UNITY INOVA Solids Hardware Installation

UNITY INOVA NMR Spectrometer Systems

Pub. No. 01-999044-00, Rev. A0398

VARIAN

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Revision history:
A0896 – Initial release as Pub. No. 87-195482-00
A0297 – Updated for VNMR 5.3
A0398 – Initial release as Pub. No. 01-999044-00

Applicability of manual:
Installatioon of the solids modules on Varian UNITY*INOVA* NMR spectrometers:
Rotor Synchronization (Part No. 01-903728-00)
Wideband NMR Module (Part No. 01-903007-00)
Wideline Solids Module (00-990488-0x)
CP/MAS Solids Module (Part Nos. 00-990402-04, 00-990403-04, 00-990404-04)
CRAMPS/Multipulse Solids Module (Part No. 00-990487-0x)

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SAFETY PRECAUTIONS

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

The following warning and caution 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 permanent damage to equipment or data.**

WARNINGS

Cardiac pacemaker and metal prosthetics wearers must remain beyond the 5-gauss stray fields, as shown in the table below, until safety is clearly established; for example, remain more than 4.6 meters (15 feet) from 600-MHz magnet.

The magnet system generates strong magnetic and electromagnetic fields that can affect operation of some cardiac pacemakers or harm a metal prosthesis. 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 always notify their physician and discuss the health risks of being in proximity to magnetic fields. Wearers of metal prosthetics should contact their physician to determine if a danger exists. The following table may help determine the effect of a system on pacemakers or a metal prosthesis. The table shows the radial (horizontal) and axial (vertical) extent of a 5-gauss-level stray magnetic field as measured from the center of the magnet:

Proton Frequency (MHz)	*Bore (mm)*	*Radial Extent (m)*	*Axial Extent (m)*
200	51	1.5	1.5
200	89	2.0	2.0
300	51	1.7	1.7
300	89	2.2	2.8
400	54	2.2	2.8
400	89	3.0	3.8
500	51	2.8	3.6
600	51	3.7	4.6
750	51	6.1	7.6

WARNINGS *(continued)*

Refer to the *Installation Planning Guide* for your system for additional stray magnetic field plots and the effect of the stray field on electronic equipment. Varian provides signs containing this warning with each system. Post the signs according to the directions on the sign. Upon request, additional signs are available at no charge.

Keep metal objects beyond the 5-gauss field of the magnet.

The strong magnetic field of the dewar attracts objects containing steel, iron, or other “magnetic” materials, such as tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Unless restrained, such objects can suddenly fly towards the magnet, causing personal injury and extensive damage to the probe, the dewar, and the superconducting solenoid. Only nonferromagnetic materials (such as plastics, aluminum, wood, and stainless steel) should be used in the area around the magnet dewar.

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

Dangerous high voltages exist inside the equipment that can kill or injure.

Do not substitute parts or modify the instrument.

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

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.

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.

Avoid helium or nitrogen contact with any part of the body.

In contact with the body, 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 helium or nitrogen contacts the body, seek immediate medical attention, especially if the skin is blistered or the eyes are affected.

WARNINGS *(continued)*

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

Varian 200- and 300-MHz standard bore (51 mm) superconducting magnets include removable helium vent tubes. If the magnet dewar should quench (sudden appearance of gasses 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, follow carefully the instructions and safety precautions given in the *Varian Magnet Installation and Maintenance Manual.*

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. We recommend that you always wear safety glasses when working with the probe.

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.

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 gases from the dewar. To prevent tip-over, either bolt the magnet to the floor or support the magnet from the ceiling with ropes. Refer to the *Installation Planning Guide* for your system.

Solids equipment such as CRAMPS and CP/MAS require high pneumatic pressures.

Fittings must be secure. Wear safety glasses when working around the probe. Follow the warnings about maximum spin rates for particular rotors.

CAUTIONS

Prevent the sample tube from breaking.

The top of the sample tube can break off when the probe is removed. It is best to eject the sample before removing the probe from the magnet. Otherwise, use extreme caution when removing the probe if the sample is still in the probe.

Keep magnetic tapes, credit cards, and watches away from the magnet dewar.

Most cards, such as ATM cards, credit cards, and some new drivers licenses, contain a strip of magnetic media that can be damaged by a strong magnetic field. Many wrist and pocket watches are also susceptible to damage from intense magnetism.

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.

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 will contain moisture that can freeze, causing blockage of the vent tubes and possibly extensive damage to the magnet. Except when transferring nitrogen or helium, be certain that the relief valves are secured on the vent tubes.

Never operate high-power amplifiers with liquids probes.

On systems with high-power amplifiers for solids or microimaging, 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.

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.

Introduction

This manual describes the procedures used for installing and testing the following solids modules on UNITY*INOVA* NMR spectrometers:

- Rotor Synchronization Accessory
- CP/MAS Solids Module
- Wideline Solids Module
- CRAMPS/Multipulse Solids Module

Other Applicable Manuals

Procedures for installing and tuning solids probes are contained in the *NMR Probes Installation* manual.

Information for operating solids systems is contained in the *User Guide: Solid-State NMR*.

For solids systems schematics, see the publication UNITY*INOVA Solids Schematics*.

Conventions Used in the Manual

The following notational conventions are used throughout all VNMR manuals:

- Characters in `courier` (typwriter-like characters) are used for UNIX or VNMR commands, parameters, directories, and file names in the text of the manual; for example:

  The `shutdown` command is in the `/etc` directory.

- Characters in `courier` are also used to show screen output; for example:

  `Self test completed successfully`

- Italic characters are used for text displayed on the screen that is not the same every time; for example,

  `Abort at` *`some_address`*

  means the value of *`some_address`* depends upon when the abort command is made—what you might see on the screen is a message like this:

  `Abort at 47F82`

- Because pressing the Return key is required at the end of almost every command or line of text you type on the keyboard, use of the Return key is mentioned only in cases where it is *not* used. This convention avoids repeating the instruction “press the Return key” throughout most of this manual.

Except for a few commands entered from UNIX, the commands in this manual are invoked in the VNMR window environment. Although some VNMR commands (such as `echo` and `vi`) have the same name as UNIX commands, the VNMR and UNIX syntax is always different.

Chapter 1. Getting Started

This manual describes the procedures used for installing and testing the following solids modules on UNITY*INOVA* NMR spectrometers:

- Rotor Synchronization Accessory
- CP/MAS Solids Module
- Wideline Solids Module
- CRAMPS/Multipulse Solids Module

Figure 1 shows the UNITY*INOVA* NMR console next to the solids cabinet.

This chapter provides information about how to proceed through this manual for different combinations of solids hardware. Also in this chapter is information about what to do before the installation is started.

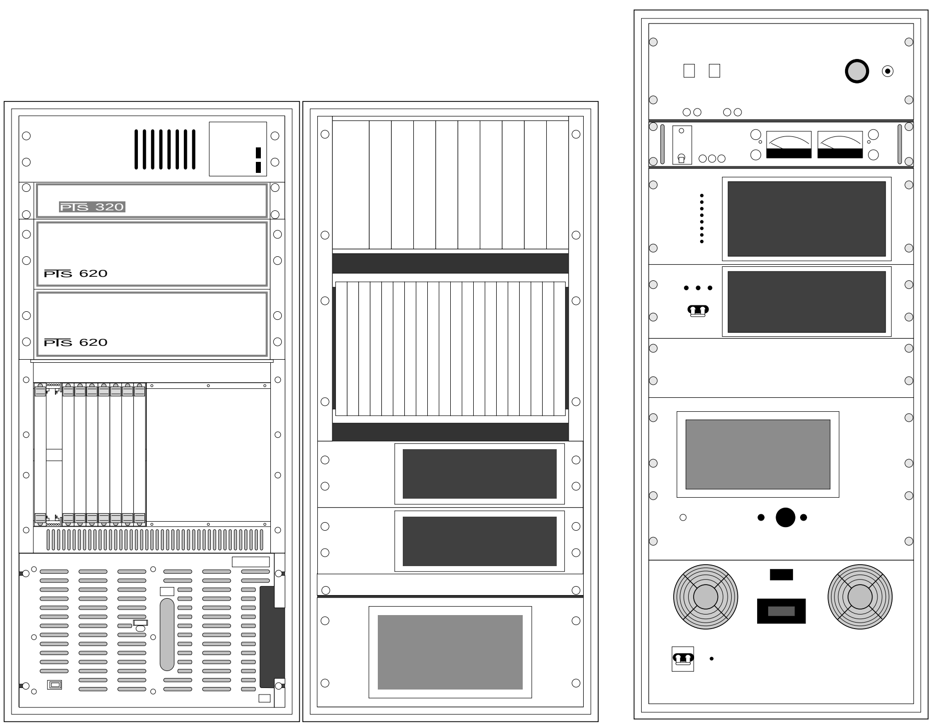


Figure 1. UNITY*INOVA* NMR Console with Solids Cabinet

1.1 Solids Modules Installation Guide

Listed below are the chapters in this manual that guide you in installing the different solids modules. Work through the chapters, in the order listed, under the solids module you want to install.

Rotor Synchronization and Rotor Speed Controller Accessory

- Complete all of Chapter 2, "Rotor Synchronization and Rotor Speed Controller Installation," on page 19.

CP/MAS Module

- Complete all of Chapter 2, "Rotor Synchronization and Rotor Speed Controller Installation," on page 19.
- Complete all of Chapter 4, "CP/MAS Solids Module Installation," on page 39.

Wideline Module

- Complete all of Chapter 3, "Wideband NMR Module," on page 35
- Complete all of Chapter 5, "Solids Cabinet Preparation and Installation," on page 45.
- Complete all of Chapter 6, "Wideline Module Tests," on page 53.

CRAMPS/Multipulse Module

- Complete all of Chapter 2, "Rotor Synchronization and Rotor Speed Controller Installation," on page 19.
- Complete all of Chapter 3, "Wideband NMR Module," on page 35
- Complete all of Chapter 5, "Solids Cabinet Preparation and Installation," on page 45.
- Complete all of Chapter 7, "CRAMPS/Multipulse Module Tests," on page 67.

Complete Solids Module

- Complete all of Chapter 2, "Rotor Synchronization and Rotor Speed Controller Installation," on page 19.
- Complete all of Chapter 3, "Wideband NMR Module," on page 35
- Complete all of Chapter 5, "Solids Cabinet Preparation and Installation," on page 45.
- Complete all of Chapter 6, "Wideline Module Tests," on page 53.
- Complete all of Chapter 7, "CRAMPS/Multipulse Module Tests," on page 67.

1.2 Saving the Current Experiment

Before starting the installation, we recommend saving the current experiment, which is `exp1`. Enter `jexp1 svf(`*`file`*`)`, where *`file`* is the name of the file to be saved.

1.3 Installing the Software

1. If the system is currently running the latest version of Solaris (2.4 , 2.5, or 2.6) or VNMR (5.2 or 6.1) software, skip to the next section and log in as `vnmr1`.

   If the system is not running the latest versions of Solaris and VNMR software, follow the procedures in *VNMR, Solaris, and SunOS Software Installation Manual* to upgrade to the latest versions.

2. Boot up the system.

1.4 Configuring the Software

The rotor synchronization, wideline, CRAMPS/multipulse, and complete solids modules require you to configure the NMR system as described below.

1. When bootup is finished, log in as `vnmr1` (you may need to request a password from the user):

   ```
   login: vnmr1
   password:
   ```

2. After VNMR has started, enter the VNMR command `config`.
3. Use the VNMR CONFIG window to configure the system. VNMR configuration, including details on changing the “Max Spectral Width” and “Rotor Sync” system parameters, is described in the *VNMR and Solaris SunOS Software Installation* manual.
4. Enter `rts(`*`file`*`) su load='n'`, where *`file`* is the name of the file holding the shim values, to retrieve the shim values. Use the same file name that was used for the `svf` command during shutdown.

1.5 When the Installation is Finished

All replaced items from the system and all unused items from the hardware installation kit must be packaged and returned to Varian.

Chapter 2. Rotor Synchronization and Rotor Speed Controller Installation

This chapter provides instructions for installing the rotor synchronization and rotor speed controller hardware on a UNITY*INOVA* NMR spectrometer system. The rotor synchronization and rotor speed controller accessory (01903728-00) consists of the following:

- Rotor synchronization accessory (01-903729-00)
- Rotor speed controller accessory (01-903732-00)

The rotor synchronization accessory provides access to three additional pulse sequence elements: `xgate`, `rotorsync`, and `rotorperiod`. These elements are used to synchronize pulses and delays with the tachometer signal of the pneumatics/ tachometer box.

The rotor speed controller accessory provides computer control and stabilization of the spin rate of a CP/MAS sample. Software is built into the Magnet Sample Regulation (MSR) board and is controlled through the `spinner` command in VNMR.

Both of these accessories require a pneumatics/tachometer box. The rotor synchronization accessory requires one of the following:

- Pneumatics/tachometer box for VT (00-992090-01)
- Pneumatics/tachometer box for VT and rotor speed controller (01-903863-00)
- Pneumatics/tachometer box for VT that is upgraded with the rotor speed controller upgrade kit (01-903862-00)

The rotor speed controller accessory requires one of the following:

- Pneumatics/tachometer box for VT and rotor speed controller (01-903863-00)
- Pneumatics/tachometer box for VT that is upgraded with the rotor speed controller upgrade kit (01-903862-00)

2.1 Installing the Rotor Synchronization Accessory

This section describes how to install the rotor synchronization accessory. Please read the following cautions before beginning the installation.

CAUTION: Use only dry nitrogen (N_2) for variable temperature work.

While working near the magnet, prevent tools and other hardware from being drawn into the magnet bore. Plug the magnet bore and use nonmagnetic tools.

To prevent damage to the boards and components from electrostatic discharge, wear a grounded wrist strap while handling printed circuit. boards

The rotor synchronization accessory consists of the following:

- Rotor sync FPGA chip (Part No. 01-903362-01)
- Pneumatics/tachometer box for VT with mounting hardware
- Pneumatics/tachometer box power supply
- Control and power cable

The accessory works with Varian high-speed CP/MAS probes and Doty probes that have an optical tachometer. The accessory does not work correctly with Doty probes that have a triboelectric tachometer.

Doty probes must have the following additional hardware:

- Tachometer amplifier
- Tachometer cable, probe to amplifier
- Tachometer amplifier power supply
- BNC cable, tachometer amplifier to pneumatics/tachometer box EXTERNAL INPUT

The following procedures describe how to install the rotor synchronization accessory hardware.

To Install the Rotor Synchronization FPGA Chip

1. Power down the UNITY*INOVA* console. Follow the shutdown procedures in the *User Guide: Solid-State NMR*.
2. Remove the cables from the DAC (Data Acquisition Controller) board.
3. After placing a grounding strap on your wrist, remove the DAC board.
4. Insert the rotor synchronization FPGA chip into socket U12T of the DAC board. Make sure the chamfered corner of thee chip matches the chamfered corner of the socket and board silkscreen. See Figure 2.
5. Insert the DAC board into the card cage.
6. Reconnect the cables from to the DAC board using the cable labels as a reference.
7. Turn on the power to the UNITY*INOVA* console.

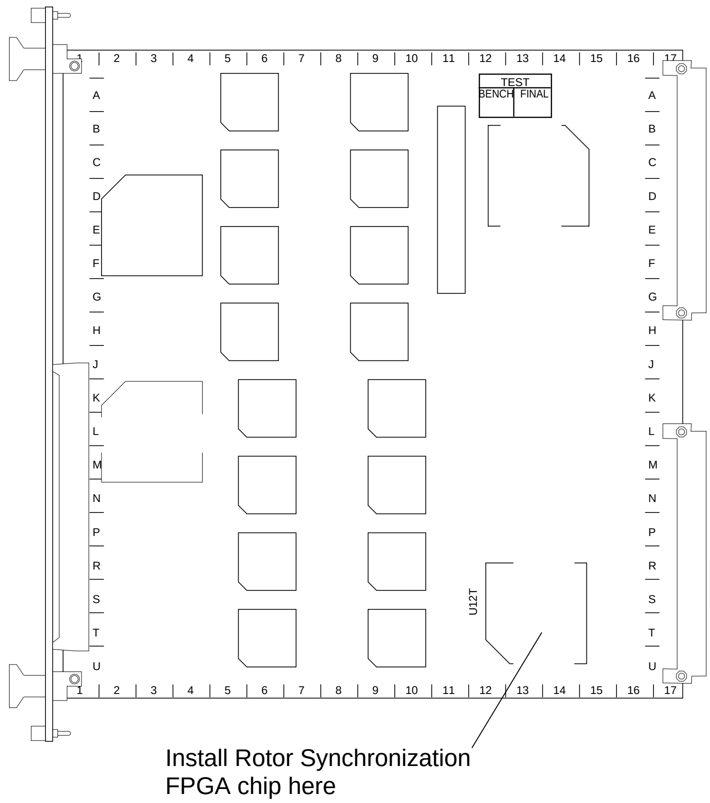


Figure 2. DAC Board Layout Showing Location for Rotor Sync Chip

To Install the Pneumatics/Tachometer Box

This procedure describes how to install the pneumatics/tachometer box for VT or the pneumatics/tachometer box for VT and rotor speed controller, shown in Figure 3. Rotor speed controller installation is described in "Installing the Rotor Speed Controller Accessory" on page 24.

1. Install the pneumatics/tachometer box on the standard (round) magnet leg using the hardware provided in the kit. If the antivibration leg system is installed (square legs), install the box on the leg using the pneumatics box mount kit if available (01-901479-00), or rest the pneumatics/tachometer box on the floor.

2. Connect the inlet hose of the pneumatics/tachometer box to a clean dry supply of compressed air or nitrogen. This supply should be at 90 psig continuous. Close both drive and bearing pressure regulators, turn on the supply. Make sure the flow meters are full open.

   Note that under normal circumstances the flow meter needle valves are left in the open position—they are not used for control.

3. Check for air leaks by slowly opening both pressure regulators until a pressure of 10 psig shows on the gauges. No air should escape through the hoses.

   If air does escape, check for grit in the probe connectors.

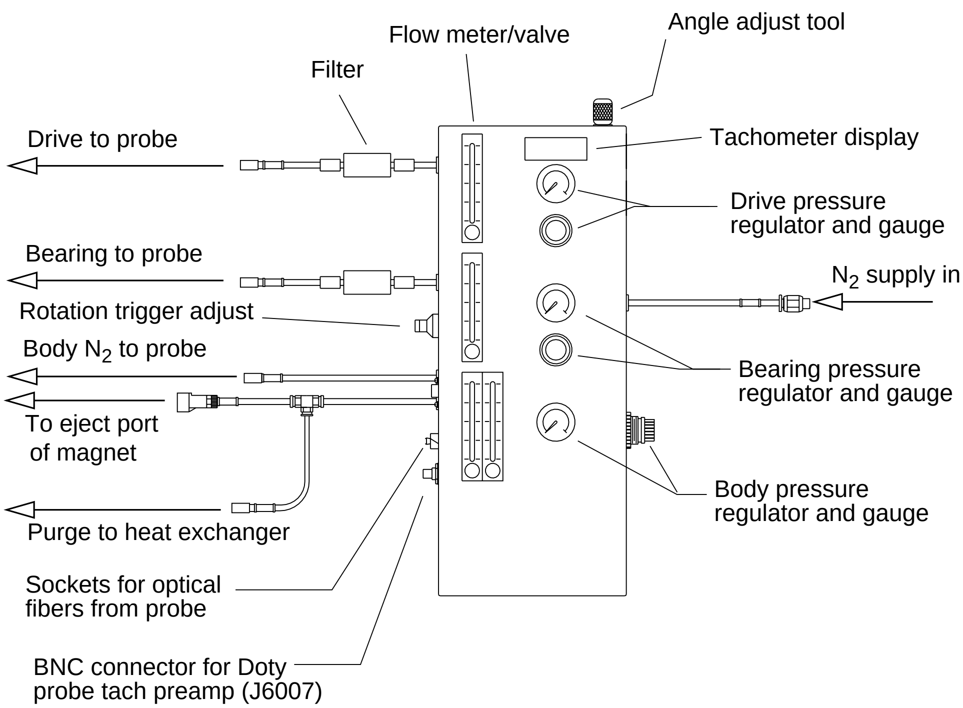


Figure 3. VT CP/MAS Pneumatics/Tachometer Box

When this test is finished, close both regulators.

4. Open the check valve of the air connector on each hose from the pneumatics/tachometer box to the probe. This is done by inserting a thin tool (such as a small flat-head screwdriver) into the connector and pressing down until it clicks. Turn the bearing regulator to show 10 psig. The flow meter should show a good flow rate. If not, check that the valve on the flow meter is in the full open position. Repeat this step for the drive supply.
5. Install the pneumatics/tachometer box power supply in the back of the rf cabinet, or another convenient location
6. Plug the supply cord from the pneumatics/tachometer box power supply into the 110 Vac outlet at the rear of the system power supply.
7. Install the cable from the power supply to the pneumatics/tachometer box, routing it with the other cables running to the magnet. Lead the coax terminated with a SMB connector through the cabinet to the front of the digital card cage.
8. Plug the SMB connector into the J2 at the EXT TIME BASE position.
9. Turn on the power and make sure that power is supplied to the pneumatics/tachometer box by checking for light from the optical connector of the pneumatics/tachometer box. The LCD display on the pneumatics/tachometer box should also display a decimal point.
10. Connect a CP/MAS probe to the pneumatics/tachometer box drive and bearing hoses.

11. Connect the body nitrogen air hose to the probe and adjust to 20 lpm. for VT operation.

    For VT operation, clean and dry body nitrogen (20 lpm) is required. For ambient operation, 5 to 20 lpm body air is recommended.

12. **For Doty probes only:**
    a. Connect the Doty tachometer amplifier OPTICAL OUTPUT position to the pneumatics/tachometer box EXTERNAL INPUT.
    b. Plug the power supply of the tachometer amplifier into a source of 110 Vac.
    c. Move the switch on the tachometer amplifier marked OPTICAL OFF to the position away from its label.
    d. On the pneumatics/tachometer box, set the 3-position switch, near the external input, to EXT NEG or EXT POS.

13. **For Varian VT CP/MAS probes only:**
    a. Connect the optical lines to the fiber optic lines of the probe to the sockets on the pneumatics/tachometer box.
    b. Set the 3-position switch near the external input to VA OPTIC.

To Replace the Air/Gas Filter

Replace the blue disposable 0.6 micron particle filter (Part No. 27-180354-00) after it has become discolored (turned red).

1. Disconnect the filter by pressing the movable sleeve on the gas hose fitting toward the tube fitting (you may need a small crescent wrench) and pulling the filter out of the tube fitting.
2. Insert a new filter, with the arrow on the filter pointing towards the probe.
3. Return the movable sleeve to its original position.

2.2 Installing the Rotor Speed Controller Accessory

This section describes how to install the rotor speed controller accessory. The rotor speed controller accessory consists of the following:

- Pneumatics/tachometer box for VT and rotor speed controller (01-903863-00) or a pneumatics/tachometer box that has been modified with the rotor speed controller upgrade kit (01-903862-00).
- Cable to the UNITY*INOVA* console breakout panel.
- Cable from the Breakout Panel to the Magnet-Sample Regulation (MSR) board.

To Upgrade the Pneumatics/Tachometer Box

This procedure describes how to modify the pneumatic/tachometer box by installing the plumbing, the wiring, and the two valves from the upgrade kit.

If you are installing a previously modified pneumatic/tachometer box, or if you are using the pneumatics/tachometer box for VT and rotor speed controller, first follow the procedure "To Install the Pneumatics/Tachometer Box" on page 21, and then skip to step 18 on page 27.

1. Disconnect the pneumatic/tachometer box from the system. Remove the rear cover from the pneumatic/tachometer box.
2. Remove the tubing elbow from the input side of the drive pressure regulator, located near the top of the pneumatic/tachometer box, and replace the elbow with the swivel tee from the kit. To release the tubing from the fitting, push the plastic gray ring toward the fitting
3. Make new holes in the pneumatic/tachometer box housing as follows:
   a. Make two holes in the side of the housing, below the Body Pressure regulator, as shown in Figure 4.
   b. Make six holes in the bottom of the housing, as shown in Figure 5. If weld studs are present on the bottom wall of the housing, punch or drill them out before creating the new holes.
4. On the electro-pneumatic regulator, install the tubing elbow, previously removed from the Drive Pressure regulator, into the SUP port. Use Teflon pipe tape to seal the treads of all tube fittings installed during this procedure. Install the 1/4-inch tube elbow into the OUT port. Plug the G port of the electro-pneumatic regulator.
5. Remove the black plastic cover, wire entry nut, metal washer, and rubber spacer from the electro-pneumatic regulator. Cut the rubber spacer and wrap it around the BRN, ORN, and RED wires from the cable harness. Thread the BRN, ORN, and RED wires through the wire entry nut and cable hole in the side of the housing. Replace the cover and secure it using the original two screws and O-rings.
6. Mount the electro-pneumatic regulator on the bottom wall of the housing using the screws and washers provided in the kit. The inlet port and cable hole of the regulator mount nearest the side wall of the housing.
7. Install connector J3 of the cable harness into the new hole in the side of the housing. Mount the BNC connector in the new hole above J3. Place short lengths of shrink tubing over the short YEL and GRN wires of the harness. Solder the

short YEL wire to the center conductor and the GRN wire to the outer conductor of the BNC connector according to the diagram in Figure 6. Cover the solder joints on the connector with shrink tubing and shrink it with a heat gun or hair dryer.

