete the experiment workspace), execute a simple experiment that uses little disk space (`s2pul` with `np=8k` for example), or load a previously saved data set that has a small number of data points defining the FID.

Saving the data to a directory that is on the same slice or partition as the user's `vnmrsys` will not improve matters. The saved data should be placed in a directory on a different slice, a different physical hard disk, a network server disk, or copied to some archive such as a tape. The contents of directories such as `maclib`, `seqlib`, etc. do not tend to fill up with old files that quickly except for users that are developing pulse sequences, macros, etc. Old shim files tend to accumulate and can be cleared out from time to time. Periodic archiving of important spectra to tape, central file servers, etc. and removal of these files from the disk is necessary. Small data files can be copied to floppy disks. The use of tapes and floppy disks is discussed in detail in the *System Administration* manual.

## 4.5 Tuning Probes

Probe tuning is covered in [Chapter 6](#) of the *Getting Started* manual.

Tuning operations and tuning ranges specific to each probe are covered in the manual provided with the probe.

## 4.6 Data Acquisition – Calibration and Indanone Spectra

This section covers the calibration of the spectrometer and the acquisition of several 1D and 2D data sets using the 2% 2-ethyl-1-indanone sample, a typical small molecule with a formula weight of 160 g/mole. Step-by-step instructions for calibration and acquiring spectra of the 2-ethyl-1-indanone sample are given in [2.1 “AutoCalibration and GLIDE Operation Demonstration” page 21](#) and [2.2 “Automated Data Acquisition” page 26](#) of this manual and will not be repeated here. This section examines the order in which the calibration data is acquired and resulting spectra.

### The Calibration Process

Before acquiring spectra of a sample the spectrometer must first be calibrated. Calibration is necessary if one of the following is true:

- The probe has been changed
- The experiments are to be run at a different temperature than the last calibrations
- Solvent changes significantly (i.e., an organic lock solvent vs. deuterium oxide lock solvent)
- The calibration has not been run for several weeks (in this case it is simply a matter of good laboratory practice to check the calibration).

All calibration spectra are saved in:

```
/export/home/vnmr1/vnmrsys/data/probe_calibs/  
probenname_calib_date.
```

Probe calibrations executed by vnmr1 are written to probe files one of two places.

- If the probe name *was created as a system probe and the probe name is unique* (it does not exist in /export/home/vnmr1/vnmrsys/probe) it is written to /vnmr1/probes/probe\_name.
- If the probe name *was not created as a system probe or the probe name is not unique* (it does exist in /export/home/vnmr1/vnmrsys/probe) it is written to /export/home/vnmr1/vnmrsys/probes/probe\_name.

Before you begin the calibration procedure, check the probe parameter. This parameter should be set to the probe that is currently installed in the magnet. If this is not true, set the parameter to the name of the probe that is currently installed. The names of the installed user level and system level probes can be obtained as shown in the following example:

Open a terminal window.

- User level probes:
 

```
> cd ~/vnmrsys/probes  
> lf  
> hcn/ HFCN/...
```
- System level probes:

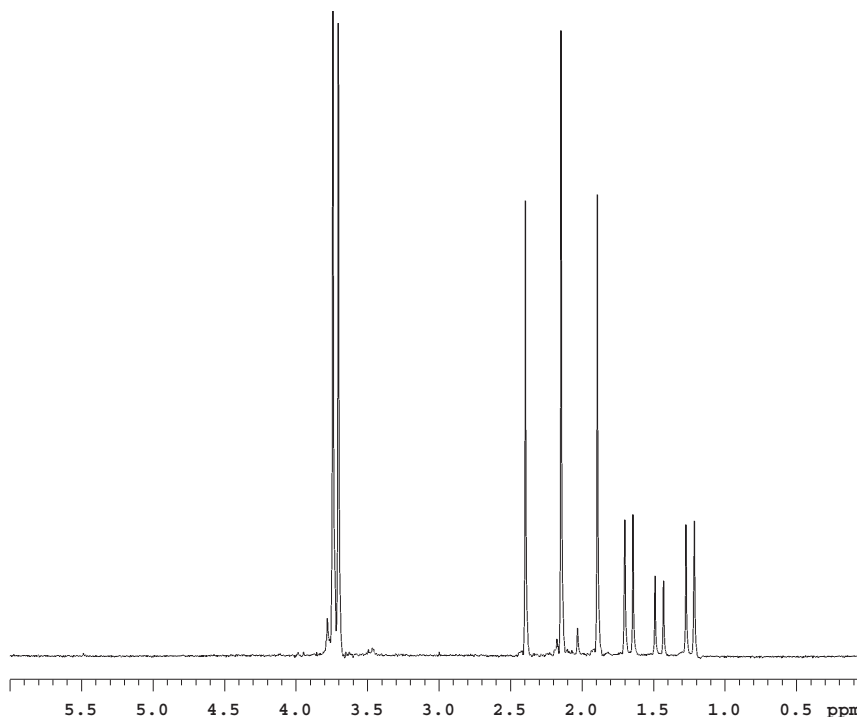
```
> cd /vnmr/probes
> lf
> GHX/ BB10mm/ probe.tmplt...
```

The actual probe directory names on your system might be different. The probe parameter must match one of the these directory names. If no calibration file exists for the probe or if the probe is new, add the probe using the `addprobe` command.

At this point you are ready to begin the calibration process. Upon completion of the calibration, 1D and 2D spectra of the test sample can be obtained using *GLIDE*.

After the 1D and 2D spectra are obtained, continue with the Automated Data Acquisition described in 2.2 “Automated Data Acquisition” page 26. A series of spectra is acquired.

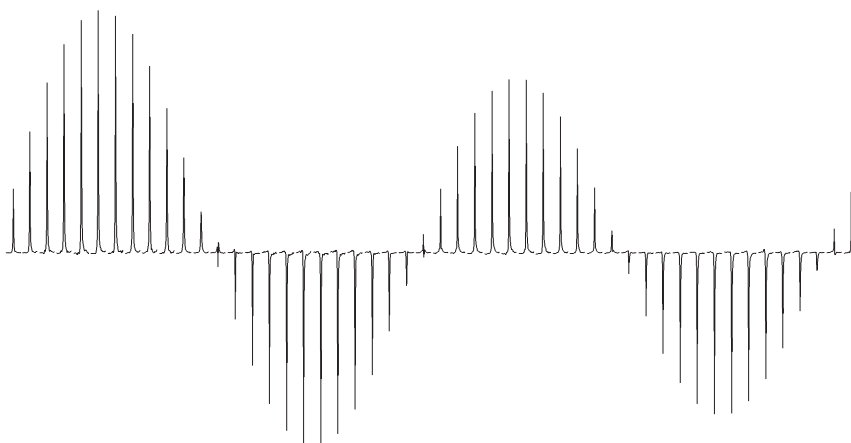
The first of the spectra, shown in Figure 16, is a  $^1\text{H}$  observe with  $\text{CDCl}_3$  as the lock solvent. This data is saved as `H1ref`. This spectrum contains three  $^{13}\text{CH}_3\text{I}$  resonances centered at about 2.2 ppm. The center resonance is from the protons attached to carbon-12 and the two outer resonances are from the protons attached to carbon-13. The other resonances in the sample are from trimethylphosphite that has reacted with the methyl iodide (which was initially 100%  $^{13}\text{C}$  enriched).



