

ARTISAN®
TECHNOLOGY GROUP

Your **definitive** source for quality pre-owned equipment.

Artisan Technology Group
(217) 352-9330 | sales@artisantg.com | artisantg.com

Full-service, independent repair center
with experienced engineers and technicians on staff.

We buy your excess, underutilized, and idle equipment
along with credit for buybacks and trade-ins.

Custom engineering
so your equipment works exactly as you specify.

- Critical and expedited services
- In stock / Ready-to-ship
- Leasing / Rentals / Demos
- ITAR-certified secure asset solutions

Expert team | Trust guarantee | 100% satisfaction

All trademarks, brand names, and brands appearing herein are the property of their respective owners.

Visit our website - *Click HERE*

Indirect Detection NMR Probe Installation

Varian NMR Spectrometer Systems

Pub. No. 01-999123-00, Rev. E0501

VARIAN

VARIAN

Pub. No. 01-999123-00, Rev. E0501

Indirect Detection NMR Probes

Installation, Testing, and Specifications

- Overview, page 2
- Safety Precautions, page 3
- Installing the Probe, page 3
- Connecting to UNITY-Series, page 6
- Connecting to MERCURY-Series, page 7
- Tuning the Probe, page 8
- Returning to Normal Operation, page 9
- Tuning Ranges for Indirect Probes, page 9
- Testing Indirect Detection Probes on UNITYINOVA, page 11
- Testing Indirect Detection Probes on MERCURY-Series, page 20
- Specifications for Indirect Detection Probes, page 31

Overview

Designed for inverse detection experiments, indirect detection probes have optimal proton sensitivity and capability for decoupling irradiation for a broadband frequency range.

Magnet (MHz)	*Bore (mm)*	*Sample Size (mm)*	*Range*	*Probe Part No.*
300 PFG	54	5	$^{1}H\{^{15}N–^{31}P\}$	00-901980-00
300 PFG	89	5	$^{1}H\{^{15}N–^{31}P\}$	00-901981-00
400 PFG	54	5	$^{1}H\{^{15}N–^{31}P\}$	00-993520-01
400 PFG	89	5	$^{1}H\{^{15}N–^{31}P\}$	01-901583-01
500 PFG	54	5	$^{1}H\{^{15}N–^{31}P\}$	00-993373-03
600 PFG	54	5	$^{1}H\{^{15}N–^{31}P\}$	00-993374-03

The $^{1}H\{^{15}N–^{31}P\}$ indirect detection probe is specifically designed for demanding applications in high-field NMR, for example, heteronuclear 2D, 3D, and 4D experiments.

The PFG version of the Indirect probe adds rapid recovery gradients, up to 60 G/cm along the Z axis, to the probe features. The PFG probe requires the Performa I or the Performa II Pulsed Field Gradient module (Part No. 00-901858-00 for Performa I or 00-993854-00 for Performa II).

To achieve the level of performance required for these specialized experiments, the probe has been optimized in the following ways:

- *High-power handling* – The probe can typically handle the full pulse power (200–300 W) delivered by a spectrometer for up to a 180° pulse for nuclei between ^{15}N and ^{13}C. The probe is not optimized for ^{31}P and must be operated at ^{31}P with the reduced power (you can use `tpwr < 54` as a guideline) and a 90° pulse width longer than 30 µs.

  For longer pulses (for example, a composite 360° pulse), the power should be decreased (2 to 4 dB) to minimize the chance of arcing. The amount of power reduction required is best determined by experimentation. The probe is not usually damaged by short pulse arcing (under 80 µs), but arcing during a long pulse can damage the probe.
- *Spin-locking conditions* –

Channel	*Gamma B_x*	*Duration*	*Approximate Power*
^{13}C	9 kHz	30 ms	30 W
^{15}N	4 kHz	30 ms	30 W

- *Decoupling conditions* –

Channel	*Gamma B_x*	*Duration*	*Approximate Power*
^{13}C	7 kHz	150 ms	15 W
^{15}N	2 kHz	150 ms	7.5 W

- *Tuning* – Proper tuning is vital to short pulse width performance. (Always tune the X channel with no filter in line.)