8. Connect the longer YEL wire to terminal 4and the GRN wire to terminals 3 of the tachometer. Replace the terminal screws on the tachometer with the new screws provided in the kit—these are longer than the originals.
9. Install tube elbows in the SUP and OUT ports of the control valve.
10. Mount the control valve to the bottom of the housing—using metric screws and spring and flat washers—in the two newly created holes (0.120 dia). Orient the OUT port of the control valve away from the electro-pneumatic regulator previously installed.
11. Check that the EXH (exhaust) port is free and clear. If this port is sealed with a set screw, remove the screw.

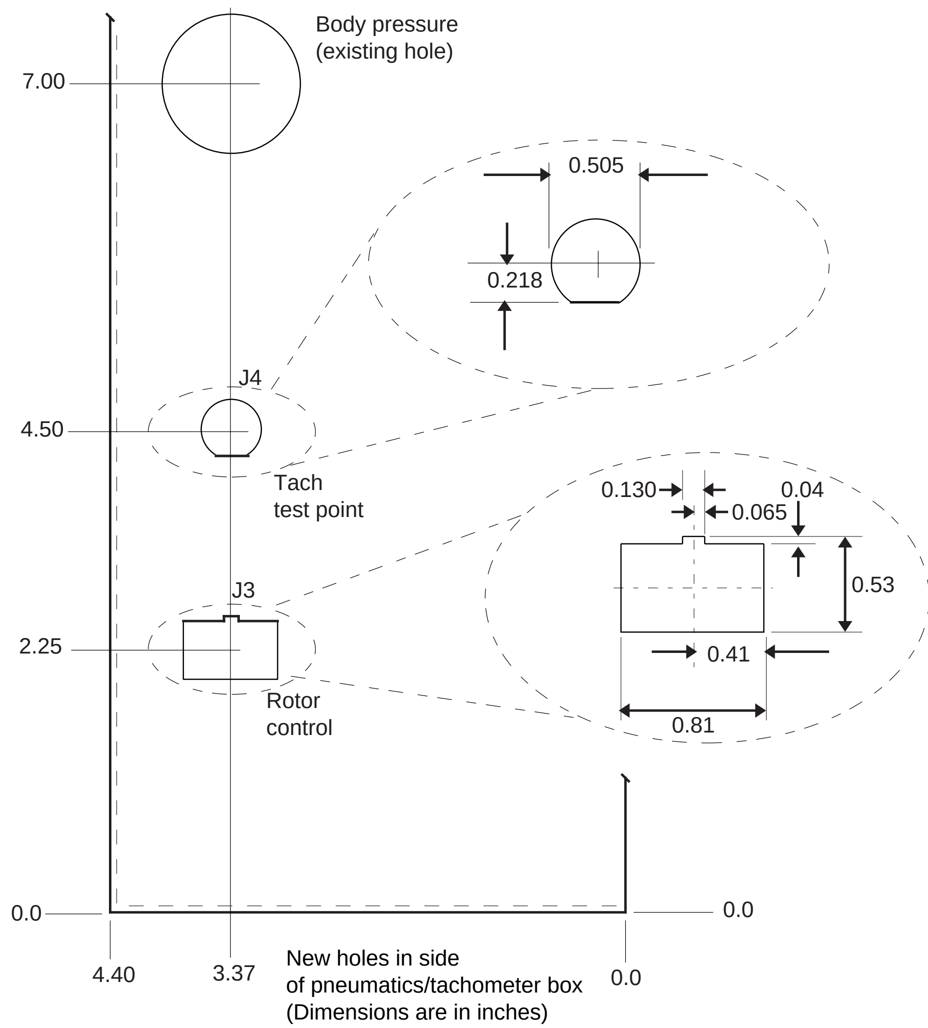


Figure 4. New Holes in the Side of the Pneumatics/Tachometer Box

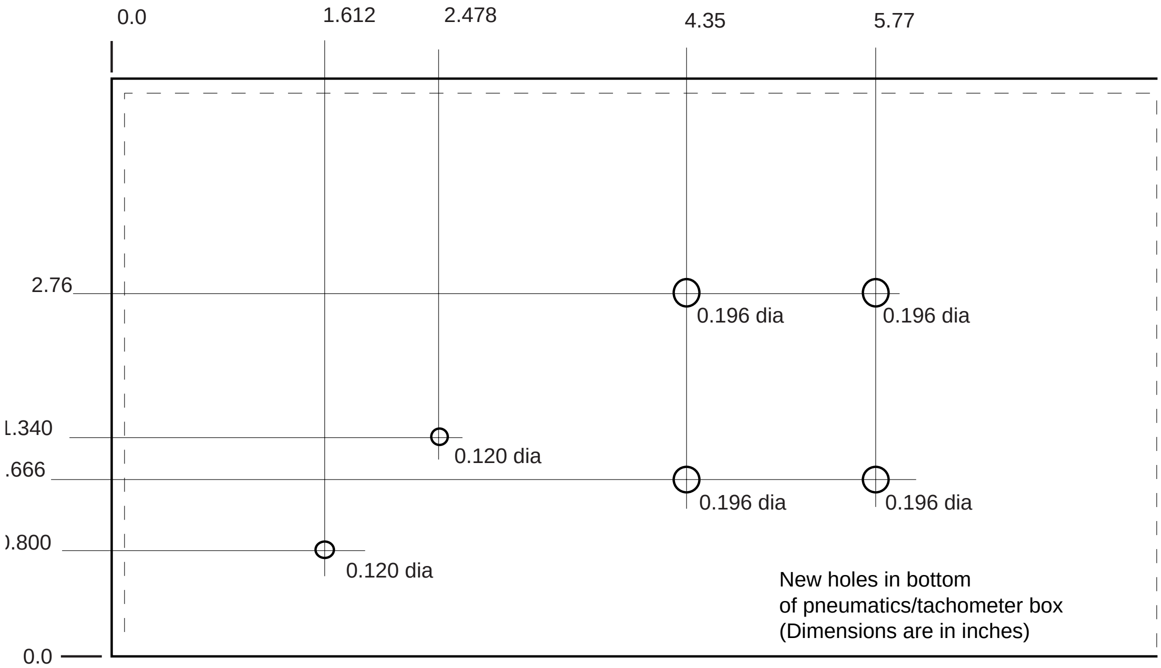


Figure 5. New Holes in Bottom of Pneumatics/Tachometer Box

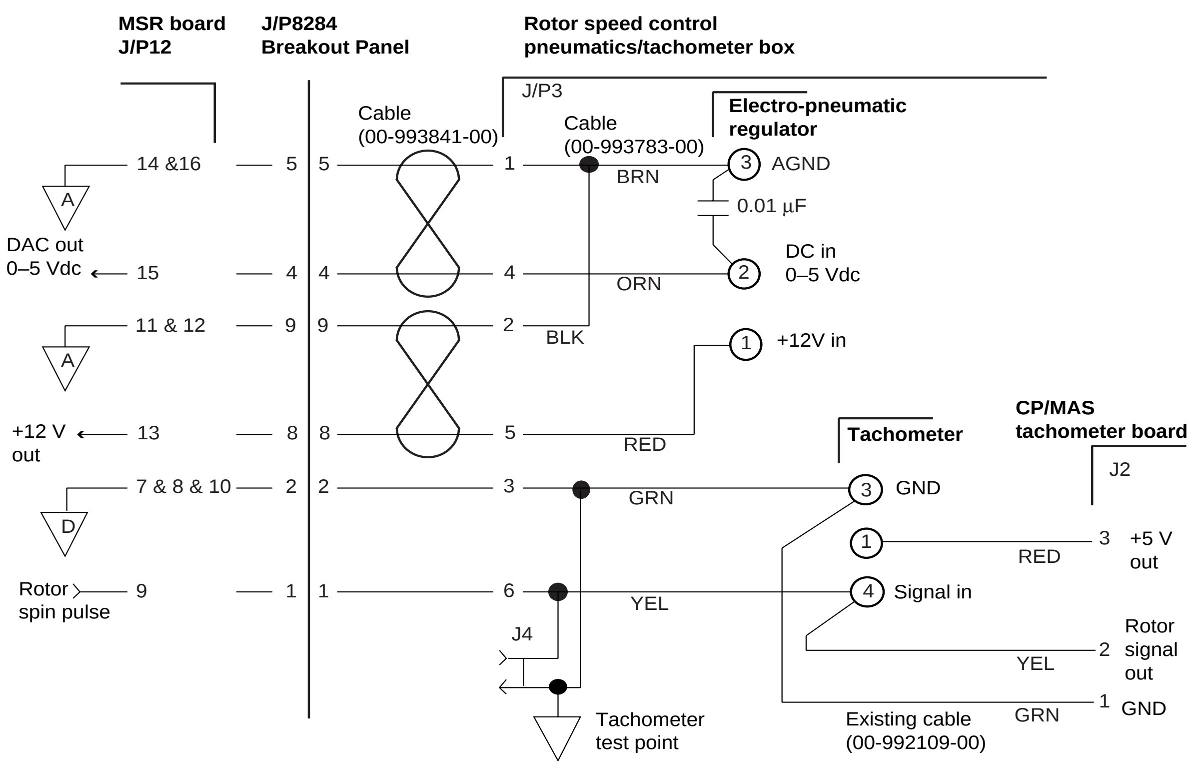


Figure 6. Rotor Speed Controller Interface Diagram

12. Install a 26-inch length of 3/8-inch O.D. tubing between the SUP port of the electro-pneumatic regulator and the top of the newly installed tubing tee (step 2 above) at the drive pressure regulator.
13. Install a 9-inch length of 1/4-inch O.D. tubing between the OUT port of the electro-pneumatic regulator and the Drive air. Refer to Figure 7 for pneumatic connections.
14. Disconnect and discard the 26-inch length of 1/4-inch O.D. tubing from the upper (outlet) fitting of the drive flowmeter (the upper one) and from the union at the filter outside of the pneumatic/tachometer box.
15. Install a 45-inch length of 1/4-inch O.D. tubing between the A port of the control valve and the drive filter outside of the pneumatic/tachometer box. Route the tubing through the grommet near the outlet of the drive flowmeter.
16. Install a 19-inch length of 1/4-inch O.D. tubing between the P port of the control valve and the elbow at the outlet of the drive flowmeter.
17. Check the installations of the wiring harness and tubing and fittings for accuracy. Reinstall the cover on the pneumatic/tachometer box and reconnect the box to the nitrogen supply and probe.
18. Reconnect the pneumatic/tachometer box to the nitrogen supply and probe. Refer to the procedure “To Install the Pneumatics/Tachometer Box” on page 21.

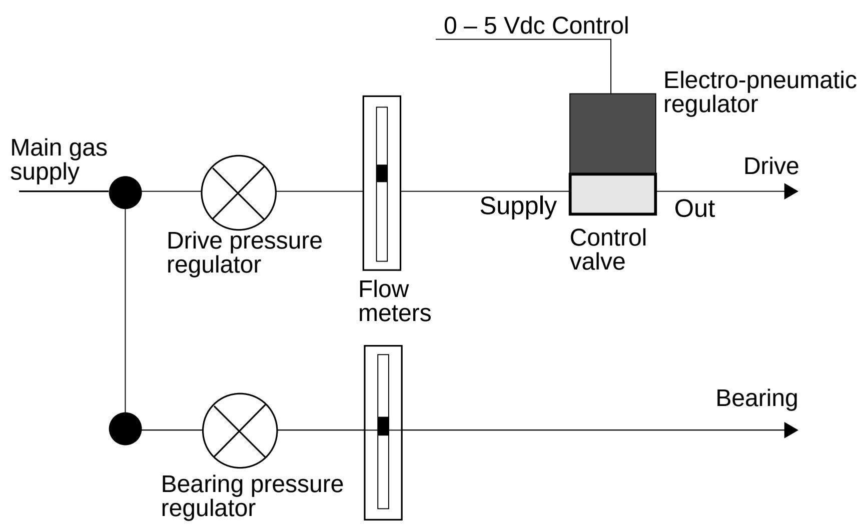


Figure 7. Rotor Controller Pneumatics Block Diagram

To Connect the Cables

This procedure describes how to connect the cables associated with the rotor speed controller accessory. Use Figure 8 as a guide during the following procedure.

1. Power down the UNITY*INOVA* console. Follow the shutdown procedures in the *User Guide: Solid-State NMR*.
2. Partially remove the MSR board from the digital card cage.
3. Plug the ribbon cable end of the cable into J12 on the MSR board.
4. Reseat the MSR board.
5. Run the D-shell end of the cable 01-903730-00 along the lower front of the digital card cage to the left cable access.
6. From the rear of the console, get the D-shell end and run it to the hole for J8242 to the left (from the back of the Breakout panel). Punch out the hole cover if necessary. Fasten the connector to the Breakout panel with the bolts provided.
7. Connect the cable 00-993841-00 to J3 of the pneumatic/tachometer box and to the newly installed J8242.
8. Turn on the power to the UNITY*INOVA* console.

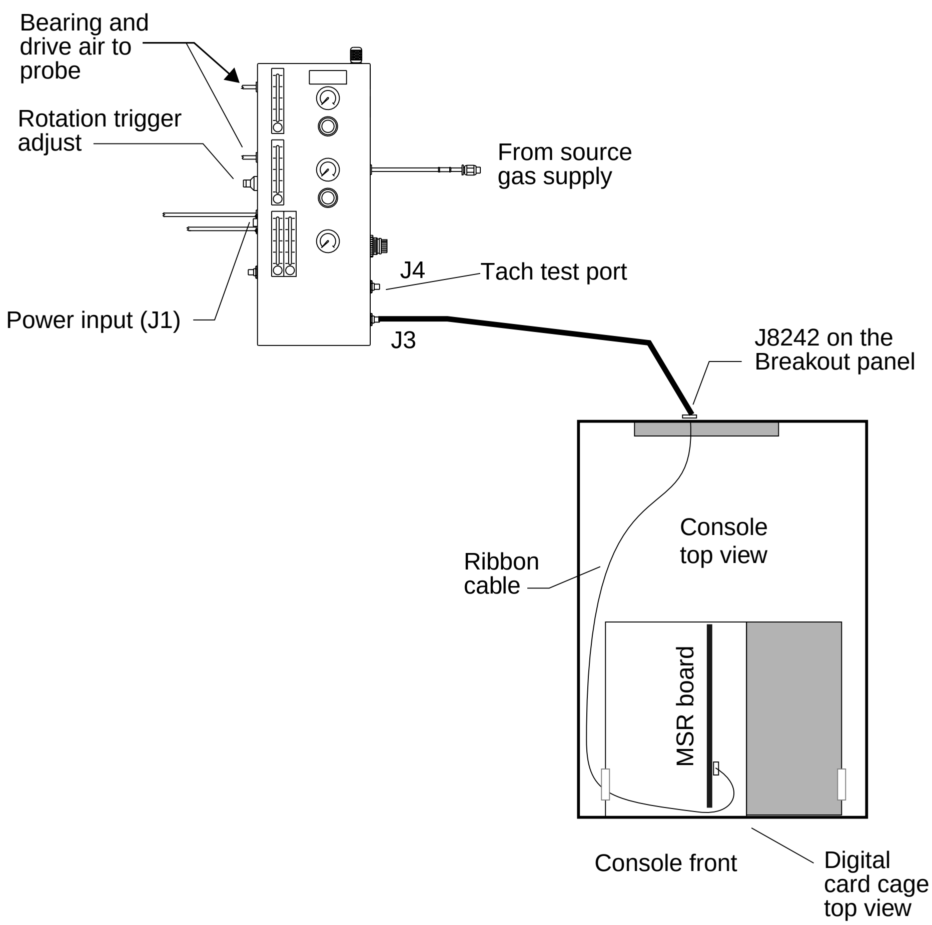


Figure 8. Rotor Controller Connection Diagram

2.3 Testing and Using the Rotor Speed Controller

This section provides instructions for testing and using the rotor speed controller. The rotor speed controller can be operated with spinning speed regulation (closed-loop mode) or with a specific airflow setting (open-loop mode).

Alternatively, the air flow can be set to a maximum (65535) and the drive-pressure regulator on the pneumatics/tachometer box can be used for manual control the spinning speed.

To Start the Rotor Speed Controller

1. Place a painted, good quality, empty, silicon nitride rotor in the CP/MAS probe.
2. Connect the bearing and drive air supplies of the pneumatics/tachometer box to the probe.
3. Connect the black fiber optic cable to the pneumatics/tachometer box (lower left panel) and set the toggle switch below to V.A. for the Varian CP/MAS probe.

   See “To Use Rotor Speed Control with a Doty Probe” on page 31, if a Doty probe is to be installed.
4. Adjust the bearing pressure to 30 psig.

   The rotor should begin to spin at about 200 Hz. If speeds greater than 5000 Hz will be desired, the bearing pressure will have to be set higher than 30 psig. Consult the *NMR Probes Installation* manual or the test data supplied with the probe to determine the proper bearing pressure for a particular spinning speed and probe.
5. Enter `spinner` in the VNMR input window to open the Spinner Control window (see Figure 9).
   a. Click on the High Speed Spinner (Solids Style) control button.
   c. Click the Set spinner airflow instead of speed, adjust the two slider bars to zero, and click the Turn Spinner Off button.

      The spinner is now in an unregulated or open loop mode in which the airflow is set directly.

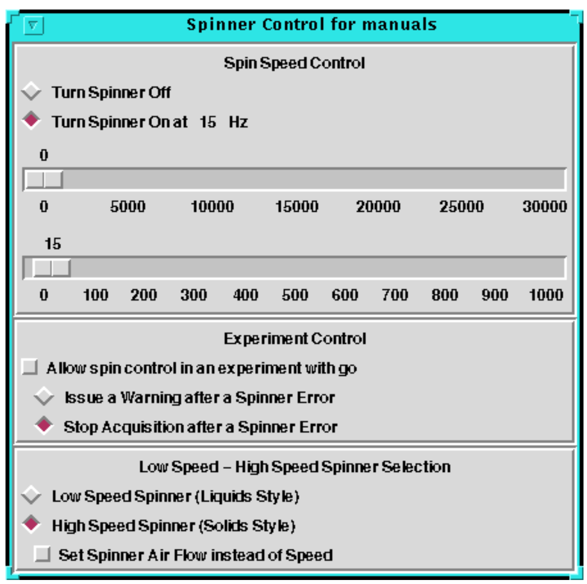


Figure 9. Spinner Control Window

6. Adjust the drive pressure *slowly* to a value of 60 or 70 psig maximum.

The rotor initially begins to spin and eventually reaches a speed of about 3000 to 6000 Hz at about 40 psig. The speed then drops to zero as the electro-pneumatic regulator engages.

7. Once engaged, continue to increase the pressure to the maximum value.

   If the regulator does not engage repeat step 5 and recheck the connections to the pneumatics/tachometer box. In any event, *do not let a rotor and end cap exceed their maximum rating during this process*. Steps 6 and 7 must be repeated anytime the drive-pressure regulator has been set to zero.

The rotor speed should be displayed on the pneumatics/tachometer box. If not, adjust Rotation Trigger Adjust on the lower left panel until the speed is displayed. If the speed cannot be observed, check the rotor painting and the fiber optic connections. Also, one can observe the tachometer pulses with an oscilloscope at J4 (see Figure 8 on page 28). The duty cycle should be about 50% but may vary if the rotor paint has worn thin.

To Set the Air Flow

This procedure describes how to set the air flow

1. Set the airflow to 1000 by moving the slider bar to the right. The air flow engages after several seconds.
2. Continue to increment the air flow setting in steps until the sample is spinning at about 2500 Hz.

   The fine slider bar can be incremented by one when the mouse pointer is clicked in the bar.

   The speed displayed in the Acquisition Status window is correct. The speed displayed on the pneumatics/tachometer box tends to be slower.

Move the slider bar slowly. If the rotor is spinning, changing the air flow in small steps is good practice. *Never set an air flow value that causes a speed that exceeds the rated speed of the rotor, cap and probe*. The air flow can also be set before setting the drive pressure. In this case the electro-pneumatic regulator engages at the specified airflow setting.

To Regulate Spinning Speed

If the rotor checks out, return the airflow setting to zero, click on Turn Spinner Off and click to disengage the button Set Spinner Airflow Instead of Speed. The spinner is now in regulation or closed-loop mode. Enter a spinner speed of 2500 on the slider bars. The spinner should regulate to 2Hz or better within about 30 seconds to 1 minute. Experiment with a number of set points within the rated speeds of the rotor, endcap and probe.

In closed-loop mode, the speed can be increased or decreased by any value. The speed is safely ramped to the new value. However, making small changes in speed (1000 to 2000 Hz) is good practice, until you are comfortable with the operation and reliability of a particular rotor and endcap combination. *Never exceed the rated speed of the rotor, cap, and probe.*

To Manually Control the Spinner

This procedure describes how to manually control the spinner with the drive-pressure regulator.

1. Start the rotor speed controller as described in the procedure "To Start the Rotor Speed Controller" on page 29.
2. Set the drive-pressure regulator to zero and move the slider bars fully to the right to set a value of 65535. *Slowly* adjust the spin speed to the desired value by increasing the drive-pressure regulator.

Under manual control or with an airflow setting the spinning speed will typically drop by about 100 to 300 Hz over five to ten minutes (due to heating of the bearings) and then stabilize.

To Regulate Spinning Speed within VNMR

This procedure describes how to regulate spinning speed with VNMR input window.

1. Start the rotor speed controller as described in the procedure "To Start the Rotor Speed Controller" on page 29.
2. Click on the button next to Allow spin control in experiment with go.

   This button disables the speed in the Spinner Control window and transfers spin rate control to the `spin` parameter in VNMR.
3. Set up a typical solids experiment, set the value of `spin` to the desired speed, and enter `go`.

   The spinner regulates at the value of `spin`. Include a preacquisition delay `pad` to give the spinner time to stabilize. The parameter `spin` can be included in an array to obtain multiple spin rates in a single experiment. In an array of `spin`, the preacquisition delay is applied before each FID.
4. After the experiment is complete, click next to Allow spin control in experiment with go to return control to the Spinner Control window.

To Use Rotor Speed Control with a Doty Probe

Using the rotor speed controller with a Doty probe is similar to that with a Varian probe. Consult the manual from Doty Scientific for the required bearing and drive pressures. Regulation of 2 Hz may or may not be attainable, but experience to date suggests that operation with a supersonic probe is similar to that with a Varian probe. Be sure to use a well-painted supersonic rotor. If in doubt, consult Varian for any specific recommendations and procedures before proceeding.

1. Connect the drive and bearing lines to the probe.
2. Connect the multi-pin cable between the probe and the small, blue spin-rate tachometer box that is supplied with the probe.
3. Connect a BNC cable between spin-rate tachometer box and the BNC connector on the lower left panel of the pneumatics/tachometer box.
4. Set the toggle switch on the pneumatics/tachometer box to one of the two EXT connections.

5. Set the toggle switch on the spin-rate tachometer box to optical detection.

To Calibrate the Rotor Speed Controller

The electro-pneumatic regulator has two set screws, accessible from the sliding window, for adjusting the zero and the span of the pressure output. The zero of electro-pneumatic regulator may need calibration if the rotor continues to spin (at an airflow setting of zero) with zero bearing pressure. A small amount of bearing air is shunted to the drive stream in the stator, causing a minimum speed that depends on the bearing pressure. However, the drive should provide no airflow when set to zero. Also, it may be necessary to adjust the span if the maximum rotor speed can not be obtained, or to limit the maximum speed. Use the following steps for the calibration:

Repeating this procedure several times may be necessary.

1. Use a painted, good quality, empty, silicon nitride rotor. Install the probe and rotor and set the airflow to zero.
2. With a bearing pressure of 30 psig, adjust the zero set screw for a minimum sample spinning rate (usually about 200 Hz).

   The electro-pneumatic regulator responds slowly so allow time for settling after each change.
3. After the zero is adjusted, briefly turn the bearing-pressure regulator to zero with the drive pressure engaged. The speed should fall to zero. If it does not, quickly return the bearing pressure to 30 psig and repeat the adjustment. *Do not leave the rotor spinning with no bearing pressure.*
4. To adjust the span, while the rotor is spinning, increase the airflow setting in a series of steps of 5000 or less until either the maximum airflow setting of 65535 is obtained or until the maximum rated speed of the probe is obtained.
5. Adjust the span set screw to obtain the maximum spinning speed with an airflow setting of 65535.

   Do not exceed the maximum rated speed for rotor, caps and probe. The electro-pneumatic regulator responds slowly so allow time for settling after each change.

If the maximum speed can not be obtained, be sure the drive pressure is 70 psig. It may be necessary to increase the supply pressure to the pneumatics/tachometer box to achieve this. If maximum speed can not be obtained at 70 psig, the rotor or stator may have a problem—repeat with a new rotor. If necessary, use manual control to compare the drive-pressure dependence of the spinning speed with the test data supplied with the probe.

To Change Rotors

This procedure describes how to change a rotor.

1. Set either the airflow or the rotor speed to zero and click Turn Spinner Off. *Do not* lower the drive-pressure regulator on the pneumatics/tachometer box. The sample will remain spinning at about 200 Hz.

2. Turn the bearing-pressure regulator on the pneumatics/tachometer box to zero, and the rotor should now stop spinning. If the rotor remains spinning, repeat step 1. Set a 0 in open-loop mode if necessary.
3. Lower the probe into its stand. The rotor can be removed with a small loop of tape about the index finger.
4. Place the new rotor and the probe in the magnet. Return the bearing pressure to 30 psig (or the desired setting) and continue.

Chapter 3. Wideband NMR Module

Install the Wideband NMR Module (01-903007-00) according to the following procedure. The Wideband NMR Module is a board that includes two 5-MHz 12-bit ADCs and 2 MB of onboard memory.