**Figure 16.**  $^1\text{H}$  Spectrum of  $^{13}\text{C}$ -Methyl Iodide

All trimethylphosphite has reacted to form a phosphonate ester  $(\text{CH}_3)_2\text{P}(=\text{O})(\text{OCH}_3)_2$ . This phosphonate ester has a doublet at about 1.5 ppm, methyl group attached directly to  $^{31}\text{P}$  and a triplet of doublets centered around 4 ppm that arise from  $^{13}\text{C}$  (outer pair of doublets) and  $^{12}\text{C}$  inner doublet of the methyl ester. The analysis of this sample is fully discussed in an article by Paul Keifer in *Magnetic Moments* (Keifer, P.A., *Magnetic Moments*, 1996, 8 (#2), 18–20). The reaction results in a sample is partially enriched to give approximately 60% abundance of carbon-13 in methyl iodide. The natural abundance of carbon-13 is 1.1% so this level of enrichment is more than adequate for the purposes of calibration.

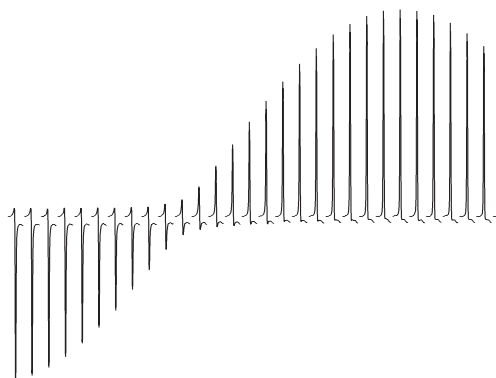
The next spectrum, shown in **Figure 17**, is an array of increasing  $^1\text{H}$  pulse widths based on the  $^1\text{H}$  pulse you specified in the Acquire window. If you did not enter a value for the pulse width it is set to the default targets pw90 is set to 15  $\mu\text{s}$  and  $\tau\text{pwr}$  of 51.



**Figure 17.**  $^1\text{H}$  pw Array

The observe transmitter power is set to the value you specified and reduce by 3 for the first test. If the resulting pw90 is shorter then the value you specified (or the default, if you did not specify a pw90 target) the next test is started. If the pw90 is longer then the target, the observe power is increased. Two attempts are made. If the calibration fails to achieve a pw90 that is less than the specified pw90 the autocalibration exits. If either attempt yields a pw90 that is less then the specified value the autocalibration routine adjusts the observe power the remaining tests are aborted. If the pw90 is less then the specified value, the autocalibration then adjusts the power until the measured pw90 is no greater then the specified value but not more then 0.5  $\mu\text{s}$  less than this value. The spectra from a successful calibration are saved as H1pw90.

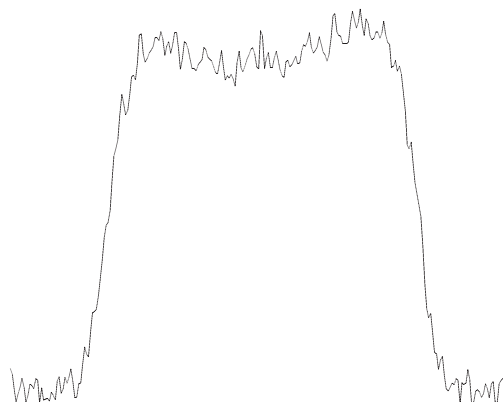
The carbon pw90 calibration is the next experiment. The pulse sequence changes from s2pul to PWXCAL. The specification for the carbon pw90 and  $\tau\text{pwr}$  are used as the target values. If no values were specified, the default values of 15  $\mu\text{s}$  at power, in this case  $\text{pw} \times 1.1$ , of 51 are used as the target values. Just as with the proton pw90 calibration, the autocalibration makes two attempts to achieve the specification and exits the autocalibration if the target specification is not reached after the second attempt. The data from the PWXCAL are saved as C13pwx and shown in **Figure 18**.



**Figure 18.**  $^{13}\text{C}$  pwx Array

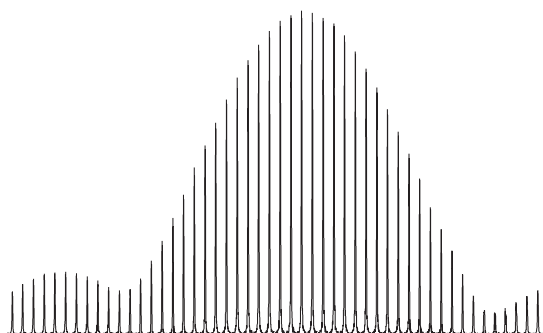
The next two experiments are run only if you have gradients. The first experiment calibrates the Z-gradient strength, produces the profile shown in **Figure 19**, and stores this information in the parameter `gcal`.

The next experiment calculates the ratios of the gradients to be used in various indirect detection experiments and stores this information in the parameter `Cgrad`, **Figure 20**.



**Figure 19.** Gradient Profile

The next calibration is carbon observe pulse width and the pulse sequence is changed to `s2pul` for direct observation of the carbon. The calibration will follow the same pattern as the calibration of the proton `pw90` and the carbon `pw90` using default values for target values if no target specification is given. A reference carbon spectrum is obtained first. The full reference spectrum contains three sets of resonances, at the far right (approximately -22 ppm) is the



**Figure 20.** Gradient Calibrations

(approximately -22 ppm) is the  $^{13}\text{C}$  resonance from methyl iodide, the doublet at 10 ppm is from the  $^{13}\text{C}$  resonance from the phosphonate methylester, and the 1:1:1 triplet (far left) at 78 ppm is the  $^{13}\text{C}$  resonance of chloroform-d,  $^2\text{HCCl}_3$ . The carbon `pw90` calibration is analogous to the proton calibration. The reference carbon spectrum, shown in **Figure 21**, is saved as `C13ref`.

The carbon observe `pw90` is determined using a `pw` array, see **Figure 22**, and saved as `C13pw90`.

The final calibration is of the proton decoupler. The first calibration step determines the value of  $\gamma\text{H}_2$  at a decoupler power of 40 (the default value).

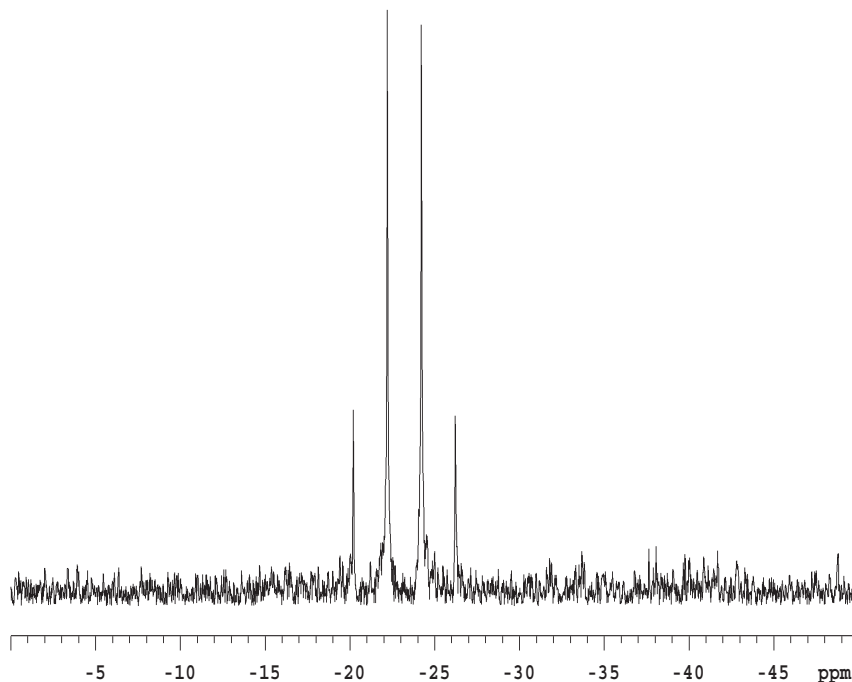
This measurement is made using continuous wave, `cw`, decoupling. The pulse sequence is the same as in the previous experiment, carbon observe with proton decoupling, except `pw` is now set to a fixed value, decoupler modulation mode; `dmm` is set to 'c', decoupler mode `dm`; is set to 'yyy', and the decoupler offset; `dof` is arrayed to produce the spectra shown in **Figure 23**.

From these spectra, the first estimate of the proton decouple `pw90` is made. Using the relationship between the pulse width and the decoupler field strength,  $\gamma\text{H}_2$ , shown in the equation below, the decoupler `pw90` is determined.,

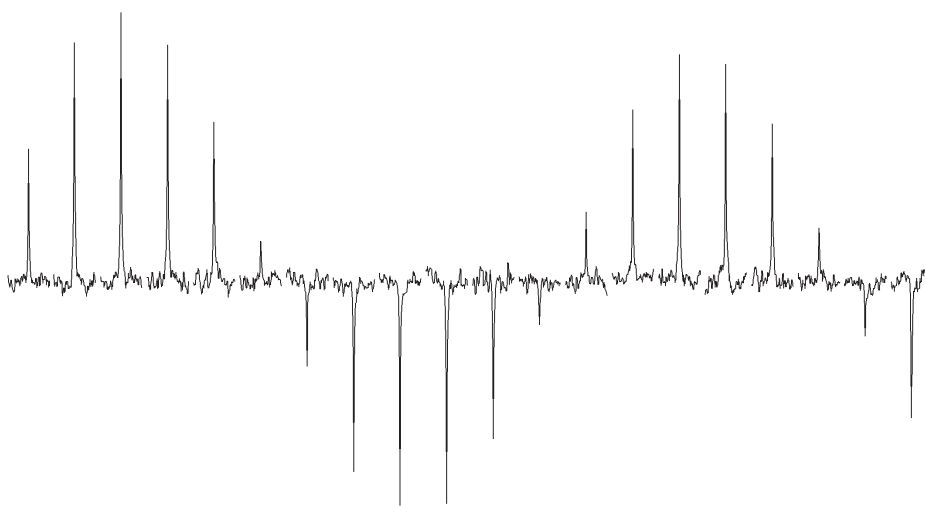
$$\gamma\text{H}_2 = \frac{1}{4(\text{pw90})}$$

The sequence is now set to `ppcal` and the proton decoupler 90° pulse, `pp`, is determined. These spectra, shown in **Figure 24**, are saved as `Hdec_dept`.



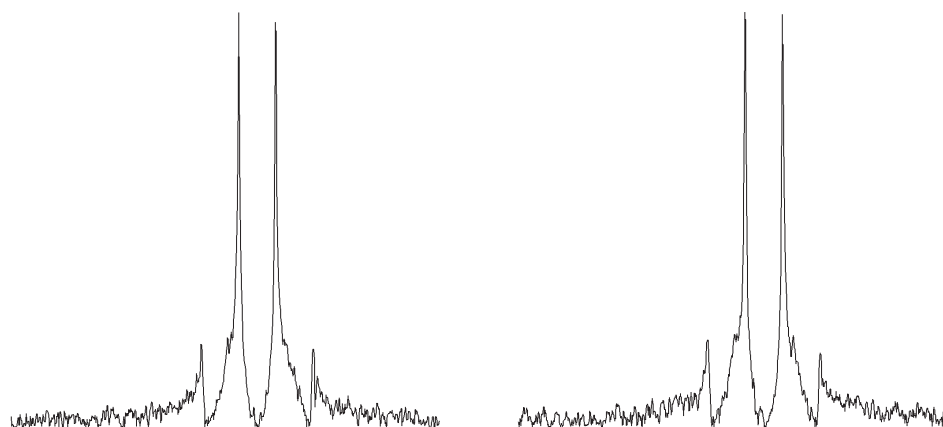


**Figure 21.** Proton Coupled  $^{13}\text{C}$  Spectrum of  $^{13}\text{C}$ -Methyl Iodide

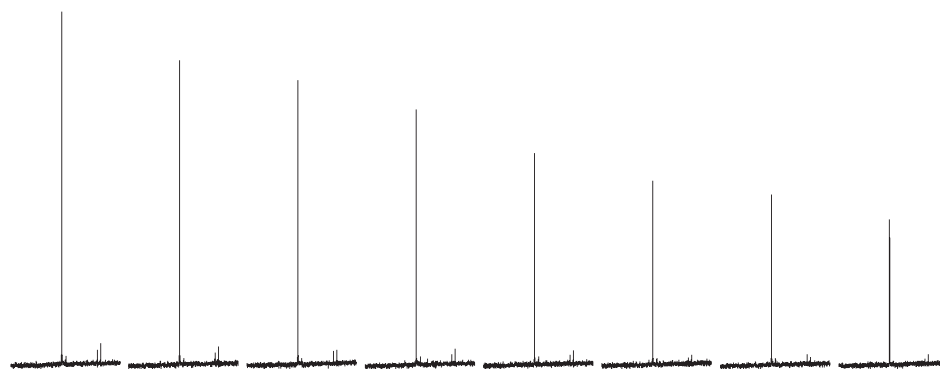


**Figure 22.**  $^{13}\text{C}$  Observe pw Array of Proton Coupled Spectra

These parameters and calibrations are used to setup WALTZ decoupling. This completes the calibrations. During the calibration procedure, spectra and the array values are plotted to provide a permanent record of the calibrations.