CAUTION: **The following installation involves handling static sensitive equipment—PROMs and printed circuit boards. Take all precautions necessary to suppress electrostatic spikes and discharges near the devices: stand on antistatic pads, wear natural fiber materials, and use a grounded antistatic wristband before touching any equipment, and do not place other boards or paper on top of boards and so on. Failure to suppress electrostatic discharges can result in permanent damage to components.**

3.1 Installing the Wideband NMR Module

1. Make sure the power is off in both the digital and rf card cages.
2. Install the Wideband NMR Module in the digital card cage in the slot to the right of the 500-kHz ADC board.

   Use the usual antistatic precautions and be careful that the small surface-mount components on the solder side of the board do not catch on 500-kHz ADC board front panel.
3. Route the two BNC cables (00-958298-10 provided in the kit) through the cable path on the left of the rf card cage and through the right of the digital card cage. Connect the cables as follows:
   - Connect one cable from J1 on the Wideband NMR Module to WB CH A (J246) on the Observe Receiver module.
   - Connect the other cable from J2 on the Solids ADC to WB CH B (J247) on the Observe Receiver module.
4. Make the following connections:
   a. Remove the CTC cable from the DAC board and connect the SMB Tee (58-180018-00) in its place on the DAC board.
   b. Connect the CTC cable to one side of the SMB Tee.
   c. Connect cable (00-992897-04) between the other side of the SMB Tee and J5 on Solids ADC board.
5. Turn the power back on to the digital and rf card cages.
6. For VNMR 5.2F only, install the software patch as follows:

a. Log in a `vnmr1` and insert the software patch CD-ROM.

b. Open File Manager and click on the File Manager window for the CD-ROM.

c. Double click on the `loadpatch` icon. Read the instructions associated with the installation. The installation may take several minutes.

An alternative to using File Manager is to use a Shell Tool. Open a Shell Tool, change to the `/cdrom` directory and enter `./loadpath`.

7. Configure VNMR (CONFIG) to have the maximum spectral width of 5 MHz.
8. Go to the next section to test the Wideband NMR Module.

3.2 Testing the Wideband NMR Module

For the following tests disconnect the probe from the preamplifier and terminate the port on the preamplifier with a 50 ohm load. Set `tpwr=0.0` and `pw=0` to avoid applying power to the load.

500 kHz ADC Test

Recall an `s2pul` parameter set. Enter `nt=1 sw=100 np=8k tpwr=0 pw=0 go`.

If the run terminates with an error "`np not equal to number of points`" check the cable connections that were just made.

Wideband Module Test

Enter `sw=5e6 np=8k go`. If no errors occur, enter `wft` and inspect the spectrum.

The noise pattern should be approximately symmetric and contain no spurious signals. The noise level at 2.5e6 Hz should be about 1/3 that at the center

If errors occur check the cable connections. If no errors or spurious signals occur the installation is complete.

Data Transfer Test

The wideband module makes it possible to acquire data faster than the computer can store it. A fifo underflow will then occur. VNMR 5.3 and later software warns when this might happen and suggests a `d0` or `d1` value that will avoid the problem.

For a 1D acquisition with `bs='n'`, data transfer from the Wideline NMR Module to the Sun occurs at the end of the run. Acquisition of data with the maximum `np` (256k for the beta boards) and `d1=0` is possible.

For UNITY*INOVA* when `bs>0`, during arrayed or 2D operation or during real-time display of acquisition, the data-transfer between the Wideband NMR Module and the Sun takes place during the `d1` delay period. The minimum value of `d1` (required to complete the transfer) is determined by the number of points, `np`, and the speed of data-transfer, which depends on the amount of memory on the Wideline NMR Module. Incomplete transfer causes a fifo-underflow error. The table below shows typical values of `d1` versus `np` for the current beta boards (1 MB of memory). Released boards will have 2

MB. They will run with faster data-transfer rates and trap for most fifo-underflow conditions.

np	*bs*	*d1(s)*
>64k	>0, acqi, 2D, or array	operation unreliable
64k	>0, acqi, 2D, or array	0.4
32k	>0, acqi, 2D, or array	0.2
16k	>0, acqi, 2D, or array	0.1
8k	>0, acqi, 2D, or array	0.05

Operation with a shorter `d1` may cause an error (fifo underflow) and halt the acquisition. If an underflow error occurs, decrease `np` or increase `d1`. Run with `bs='n'` if possible. Once a fifo underflow error has been generated it is sometimes necessary to run one scan successfully with a small number of points (say 2k) before continuing.

3.3 Pulse Sequences Proton Multipulse NMR

The pulse sequence source files `flipflop.c`, `br24.c`, `cylbr24.c`, `mrev8.`c, and `cylmrev8.c` must be modified to function with UNITY*INOVA* and VNMR 5.2F.

For UNITY*INOVA*, the receiver is off by default (it is on for UNITY*plus*). When using explicit acquisition, the receiver is turned on with the beginning of the `'acquire'` statement. For CRAMPS, this is too late—the receiver must be turned on at the beginning of the delay `'dtau'`.

Place `rcvron()` before the `delay(dtau)` or `delay(dtau + tau)` and before `rcvroff()` after the acquire statement in each sequence.

Chapter 4. CP/MAS Solids Module Installation

The CP/MAS solids module enables Varian NMR spectrometers to run CP/MAS and MAS experiments.

This chapter covers the installation of the CP/MAS amplifier on the NMR spectrometer. The two sections of this chapter are the following:

- Installing the CP/MAS solids module
- Testing the CP/MAS solids module

4.1 Installing the CP/MAS Solids Module

The steps below cover installing the solids decoupler pulse amplifier and connecting the cables. Note that 400-MHz systems do not use the Solids Control Box.

1. Enter `svs(`*`file`*`)`, where *`file`* is the name of the file to save existing shim settings.
2. Power down the system using the standard procedure.
3. Turn off the power to the console and remove the power cable from its socket.
4. Remove the rear panel and vent outlet from the top AMT linear amplifier in the rear of the console. See Figure 11 on page 46 for the location.
5. Disconnect all cables—two coax input cables, two N-type output cables, the 25-pin D-type connector, and the power cord. If coaxial attenuators are connected to the input of the amplifier, leave them attached to the cables.
6. Remove the 4 screws from the front of the top AMT linear amplifier and slide the amplifier out of the cabinet. **Be careful because the amplifier is heavy**.
7. Install the 100-W AMT amplifier in the top slot:
   a. Remove the slides and shims from the 50 W AMT amplfier(3900-12) and attach them to the 100 W AMT (3900-15).
   b. Slide the 100 W AMT amplifier (00-993200-00) into the top slot.
   c. Secure the front panel of the amplifier to the front rails with the original screws.
8. Reattach the cables removed in step 5 to the back of the 100 W AMT amplifier and turn the amplifier on.
9. Remount the rear panel and vent duct in the rear of the cabinet.
10. Close the front cover, reconnect the power, and turn the system power on. Start the data system as described in the *User Guide: Solid-State NMR*.

11. Recall shim values by entering `rts(file) su`, where `file` is the name of the file containing the shim values you saved in step 1.

Refer to the *User Guide: Solid-State NMR* for solids operating instructions and sample preparation procedures. Refer to the manual *NMR Probes Installation* for information specific to CP/MAS probes.

The fine attenuators are controlled by `dpwrf` or, in the XPOLAR pulse sequence by, `level1f` and `level2f`, which range from 0 too 4095 (where 4095 is the maximum). The parameter `dpwr` has a maximum value set at 49; `level1` and `level2` have maximum values of 63. The observe fine attenuator is controlled by `tpwrf` in the range 0 to 4095. The maximum value for `dpwr` can be set in the `config` program, using the "Attenuator" and "Upper Limit" labels.

You may have to create the parameters for fine control; for example,
`create(parameter_name,'integer')`
`setlimit(parameter_name,4095,0,1)`. `tpwrf` can be created similarly.

4.2 Testing the CP/MAS Solids System

The CP/MAS Solids Module for Varian NMR spectrometers is used to acquire NMR data from solid samples. The sample, usually a powder, is packed into a hollow rotor. The rotor is placed in the probe at the "magic angle" of 54.7 from the magnetic field and spun at a variety of speeds of up to 24 kHz. High-speed spinning at the magic angle, coupled with high power decoupling, narrows the broad lines that are characteristic of solid samples (typically 20 kHz) to about 1 ppm.

CAUTION: **Read this before doing any tests:**

1. **Run all tests with the XPOLAR pulse sequence and `dm='nny'` to provide some protection to the probe from excessive decoupler duty cycle.**
2. **Do not turn on the 100 W amplifier unless the output is connected to a properly tuned probe (with a high-pass filter at J6003), to a large 50-ohm load, or to a large 30-dB attenuator terminated in 50 ohms.**

Refer to the manual *NMR Probes Installation* for tuning the decoupler and observe channels on the probe. The probe must be installed and tuned before you can run a CP/MAS solids experiment.

WARNING: **Dangerous high rf voltages exit that can cause serious injury or death. Avoid electric shock by completely turning off the rf and disconnecting rf cables before removing the probe.**

Testing the Amplifier

Typical power outputs are given as a guide to troubleshooting. Pulse droop should be recorded on all systems, but accurate power measurements are only needed if the system fails to make pulse width or γB_2 specifications. Power measurements should be made through a 30-dB, 100-W attenuator on an oscilloscope or spectrum analyzer. A spectrum analyzer provides more accurate power measurements.

1. Check the observe amplifier (Channel B) as follows:
   a. Enter `rt('/vnmr/tests/hmb')` to recall parameters.

b. Enter `xpol='y' p2=1e4 at=0.01 d1=.05 nt=1e6 level1=63 level1f=4095` to set up for rapid pulsing.

c. Attach the observe cable from the magnet leg to a large 30-dB attenuator. Attach the output of the attenuator to a oscilloscope or spectrum analyzer.

d. Set the decoupler to OFF by entering `dm='nnn' tpwr=63 tpwrf=4095 go`; then measure the power and droop of observe pulse amplifier. It should meet the following values:

- Minimum power: 300 W, 220 Vpp, +54 dBm (6.3 Vpp, when measured through a 30-dB attenuator).
- Maximum droop: 2% over 10 ms.

e. Enter `aa` to stop the acquisition and then reattach the observe cable to the probe.

2. Check the AMT decoupler amplifier (channel A) according to the following steps. The maximum drive to the AMT should not exceed +2 dBm.

   a. Disconnect the input to Channel A of the AMT amplifier from the ATTN/SW board and then connect OUT 1 to a power meter.

   b. Set `level2=63 level2f=4095 nt=1e6 dm='nny' sw=1e5 np=128 d1=0.1`

   c. Enter `go` and add sufficient in-line attenuation at the input of the AMT amplifier so that the peak power does not exceed 2 mW (+3 dBm).

   d. Enter `aa` to stop the acquisition, `dm='n' su` to reset the power, and then reconnect the AMT amplifier.

Adjusting Decoupler Field Strength

1. Fill a rotor with dioxane (00-957734-01) and cap it with a Torlon end cap (brown). Note that any other type of end cap will dissolve in the dioxane.

WARNING: **The 1,4-dioxane sample (00-957734-01) is a cancer suspect agent containing flammable liquid oxidizers. See the enclosed MSDS for additional information and handling precautions.**

2. Spin the sample slowly (200–300 Hz).

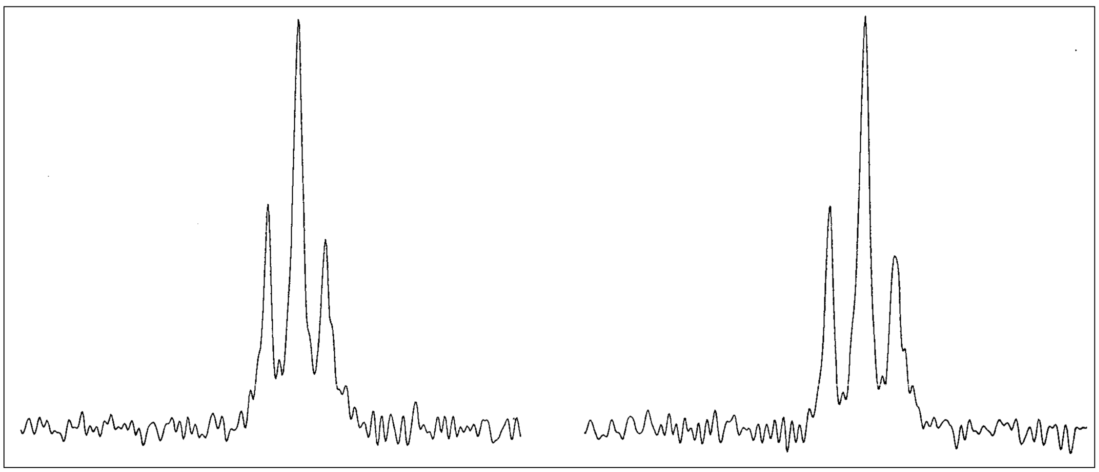
3. Enter `rtp('/vnmr/tests/hmb') seqlib='xpolar'`. Use XPOLAR with `xpol='n'`.

CAUTION: **High spinning speeds can cause the rotor to shatter, creating a potential physical hazard to the operator. Never spin 7-mm PSZ (zirconia) rotors (white or off-white in color) above 7.2 kHz or silicon nitride rotors (gray) above 9.5 kHz.**

Decoupler power greater than 2 W will damage a switchable probe. The maximum value for `dpwr` on a 200, 300, or 400 MHz system with a linear amplifier on the decoupler channel has been set to 49, which corresponds to approximately 2 W of power. For this reason, do not use or alter `dpwr` for solids operation. Use the XPOLAR pulse sequence rather than S2PUL.

Follow all pulse duration, pulse power and duty cycle warnings if given for a particular probe. Generally, pulse durations can be increased with a proportional decrease in pulse power.

4. Tune the probe according to the instructions in the manual *NMR Probes Installation*. Tune the decoupler channel before the observe channel and with a high-pass filter in place for a return ratio of 50 dB or better.
5. Check that `dm=nny`, `level2=63`, and `level2f=4095`.
6. Record a pair of ^{13}C spectra by entering `dof=50000, -50000 ga` (see Figure 10).



```
 ACQUISITION          SAMPLE               PROCESSING          FLAGS
sfrq   100.57      date     April 1     lb          10.00     il             n
tn        C13      file         exp     sb       not used     toss           n
at      0.100      DECOUPLING           gf       not used     pdp            n
np       6016      dn            H1     awc      not used     dp             y
sw     3000.0      dof      arrayed     lsfid    not used     SPECIAL
xpol        n      dm           nny     phfid    not used     srate        415
tpwr       55      dmm            c     wtfile                temp    not used
tpwrf      51      dpwr          63     proc           ft
pw        8.0      level1        57     fn          32768
p1          0      level1f      200
d1     10.000      level2        63
tof         0      level2f     4095     werr
nt          1                           wexp
ct          1                           wbs
                                        wnt
                                           FLAGS
                                        ai no ph


                   Gamma-H2 calculation
                   See J. Magnetic Resonance 7:442 (1972)
   jo =  284.00 Hz,   dof[1] =  -50000.00 Hz,   dof[2] =  50000.00 Hz
jr[1] =  165.00 Hz,    jr[2] =  167.00 Hz
Gamma-H2          =      69404.97 Hz
90 degree pulse =           3.6 usec
coalescence frequency =     -457.51 Hz
```

Figure 10. Decoupler Field γB_2

7. Measure the residual splitting of the dioxane triplet. Use `h2cal` to calculate γB_2. Minimum values are shown in Table 1.

Table 1. Maximum γB_2 Values

Magnet	*Varian RT Probe, 7mm*	*Varian VT Probe, 7mm*	*Doty Probe, 7mm HS*	*Other Probes*
200 MHz	75 kHz	—	60 kHz	See individual specs
300 MHz	75 kHz	80 kHz	60 kHz	
400 MHz	60 kHz	70 kHz	55 kHz	

Set `level2` to achieve a γB_2 as close to these minimum values as possible. Do not exceed this power level for any other experiment. Note this value.

8. If the probe does not make specification, check for rf losses in the decoupler circuitry going to the probe.

Chapter 5. Solids Cabinet Preparation and Installation

This chapter describes preparation and installation procedures for the solids cabinet and installation procedures for the Solids Relay Driver board.

5.1 Preparing to Install the Solids Cabinet

Before starting to install the solids cabinet, make sure you have the correct power supply, tools, and sufficient space for the installation. Refer to Table 2 for weights and dimensions and to the UNITY*INOVA Installation Planning Guide* for additional planning details, such as air conditioning requirements.

1. The solids cabinet contains Class A/AB rf amplifiers requiring a high-current power source. Verify that the main power supply is 240 Vac single-phase, 30 A dedicated line, separate from the 20 A power line used by the UNITY*INOVA* console. Figure 11 illustrates the solids cabinet with the front panel removed.
2. The following tools and equipment are needed to perform the upgrade and performance testing. Collect these before starting.
   - Dual-trace oscilloscope capable of accurate voltage measurements from dc to 400 MHz
   - 500 W, 30 dB attenuator
   - 2 W rotary attenuator, 0 to 100 dB in 10 dB and 1 dB steps
   - Spectrum analyzer
   - Complete Varian field service engineer tool kit
3. Check the items in the shipping kit against the parts list. If all the parts on the list are included in the kit, proceed to the next step. If anything is missing, obtain it before proceeding.

Table 2. Dimensions of Complete Solids Cabinet and UNITY*INOVA* Cabinet

Component	*Height*	*Width*	*Depth*	*Weight*
Complete Solids Cabinet	142.3 cm 56 in.	55.3 cm 21.8 in.	78 cm 30.7 in.	< 272 kg < 600 lb
UNITY*INOVA* Standard Cabinet	124.5 cm 49 in.	110.7 cm 43.6 in.	78 cm 30.7 in.	~ 272 kg ~ 600 lb

WARNING: **Remember that during the installation the magnet is up to field. Equipment and tools used by the moving crew must be evaluated for safety. Install a plastic chain (or chain of other nonmagnetic material) at about 42 in. (107 cm) from the magnet body. This will serve as a reminder to prevent magnetic materials (e.g. , consoles) from being pulled into the magnet body.**

4. Verify that there is enough room to accommodate the solids cabinet on the floor plan of the laboratory by following the guidelines below:

   a. Measure the distance between the edge of the UNITY*INOVA* console and the centerline of the magnet.

      If this distance is greater than the distance indicated in Table 3 for the specific system, go on to step 5:

   b. If the distance is less than the distance indicated above, determine how much distance must be added to accommodate the solids cabinet:

______________	–	______________	=	______________
distance from above table		actual distance		additional distance needed

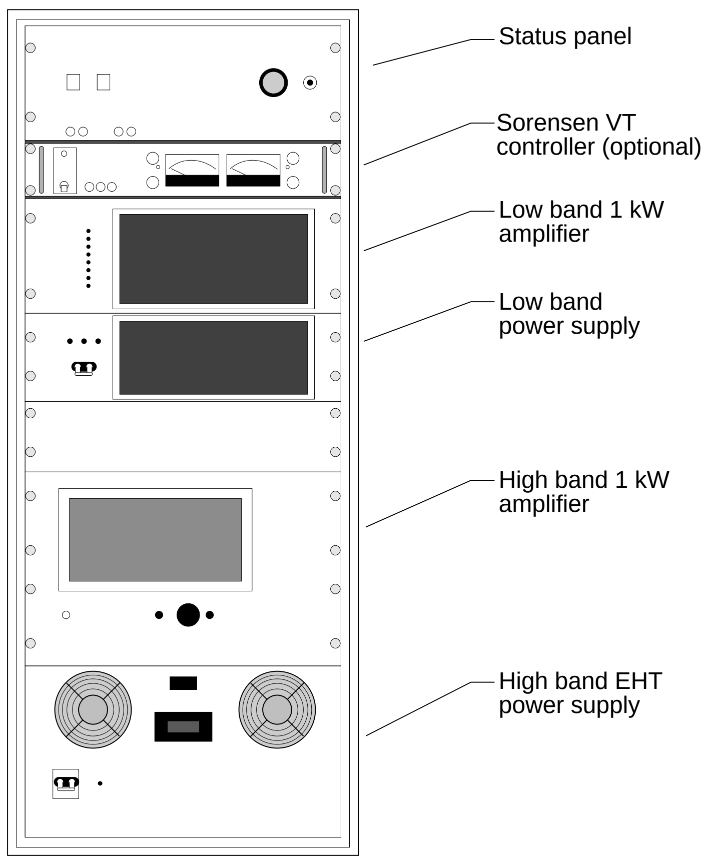


Figure 11. Solids Cabinet Open Front View

Table 3. Minimum Distances of UNITY*INOVA* Console from Magnet Centerline

System (MHz)	*Distance*
300/51	170 cm (67 in.)
300/89	185 cm (73 in.)
400/54	185 cm (73 in.)
400/89	185 cm (73 in.)

c. Place the acquisition console at the correct distance. Disconnect all cables from the magnet leg and move the cables away from the right side of the console.

5. Shut down the data system using the steps below:

a. Exit VNMR by entering `svs(`*`file`*`)`, where *`file`* is the name of the shim file. Then enter `exit`.

CAUTION: **Shut down the system according to the instructions provided in the manual *User Guide: Solid-State NMR*. Failure to shut down the system properly can corrupt system files.**

b. Shut down VNMR and UNIX system according to instructions in the *User Guide: Solid-State NMR.*.

c. After VNMR and UNIX are shut down, turn off the power switch located at the rear, center of the acquisition console.

d. Remove the power connector from the rear of the acquisition console.

6. For convenience, remove the back panel of the acquisition console, which covers the Relay Driver board as shown in Figure 12 on page 48. Set the back panel aside until the modification is complete.

7. Carefully push the solids cabinet up towards the right side of the UNITY*INOVA* console (looking from the front) so that there is 6 in. (15 cm) between them and both front panels face the same directions. The minimum distance between the edge of the solids cabinet and the magnet must be at least 42 in. (107 cm) for 300 MHz magnets and 48 in. (122 cm) for a 400 MHz or any widebore (89 mm) magnet.

5.2 Installing the Solids Relay Driver Board

This section describes replacing the Relay Driver board (00-992962-00) with the Solids Relay Driver board (00-992962-01). The solids cabinet is connected to the UNITY*INOVA* console through the Solids Relay Driver board, which is located in the rear of the UNITY*INOVA* console, behind the rf control card cage. The illustrations in Figure 12 shows the location of the Relay Driver board as well as the front and rear layouts of the two UNITY*INOVA* console cabinets.

Before you begin, shut down VNMR and power down the system as described in the manual *User Guide: Solid-State NMR*.

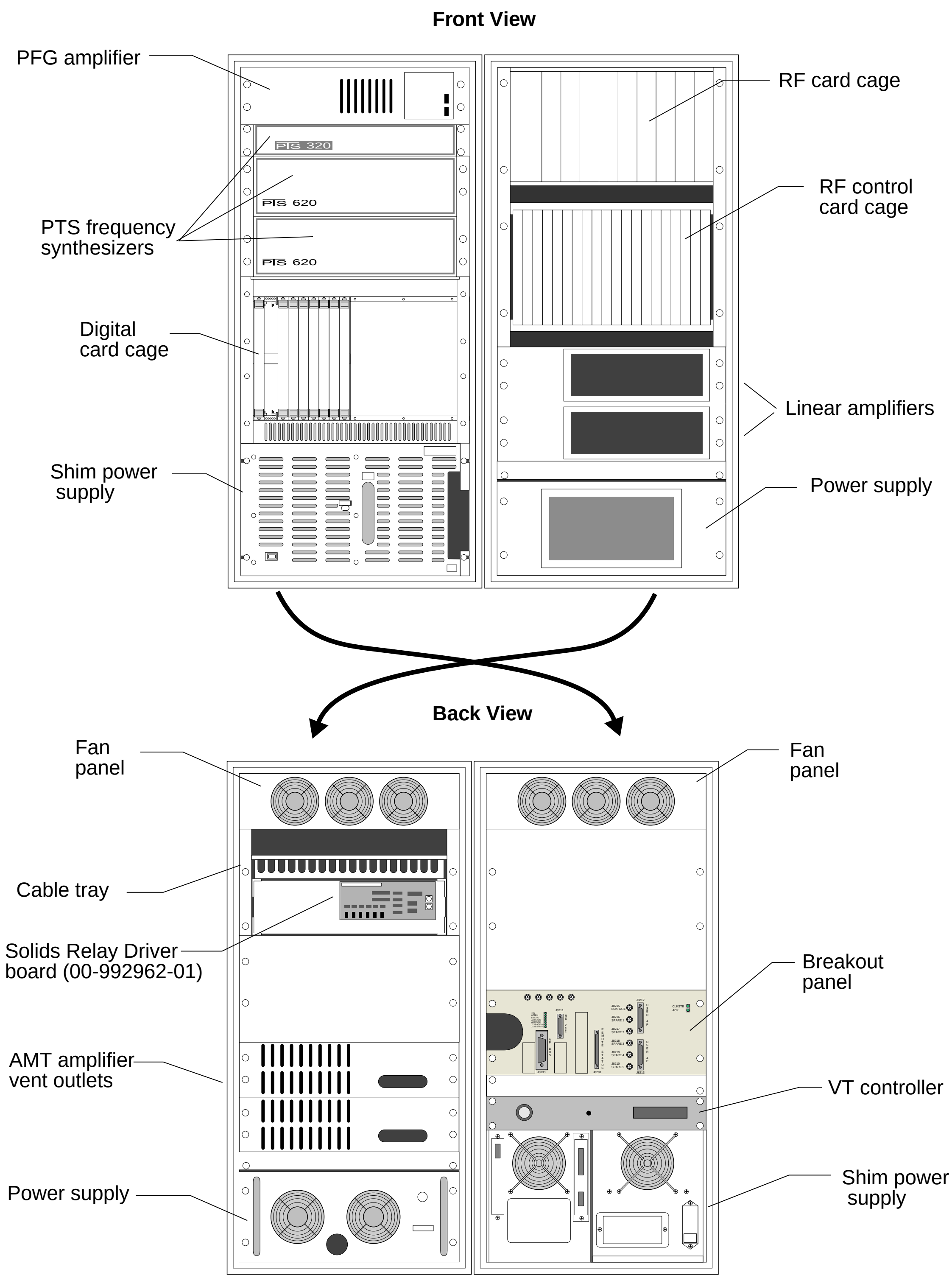


Figure 12. UNITY*INOVA* Spectrometer, Front and Back Views

1. If not already done, remove the appropriate panel at the rear of the UNITY*INOVA* console to gain access to the Relay Driver board, see Figure 12.
2. Disconnect the ribbon cable at J7001. Disconnect the power cable at J7004. Disconnect the tune relay cable at J7301
3. Remove the Relay Driver board (00-992962-00) by removing the four screws that hold the board in place.
4. Install the Solids Relay Driver board (00-992962-01) by inserting the four screws removed step 3.
5. Connect the ribbon cable at J7001 and connect the power cable at J7004. Reconnect the tune relay cable at J7301.
6. Connect one end of the second power cable (00-993140-00) to J7003 on the Solids Relay Driver board. Connect the other end to the +5 V rail (upper, labeled TBxx) and GND rail (lower, labeled TB2x) on the control card cage.