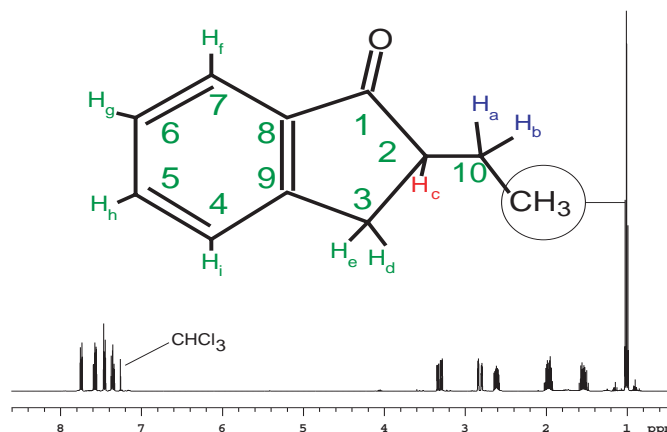
**Figure 23.** Proton Decoupler dof Array



**Figure 24.** Calibration of the Decoupler 90° Pulse Width, pp

## 2-Ethyl-1-Indanone Spectra

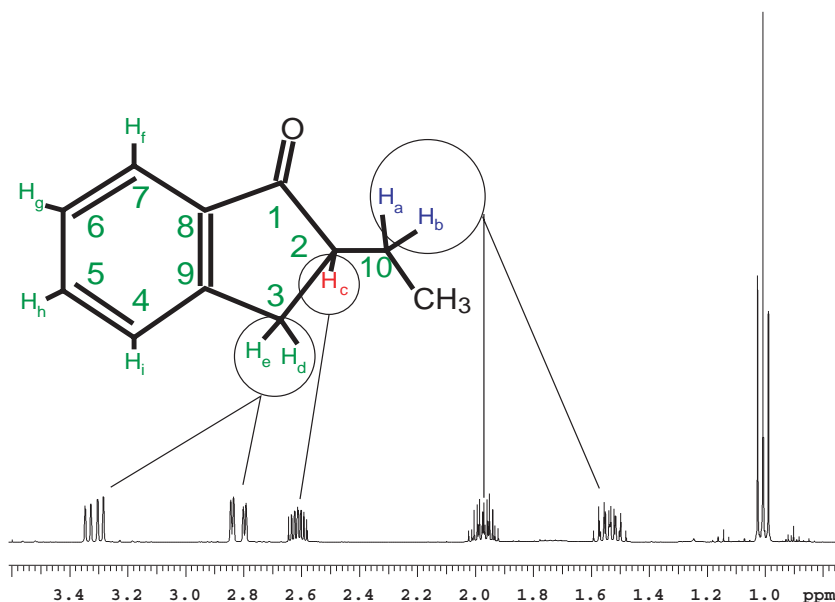
The proton NMR shows several distinct features. First, there are some impurities in the sample. These impurities, shown in **Figure 25**, are at the 2% level and some crosspeaks will show up in the 2D. The very large triplet for the methyl group has  $^{13}\text{C}$  satellites at  $J=125$  Hz. The singlet at 7.24 ppm is the residual  $\text{CHCl}_3$  in the  $\text{CDCl}_3$  solvent.



**Figure 25.** Proton Spectrum of 2-Ethyl-1-Indanone

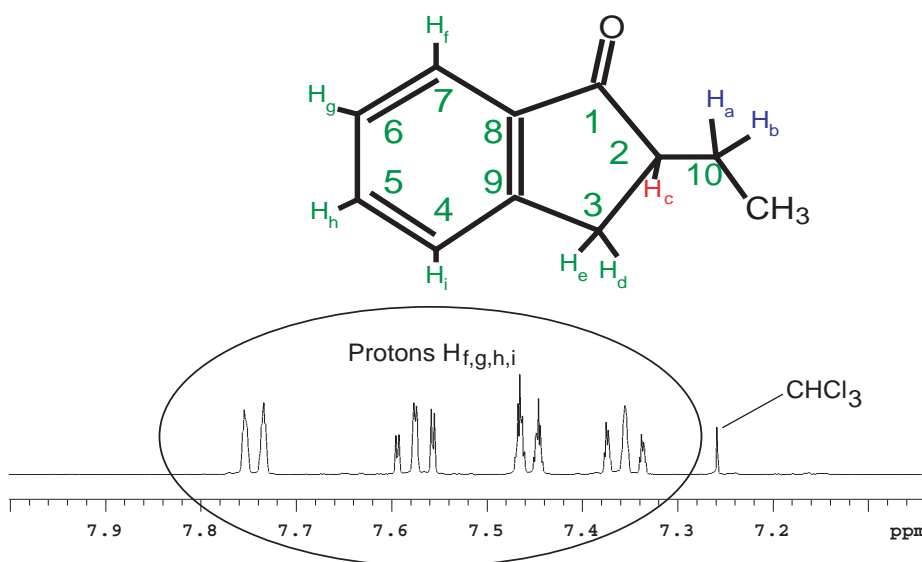
The protons are assigned in the two expansions.

The assignments are based upon the 2D data for the compound. The protons of the two  $\text{CH}_2$  groups in the molecule, shown in **Figure 26**, are magnetically nonequivalent and show up as individual multiplets. The signals at 1.9 ppm and 1.55 ppm belong to protons on carbon 10. The two double doublets at 2.8 ppm and 3.26 ppm belong to protons on carbon 3. A complex multiplet at 2.6 ppm is the single proton on carbon 2.



**Figure 26.** Aliphatic Region of the 2-Ethyl-1-Indanone Spectrum

The protons of the aromatic ring, shown in **Figure 27**, are assigned based on the gHMBC and gCOSY data. Some minor impurities can be seen as well as the residual  $\text{CHCl}_3$  signal. If the sample is shimmed very well there may be some truncation artifact on the  $\text{CHCl}_3$  signal.



**Figure 27.** Aromatic Region of the 2-Ethyl-1-Indanone Spectrum

The gradient COSY shows cross peaks describing the coupling pathways. Some smaller cross peaks are also present in the spectrum which actually arise from the impurities shown in [Figure 28](#). An example of this is the cross peak at 3.5 ppm.

The methyl triplet in [Figure 29](#) shows major cross peaks to the H10 protons. The multiplicity of the H10 and H2 protons cross peaks show that they are weakly coupled (the J value is small).

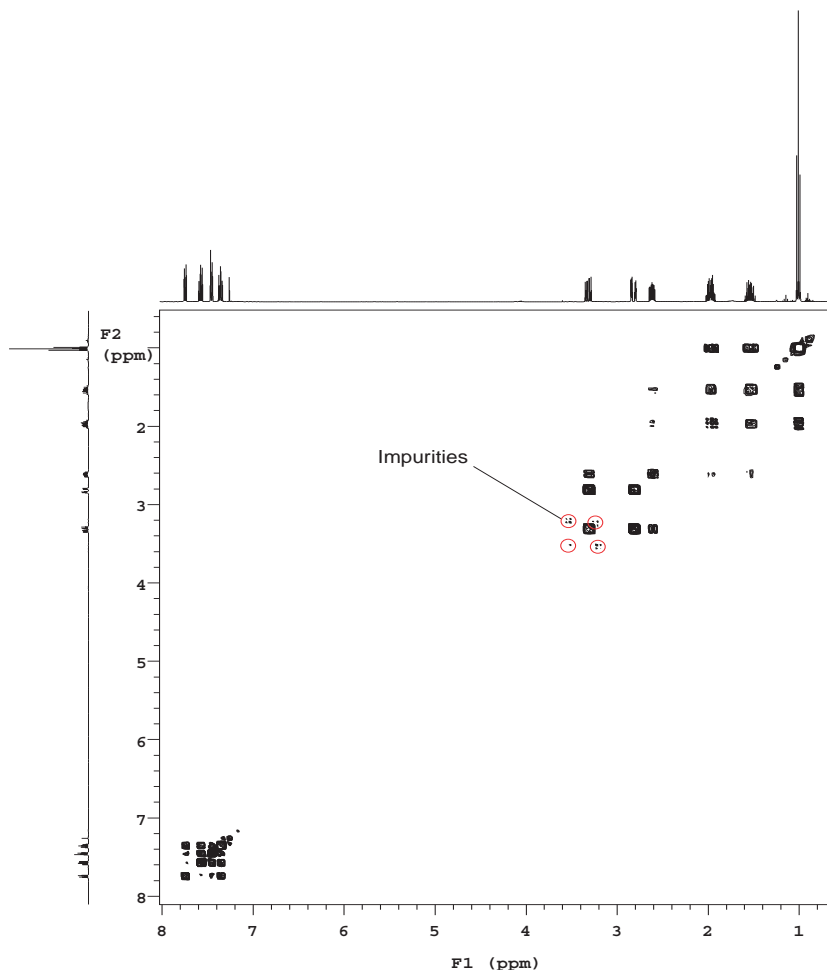
Assignment of the aliphatic region, begins with H7, the most deshielded proton, [Figure 30](#). From H7 direct connectivity is apparent to H6. The rest of the assignment is H6 to H5 (the other triplet) and then to H4.

The assignment of H7 to the signal at 7.72ppm is confirmed by the gHMBC data.

TOCSY is a phase sensitive experiment. The cross peaks are narrower than in the COSY giving higher “resolution”. Correlations among all protons in a spin system are observed in the TOCSY spectrum, see [Figure 31](#). The critical parameter is mix. In this case mix is 0.08 seconds which is sufficient to show correlations throughout the entire spin system. Shorter mix times will reveal fewer correlations.

The expansion shows the completely defined spin system starting with the CH3 group and ending with protons on C10, [Figure 32](#). A total of 5 crosspeaks are seen in the row]

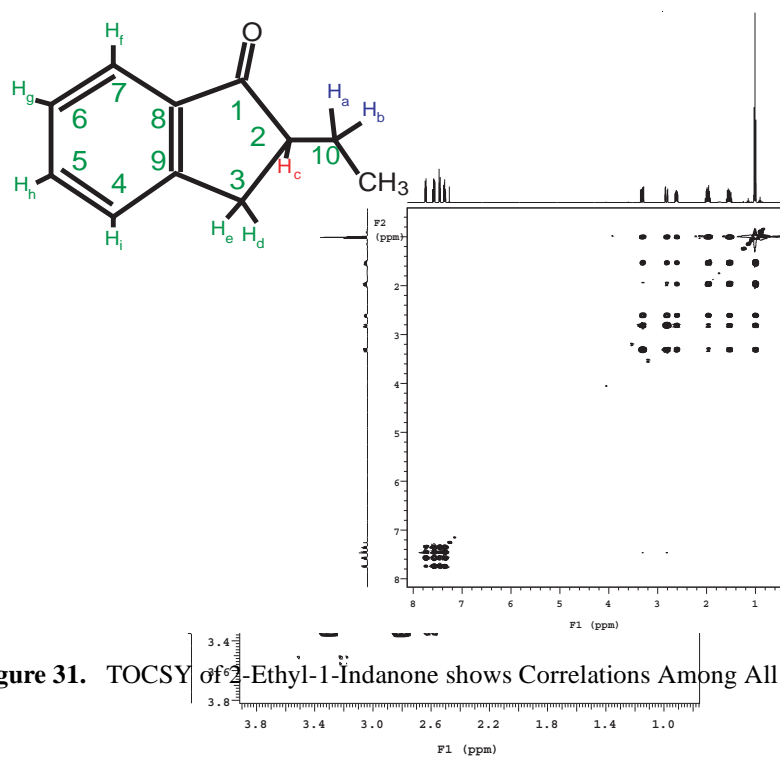
The indanone sample does not have any significant NOE crosspeaks, [Figure 33](#). The main area of interest in this spectrum is to note that the diagonal will be negative and the NOESY crosspeaks will be positive. Crosspeaks which appear to have both positive and negative components are actually not NOE correlations but coupling artifacts.



**Figure 28.** Gradient COSY of 2-Ethyl-1-Indanone

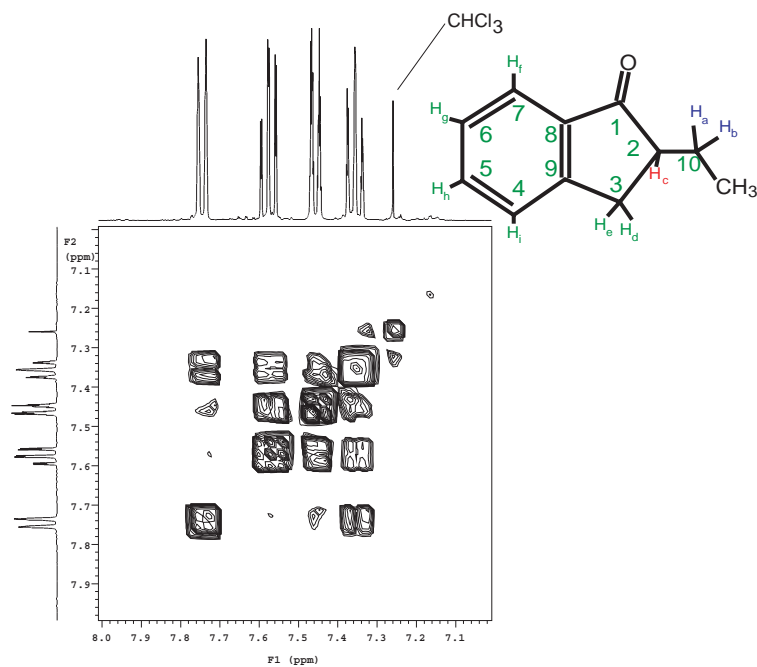
In the gHSQC (and HSQC) experiment, see [Figure 34](#) the protons correlate with the carbons to which they are attached. The detected nucleus is  $^1\text{H}$  and this results in a higher signal to noise than the  $^{13}\text{C}$  detected heteronuclear experiment. When compared to the HMQC experiment, the HSQC experiment has the advantage that the  $^1\text{H} - ^1\text{H}$  homonuclear coupling does not evolve. As a result the resolution in the 2D plane is higher in the HSQC experiment. The higher resolution has the added advantage of improving the signal to noise. The version of the HSQC experiment supplied with the *MERCURY* has the added benefit that it will distinguish  $-\text{CH}$ ,  $-\text{CH}_2$ , and  $-\text{CH}_3$  groups. In this case phase is indicated by whether the crosspeak is filled in with multiple contours (above the plane) or is a single contour (below the plane).