5.3 Installing the Solids Cabinet

The following steps describe how to install the solids cabinet. Figure 13 illustrates the open back of the solids cabinet and the relay panel. The table shows which steps to follow for each solids module.

When Installing	*Do These Steps*
Wideline	1 through 5
CRAMPS/Multipulse	5 through 10
Complete solids	1 through 10

1. Locate the cable connected between J4072 (liquids AMT channel B out) and K5004-2 (preamplifier housing). Remove this cable.
2. Locate the cable (00-990626-16) connected to K7200-C in the solids cabinet. Connect the other end to J4072 (liquids AMT channel B out).
3. Locate the cable (00-990626-17) connected to K7202-C in the solids cabinet Connect the other end to K5004-2 in the preamplifier housing.
4. Connect J7022 on the Solids Relay Driver board to J26 on the AMT 1 kW amplifier (RF Blanking, 00-958297-16).
5. Connect harness (00-990729-00) between the UNITY*INOVA* console and the solids cabinet as follows:

   Standard cabinet: Solids Relay Driver board: J7009, J7011, J7030
   Solids Status board: J7101, J7103, J7104, J7106, J7107, J7108, J7109, J7110

   Solids cabinet: Solid relay panel: J7206
   1 kW high-band amplifier: J7703

6. Connect J7023 on the Solids Relay Driver board to J7714 on the 1 kW Hi band amplifier (RF Blanking, 00-958298-16).
7. Locate the cable connected between J4073 (liquids AMT channel A out) and K5004-3 (preamplifier housing). Remove this cable.

8. Locate the cable (00-990626-16) connected to K7201-C in the solids cabinet. Connect the other end to J4073 (liquids AMT channel A out).
9. Locate the cable (00-990626-17) connected to K7203-C in the solids cabinet. Connect the other end to K5004-3 in the preamplifier housing.
10. Connect the solids cabinet to the mains (208, 220, or 240 Vac 30A). Do not switch the instrument on at this time. Wait until the hardware installation is completed.

5.4 Setting the AMT Amplifier Main Supply

The facility voltage entering the amplifier can be set to 230 or 208 Vac. Selection is made by connecting a jumper on the M3000 module at the appropriate taps. This is illustrated in Figure 14.

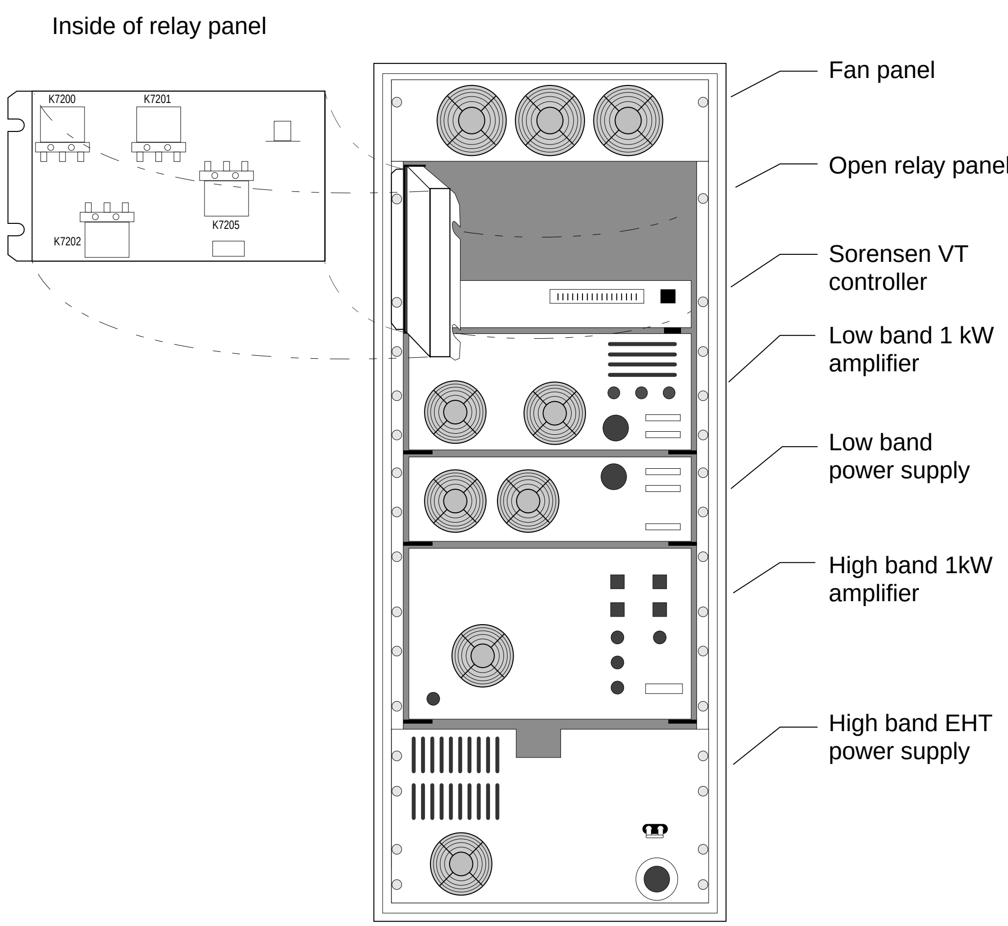


Figure 13. Solids Cabinet, Open Back View and the Relay Panel

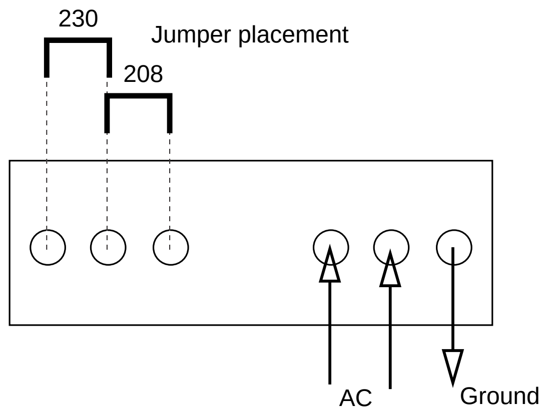


Figure 14. AC Input at Rear of AMT M3000 Amplifier

Chapter 6. **Wideline Module Tests**

This chapter contains test procedures for the wideline module. The tests are broken into two groups as follows:

RF transmitter performance tests:

- Liquids observe transmitter rf signal path
- Solids observe transmitter rf signal path
- Decoupler transmitter performance
- Solids cabinet status light and safety switch performance

NMR tests:

- Wideline solids test sample kit
- Deuterium 90 pulse and system sensitivity measurement
- Sodium 90 pulse and sensitivity measurement
- System recovery time
- Wideline spectral appearance
- Liquids performance

CAUTION: **Read the following cautions:**

- **Make sure that all amplifiers are terminated in appropriate 50-ohm loads at all times.**
- **The high power available from the amplifier in the solids cabinet is intended only for solids experiments. Applying this power to a liquids probe will destroy the probe. Before turning on the amplifier in the solids cabinet, check that the appropriate solids probe has been put in place.**
- **Always disable all high-power solids amplifiers before installing any probe.**
- **Confirm that the rf signal being measured can never exceed the maximum input signal of the measuring device.**
- **Do not block any of the ventilation holes in the solids cabinet. Even on standby, the amplifiers radiate considerable heat.**

6.1 Testing RF Transmitter Performance

This series of tests evaluates the solids cabinet relay and attenuator operation as well as the 1 kW amplifier power output and performance.

Liquids Observe Transmitter RF Signal Path

1. Recall the parameters: `rtp('/vnmr/stdpar/H2')`.
2. Enter the following parameters to observe the transmitter pulse along the transmitter pathway: `nt=1e5 pw=100 dm='n' rof1=30 rof2=0.2 alfa=0 sw=1e5 at=0.001 tpwr=63`.
3. On the solids cabinet control panel (see Figure 15), set the LO BAND switch to HI POWER. Enter `go`.
4. Confirm that the transmitted signal level at J25 rf input on the AMT M3201 is 54 dBm or less.
5. Halt the acquisition with `aa` and replace the cables.

Testing the Solids Observe Transmitter RF Signal Path

1. The broadband amplifier requires no tuning but must be properly terminated by a 50-ohm load at all times. To verify its operation, switch the LO BAND to HI POWER and observe the rf using a 500-W, 30-dB attenuator and additional attenuation as required. Check the output at the magnet observe port as described in steps 5 and 6 below.
2. Confirm that the blanking gate cable is connected as described below for the appropriate amplifier and required gate logic.

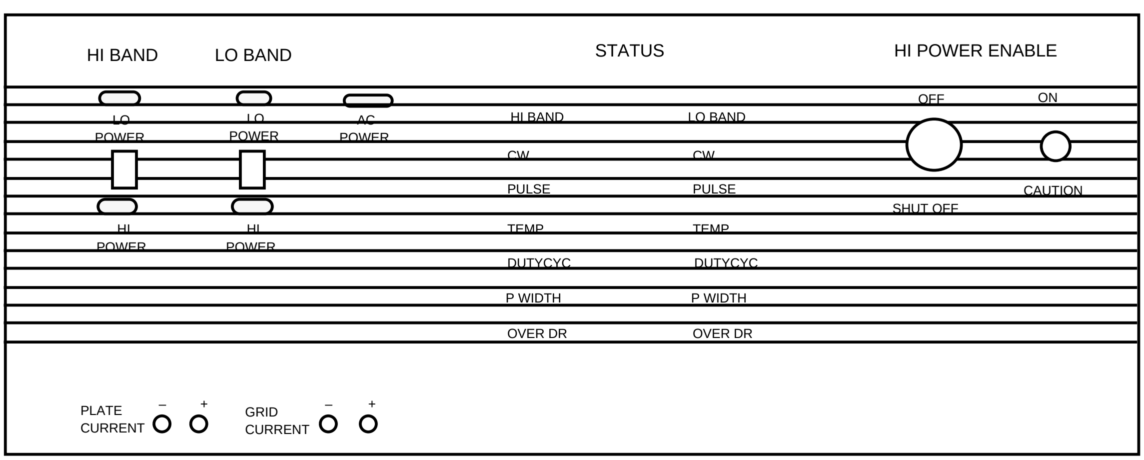


Figure 15. High-Power Amplifier Control Panel on Solids Cabinet

3. Turn on the AMT amplifier at the front panel and check that all interlocks go out. Only the light labeled POWER should be lit. If the status lights labeled PULSE WIDTH and/or DUTY CYCLE are lit, move jumper J2 on the Solids Relay Driver board to short the middle and bottom pins.
4. Confirm that the minimum full power output in the pulsed mode is 1 kW (60 dBm). This should not require adjustment of the amplifier gain.
5. Observe the pulse shape using a 8-ms pulse width. *Do not* confuse the rf pulse rise and fall times with the amplifier turn-on time of 15 sand turn-off time of 3 s. The pulse droop for an 8-ms rf pulse would be 3% or less.
6. Confirm the absence of any amplifier gating/blanking transient pulses coincident with the external amplifier gating signal.

Decoupler Transmitter Performance

Confirm that the decoupler transmitter output to the probe normal as for liquids high band when the amplifier switch is in the LO POWER position. The decoupler should perform as in a normal, liquids-only NMR spectrometer and should be under the control of `dpwr` as usual.

Some parameters in the acquisition group may not be displayed in `dg` if they are set to zero. Be aware of power parameters like `tpwr`, `dpwr`, and pulse lengths. Check their values at experiment setup!

Solids Cabinet Status Light and Safety Switch Performance

1. Make sure that there is no observe or decoupler output to the probe with the amplifier output switch in the OFF position.
2. Make sure that rf output is restored when the ON switch is toggled. The status light goes off when the amplifier output switch is on.

6.2 Wideline NMR Tests

This section procedures for testing the wideline module.

Wideline Solids Test Sample Kit

The wideline solids test sample kit (00-949065-23) consists of the following samples:

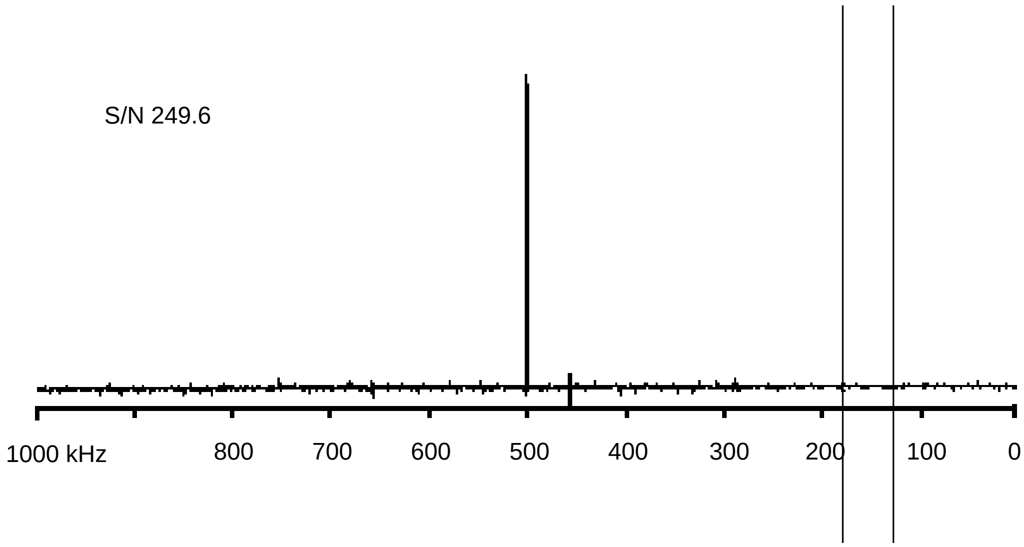
Part No.	*Description*
969104-00	100% malonic acid-d_4
969104-01	100% $NaNO_3$
969104-03	1 M NaCl, 2% D_2O, 98% H_2O

Verify the following NMR performance using the 1 M NaCl, 2% D_2O, 98% H_2O standard sample (Part No. 00-969104-03).

Measuring 90 Pulse and System Sensitivity

1. Enter `rtp('/vnmr/stdpar/H2')` to recall parameters.
2. Set up the spectrometer for data acquisition as follows: `nt=1 dm='n' dp='y' lb=50 gain=0 rof1=30 alfa=0 sw=1e5 np=max fn=65536`
3. Enter `su`.
4. Tune the probe to the deuterium resonance frequency (refer to the *NMR Probes Installation* manual).
5. Determine the 90 pulse for ^{2}H. Enter this value for `pw90`.
6. Enter `sw=1e6`. Set the transmitter offset *tof* such that the deuterium signal resonates 250 kHz from the transmitter frequency. Acquire one transient by entering `ga`. Display the resultant spectrum in the absolute value mode, with `vs=1000`, normalized, `nm`. Confirm that the quadrature image is less than 1% by height as compared to the main resonance.
7. Set `sw=1e5`.

   Set `tof` such that the deuterium signal resonates 25 kHz from the transmitter frequency. Acquire one transient by typing `ga`. Display the resultant spectra in the absolute value mode, with `vs=1000`, normalized, `nm`. Confirm that the quadrature image is less than 1% by height as compared to the main resonance.
8. Set `tof` to 1 kHz off-resonance.
9. Measure ^{2}H sensitivity. Use a spectral noise region of 20 kHz, centered approximately 10 kHz off-resonance. Refer to Figure 16 for sample spectrum and to Table 4 for specifications.




```
 ACQUISITION          SAMPLE              PROCESSING            FLAGS
sfrq   61.395      date      Apr 1     lb        50.00       il            n
tn         H2      solvent    none     sb     not used       in            n
at      0.008      file    /vnmr/~     gf     not used       dp            y
np      16384           stdpar/H2      awc    not used       hs           nn
sw      1e+06      DECOUPLING          lsfid  not used
fb        1e6      dn           H1     phfid      90.0
bs   not used      dof           0     wtfile
ss          0      dm            n     proc         ft          SPECIAL
tpwr       57      dmm           c     fn        65536       temp   not used
pw        2.2      dmf        4000     math          f
p1          0      homo          n
d1      4.000                          werr
d2          0                          wexp
tof         0                          wbs
nt          1                          wnt
ct          0
```

Figure 16. Sample Spectrum, S/N = 249.6, 300-MHz System

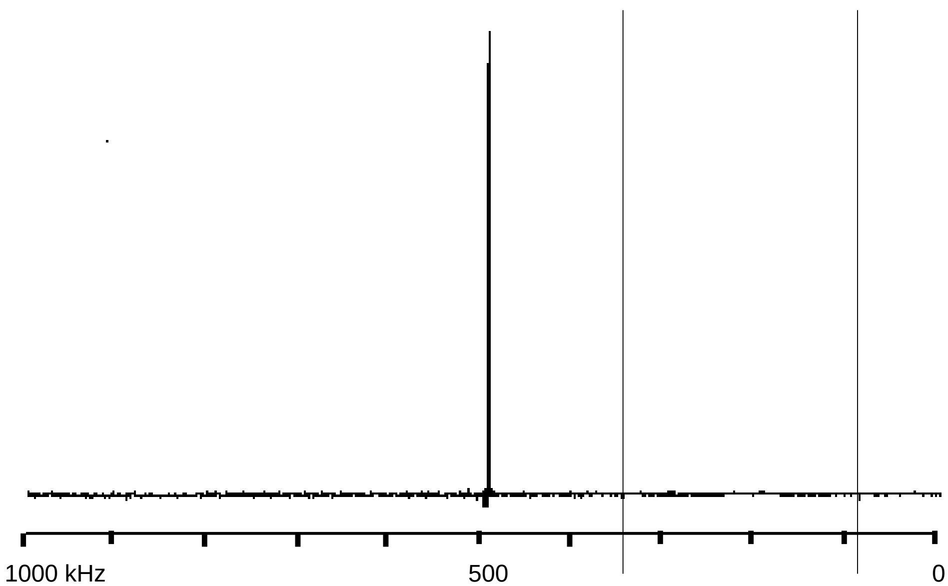
Table 4. 90 Pulse and Signal-to-Noise Specifications

System	*Nucleus*	*PW*$_{90}$	*Frequency*	*S/N*
400 MHz	^{2}H	≤ 3.0 s	61.4	300:1
	^{23}Na	≤ 3.0 s	105.8	630:1
300 MHz	^{2}H	≤ 2.5 s	46.1	200:1
	^{23}Na	≤ 2.5 s	79.4	475:1
200 MHz	^{2}H	≤ 2.2 s	30.7	110:1
	^{23}Na	≤ 2.2 s	52.9	315:1

Sodium 90 Pulse and Sensitivity Measurement

Use the standard liquid sample(00-969104-03), the same as for ^{2}H.

1. Enter `rtp('/vnmr/tests/Na23wl')` to recall parameters.
1. Set up the spectrometer for data acquisition as follows: `seqfil='s2pul' nt=1 sw=1e5 np=max dm='n' dp='y' lb=50 gain=0 rof1=30 alfa=0 fn=65536`.
2. Tune the probe to the sodium resonance frequency (refer to the *NMR Probes Installation Manual*).
3. Determine the 90 pulse for ^{23}Na. Enter this value for `pw90`.
4. Set `tof` to 1 kHz off-resonance.
5. Measure ^{23}Na sensitivity. Use a spectral noise region of 20 kHz, centered approximately 10 kHz off-resonance. Refer to Figure 17 for sample spectrum.




```
 ACQUISITION          SAMPLE               PROCESSING            FLAGS
sfrq  105.800   date        Apr 1   lb         50.00    il              n
tn       Na23   solvent      none   sb      not used    in              n
at      0.004   file      /vnmr/~   gf      not used    dp              y
np      16384     tests/Na23wl      awc     not used    hs             nn
sw     2e+06    DECOUPLING          lsfid   not used
fb     1e+06    dn             H1   phfid       90.0
bs   not used   dof             0   wtfile
ss          0   dm              n   proc          ft       SPECIAL
tpwr       57   dmm             c   fn         65536    temp   not used
pw        2.2   dmf          4000   math           f
p1          0   homo            n
d1      4.000                       werr
d2          0                       wexp
tof         0                       wbs
nt          1                       wnt
ct          0
```

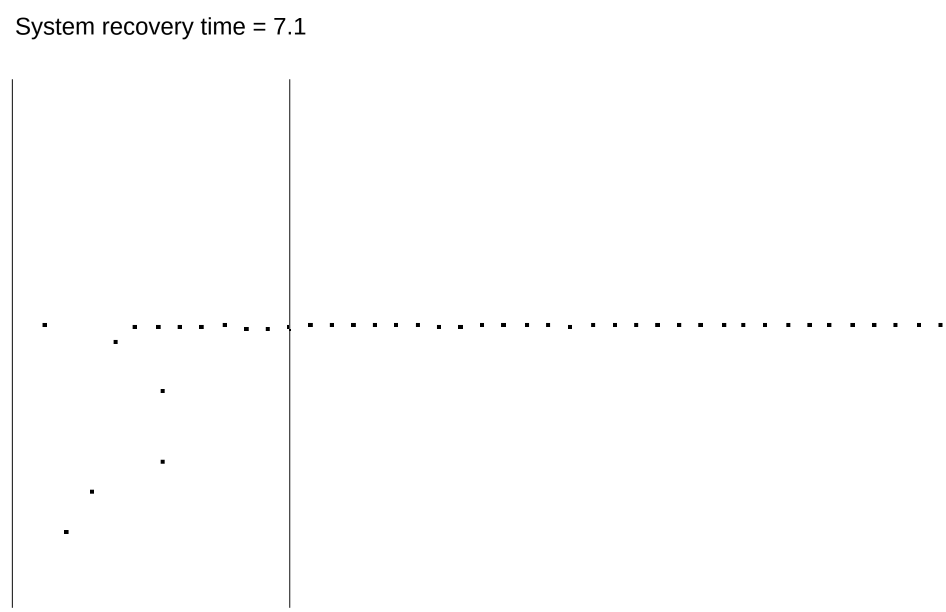
Figure 17. Sample Sodium 90 Pulse and Sensitivity Spectrum, 300-MHz System

System Recovery Time

This test is run with no sample in the probe.

1. Remove the sample and retune the probe.
2. Set the spectrometer as follows: `rof2=0 sw=2e6 np=16384 nt=1 alfa=0`.
3. Set `pw` to the value of `pw90`.
4. Enter `go`.

   Display the resulting FID. Set the cursors such that the left-hand cursor is at the very beginning of the FID and the right-hand cursor is at the point in the FID where the normal noise level returns. The difference between the cursors should be less than 12 s. Refer to Figure 18 for a sample FID display.




```
 ACQUISITION        SAMPLE             PROCESSING           FLAGS
sfrq  105.800    date       Apr 1   lb         50.00    il            n
tn       Na23    solvent     none   sb      not used    in            n
at      0.004    file     /vmnr/~   gf      not used    dp            y
np      16384      tests/Na23wl     awc     not used    hs           nn
sw     2e+06     DECOUPLING         lsfid   not used
fb     1e+06     dn            H1   phfid       90.0
bs   not used    dof            0   wtfile
ss          0    dm             n   proc          ft      SPECIAL
tpwr       57    dmm            c   fn         65536    temp   not used
pw        2.2    dmf         4000   math           f
p1          0    homo           n
d1      4.000                       werr
d2          0                       wexp
tof         0                       wbs
nt          1                       wnt
ct          0
```

Figure 18. System Recovery Time, Sample FID Display

Wideline Spectral Appearance

1. Insert wideline solids sample (Part No. 00-969104-00). Tune the probe carefully for the ^{2}H frequency. If you need to, refer to the *NMR Probes Installation* manual for tuning instructions.
2. Acquire a quadrupolar signal of the malonic acid-d_4 sample using the SSECHO pulse sequence and following the instructions in the *User Guide: Solid-State NMR*. Be sure to center the sample in the coil.
3. Enter `rtp('/vnmr/tests/H2wl')` to recall parameters.
4. Set the following parameters: `nt=16 d1=10 rof1=10 rof2=5 alfa=0.0 tau1=20 tau2=15 seqfil='ssecho' np=16384 lb=1000`.

   The quality of the spectrum is very sensitive to acquisition and processing parameters. Be sure that spectrum is processed in the phased (`ph`) mode, not the absolute value (`av`) mode. If all previous tests have been successful, any spectral imperfection is most likely due to these parameter settings and not hardware malfunctions. When phased properly, the height of the two horns of the pattern should differ by less than 5%.

   The sample has D_2O; therefore, you get a center spike that will phase correctly only if `np=16384`.
5. Set `pw` to the 90 pulse for ^{2}H.

   Figure 19 shows an example SSECHO FID using `lsfid` and correct phase. Refer to Figure 20 for all other spectral parameters. Set `tof` on-resonance for the wideline sample.
6. Acquire 16 transients of data.
7. Look for the following spectral characteristics:
   a. The amplitude of the two "horns" (perpendicular singularities) should be within 5% of each other. If the horns are not within the specification, the `tof` is not truly on resonance. To set the `tof` on-resonance, set the two cursors on the horns of the wideline spectrum and enter `split`.

      One cursor will be set at the middle of the broad peak. Enter `movetof` to set the transmitter offset to this middle position of the broad resonance.
   b. The "shoulders" to the very outside of the broad resonance should be clearly visible and well-defined.
   c. The broad resonance should be very symmetrical in appearance.

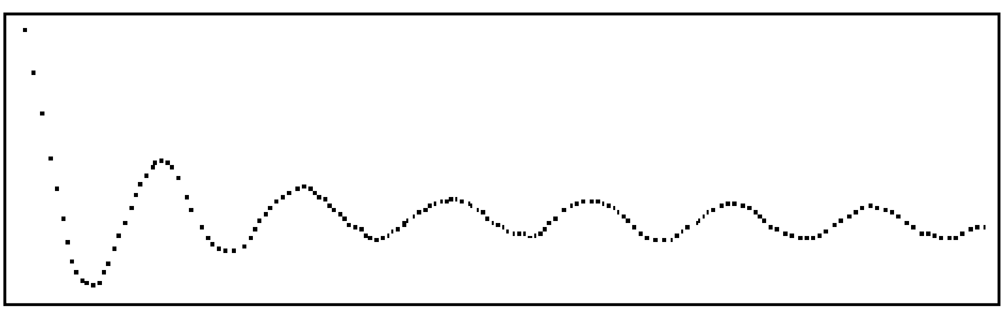


Figure 19. SSECHO FID for Malonic Acid-d_6 Using `lsfid`

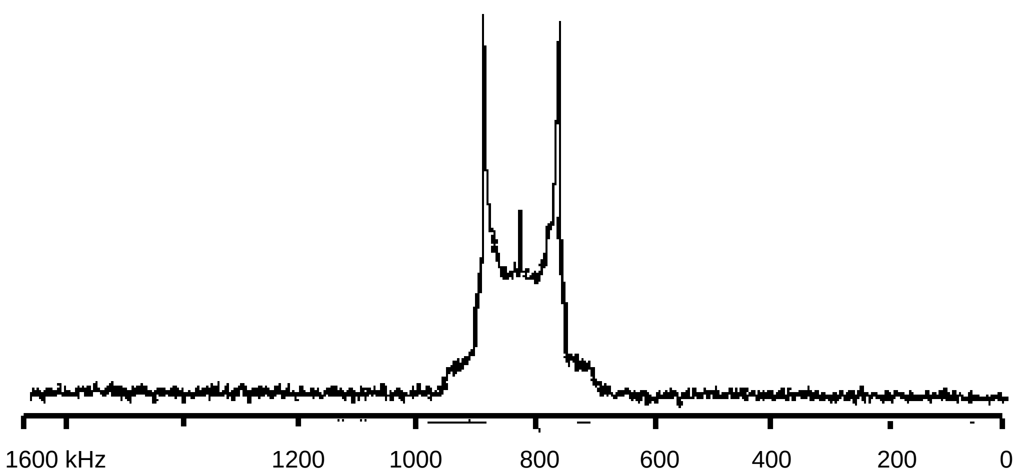
8. Set `compul='y'`.

   Repeat steps 1 to 5 above. Although the echo maximum may change position, symmetry of the horns should be the same. The edges of the spectrum will alter and become sharper. This flag (i.e., `compul`) changes the $\pi/2$ pulses from single to composite and so checks the 90 phase shifting speed of the spectrometer Refer to Figure 20 and Figure 21.

9. Repeat steps 1 to 4 above for sample 00-969104-01, the sodium solid standard sample; tune the probe to the sodium frequency. Recall the standard test parameter by entering `rt('/vnmr/tests/Na23wl')`. The sodium solid standard FID appears similar to the example in Figure 22.

   Refer to Figure 23 for the spectral appearance and spectral parameters. Again, the criteria in step 5 should be observed.

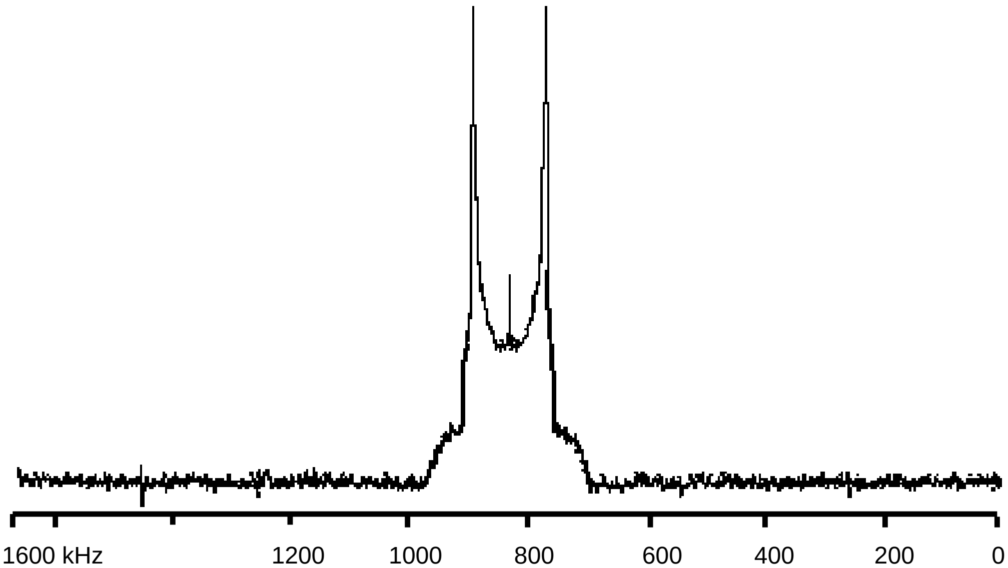
   Apply the symmetry test to the two horns. You should be able to see edges at 200 kHz. (Notice to the left of the pattern is another peak due to aluminium background that may not phase correctly. Ignore it.)




```
ACQUISITION          SAMPLE              PROCESSING            FLAGS
sfrq    61.395     date      Apr 1     lb       1000.00     il           n
tn          H2     solvent    none     sb      not used     in           n
at       0.082     file     /vnmr/~    gf      not used     dp           y
np       16384         tests/H2wl      awc     not used     hs          nn
sw      2e+06       DECOUPLING         lsfid         10     compul       n
fb      1e+06      dn           H1     phfid       90.0
tof          0     dof           0     wtfile
tpwr        57     dm            n     proc          ft       SPECIAL
pw         2.2     dmm           c     fn         65536     temp   not used
p1           0     dmf        4000
p180         0     homo          n
d1      10.000      TRANSIENTS         werr
tau1      20.0     bs     not used     wexp
tau2      15.0     ss            0     wbs
                   nt           16     wnt
                   ct            0
```

Figure 20. Wideline SSpectrum of Malonic Acid-d_6 With `compul='n'`

10. Verify that there is no acoustical ringing at the deuterium frequency using `tau1` and `tau2` values of 20 s.Test the probe with no sample present. Tune the probe to the deuterium frequency and acquire 16 transients. The spectrum should have a flat noise baseline with no broad peaks. Refer to Figure 24 for test parameters and results with a probe that does exhibit acoustical ringing.




```
 ACQUISITION           SAMPLE              PROCESSING             FLAGS
sfrq   61.395      date       Apr 1      lb      1000.00      il            n
tn         H2      solvent     none      sb     not used      in            n
at      0.082      file     /vnmr/~      gf     not used      dp            y
np      16384         tests/H2wl         awc    not used      hs           nn
sw     2e+06        DECOUPLING           lsfid        10      compul        y
fb     1e+06       dn            H1      phfid      90.0
tof         0      dof            0      wtfile
tpwr       57      dm             n      proc         ft        SPECIAL
pw        2.2                            fn        65536      temp   not used
p1          0
p180        0
d1     10.000       TRANSIENTS           werr
tau1     20.0      bs      not used      wexp
tau2     15.0      ss             0      wbs
                   nt            16      wnt
                   ct             0
```

Figure 21. Wideline Spectrum With `compul='y'`

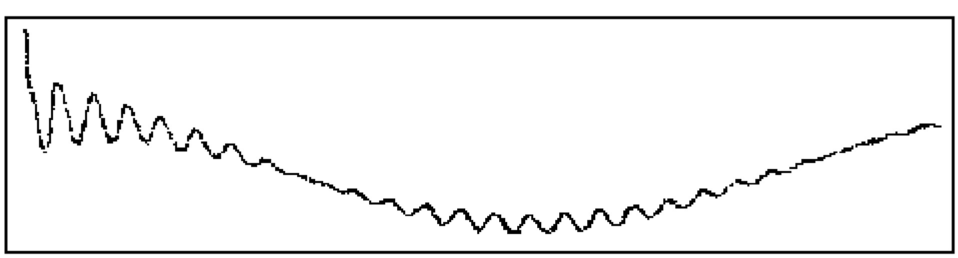
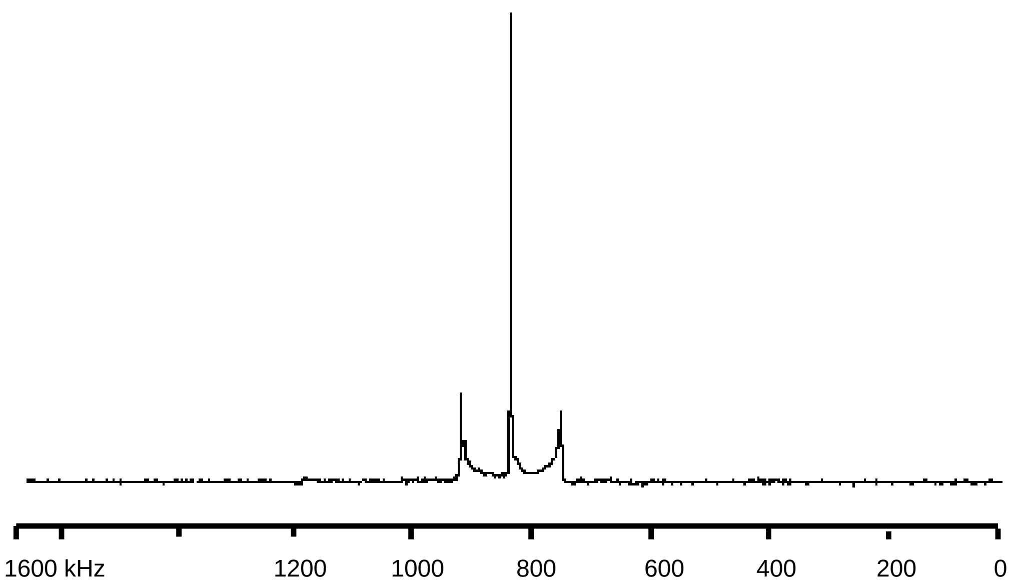


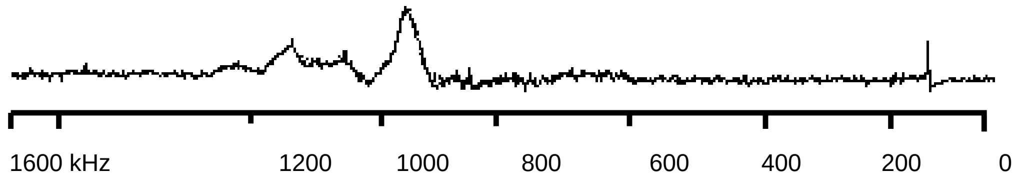
Figure 22. ^{23}Na FID of Sodium Standard ($NaNO_3$) Starting at Top of Echo

11. The wideline probe does exhibit ^{23}Na and ^{27}Al background due to the glass rods in the coil support. Refer to Figure 25 and Figure 26 for an example of the expected sodium and aluminum background.



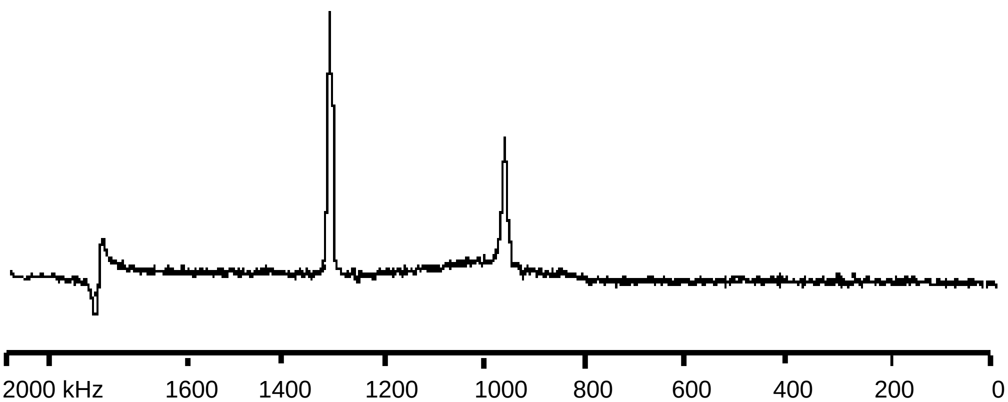

```
 ACQUISITION         SAMPLE              PROCESSING           FLAGS
sfrq  105.800      date      Apr 1     lb      1000.00      il           n
tn       Na23      solvent    none     sb     not used      in           n
at      0.082      file     /vnmr/~    gf     not used      dp           y
np      16384        tests/Na23wl      awc    not used      hs          nn
sw      2e+06       DECOUPLING         lsfid        10      compul       n
fb      1e+06      dn            H1    phfid      90.0
tof         0      dof            0    wtfile
tpwr       57      dm             n    proc         ft        SPECIAL
pw        2.7                          fn        65536      temp  not used
p1          0
p180        0
d1      1.000       TRANSIENTS         werr
tau1     20.0      bs      not used    wexp
tau2     15.0      ss             0    wbs
                   nt            16    wnt
                   ct             0
```

Figure 23. Wideline Spectrum With Sodium Solid Standard



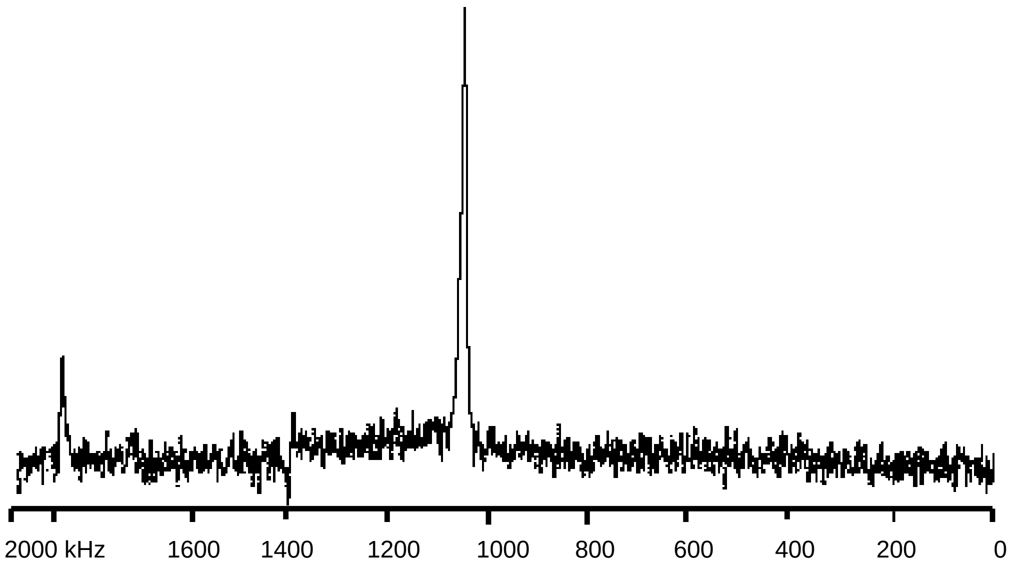

```
 ACQUISITION         SAMPLE              PROCESSING              FLAGS
sfrq  61.395     date      Apr 1     lb       1000.00     il            n
tn        H2     solvent    none     sb      not used     in            n
at     0.082     file     /vnmr/~    gf      not used     dp            y
np     16384         tests/H2wl      awc     not used     hs           nn
sw    2e+06       DECOUPLING         lsfid         10     compul        n
fb    1e+06      dn           H1     phfid       90.0
tof        0     dof           0     wtfile
tpwr      57     dm            n     proc          ft        SPECIAL
pw       2.2                         fn         65536     temp   not used
p1         0
p180       0
d1     1.000      TRANSIENTS         werr
tau1    20.0     bs     not used     wexp
tau2    15.0     ss            0     wbs
                 nt           16     wnt
                 ct            0
```

Figure 24. Sample Spectrum From a Probe With Acoustical Ringing




```
 ACQUISITION         SAMPLE              PROCESSING              FLAGS
sfrq 105.800     date      Apr 1     lb       1000.00     il            n
tn      Na23     solvent    none     sb      not used     in            n
at     0.082     file     /vnmr/~    gf      not used     dp            y
np     16384       tests/Na23wl      awc     not used     hs           nn
sw    2e+06       DECOUPLING         lsfid         10     compul        n
fb    1e+06      dn           H1     phfid       90.0
tof        0     dof           0     wtfile
tpwr      57     dm            n     proc          ft        SPECIAL
pw       2.7                         fn         65536     temp   not used
p1         0
p180       0
d1     1.000      TRANSIENTS         werr
tau1    20.0     bs     not used     wexp
tau2    15.0     ss            0     wbs
                 nt          100     wnt
                 ct            0
```

Figure 25. Sample Spectrum With Sodium Background




```
 ACQUISITION         SAMPLE              PROCESSING           FLAGS
sfrq  105.800      date      Apr 1     lb      1000.00      il            n
tn       Na23      solvent    none     sb     not used      in            n
at      0.082      file     /vnmr/~    gf     not used      dp            y
np      16384        tests/Na23wl      awc    not used      hs           nn
sw     2e+06        DECOUPLING         lsfid        10      compul        n
fb     1e+06       dn            H1    phfid      90.0
tof         0      dof            0    wtfile
tpwr       57      dm             n    proc         ft        SPECIAL
pw        2.7                          fn        65536      temp   not used
p1          0
p180        0
d1      1.000       TRANSIENTS         werr
tau1     20.0      bs      not used    wexp
tau2     15.0      ss             0    wbs
                   nt           100    wnt
                   ct             0
```

Figure 26. Sample Spectrum With Sodium And Aluminium Background

Chapter 7. CRAMPS/Multipulse Module Tests

The CRAMPS/Multipulse Solids Module enables Varian UNITY*INOVA* spectrometers to run CRAMPS and multipulse experiments. The CRAMPS/multipulse experiment is normally used to observe ^{1}H. In CP/MAS, the ^{1}H amplifier is used as a heteronuclear decoupler, while in CRAMPS the ^{1}H amplifier is used as an high-power observe channel. This chapter contains system tests to check the performance of the CRAMPS/ Multipulse module. The following is a list of the tests that are described:

RF transmitter performance tests

- 1 kW amplifier tuning
- Liquids observe transmitter rf signal path
- Solids observe transmitter rf signal path
- Third cabinet status light and safety switch performance
- AMT amplifier calibration
- 1 kW amplifier calibration gating

CRAMPS/Multipulse NMR tests

- Decoupler calibration
- FLIPFLIP
- FLIPFLOP
- MREV8
- ^{1}H wideline

CRAMPS probe test (Varian-supplied probe only)

- MREV8 narrowing at magic angle
- BR24 narrowing at the magic angle

CAUTION: **Read the following cautions:**

- **Make sure that all amplifiers are terminated in appropriate 50-ohm loads at all times.**
- **The high power available from the amplifier in the third cabinet is intended only for solid state. Applying this power to a liquids probe will destroy the probe. Before turning on the amplifier in the third cabinet, check that the appropriate solids probe has been put in place.**
- **Disable all high-power solids amplifiers before installing any probe.**
- **Confirm that the rf signal being measured can never exceed the maximum input signal of the measuring device.**
- **Do not block any of the ventilation holes in the third cabinet. Even on standby, the amplifiers radiate considerable heat.**

7.1 Tuning the High-Power (Cavity) Amplifier

The most reliable method for tuning the cavity amplifier (shown in Figure 27) requires a Bird through-line wattmeter on the input to the cavity and some other power measuring device on the output from the cavity. The output power can be measured using a 30-dB, 500-W attenuator with an additional 30-dB, 5-W attenuator coupled to a Bird meter or spectrum analyzer, or, if necessary, by performing NMR pulse width calibrations. Grid current should be monitored at the status panel using an oscilloscope.

The cavity amplifier has five adjustments that must be optimized for best performance. While making these adjustments, the grid current can rise dangerously if the rf power input is too high and/or the output coupling is not adjusted correctly—therefore, **always be aware of the grid current value**.

WARNING: **Dangerous high voltages over 2200 Vdc exist inside the high voltage section. Contact with a bare terminal can result in serious injury or death. The high-voltage section has no tuning adjustments. Only qualified service personnel shall remove the cover.**

CAUTION: **To prevent the cavity amplifier from failing, never let the grid current exceed 160 mA pulsed or 60 mA CW (continuous wave).**

The tuning procedure is broken into three sections: initial tuning and fine tuning, which are only performed on installation of a new power tube in the cavity, and routine tuning, which is performed at system installation as well as on other occasions. The user should only be concerned with the routine tuning procedure.

Initial Tuning of the Cavity Amplifier (CW Mode)

1. Place a Bird through-line wattmeter in line with the input to the cavity amplifier. Direct the cavity amplifier output through a 30-dB, 500-W and a 30-dB, 5-W attenuator into a spectrum analyzer. Remove the left (low-voltage) top cover of the amplifier.

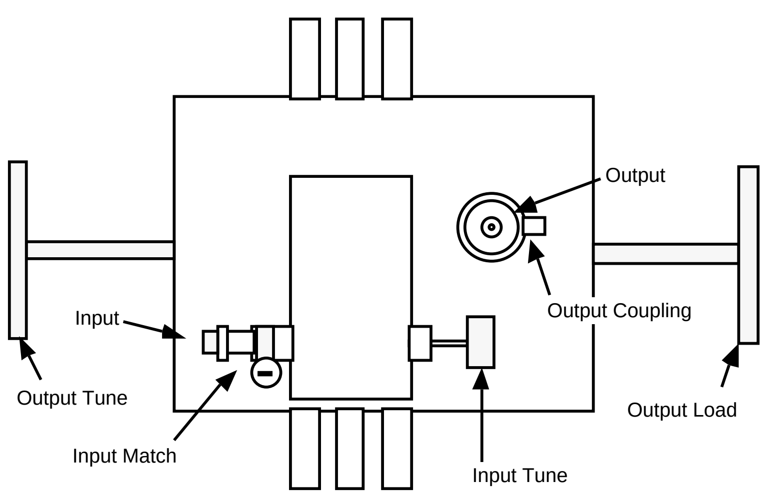


Figure 27. High-Power (Cavity) Amplifier

WARNING: This procedure exposes the operator to high voltages. Take all appropriate safety precautions! Exercise extreme care!

2. Turn on the low voltage power supply to energize the tube filament.

CAUTION: Do not apply rf input or high voltage supply until at least 3 minutes after turning on the low voltage power supply.

3. **After 3 minutes**, energize the high voltage supply.
4. Enter `tn='C13' dn='H1' dm='n' su` to switch to CW unblanking.
5. Apply 37 dBm (5 W) of rf drive CW by setting `dpwr` to an appropriate value and entering `dm='y' su`.

   You can check the upper limits of `dpwr` by entering `dpwr=63` and pressing Return. Check in `dg` for the value of `dpwr`. If `dpwr=49` appears, the upper limit on the `dpwr` parameter can be changed using `config` (refer to the *VNMR and Solaris Software Installation Manual*).

   Adjust the input tuning and matching (slide coupling with hose clamp, see Figure 27) for minimum reflected power on the thru-line wattmeter. This is an repetitive procedure, much like probe tuning.
6. Adjust the TUNE and LOAD tuning controls repetitively for maximum output power. Check that the grid current is well within the limits of 160 mA.

CAUTION: To avoid damaging the internal sense antenna, do not exceed the 3-o'clock position shown in Figure 27 in a counter-clockwise direction. The antenna is damaged when it contacts the moving load wall of the cavity. Also, do not over-loosen the locking ring because the antenna will become loose, move freely, and be damaged.

7. Rotate the output coupling lever to achieve maximum power output and then tighten the locking disk to hold this position.
8. Again adjust the TUNE and LOAD tuning controls repetitively for maximum output power. Check that the grid current is well within the limits of 160 mA.
9. Enter `dm='n' su`.

Fine Tuning the Cavity Amplifier (Pulsed Mode)

1. Apply 46 dBm of power in 1 ms pulses with 10% duty cycle to the amplifier by setting the following parameters: `seqfil='s2pul' pw=1e3 tn='H1' sw=1e5 at=.01 d1=0 nt=1e6`

   If the amplifier trips off with an OVERDR indication, lower the input power until the protection circuit does not activate.
2. Monitor the grid current at the test point on the status panel with an oscilloscope. The scope shows 1 mV for each mA grid current. If necessary, raise the current set point of the protection circuit (R83 on the cavity control board 990556), as described in "Adjusting the Overdrive Interlock" on page 70, to allow for 46-dBm pulses.
3. Attempt to maximize the output power while minimizing the grid current. Rotate the output coupling to maximize the output—typically the 6 o'clock position.