By contrast to the gHSQC experiment, the gHMBC (and HMBC) experiment shows long range (mostly 2 and 3 bond)  $^1\text{H} - ^{13}\text{C}$  correlations. This shows connectivity between the non-protonated carbonyl and the protons on C-2.

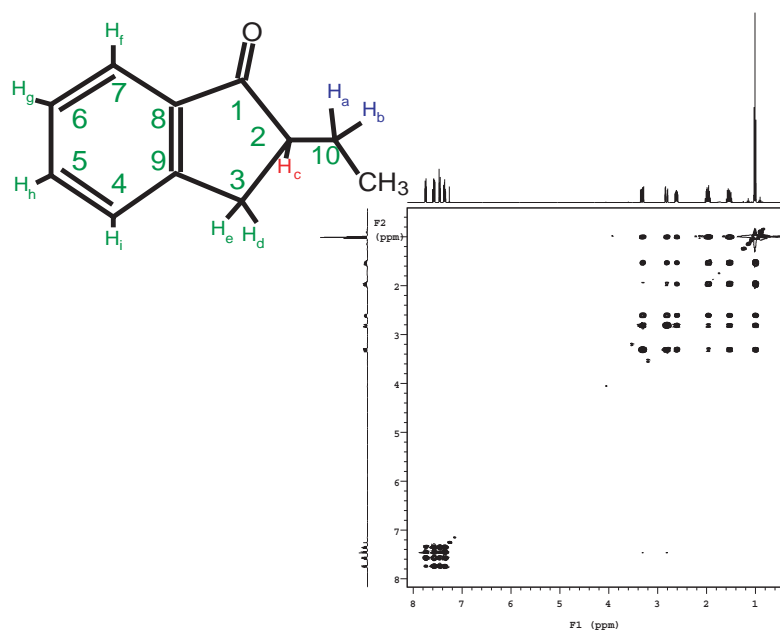


**Figure 31.** TOCSY of 2-Ethyl-1-Indanone shows Correlations Among All Protons

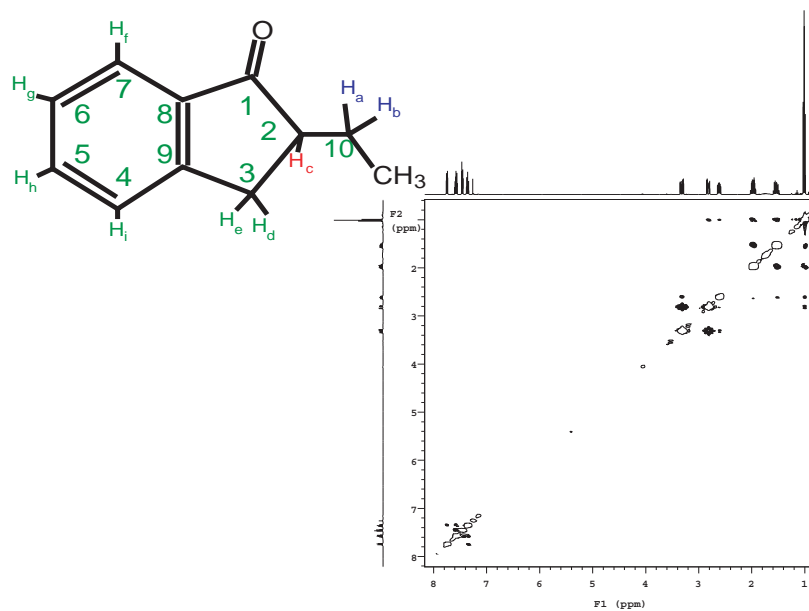
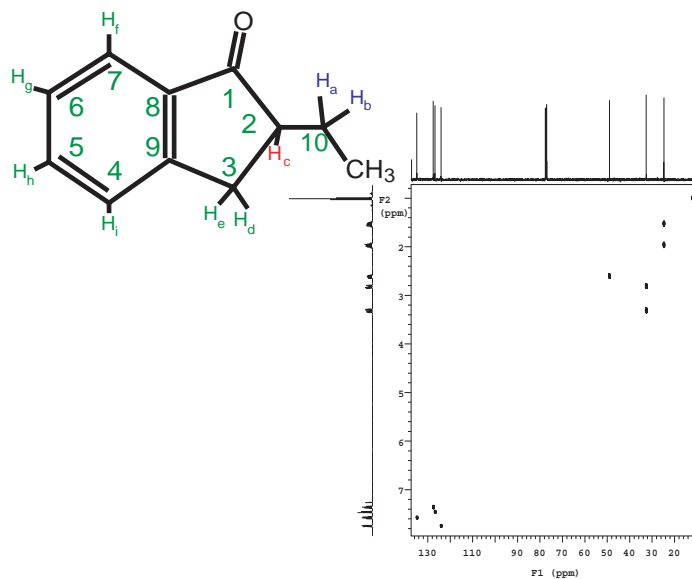
**Figure 29.** Gradient COSY (gCOSY) of Aliphatic Region of 2-Ethyl-1-Indanone



**Figure 30.** Gradient COSY (gCOSY) of the Aromatic Region of 2-Ethyl-1-Indanone



**Figure 31.** TOCSY of 2-Ethyl-1-Indanone shows Correlations Among All Protons

**Figure 33.** NOESY Spectrum of 2-Ethyl-1-Indanone**Figure 34.** HSQC Spectrum of 2-Ethyl-1-Indanone



## 4.7 Magnet Maintenance

Magnet maintenance is described in *Chapter 22* of the *System Administration* manual. Magnet maintenance consists of three basic elements:

- Periodic checks of the cryogen levels and boil off rates
- Liquid nitrogen fill
- Liquid helium fill.

**WARNING:** The extremely low temperature of liquefied helium and nitrogen can cause skin damage similar to high-temperature burns. Contact with the cold gas evolving from the liquid may produce the same effect. Delicate body tissues, such as the eyes, are easily damaged by exposure to cold gas or liquid. Skin can stick to metal that is refrigerated by liquid helium and can tear when pulled away. Immediately flood with large quantities of unheated water any area of the body that is “burned” by liquid or cold gas, and then apply cold compresses. If the skin is blistered or there is any chance the eyes are affected, immediately seek medical treatment.

**WARNING:** Wear goggles and loose-fitting protective gloves while working with cryogens.

### Periodic checks

A record or log book should be kept with dates, flow rates, cryogen levels, and notes on changes made to or observed in the magnet. A typical schedule of checks would be:

#### *Weekly*

1. Check air line traps for dirt or condensed water.
2. Record the following readings (note that the flow rates of nitrogen and helium depends on a variety of factors such as atmospheric pressure and a high reading is not necessarily an indication of a problem, but it is worth investigating):
  - Pressure at the air valve for the magnet.
  - Liquid nitrogen level.
  - Readings on the nitrogen and helium flow meters.

#### *Twice Each Month*

- Check liquid helium level.