Adjust both TUNE and LOAD to maximize the output. The gain should be 14 dB at 46 dBm input.

To find the amplifier input/output match, begin with the TUNE at one extreme and then adjust the LOAD for a maximum. Then move the TUNE 20% towards the other extreme and readjust the LOAD for a new maximum. Continue in this way (40%, 60%, . . .) noting each apparent maximum. The greatest maximum is in the region of the match. Continue to refine the adjustment in this region to achieve maximum output power.

4. Make fine adjustments to the input tuning and matching for maximum output.
5. If a gain of 14 dB is not achieved, it may be necessary to repeat step 3 and 4 with the output coupling in a new orientation.
6. Adjust the protection circuit current set point to approximately 20% above the grid current level measured with a scope at 1 kW output.
7. Reassemble the amplifier.

Tuning the Cavity Amplifier During Normal Operation

Routine tuning is done during the initial installation in the field and on a regular basis during normal operation. The routine tuning procedure involves neither access to high voltages nor disassembly of the amplifier.

1. Apply 46 dBm of power in 1 ms pulses with 10% duty cycle to the amplifier by setting the following parameters: `seqfil='s2pul' pw=1e3 tn='H1' sw=1e5 at=.01 d1=0 nt=1e6`
2. Attempt to maximize the output power while minimizing the grid current. Adjust both TUNE and LOAD to maximize the output. The gain should be 14 dB at 46 dBm input.

   To find the amplifier input/output match, begin with the TUNE at one extreme and then adjust the LOAD for a maximum. Then move the TUNE 20% towards the other extreme and readjust the LOAD for a new maximum. Continue in this way (40%, 60%, . . .) noting each apparent maximum. The greatest maximum is in the region of the match. Continue to refine the adjustment in this region to achieve maximum output power.
3. Make fine adjustments to the input tuning and matching for maximum output.

Adjusting the Overdrive Interlock

WARNING: **Dangerous high voltages over 2200 Vdc exist inside the high voltage section. Contact with a bare terminal can result in serious injury or death. The high-voltage section has tuning adjustments. Only qualified service personnel shall remove the cover.**

1. Power down the 1 kW high-band amplifier EHT.
2. Remove the 1 kW high-band amplifier from the cabinet.
3. Remove the top cover from the low-voltage side (wider cover).
4. Locate on the control board (see schematic 87-195723-00) the test point TP2, which is on the lower right side of the board (zone P5).
5. Connect a Fluke DVM (or equivalent) to the test point on 20 Vdc range.

6. Determine the current interlock level by measuring the voltage from the test point to the ground. Using A = V/62, verify that for 120 mA level, V = 7.44.

   If this is not the value, adjust the potentiometer R83 (in zone P4) on the board to obtain it. (The range is from 80 mA (4.96 V) to 160 mA (9.92 V)).
7. Reassemble the amplifier and return it to the cabinet.

7.2 Testing RF Transmitter Performance

This series of tests evaluates the third cabinet relay and attenuator operation as well as the 1 kW amplifier power output and performance. Figure 28 illustrates the third cabinet status panel.

1. The blanking signal from the connector J7023 should show correct levels. During REC ON the level is low and during REC OFF the blanking signal is high. If this not the case, you can change each output to the correct level by moving the blanking polarity select jumper J1 on the Solids Relay Driver board. Confirm that the blanking gate cable is connected to the amplifiers.
2. Using a 30-dB, 500-W attenuator, check the input at J7710 as described in step 7 below. Add at least a 20-dB attenuator in series if you are using a spectrum analyzer.
3. Make sure that the power to the third cabinet and the low-voltage power supply are on. Wait 3 minutes.
4. **After three minutes**, switch on the EHT supply and make sure of the following:
   - The EHT voltage readout is 2.2 kV or greater.
   - The EHT current is no more than 2.2 mA (pulsed mode) or 55 mA (CW mode).
5. Carefully tune the cavity amplifier. See "Tuning the High-Power (Cavity) Amplifier" on page 68 for the tuning procedures.
6. Perform the following amplifier tests:
   a. With both LO BAND and HI BAND set to LO POWER, enter `rtp('/vnmr/stdpar/H1')` to recall parameters.

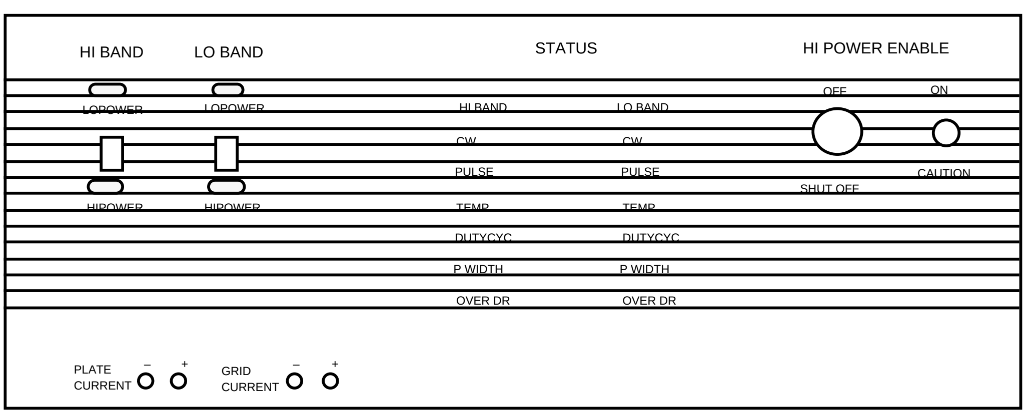


Figure 28. High-Power Amplifier Control Panel on the Solids Cabinet

d. Set the following parameters: `nt=1e6 sw=1e5 np=128 pw=2e3 d1=0.1 gain=0 tpwr=63`.

e. Enter `go`. Check that no rf is present at the input of the amplifier.

f. Enter `aa` and wait for the `abort` message. Set the HI BAND switch to HI POWER. Verify rf power of at least 44 dBm.

g. Stop the acquisition. Move the spectrum analyzer input with the 500-W attenuator to transmitter cable at the preamplifier. Set the HI BAND switch to LO POWER.

h. Enter `go`. Check rf power of about 44 dBm.

i. Stop the acquisition. Set `tpwr=50` and set HI BAND to HI POWER. Confirm rf power of about 50 dBm at the preamplifier.

j. Set `tpwr=63`. Restart and check for 900 W (59.5 dBm) or more at the preamplifier.

   If the "overdrive" interlock comes on, check the interlock adjustment. Otherwise, stop the acquisition.

k. Reconnect the transmitter cable to the preamplifier. Connect the spectrum analyzer to the PROBE port on the preamplifier. Repeat step g above, making sure that loss through the preamplifier is less than 1.0 dB.

7. Observe the pulse shape using a 8 ms pulse width. *Do not* confuse the rf pulse rise and fall times with the amplifier turn-on and turn-off times. The pulse droop for an 8 ms rf pulse would be 3% or less.

8. Confirm the absence of any amplifier gating/blanking transient pulses coincident with the external amplifier gating signal.

Testing the Gating

1. Stop the acquisition. Select LO POWER on both HI BAND and LO BAND. The scope should still be connected to the probe port.

2. Set the following parameters: `rof1=0.2 pw=2e3`

3. Enter `go`. Trigger on blanking signal J7024 and observe rf. Determine the gating delay by measuring the time at which the rf is fully on. Add 200 ns to this value. The liquids amplifier should gate on in less than 2 µs.

4. Repeat steps 1 through 3 with both HI BAND and LO BAND set to HI POWER. The rf should gate on in less than 2 µs.

Checking Decoupler Transmitter Performance

Confirm that the decoupler transmitter output to the probe is normal (as for liquids) when the LO BAND switch is set to LO POWER. The decoupler should perform as in a liquids-only NMR spectrometer and should be under the control of `dpwr` as usual.

Some parameters in the acquisition group may not be displayed by the `dg` command if they are set to zero. Be aware of power parameters like `tpwr`, `dpwr`, `dlp`, `tpwrf`, `dpwrf`, and pulse lengths. Check their values at experiment setup!

Testing the Amplifier

Typical power outputs are given as a guide to troubleshooting. Pulse droop should be recorded on all systems, but accurate power measurements are only needed if the system fails to make pulse width or γB_2 specifications. A spectrum analyzer should be used for accurate power measurements.

To Test the AMT Decoupler Amplifier (Channel A)

The maximum drive to the AMT amplifier should not exceed +2 dBm. This should be checked for the decoupler as follows:

1. Disconnect the input to Channel A of the AMT amplifier with fixed attenuators in-line and connect it to a power meter.
2. Set the following parameters: `level2=63 level2f=4095 nt=1e6 dm='nny' sw=1e5 np=128 d1=0.1`
3. Enter `go` and add sufficient in-line attenuators so that the peak power does not exceed 2 mW (+3 dBm).
4. Enter `aa` to stop the acquisition and then reconnect the AMT amplifier.
5. At the input to the high-power decoupler amplifier, calibrate power as a function of `level2` when `level2f=4095`. The maximum should be at least 44 dBm (25 W).
6. Enter `aa` to stop acquisition.

To Test the High-Power Decoupler Amplifier

CAUTION: **For CP/MAS, the maximum decoupler power at the probe should be no more than 100 W. If this is exceeded, damage to the CP/MAS probe could occur.**

Perform this calibration as follows.

1. Connect a 30-dB, 500-W attenuator to the DEC port on the magnet leg, replacing the probe. Connect the other end of the attenuator to a scope or spectrum analyzer.
2. Set `level2=63 level2f=4095 nt=1e6 xpol='n'` and enter the `go` command.
3. Measure the output level in watts.
4. Decrease the value of `level2` until this output is no more than 100 W. Note that you must enter `aa level2=# go`, where # is a new value for `level2`, each time you change the value of `level2`.
5. When the `level2` value is determined, halt the acquisition and write down the value so you can refer to it later.
6. Enter `aa` and return the system to normal configuration.

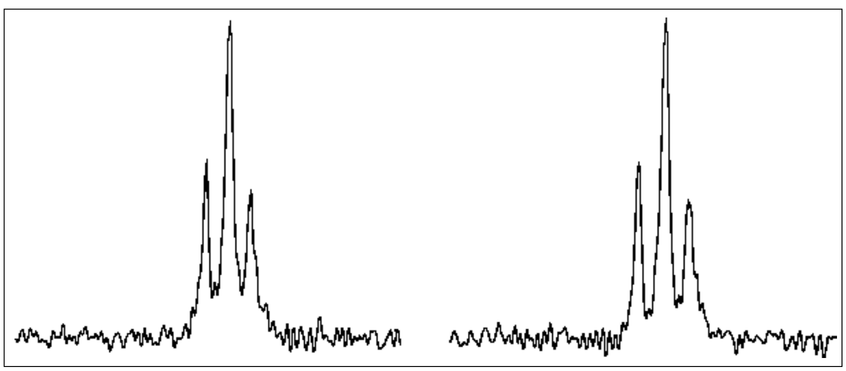
Note: Whenever `level1=63` or `level2=63` is used in the next few pages, use the value determined step 4 above instead of 63.

Adjusting Decoupler Field Strength

1. Use the dioxane sample (00-957734-01) spinning at 200 to 300 Hz.

WARNING: **The 1, 4-dioxane sample (00-957734-01) is a cancer suspect agent with flammable liquid oxidizers. See the enclosed MSDS for additional information and handling precautions.**

2. Enter `rtp('/vnmr/stdpar/C13') su` to select the ^{13}C observe parameters.
3. Set HI BAND to LO POWER.
4. Tune the decoupler channel of the probe (refer to the *NMR Probes Installation* manual)
5. Check limits of `dpwr`. Enter `dpwr=63`. Check for the value of `dpwr`. If `dpwr=49` appears, the upper limit on the `dpwr` parameter can be changed (refer to the *VNMR System Operation* manual).
6. Change the following parameters: `at=0.10 d1=30 dm='nny' dpwr=63`. Set `pw` and `tpwr` to the 90 value. Set up a `dof` array by entering `dof=–2e4,2e4`.
7. Enter `ga`, record the spectra, and measure the residual splitting of the dioxane triplet. Figure 29 shows a spectrum.



```
 ACQUISITION          SAMPLE             PROCESSING            FLAGS
sfrq   100.57     date       Apr 1     lb          10.00     il            n
tn        C13     file         exp     sb      not used      toss          n
at      0.100     DECOUPLING           gf      not used      pdp           n
np       6016     dn            H1     awc     not used      dp            y
sw     3000.0     dof      arrayed     lsfid   not used       SPECIAL
xpol        n     dm           nny     phfid   not used      srate       415
tpwr       55     dmm            c     wtfile                temp   not used
tpwrf    4095     dpwr          63     proc           ft
pw        8.0     level1        45     fn          32768
p1          0     level1f      200                              FLAGS
d1     30.000     level2        50     werr                  ai no ph
tof         0     level2f     4095     wexp
nt          1     dpwrf       4095     wbs
ct          1                          wnt
                  Gamma-H2 calculation
                  See J. Magnetic Resonance 7:442 (1972)
   jo =  284.00 Hz,   dof[1] =  –70000.00 Hz,   dof[2] =  70000.00 Hz
jr[1] =  165.00 Hz,    jr[2] =  167.00 Hz
Gamma-H2          =      69404.97 Hz
90 degree pulse =           3.6 usec
coalescence frequency =     –457.51 Hz
```

Figure 29. Decoupler Field Strength γB_2

8. Use `h2cal` to calculate γB_2. No specification exists, but a value of about 25 kHz is needed for the 100 W test to be successful.
9. Set HI BAND to HI POWER. Be sure that `dpwr=63` is calibrated for about 100 W at the HI POWER output, or use the value of `dpwr` that gives about 100 W.
10. Enter `ga`, record the spectra, and measure the residual splitting of the dioxane triplet with `dof=–7e4,7e4`.

CAUTION: **Avoid damaging the probe, follow all pulse duration, pulse power, and duty cycle cautions and warnings when given for a particular probe. Generally, pulse durations can be increased with a proportional decrease in pulse power.**

11. Use `h2cal` to calculate γB_2. Set `dpwr` to achieve a γB_2 that is as close as possible to the minimum values listed in the following table. Do not exceed this power level for any other experiment. For the XPOLAR pulse sequence, the maximum `level1` and `level2` correspond to this `dpwr` value.

Magnet	*Varian RT CP/MAS*	*Doty CP/MAS*	*Varian VT CP/MAS*
200 MHz	75 kHz	—	—
300 MHz	75 kHz	60 kHz	80 kHz
400 MHz	60 kHz	55 kHz	70 kHz

If the probe does not make the specification, check for rf losses in the decoupler circuitry.

12. Note the value of `dpwr` because it is used for `level2` during future tests.

7.3 Testing CRAMPS NMR

For CRAMPS installations, the NMR tests break into two parts: the tune-up pulse sequences and probe-dependent multipulse experiments. The following list of tune-up pulse sequences should be performed on a liquid sample of benzene and $Cr(acac)_3$ (Part No. 00-958984-87) at all installations:

- *FLIPFLIP*
- *FLIPFLOP*
- *MREV8*
- ^{1}H wideline

If the CRAMPS probe is supplied by Varian, two further spinning tests should be performed on adipic acid:

- *MREV8*
- *BR24*

Before operating the cavity amplifier, it must be tuned as described in “Tuning the High-Power (Cavity) Amplifier” on page 68.

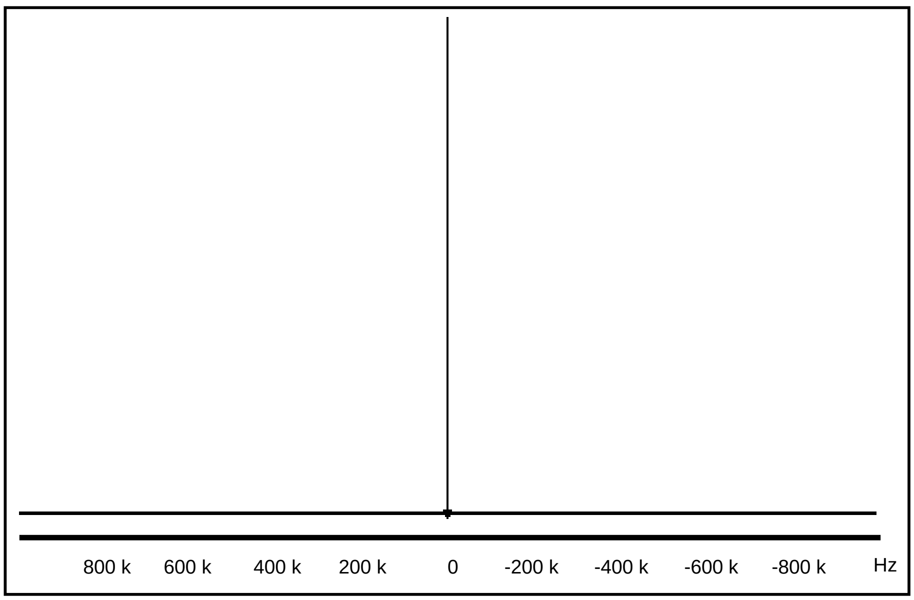
Testing Multipulse NMR

Use a CRAMPS probe, if available, or a wideline probe tuned to the ^{1}H frequency. The following tests are performed using a test sample of benzene and $Cr(acac)_3$ (Part No. 00-958984-87) in a spherical bulb. This sample, provided with the kit, is inserted into the coil of the probe so that it is at the center of the coil. Note that is essential that the spherical portion of the sample has no air bubbles.

Testing Basic NMR

An example spectrum of the standard ^{1}H observe using the S2PUL pulse sequence is shown in Figure 30.

1. Enter `rtp('/vnmr/stdpar/H1')` to recall parameters.




```
 ACQUISITION         SAMPLE             PROCESSING            FLAGS
sfrq  399.952      date      Apr 1     lb     not used     il             n
tn         H1      file        exp     sb     not used     in             n
np      16384        DECOUPLING        gf     not used     dp             y
sw     1e+04       dn           H1     awc    not used     trig           y
at        0.2      dof           0     lsfid         0        SPECIAL
bs         16      dm            n     phfid         0     srate       1542
at       0.20      dpwr         30     fn       131072     temp        27.0
tpwr       55
tpwrf   4e+03         FLAGS            werr                tauc       3.500
pw      1.000      ai cdc ph           wexp         df     mp_at 0.000896
tau     3.500                          wbs
d1      1.000        DISPLAY           wnt                 phase1         0
tof   -2986.3      sp    -999877.9                         phase2         0
nt          1      wp       2e+06        REFERENCE         phaser         0
ct          1      vs           79     rf1    999877.9
                                       rfp           0
                                       cr      58579.4
                                       delta   99377.4
```

Figure 30. Standard ^{1}H Observe Spectrum Using the S2PUL Pulse Sequence

2. Enter `create('tpwrf','integer')` `setlimit('twprf',4095,0,1)`, and then set `tpwrf=4095`.
3. Set the following parameters for basic NMR tests: `sw=1e4 tpwr=55 pw=1 gain=0 at=.2 d1=1 nt=1 dp='y'`
4. Select LO POWER for both HI BAND and LO BAND on the solids cabinet.
5. Acquire an FID.
6. Make sure that the rf is not on-resonance and then shim on the FID to get a FWHH (full-width at half-height) of approximately 30 Hz. For more information on FID shimming, refer to the *System Operation Manual*. Note that it is essential that the spherical portion of the sample has no air bubbles.
7. After this linewidth is met, set the rf on resonance using `movetof` and reacquire a FID.
8. Check that a simple exponential decay exists for the on-resonance condition.
9. Set the HI BAND switch to HI POWER on the solids cabinet.
10. Determine the power necessary for a 180 pulse by setting: `pw=3 nt=1 tpwrf=4e3 tpwr=50,51,52,53,54,55,56,57,58,59,60` **and then entering** `go`**.**
11. Select the value of `tpwr` that gives the closest to a null. Then set `pw=1.5`. Write down this `tpwr` value for future use.
12. Enter `rof2=0 alfa=0 sw=2e6 go` to reacquire a single FID. Figure 31 shows an example of a receiver recovery FID.
13. Enter `df` and examine the beginning of the FID. Using a cursor, determine the time taken for the receiver system to recover and write down the value.

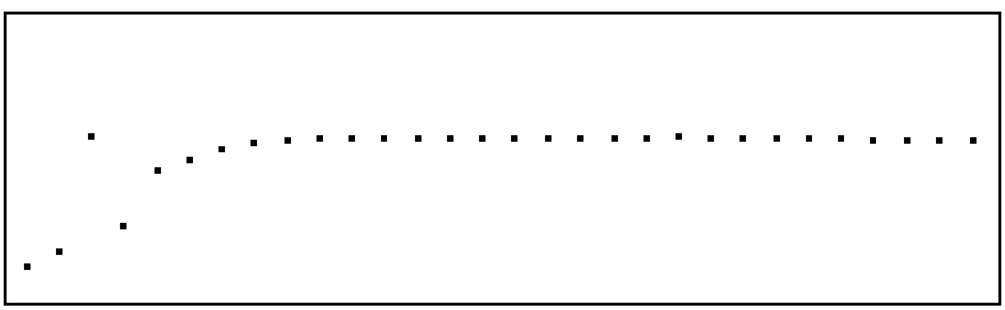


Figure 31. Receiver Recovery FID

Running the FLIPFLIP Pulse Sequence

1. Enter `flipflop` and set `d1=4 pw=1.5 fb=1e6 sw=2e6 np=128 tau=7 rof1=1.5 rof2=0`.
2. Set `phase1=0 phase2=0` (this sets up the "flipflip mode") `phaser=0 phfid=0` or `'n' trig='n' gain=0 nt=1 dp='y'`.

   Note that `trig='n'` is needed for UNITY*INOVA* systems since the 500-kHz synching signal is not available and is unnecessary.
3. Acquire a FID and enter `df` to display both real and imaginary components. Adjust the phase to minimize the imaginary signal. If this is not possible, check that the signal is on resonance.
4. Enter `phaser=phfid phfid='n'` and reacquire. The imaginary channel should be minimal. The real channel should show a FID pattern similar to that shown in Figure 32.

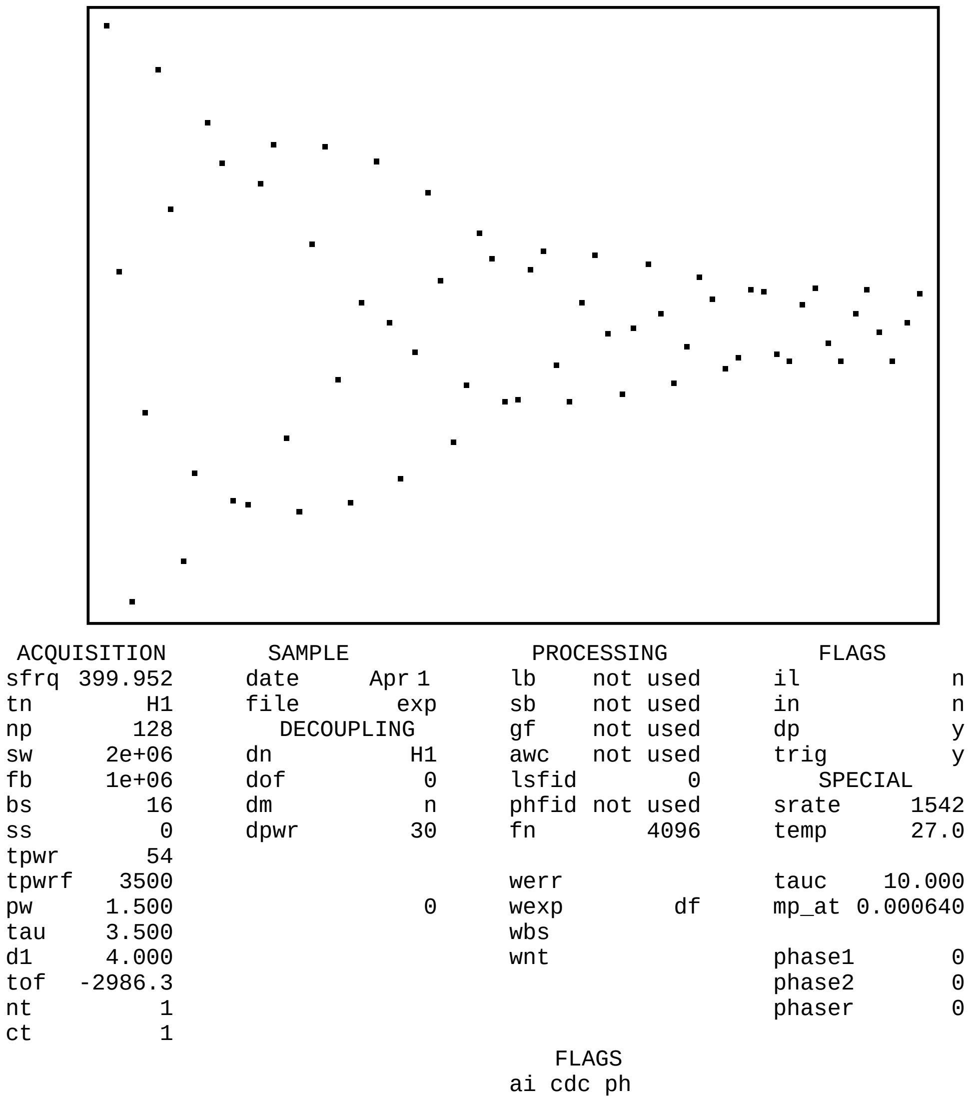


Figure 32. Real Channel FID Pattern

5. Count the number of points in one cycle of the FID and the re-estimate the `pw` for a 90 pulse from the following relationship where *N* is the number of points in 1 cycle: `pw=pw*4/N`
6. Repeat the acquisition, adjusting `pw` and then `tpwr` to get approximately a 1.5 μs 90 pulse.
7. Enter `gf` and then connect to the `acqi` window. Select FID and then IPA. Adjust `tpwrf` until a pattern similar that in Figure 33 is obtained. This occurs at the exact 90 pulse.

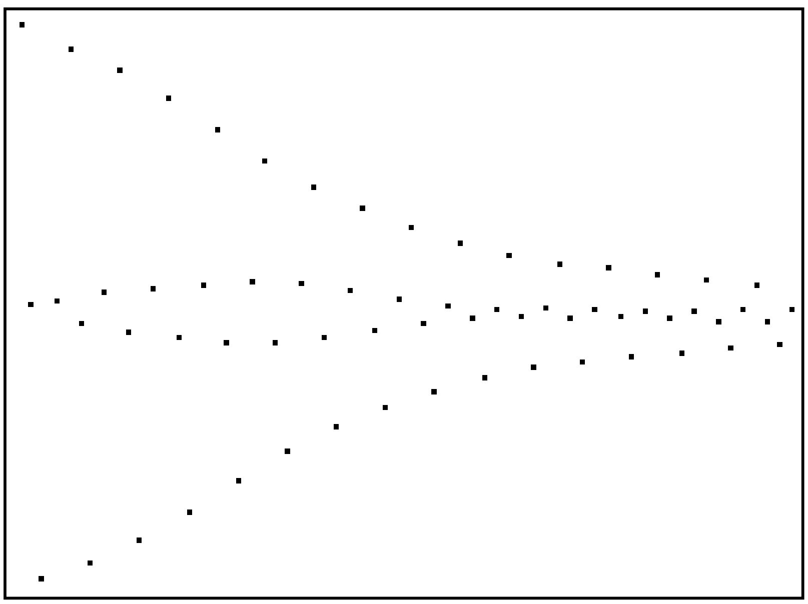


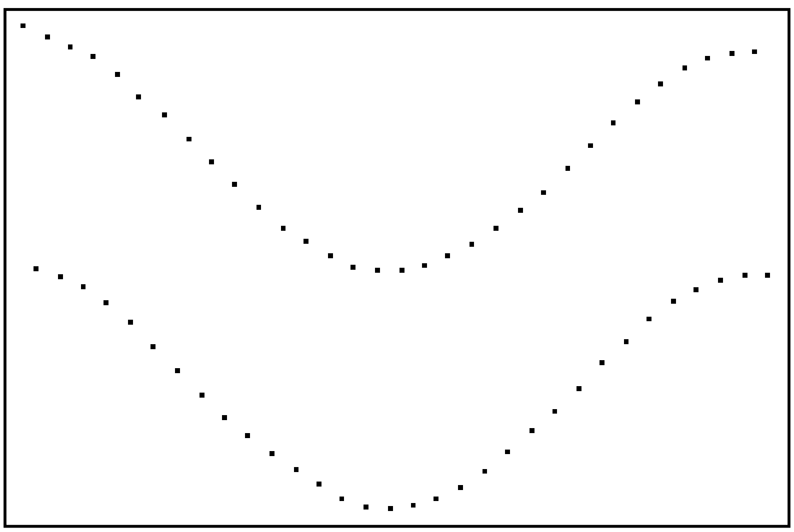
Figure 33. FLIPFLIP FID at Exact 90 Pulse

Running the FLIPFLOP Pulse Sequence

To run the FLIPFLOP pulse sequence rather that the FLIPFLIP pulse sequence, set `phase2=2`. This train of pulses alternately flips the spins into the XY plane—giving an NMR signal—and back to the Z axis—giving no NMR signal. This sequence is used for other adjustments in most CRAMPS/Multipulse spectrometers, but because of the phase shifting circuit used in the UNITY*INOVA* system, this sequence is only used to remove "phase glitch" caused by asymmetric phase transients at the beginning and end of each pulse. The resulting FID of the FLIPFLOP pulse sequence appears as a set of "tram tracks" as shown in Figure 34.

The adjustment to remove phase glitch is done either at the probe or at the 1 kW high band amplifier. After setting `phase2=2`, enter `gf` and then interactively observe the FID, using the `acqi` window.

If the FID shows a sine wave in the tram tracks, carefully adjust the probe tune or match to remove it. If this cannot be done, carefully adjust the 1 kW amplifier tune or load. The desired result is shown in Figure 35.



ACQUISITION		SAMPLE		PROCESSING		FLAGS	
sfrq	399.952	date	Apr 1	lb	not used	il	n
tn	H1	file	exp	sb	not used	in	n
np	128	DECOUPLING		gf	not used	dp	y
sw	2e+06	dn	H1	awc	not used	trig	y
fb	1e+06	dof	0	lsfid	0	SPECIAL	
bs	16	dm	n	phfid	not used	srate	1542
ss	0	dpwr	30	fn	4096	temp	27.0
tpwr	54						
tpwrf	2794			werr		tauc	10.000
pw	1.400			wexp	df	mp_at	0.000640
tau	10.000			wbs			
d1	4.000			wnt		phase1	0
tof	-2986.3			FLAGS		phase2	0
nt	1			ai cdc ph		phaser	65.7
ct	0						

Figure 34. FLIPFLOP "Tram Tracks"

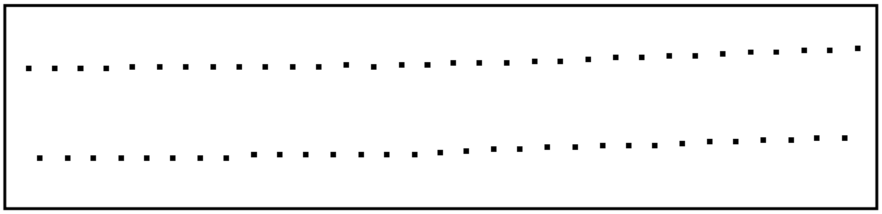


Figure 35. FLIPFLOP desired FID

Finally, set `phase2=0` and repeat the `pw90` determination as outlined above, using the flipflip process. No reexamination of gating is required, but the 90 pulse should be set as precisely as possible.

Using MREV8 to Demonstrate Multipulse Operation

The MREV8 pulse sequence demonstrates the operation of the multipulse system.

1. Enter `mrev8` to set up the sequence and parameters for the MREV8 pulse sequence.
2. Make sure that `tau=3.5`, `rof1=1.5`, and `rof2=0`.
3. Enter `go`. A FID should be obtained.

   If the FID is off-resonance, enter `gf` or `acqi` and adjust the probe tune/match to place the FID on-resonance.
4. Array `tof` from –1e4 to 1e4 in 1000 Hz steps.
5. Set `d1=20` and enter `ga`.

   The resultant displayed spectra should show a single resonance that moves with `tof`. Note that changing `tof` by 1000 Hz does not move the peak by 1000 Hz!
6. Write down the value of `tof` that gives the sharpest line. This is a guide for later use of the multipulse sequences.

Testing ^{1}H Wideline

The ^{1}H wideline test is more of a proof statement than a test with a quantitative result.

1. Use adipic acid as the sample. If using a CRAMPS probe, pack a rotor as detailed in “Preparing the Sample” on page 82 for the CRAMPS probe tests. If using a wideline probe, pack a 15 mm long piece of 5 mm NMR tube with about 3 mm in depth of adipic acid. Try to arrange this so that when placed in the probe, it is approximately in the center of the coil.
2. Install the probe and sample in the magnet. **Do not retune the probe.**
3. Change the following parameters from those used for the MREV8 test:
   `seqfil='s2pul' np=1e4 tof=0 nt=4 cp='y' d1=40 lp=0 rp=0 lb=250 ga`
4. When the experiment is finished, compare the resulting spectrum with Figure 36

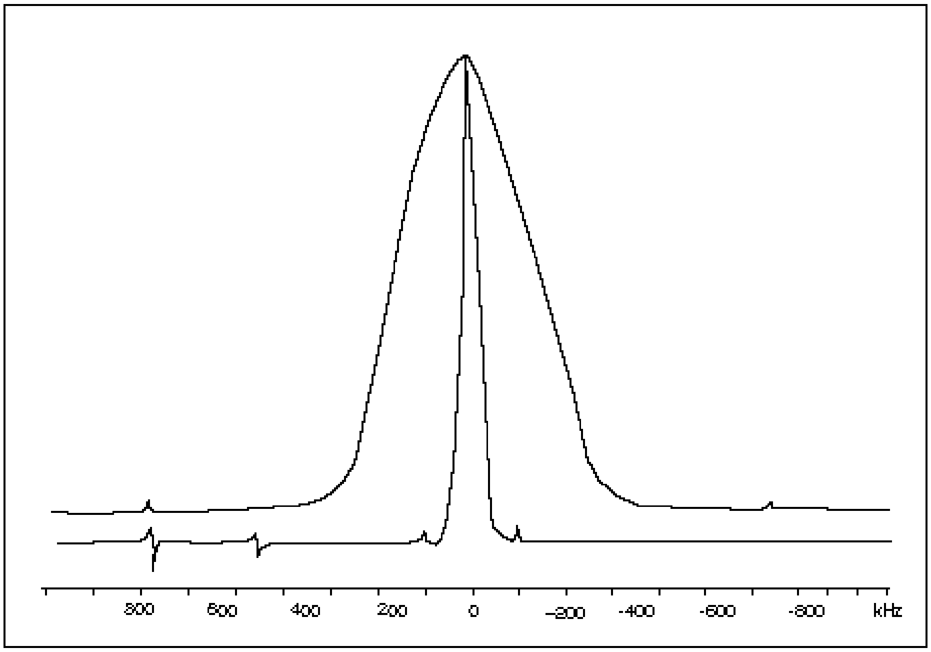


Figure 36. Adipic Acid Spectrum

The spectra should be similar. The lower trace of the wideline ^1H spectrum in Figure 36 is 2 MHz wide. The upper trace is an expansion showing the shape of the line.

Testing the CRAMPS Probe

Follow the two procedures in this section only if you are installing a Varian probe.

Preparing the Sample

The final tests are performed using a solid sample of adipic acid. This compound is a white powder. Sample preparation is crucial to obtaining the resolution specification. The sample must be packed centrally in the rotor and must be only 1 mm long; this is done as shown in the following steps.

1. Pack either a spacer or some inert material *with no protons* to the center of the rotor.
2. Tamp down this sample carefully. Then add a small amount of adipic acid and tamp the acid down.
3. Fill the rotor with more inert material and insert the cap.
4. Replace the benzene sample with the adipic acid sample. *Do not retune the probe*. Put the probe in the magnet and spin the sample at 1550 Hz.
5. Run the procedure given in the section "Using MREV8 to Demonstrate Multipulse Operation" on page 81.
6. Set `tof` to the best value as determined in the MREV8 procedure and set the following parameters: `np=512 tau=3.5 fn=4096 nt=1`.

7. Acquire a spectrum and check that it looks similar to the one in Figure 37.

   Note that the `dscale` values are wrong! Vary `tof` to get the best resolution of the right-hand peaks.
8. Place a cursor on each peak of the doublet and enter `scalesw=sfrq/delta` to set the scale factor.
9. Place the cursor on the right-most peak and enter `rl(2p)` to reference it. The spectrum should now have resonances at 2, 3, and about 14 ppm, as in Figure 37.
10. Save the FID for later reference, using `svf(`*`file`*`)`, where *`file`* is the name you select for the file to hold the FID.

Running the BR24 Sequence

The resolution specification is made using another multipulse sequence called BR24. After the MREV8 pulse sequence is working correctly, follow the steps below.

1. Enter `br24` to convert the parameters to run the BR24 pulse sequence.
2. Acquire a FID.
3. Display the FID using the `df` command and phase it so that the real channel has no on-resonance component. This is used later in step 5.
4. Transform the FID and check that the result is an adipic acid spectrum. Note that a quad image and a large zero spike appear. These are normal!
5. Array `tof` to obtain the best resolution doublet, while keeping all peaks on the same side of the center spike.
6. Set a cursor on each peak on the right-hand pair and enter `scalesw=sfrq/delta` to scale the spectrum.
7. Place the cursor on the right-most peak and enter `rl(2p)` to reference the spectrum. See Figure 38.

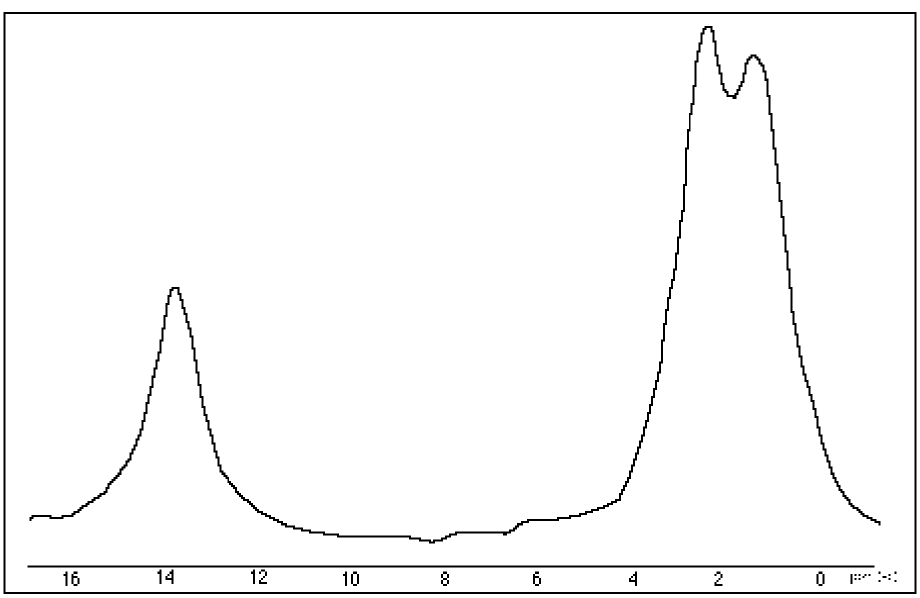


Figure 37. CRAMPS Adipic Acid Spectrum Using the MREV8 Pulse Sequence

Now you can measure the resolution by measuring the width at half height of either of the right-hand peaks. You must use the cursors to do the measurement because `dres` gives the wrong result. Save the data for future reference using the `svf(`*`file`*`)` command, where *`file`* is the name you select for the data file.

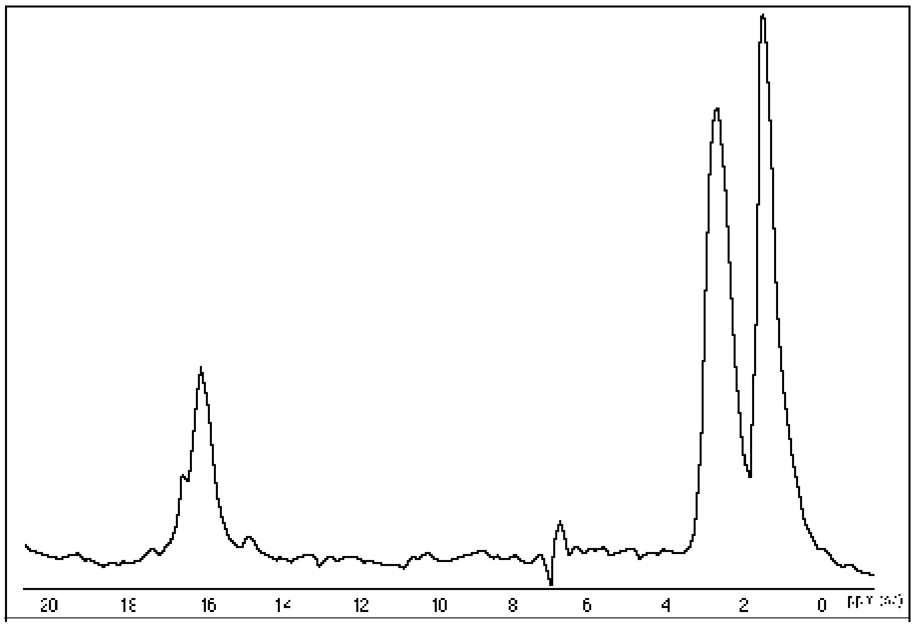


Figure 38. CRAMPS Adipic Acid Spectrum Using the BR24 Pulse Sequence

Chapter 8. Solid-State Variable Temperature System

This chapter covers the installation of the variable temperature (VT) system for solid-state NMR. The solid-state NMR VT system, shown in Figure 39, is used to cool or heat pressurized nitrogen used during VT CP/MAS experiments.

The solid-state VT system is composed of the following:

- VT transfer line hanger
- 50-liter dewar, a heat exchanger
- Booster power supply (Sorensen)
- Additional tubing connectors
- A cable to connect the Oxford VT controller to the power supply and probe
- VT CP/MAS pneumatics box

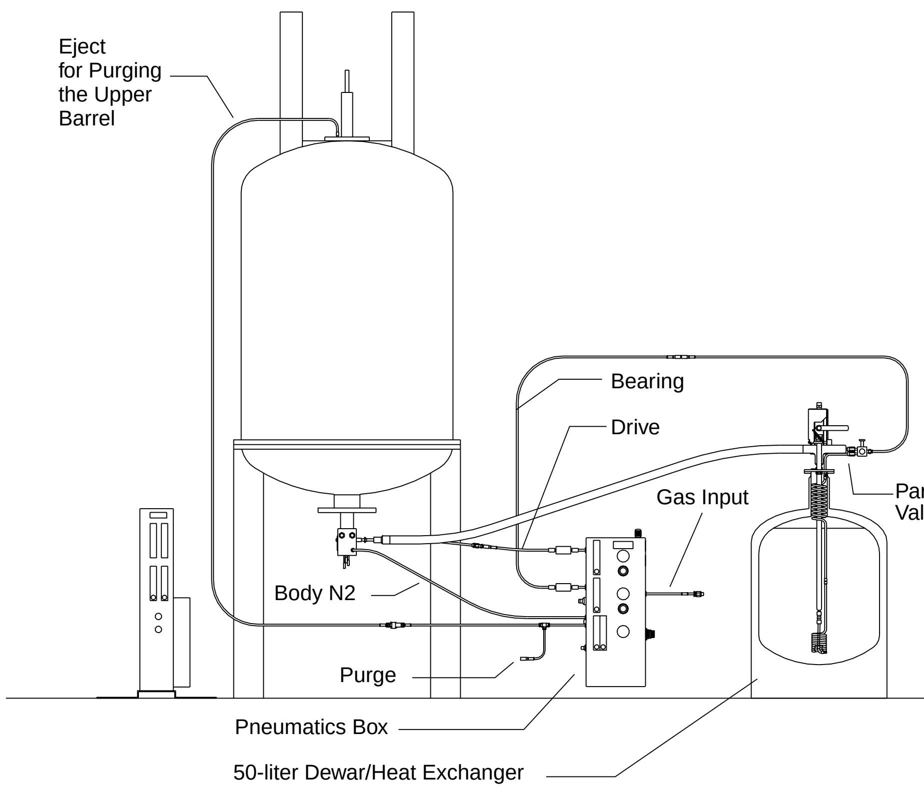


Figure 39. Solid-Sate NMR VT System Configuration

- VT CP/MAS probe
- VT CP/MAS probe start-up kit

An additional power supply is provided to boost the current supplied to the probe by the Oxford VT controller. The dewar acts as a reservoir for liquid nitrogen. The heat exchanger is in contact with the liquid nitrogen so that room temperature dry nitrogen can be cooled to around the boiling point of liquid nitrogen.

No fluid connection exists between the gas being cooled and the liquefied gas in the dewar. If the desired gas temperature is significantly higher than the boiling point of liquid nitrogen, the external gas that is to be cooled can be shunted so that part of the gas flows through the dewar and part of the gas bypasses the heat exchanger (see Figure 39), helping the liquid nitrogen last longer between refills. To prevent condensation of the pressurized room temperature gas being cooled, the liquid nitrogen coolant within the dewar is pressurized to raise its boiling point.

8.1 Installing the VT CP/MAS Pneumatics Box

Solids probes, such as the Varian VT CP/MAS probe, require several pneumatic inputs. These inputs are controlled by a pneumatics box, which also reads and displays solids sample spinning speeds. The pneumatics box controls the supply of dry gas to the input of the heat exchanger, which then supplies cold dry bearing N_2 to the probe. The pneumatics box also supplies dry N_2 for purging the probe and upper barrel. Typical purge flow rates are shown in the table below.

N2 Input	*Flow Rate (lpm)*
Body N_2	20–25
Upper barrel	5
Heat exchanger purge (standby condition)	2–5

The steps below describe how to install the VT CP/MAS pneumatics box, shown in Figure 40. Before starting, make sure that no experiments are in progress and the system is not in use.

1. Turn off the gas supply to the spectrometer.
2. Mount the pneumatics box to the magnet leg:
   - On Oxford magnets, the box is attached with two clamps and the height of the box is adjusted for convenience.
   - On Varian magnets, the box is attached with screws and the location of the box is fixed.
3. Attach the 3/8 in. supply line to the input to the pneumatics box.

CAUTION: **The VT system must be run ONLY with nitrogen gas, as specified in the *Installation Planning Guide*. Use of gases other than dry nitrogen gas will damage the equipment and create a possible safety hazard.**

4. Install the pneumatics box power supply. Connect the power supply to the box with the cable provided. Plug the power supply cord into a 120 V outlet.

5. Cut the EJECT line between the magnet leg and the magnet at a convenient place and attach the male-female, white-Delrin quick-disconnect connectors to the cut eject line (with the male on the bore side). Connect the EJECT line from the magnet to the EJECT port of the pneumatics box. During VT CP/MAS experiments, the EJECT line permits purging of the magnet bore.

   For room temperature operation, this connection is not necessary.

6. After verifying that all connections are correct, turn the gas supply back on

7. Adjust the drive and bearing flow meters so that they do not impede the flow of nitrogen.

   Always use the corresponding pressure regulators to control the drive and bearing gas—*never* adjust spin rates with the flowmeters.

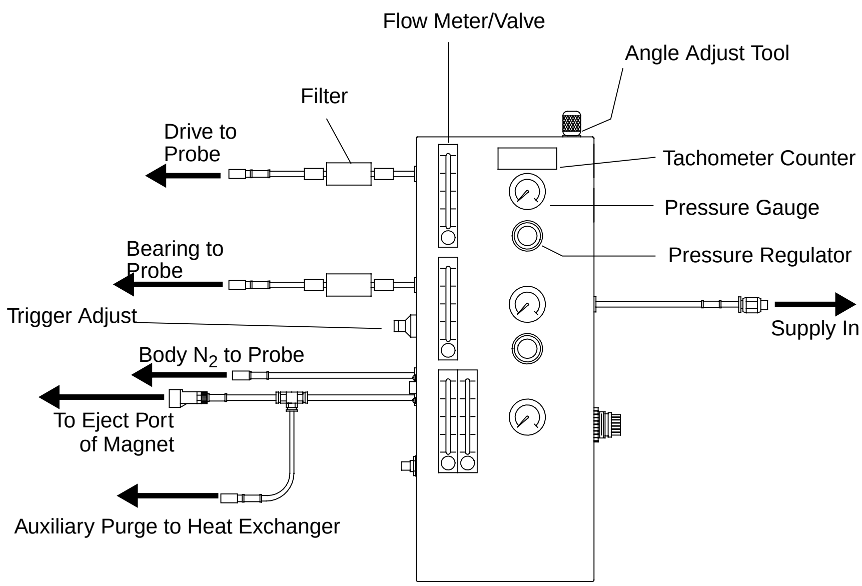


Figure 40. VT CP/MAS Pneumatics Box

8.2 Installing the 50-Liter Dewar and Heat Exchanger

The heat exchanger assembly requires dry nitrogen as an input. Before making any modifications to the system, be sure that no experiments are in progress, that the probe is disconnected from the VT system, and that the nitrogen gas supply is shut off.

1. If the magnet is equipped with a liquids VT bucket, the two gas lines going to the bucket should be cut about 1 ft away from the bucket. Install a male-female pair of quick-disconnect connectors in each line such that the male connectors are on the two lines leading to the bucket and the female connectors are on the two lines leading to the magnet leg.

   These connectors allow the original bucket to be interchanged with the 50-liter dewar for use with short-duration experiments.

2. Place the dewar near the magnet with the flexible arm oriented approximately perpendicular to the line connecting the center of the dewar with the center of the magnet, as shown in Figure 41. Be sure no undue forces be applied to the probe by the flexible line of the heat exchanger.

3. Bend the flexible arm, sliding the end through the hanger, and move the arm until the liquids adapter can be slipped over the probe dewar inlet. Allow the hanger to slip into both the corrugation closest to the hanger bracket and onto the hanger support.

4. Insulate the connection between the end of the flexible arm and the probe with some of the black closed foam.

   Note that two possible probe dewar diameters are available. The liquids adapter fits over the larger probe dewar with no modification. To fit over one of the smaller dewars, simply fold the rim of the liquids adapter over onto itself, making the inner diameter smaller.