#### *Monthly or Bimonthly*

- Check signal-to-noise and lineshape using the standard proton sample, the  $^{13}\text{C}$  90° pulse width, and the decoupler field strength. Keep the resulting spectra and parameter in a secure place for future reference.

## Filling Cryogenics

All cryogenics should be delivered in *nonmagnetic* dewars. Consult the manual supplied by Oxford complete instructions and for more detailed information on cryogen fill intervals and capacities.

### Liquid Nitrogen

The interval between liquid nitrogen fills is nominally 2 weeks. The Oxford manual that is supplied with the magnet should be consulted for the exact interval between nitrogen fills.

**CAUTION:** Failure to maintain the correct liquid nitrogen levels will lead to excessive liquid helium boil off which in turn may lead to a quench of the magnet.

### Liquid Helium

The interval between liquid helium fills is dependent upon the type of dewar. There are standard and long hold Dewars available for some magnets. The recommended refill interval is specified in the Oxford manual that is supplied with the magnet should be consulted for the exact interval between liquid helium fills and specific details on routine liquid helium fills. Depending upon the type of dewar, the exact details and procedures for liquid helium filling will vary, consult the Oxford Magnet Manual supplied with your magnet.

**CAUTION:** Failure to maintain the correct liquid helium levels may lead to a quench of the magnet.

**WARNING:** Wear goggles and loose-fitting protective gloves while working with cryogenics.

## 4.8 30-Day System Maintenance

The following procedures must be performed while logged in as `vnmr1`.

1. Updating Solvent Shims  
Reshim lineshape and then save shims to `/vnmr/shims/acetone`.  
Refer to the *Acceptance Test Procedures & Specifications* for the specific probe and to *Getting Started* – Section 6.9 Saving and Retrieving Shim Values
2. Check Calibration of `pw90` and `tpwr`, as described in 2.1 “AutoCalibration and GLIDE Operation Demonstration” page 21.
3. Update the lock frequency, as described in the *Software Installation* manual, Appendix B.
4. Check magnet drift, as described in 2.4 “Magnet Drift Test” page 32.
5. Perform hard drive maintenance: delete unused files, directories, old FIDs, macros, shims, or users.

## 4.9 Warranty and Who to Call for Assistance

The installation engineer will explain the standard warranty terms. Non standard warranty terms, if included as a condition of sale, are detailed in the sales contract and will not be covered by the installation engineer.

**Table 9.** Who to Call for Assistance

Online Customer Support and Information		Webb Addresses	
Service and Technical Support		<a href="http://www.varianinc.com/nmr/service">http://www.varianinc.com/nmr/service</a>	
Applications Support		<a href="http://www.varianinc.com/nmr/apps">http://www.varianinc.com/nmr/apps</a>	
Sales Support		<a href="http://www.varianinc.com/nmr/contact">http://www.varianinc.com/nmr/contact</a>	
Product Information		<a href="http://www.varianinc.com/nmr/products">http://www.varianinc.com/nmr/products</a>	
In warranty Service		Location	Phone Number
United States and North America		Palo Alto, California	1.800.356.4437
Europe		Local Sales Office	See <a href="#">page 17</a>
Other International		Local Sales Office	See <a href="#">page 17</a>
Technical Support		Location	Phone Number
United States and North America		Palo Alto, California	1.800.356.4437
Europe		Oxford, UK	44.1865.388.800
Other International		Local Sales Office	See <a href="#">page 17</a>
Post Warranty Support		Location	Phone Number
United States and North America		Palo Alto, California	1.800.356.4437
Europe		Local Sales Office	See <a href="#">page 17</a>
Other International		Local Sales Office	See <a href="#">page 17</a>
Applications Support		Location	Phone Number
United States and North America		Palo Alto, California	650.424.4526
		Columbia, Maryland	410.381.7229
Europe		Darmstadt, Germany	49.6152.703.253
Japan		Tokyo, Japan	81.3.5232.1211
Other International		Local Sales Office	See <a href="#">page 17</a>

## *Chapter 5.* **Acceptance Test Results**

This chapter contains the following forms for recording system information and acceptance test results:

- 5.1 “Computer Audit” page 61
- 5.2 “Installation Customer Training Checklist” page 63
- 5.3 “System Installation Checklist” page 65
- 5.4 “Supercon Shim Values” page 67
- 5.5 “Console and Magnet Test Results” page 69

*Notes:*

## 5.1 Computer Audit

Information about your site (please print):

Company/University \_\_\_\_\_  
 Address \_\_\_\_\_  
 \_\_\_\_\_  
 Principal User \_\_\_\_\_  
 Phone \_\_\_\_\_ Spectrometer type \_\_\_\_\_  
 Fax \_\_\_\_\_ Console S/N \_\_\_\_\_  
 Sales Order No. \_\_\_\_\_ Delivery (month/day) \_\_\_\_\_

Information on each computer (additional forms are on the back of this page). Include computers directly attached to the spectrometer, computers (networked or non-networked, on-site or off-site) used to process NMR data using Varian's VNMR software, and computers (on-site and off-site) used to process data collected on this spectrometer with software from other vendors.

Information on computer ____ of ____ (e.g., 1 of 3)	
Manufacturer _____	Model no. _____
Computer S/N _____	Purchased from _____
Memory (Mbytes) _____	Screen size (in.) _____
<b>Peripherals:</b> Internal hard disk (Mbytes) _____	
External hard disk (Mbytes) _____	Serial no. _____
Tape drive size _____	Serial no. _____
CD-ROM drive model _____	Serial no. _____
Printer model _____	Serial no. _____
Plotter model _____	Serial no. _____
Terminal model _____	Serial no. _____
Other peripheral _____	Serial no. _____
<b>Computer function:</b> NMR host _____	
Workstation running VNMR _____	on-site or off-site
Workstation running other NMR software _____	on-site or off-site
Workstation running VNMR and other NMR software _____	on-site or off-site
<b>VNMR version</b> _____	<b>Operating system</b> _____

The above computer audit was performed during installation of the system.

Varian Representative \_\_\_\_\_ Date \_\_\_\_\_

I certify that the information on this form is accurate and that all computers to be used to run VNMR software (including variants VnmrS, VnmrX, VnmrI, VnmrSGI, and VnmrV), or to run other software to process data obtained on this spectrometer, have been included in the audit (including those previously registered as part of purchases of other Varian NMR spectrometers).

Customer Representative \_\_\_\_\_ Date \_\_\_\_\_

Use these forms for additional computers. If more forms are needed, copy this page. Attach all copies to the Computer Audit.