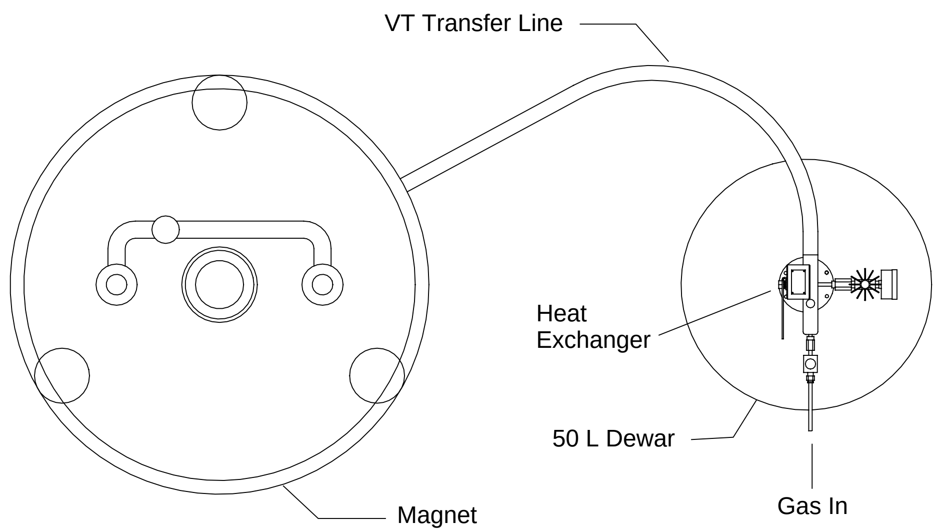


Figure 41. Approximate Orientation of Heat Exchanger Relative to The Magnet

5. If the liquids VT bucket is installed, simply detach the quick disconnects between the bucket and the magnet leg, and attach the bucket adapter to the magnet leg lines. Then attach the 50-liter dewar/heat exchanger to the bucket adapter. These connections are shown in Figure 42.

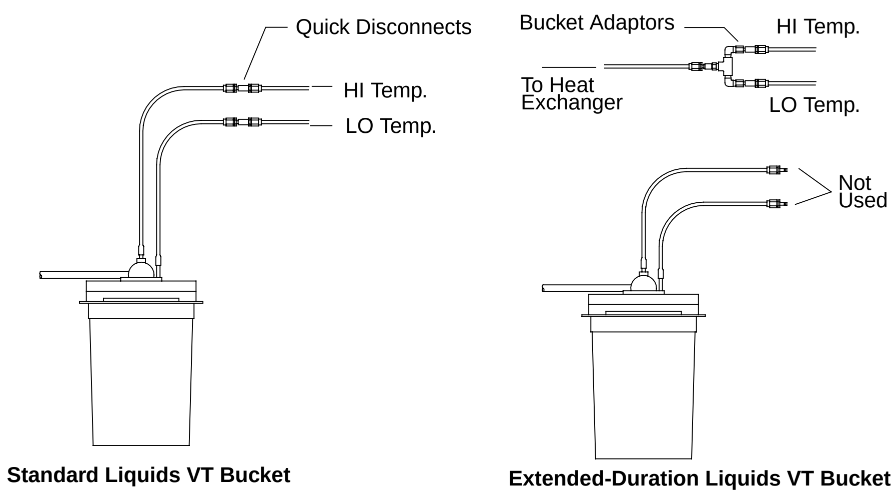


Figure 42. Standard Liquids VT Buckets Configurations

8.3 Installing the VT Transfer Line Hanger

The VT transfer line hanger is intended to support the vacuum-jacketed transfer line, thus decreasing the mechanical stresses that the line can put upon the probe.

Install the heat exchanger/dewar according to the instructions in the "Installing the 50-Liter Dewar and Heat Exchanger" on page 88.

To Install the VT Hanger on Oxford Magnets

Figure 43 shows the hanger support and hanger bracket that make up the transfer line hanger.

1. Find a convenient location for the transfer line hanger to be placed. Typically, the hanger is mounted on the same magnet leg that holds the liquids bucket, positioned slightly above the liquids bucket with the two screw holes on the right end of the support.
2. Attach the support to the leg using the U-shaped clamp, screws, and washers provided. Position the support on the outside of the leg and approximately tangent to the magnet.
3. Place a hanger bracket on the support. Two slightly different brackets are included, allowing a choice of mounting locations.
4. Insert the vacuum-jacketed transfer line into the bracket as follows:
   a. Place the dewar near the magnet with the flexible arm oriented approximately perpendicular to the line connecting the center of the dewar with the center of the magnet, as shown in Figure 41.
   b. Bend the flexible arm, sliding the end through the hanger, and allow the hanger to slip into the corrugation closest to the hanger bracket and onto the hanger support.

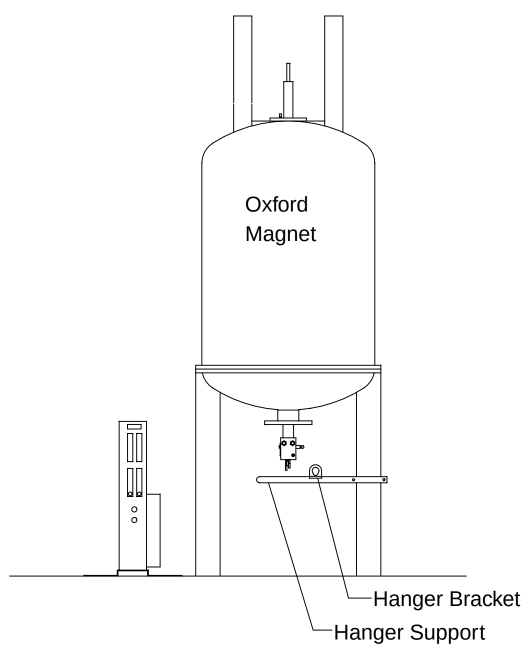


Figure 43. VT Hanger Installation on Oxford Magnets

c. Position the 50-liter dewar relative to the magnet such that no undue forces are applied to the probe by the flexible line of the heat exchanger.

The VT transfer line hanger is now installed.

To Install the VT Hanger on Varian Magnets with a Liquids Bucket

Figure 44 shows the hanger support and hanger bracket that make up the transfer line hanger.

1. Remove the styrofoam liquids bucket and verify that the upper two screws of the bucket mounting bracket are tight.
2. Remove the lower two screws and install the hanger bracket so that the hanger opening is located to the right when the magnet is viewed from the side on which the liquids bucket is installed.
3. Position the hanger bracket vertically so that the lower hanger hole is approximately in line with the probe dewar. Tighten the two screws.
4. Insert the vacuum-jacketed transfer line into the bracket as follows:
   a. Place the dewar to the right near the magnet with the flexible arm oriented approximately perpendicular to the line connecting the centers of the two magnet legs.
   b. Bend the flexible arm around the right-hand leg and insert the end of the arm through the hanger. *Be careful to avoid the coils of the liquids bucket*.
   c. Slide the arm through the hanger until the end is close to the probe and allow the hanger to slip into the corrugation closest to the hanger bracket.
   d. Position the 50-liter dewar relative to the magnet such that no undue forces are applied to the probe by the flexible line of the heat exchanger.

The VT transfer line hanger is now installed.

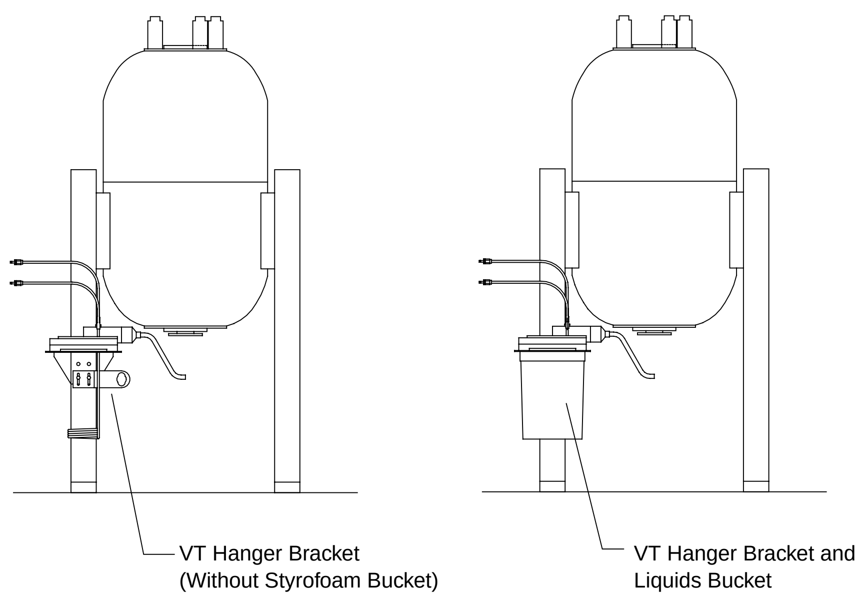


Figure 44. VT Hanger Bracket Installed on a Varian Magnet

To Install the VT Hanger on Varian Magnets without a Liquids Bucket

Figure 44 shows the hanger support and hanger bracket that make up the transfer line hanger.

1. Attach the hanger bracket to whichever side of the magnet the flexible arm of the heat exchanger is to approach from. The hanger should protrude outward from the surface of the magnet leg and toward the line perpendicularly bisecting the line between the two magnet legs. Use at least two screws, one in each slot.
2. Position the hanger bracket vertically so that the lower hanger hole is approximately in line with the probe dewar.
3. Tighten the two screws.
4. Insert the vacuum-jacketed transfer line into the bracket as follows:
    a. Place the dewar to the right near the magnet with the flexible arm oriented approximately perpendicular to the line connecting the centers of the two magnet legs.
    b. Bend the flexible arm and insert the end of the arm through the hanger.
    c. Slide the arm through the hanger until the end is close to the probe and allow the hanger to slip into the corrugation closest to the hanger bracket.
    d. Position the 50-liter dewar relative to the magnet such that no undue forces are applied to the probe by the flexible line of the heat exchanger.

The VT transfer line hanger is now installed.

8.4 Filling the 50-Liter Dewar

Nitrogen gas should be flowing through the heat exchanger at all times that liquid nitrogen is within the 50-liter dewar.

During the initial installation of the solids VT system, positioning the 50-liter dewar is easiest before it is filled. Once positioned, you may want to mark the location on the floor.

WARNING: **Fire or explosion can result when air is used for probe cooling—condensed oxygen can be explosive. Do not use air for probe cooling.**

1. Purge the heat exchanger as follows:
    a. Purge the gas lines with dry nitrogen to prevent atmospheric oxygen from liquefying within the lines and becoming a safety hazard.
    b. Connect the purge valve of the heat exchanger to the VT CP/MAS probe.
    c. Turn on the room temperature gas supply and push the valve button into the full down position, as shown in Figure 45.

       When the button is in the down position, all gas is shunted through the heat exchanger. When the button is in the up position, all gas bypasses the heat exchanger. Valve positions between the full up or full down positions allow proportional control while maintaining a nearly consistent flowrate. The valve shunts a portion of the gas through the heat exchanger and bypasses the rest around it. The two paths are later combined.

WARNING: **Blindness or frostbite can result from contact with cryogens. Observe all appropriate safety precautions when using cryogenic liquids—use eye protection and wear thermo-insulated gloves at all times!**

2. Fill the dewar with liquid nitrogen as follows:

   a. Make sure a positive flow of room temperature nitrogen is purging the heat exchanger when the dewar is being filled with liquid nitrogen. *If air is allowed to enter the heat exchanger, the oxygen condenses out and becomes a safety hazard*. The purge line of the pneumatics box can be used to avoid this hazard.

   b. Check the pressure gauge to verify that the dewar is unpressurized.

   c. Carefully crack the ball valve slightly. **Beware of residual pressure**. If the dewar has some residual pressure, the gas vents against the safety gate (see Figure 46). Let the residual gas vent to atmospheric pressure.

   d. After all gas has escaped, move the safety door aside and insert the filltube (Part No. 00-991929-00) into the ball valve of the heat exchanger (valve handle must be "up" as shown in Figure 45) and attach the liquid nitrogen source to the filltube.

      This filltube is designed to prevent overfilling the 50-liter dewar. An ice suppressor assembly and a vent that feeds dewar gas to the pressure gauge reside inside the dewar. If liquid nitrogen reaches this vent, the pressure gauge and relief valve can freeze, creating a possible safety hazard. As the dewar fills, exhaust gas exits through the sidearm portion of the filltube. This sidearm should be positioned such that the cold exhaust gas exits safely. Note that for most extended-duration liquids work, the ball valve is left in the "open" position, leaving the dewar unpressurized. If the valve is closed, the pressure within the dewar rises to approximately 30 psig. When unpressurized, the output gas temperature is lower than when pressurized.

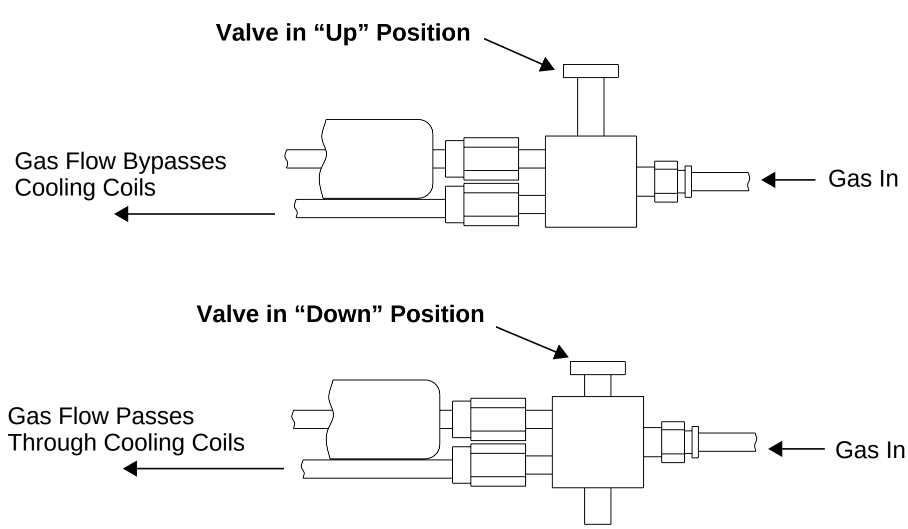


Figure 45. Partition Valve Positions

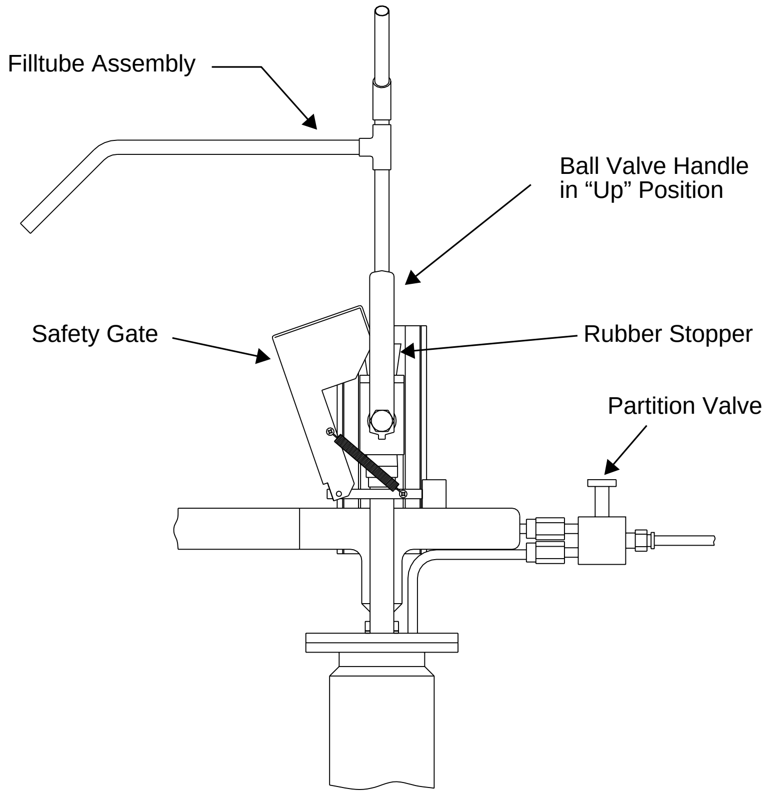


Figure 46. Assembly for Filling the Dewar With Liquid Nitrogen

8.5 Testing the Solid-State VT System

The Varian VT CP/MAS probe allows control of the sample temperature by heating the bearing nitrogen gas during VT operation. The drive nitrogen is not heated and, therefore, its input and output remains at ambient temperature.

CAUTION: **To avoid "burning out" the heater, ALWAYS provide bearing flow to the probe before turning on the main switch of the Sorensen power supply. Likewise, the Sorensen power supply MUST be off before decreasing the flow of bearing gas to the probe.**

1. For VT CP/MAS experiments, the dewar must be pressurized. Close the valve handle and allow the nitrogen reservoir to pressurize. The safety gate must be in place above the valve. Use caution when releasing pressure.
2. Make sure that 20–25 lpm of body nitrogen is flowing through the probe.
3. Prepare the system for use as follows:
   a. Place the dewar near the magnet with the flexible arm oriented approximately perpendicular to the line connecting the center of the dewar with the center of the magnet. See Figure 41.
   b. Bend the flexible arm, sliding the end through the hanger, and move the arm until the brass ball socketed end can mate with the probe dewar inlet.
   c. Allow the hanger to slip into the corrugation closest to the hanger bracket and onto the hanger support.
   d. Make sure that the location and orientation of the 50-liter dewar relative to the magnet is such that no undue forces be applied to the probe by the flexible line of the heat exchanger.
   e. To release pressure, carefully crack the ball valve slightly. **Beware of residual pressure**. If the dewar has some residual pressure, the gas vents against the safety gate (see Figure 46). Let the residual gas vent to atmospheric pressure.
4. On the VT CP/MAS pneumatics box, plug the DRIVE quick disconnect into a ball joint connector. Attach the ball end to the DRIVE input to the probe using the plastic ball joint clamp. *Do not apply any drive pressure to the probe at this time*.
5. Plug the BEARING connector of the pneumatics box to the input to the heat exchanger. Disconnect the purge line from the input to the heat exchanger.
6. To remove any ice that might have collected at the output, Lift the partition valve to the full "up" position and then wipe off the tip of the transfer line.
7. Push the switch to the half-way position, allowing some nitrogen bearing gas to pass through the heat exchanger and some to bypass the heat exchanger. Then connect the BEARING glass ball joint of the probe to the brass output port of the heat exchanger. Adjust the bearing pressure to between 2–2.5 Bar. Check that sufficient bearing gas is flowing through the probe. After connecting the glass ball joint to the drive port, adjust the drive pressure so that the spin rate is between 2.0 and 5.0 kHz.
8. Enter `dg` to display parameters on the monitor, and then enter `temp='n'` to put the VT controller in the `NOT USED` mode.

9. Enter `config` and check the value of the parameter `vttype`. If `vttype` is set to `0`, the controller is off, if it is set to `2`, the controller must be active.

10. Check that the line voltage selector is set correctly for the local power for the Oxford VT controller. Insert the input power connector and connect it to an appropriate power source. Connect the main input power connector, set the input power circuit breaker to ON. Wait 10 seconds to allow the system to stabilize and then boot up the system. On the VT controller, press the power switch to the ON position.

    The input power receptacle assembly contains the voltage selector switch and live fuses. The fuses are located behind the cover, which can be opened with a screwdriver in the slot at the top of the cover. Spare fuses are located inside the VT controller cabinet. The fuse ratings are 800 mA for 240 V input and 1.6 A for 120 V input.

11. Connect the Oxford VT cable to the Sorensen amplifier VT cable, as shown in Figure 47.

    The Sorensen DC power supply is set up and tested for operation at 100 Vac. Depending on the installation site, transformers T1 and T2 might have to be retapped according to the available line voltage. Make sure NEON DS1 remains at 110 V. Fuse F2 should be changed accordingly. Label the back panel of the power supply to show the actual operating voltage.

    If the Sorensen DC power supply is shipped with its own cabinet (Part No. 00-969330-01), make sure the power supply is mounted so that the filler panel on the bottom of the cabinet. This ensures sufficient cooling air from the bottom side. The bottom of the Sorensen is more important for cooling than the top.

12. Connect the Sorensen amplifier VT cable to the probe.

    You can connect this cable in series with the existing liquids VT cable. Connect the output of the existing liquids VT cable to the input to the Sorensen-interfaced solids VT cable. Then connect the output of the solids VT cable to the probe.

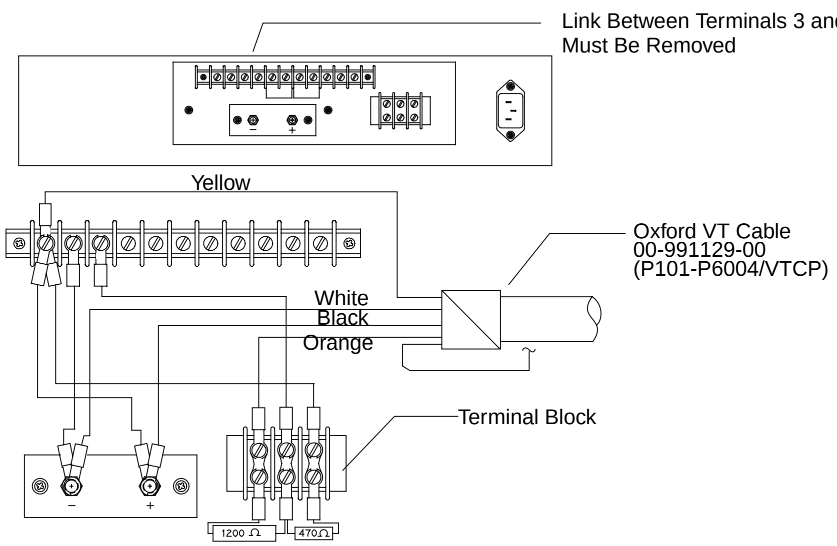


Figure 47. Connection Diagram for Oxford VT Cable and Sorensen Power Supply

13. Turn on the power to the Oxford VT controller and monitor the temperature of the probe. Turn on the power to the Sorensen amplifier. The Sorensen amplifier power supply provides four adjustable knobs —coarse voltage, fine voltage, coarse current, and fine current. The course voltage, fine voltage, and fine current knobs should be turned fully clockwise. The course current knob should turned so that the white dot is in the up, or “12-o’clock” position.
14. Before entering a temperature value for the `temp` parameter, check the `masvt` parameter. The `masvt` parameter identifies the type of variable temperature system in use. Two systems are currently available: the standard Oxford VT controller and the Oxford-Sorensen or solids VT controller system (used with the Varian VT CP/MAS probe). To select the Varian solids VT system, set the parameter `masvt='y'`. If `masvt` is set to anything other than `'mas'`, or if the parameter does not exist, the Oxford Varian VT system is assumed. To reset the system to the Oxford VT system, set `masvt=''`.
15. The parameter `masvt` is a “global” parameter. That is, it is active for each user account and it is active for all of that user's experiments. `masvt` is provided with VNMR version 4.1 or later and offers stable temperature control for VT CP/MAS probe. The current value of the parameter can be displayed in the normal way by entering `masvt?`. Note that the VT Controller option displayed by `config` must be set to Present for either VT controller system to be active. If `masvt` does not exist, it can be created with the command `create('masvt','string','global')`.
16. Adjust the partition valve downward until the temperature of the probe is 10 to 20 C below the lowest desired sample temperature. Allow the temperature to stabilize for about 5 minutes.

    As in liquids VT, the temperature is controlled by the `temp` parameter; for example, to enter a temperature of 25 Cset `temp=25 su`. If the temperature of the feedback thermocouple in the probe is much lower than the set temperature, allow sufficient time for the VT system to begin regulating.
17. Monitor the Sorensen output current and the temperature of the probe to verify that the heater is operating. Allow sufficient time for the system to begin regulating. Depending on the set temperature, thermocouple temperature, and input gas temperature, the time required for the system to regulate will vary.
18. When the system is regulating, allow sufficient time for the entire probe head to achieve thermal equilibrium. Adjust the probe tuning and match capacitors as necessary.
19. To change the temperature, set the `temp` parameter to the desired temperature and enter `su`. *Do not change the temperature by increments of more than 20 Cat a time*.
20. For high temperature (above room temperature), removing the heat exchanger out of line is optional. If the heat exchanger is left in line, move the partition valve to the full up position. You must, however, move the partition valve to the full up position.

Refilling the 50-Liter Dewar

WARNING: **When in use, the dewar contents are under pressure. Do not attempt to open the ball valve while the dewar is in use, since cracking the ball valve allows the cold pressurized gas to vent upward and severe frostbite can result. Opening the valve during an experiment may cause the condensation of the pneumatics gas feeding the probe, resulting in unpredictable spinning.**

The normal refill procedure is to stop the experiment and depressurize the dewar. Carefully and slowly, open the ball valve, allowing any residual pressure to vent. When the dewar is completely depressurized, fill the dewar as outlined above in the section "Filling the 50-Liter Dewar."

Measuring Sample Temperature

Verify sample temperature accuracy either by measuring the peak separations in known samples or by using a thermocouple at the sample position in the probe.

A set of charts in the manual *UNITY-Series Accessory Installation* shows the ^{1}H chemical shift for ethylene glycol and methanol at 200 MHz and 300 MHz. For 400 MHz, double the 200 MHz chemical shift. For 500 MHz, add the corresponding 200 MHz and 300 MHz chemical shifts. For 600 MHz, double the 300 MHz chemical shift. In order to calibrate the VT temperature on the broadband probe by the NMR method, the ^{1}H spectra of the calibration samples can be obtained by using the probe decoupler coil for the observe function. The accuracy of this calibration method as it relates to spinning solids is questionable. The user should be aware of this uncertainty and should develop temperature measurement methods pertinent to their own research.

For the VT CP/MAS probe, proton signals can be observed by connecting the observe cable to the decoupler port on the probe. The temperature calibrating compound can either be poured directly into the CP/MAS rotor or placed in a small, spherical bulb that is then inserted into the spinner.

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