<b>Information on computer ____ of ____ (e.g., 2 of 3)</b>	
Manufacturer _____	Model no. _____
Computer S/N _____	Purchased from _____
Memory (Mbytes) _____	Screen size (in.) _____
<b>Peripherals:</b> Internal hard disk (Mbytes) _____	
External hard disk (Mbytes) _____	Serial no. _____
Tape drive size _____	Serial no. _____
CD-ROM drive model _____	Serial no. _____
Printer model _____	Serial no. _____
Plotter model _____	Serial no. _____
Terminal model _____	Serial no. _____
Other peripheral _____	Serial no. _____
<b>Computer function:</b> NMR host _____	
Workstation running VNMR _____	on-site or off-site
Workstation running other NMR software _____	on-site or off-site
Workstation running VNMR and other NMR software _____	on-site or off-site
<b>VNMR version</b> _____	<b>Operating system</b> _____

<b>Information on computer ____ of ____ (e.g., 3 of 3)</b>	
Manufacturer _____	Model no. _____
Computer S/N _____	Purchased from _____
Memory (Mbytes) _____	Screen size (in.) _____
<b>Peripherals:</b> Internal hard disk (Mbytes) _____	
External hard disk (Mbytes) _____	Serial no. _____
Tape drive size _____	Serial no. _____
CD-ROM drive model _____	Serial no. _____
Printer model _____	Serial no. _____
Plotter model _____	Serial no. _____
Terminal model _____	Serial no. _____
Other peripheral _____	Serial no. _____
<b>Computer function:</b> NMR host _____	
Workstation running VNMR _____	on-site or off-site
Workstation running other NMR software _____	on-site or off-site
Workstation running VNMR and other NMR software _____	on-site or off-site
<b>VNMR version</b> _____	<b>Operating system</b> _____

## 5.2 Installation Customer Training Checklist

### Customer Information

Company/University		
Address		
Principal User		
Phone		Spectrometer type
Fax		Console S/N
Sales Order No.		Magnet S/N

### Magnet Familiarization:

Done	Topic	Reference
<input type="checkbox"/>	Overall magnet familiarization	Oxford Magnet Reference Manual
<input type="checkbox"/>	LHe and LN <sub>2</sub> top off procedures	Oxford Magnet Reference Manual and LHe refill video
<input type="checkbox"/>	Use of flow meters	Oxford Magnet Reference Manual
<input type="checkbox"/>	Antivibration system operation	Antivibration Accessory Installation Manual
<input type="checkbox"/>	Posting of magnetic field warning signs	Installation Planning Guide

### Probes:

Done	Topic	Reference
<input type="checkbox"/>	Probe installation removal, tuning, and filter setup for ID and TR experiments	Choose a specific probe manual AutoSwitchable probes Broadband probes Indirect Detection probes Nano probes Switchable probes Flow probes
<input type="checkbox"/>	VT system installation	Variable Temperature Unit Installation
<input type="checkbox"/>	VT system operation	User Guide: Liquids NMR, Chapter 8

### Console:

Done	Topic	Reference
<input type="checkbox"/>	Major component overview	System Overview
<input type="checkbox"/>	Shut down procedures	System Administration manual, 3.1, 3.2, 3.3
<input type="checkbox"/>	Host console connection and overview	System Administration manual, 3.1



**Host Computer:**

<i>Done</i>	<i>Topic</i>	<i>Reference</i>
	Host computer setup	Software Installation
	Solaris and VNMR software installation	Software Installation
	Directory structure overview	Getting Started manual, 1.4
	Using acqproc and makeuser commands	System Administration and Software Installation
	Shutdown and startup of UNIX	System Administration, 5.5
	Storage devices and accessories	System Administration, Chapter 10
	VNMR file backup and recovery	System Administration, Chapter 10

**Spectrometer Operation:**

<i>Done</i>	<i>Topic</i>	<i>Reference</i>
	Standard parameter and probe calibration setup	Walkup NMR
	Basic $^1\text{H}/^{13}\text{C}$ experiment setup using <i>GLIDE</i>	Walkup NMR, Chapter 2
	COSY demonstration <i>GLIDE</i>	Walkup NMR, 3.8
	Homonuclear and heteronuclear decoupling demonstration	Getting Started manual, 7.2 and Acceptance Test Procedures
	Manual lock and shim demonstration	Getting Started manual, 6.10
	Basic spectral display procedures	Getting started manual, 4.10 and 9.5
	Gradient shimming demonstration	User Guide: Liquids NMR, 11.6
	CP/MAS experiment information	User Guide: Solid-State NMR

**Miscellaneous:**

*ATP signoff and Walkup NMR demo, if applicable*

*Hallmark of Quality*

*Customer contacts for service, applications etc.*

*Accessory training as required*

---

 Varian Representative

---

 Date

---

 Customer Representative

---

 Date

## 5.3 System Installation Checklist

Company/University \_\_\_\_\_  
 Address \_\_\_\_\_  
 \_\_\_\_\_  
 Principal User \_\_\_\_\_  
 Phone \_\_\_\_\_ Spectrometer type \_\_\_\_\_  
 Fax \_\_\_\_\_ Console S/N \_\_\_\_\_  
 Sales Order No. \_\_\_\_\_ Magnet S/N \_\_\_\_\_

### Shipment Damage:

\_\_\_\_\_  
 \_\_\_\_\_

### Preinstallation Preparation:

Line voltage measured (Vac): console \_\_\_\_\_ accessory \_\_\_\_\_  
 Line pressure: air \_\_\_\_\_ N<sub>2</sub> \_\_\_\_\_  
 Air conditioning: \_\_\_\_\_  
 Cryogenics (liters): LHe \_\_\_\_\_ LN \_\_\_\_\_

### Testing:

1. Acceptance tests and computer audit
  - \_\_\_ Acceptance tests procedures finished
  - \_\_\_ Test results form completed and signed
  - \_\_\_ Computer audit completed and signed
2. System documentation review
  - Software Object Code License Agreement** (acceptance of product constitutes acceptance of object code license regardless of whether agreement is signed or not)
  - \_\_\_ Varian and OEM manuals
  - \_\_\_ Explanation of warranty and where to telephone for information
3. Installation Training
4. Installation Customer Training Check list

*Notes:*

## 5.4 Supercon Shim Values

Fill in the following information:

### Magnet Frequency and Serial Number

Magnet Frequency \_\_\_\_\_

Serial Number \_\_\_\_\_

### Measurement in

Helipot \_\_\_\_\_

Amps \_\_\_\_\_

### Measurements

Measurement	1. Date:	2. Date:	3. Date:
Z0			
Z1			
Z2			
Z3			
Z4			
X			
Y			
ZX			
ZY			
XY			
X2-Y2			
Drift			
Spacers			
Main Field Current			
Customer Signature:			
Varian Representative Signature:			

*Notes:*

# 5.5 Console and Magnet Test Results

Fill in the following information:

From "AutoCalibration and GLIDE Operation Demonstration," page 21

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From "Automated Data Acquisition," page 26

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From "Homonuclear Decoupling," page 31

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From "Magnet Drift Test," page 32

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From "Variable Temperature Operation (Optional Hardware)," page 33

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From "Temperature Accuracy for VT Systems (Optional Test)," page 34

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From "Stability Calibration for High-Stability VT (Optional Test)," page 36

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Varian Representative \_\_\_\_\_ Date \_\_\_\_\_

Customer Representative \_\_\_\_\_ Date \_\_\_\_\_

*Notes:*

## Numerics

- 180° pulse, 15
- 2-Ethyl-1-indanone in chloroform- d
  - automated data acquisition, 26
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