- *Tuning range* – The broadband channel (labeled X CHANNEL on the probe) can tune to nuclei between ^{15}N and ^{31}P. A capacitor stick is provided to tune to ^{15}N and an inductor stick to tune to ^{31}P. When turning the X channel on 300-MHz PFG Indirect probes, the familiar dip behavior for minimum reflected power may not be achievable on the tune meter for ^{15}N and ^{31}P. For these cases, tune the probe to the lowest meter value without forcing the matching capacitor (smooth knob) beyond its range. Adjustment to this meter value provides the maximum rated rf performance at these frequencies.

Safety Precautions

CAUTION: **The Indirect probe can be damaged by high power. Be extremely careful when applying spin-locks or high-power decoupling to the probe. Also, arcing during a long pulse can damage the probe.**

CAUTION: **Do not force tuning capacitors to turn past the end of their ranges. The end of the range is indicated by an increase in torque and not necessarily a stop to the rotation. Forced turning can cause serious mechanical damage to the probe, requiring factory repair.**

Installing the Probe

1. Make sure the upper barrel and probe mounting flange are installed and adjusted for the probe.
   - The upper barrel should be seated against the room-temperature shim coil.
   - The probe flange set screws should be backed off from the probe shield, allow the flange to slide (with some effort) along the shield.

   Probes used with widebore (89-mm) magnets should be fitted with the widebore adaptor (a white plastic ring shipped in the probe case). Position the adaptor at the appropriate midpoint of the probe shield and secure in place with the three set screws. Placing the adaptor too close to the NMR coil region causes lineshape difficulties.
2. Insert the probe into the bottom of the magnet dewar.
3. Push the probe up until contact is made with the upper barrel.
4. While continuing to apply pressure, rotate the probe until the probe shield key engages; this aligns the PFG pins with the sockets in the upper barrel.
5. Continue to push the probe up until the pins engage and the upper barrel rises slightly (this ensures a good seal between the upper barrel and probe).
6. Secure the probe flange to the magnet with the thumbscrews.
7. Press the upper barrel down until it seats into the room-temperature shim coil.
8. Tighten the three set screws in the probe flange against th probe shield. Overtightening the set screws will dent the probe shield.

Connecting to UNITY*INOVA 300 & 400-MHz*

Install the probe as described in Installing the Probe, page 3. Then, connect the PFG Indirect probe to a300 or 400-MHz UNITY*INOVA* system as follows:

1. Insert the probe into the bottom of the magnet room temp shim coil.
    a. Rotate the probe radially until it engages with the key alignment of the PFG upper barrel, and push it up so the upper barrel rises slightly.
    b. Secure the probe with the two thumb screws on the probe flange.
    c. Push the upper barrel down until it mates the room temp shim coil.
    d. Tighten the three set screws in the probe flange against the probe shield. *Overtightening the set screws will dent the probe shield.*
2. Connect the probe as follows (see Figure 1):
    a. Connect the probe PROTON channel to the PROBE (J5301) connector on the 1H/19F PREAMP.
    b. Connect the probe lock channel to the appropriate lock bandpass filter listed in the following table. The lock phase changes after the filter is added. The lock bandpass can remain in the system; however, it causes about 20% loss in lock sensitivity.

Magnet (MHz)	*^{2}H Bandpass Filter*
300	BE46-4-6BB
400	BE61-10-8BB

Filter model numbers describe the filter function. For example, the model number BE61-10-8BB, *B* indicates bandpass, the *61* indicates the center of the frequency of the filter, *10* is the width of the filter and *8* is the slope of the filter response per decade frequency.

c. Choose one of two possible configurations:

Using the broadband preamp in series with the decoupler output — Use this configuration for systematic X nuclei decoupling and observation. Connect the probe X Channel with the BNC cable to the appropriate bandpass filter. Connect the other end of the band pass filter with a BNC cable to the broadband preamp connector PROBE (J5311). Connect the correct quarter wave cable to the broadband preamp WAVELENGTH connectors for maximum rf transfer to the probe and preamp protection.

Bypassing the broadband preamp — Use this configuration to provide maximum decoupling power to the probe X channel. Since this setup is for Proton observation indirectly X decoupling experiments, X nuclei observation must be done by using the above configuration. Connect the probe X Channel with the BNC cable to the appropriate bandpass filter. Connect the other end of the band pass filter with a BNC cable to the switchable relay BB's NC (KJ5020).

Magnet (MHz)	*^{13}C Bandpass Filter*	*^{15}N Bandpass filter*
300	BE75-15-8BB	BE30.4-7.6-9BB
400	BE109-22-8BB	BE40-14-9BB

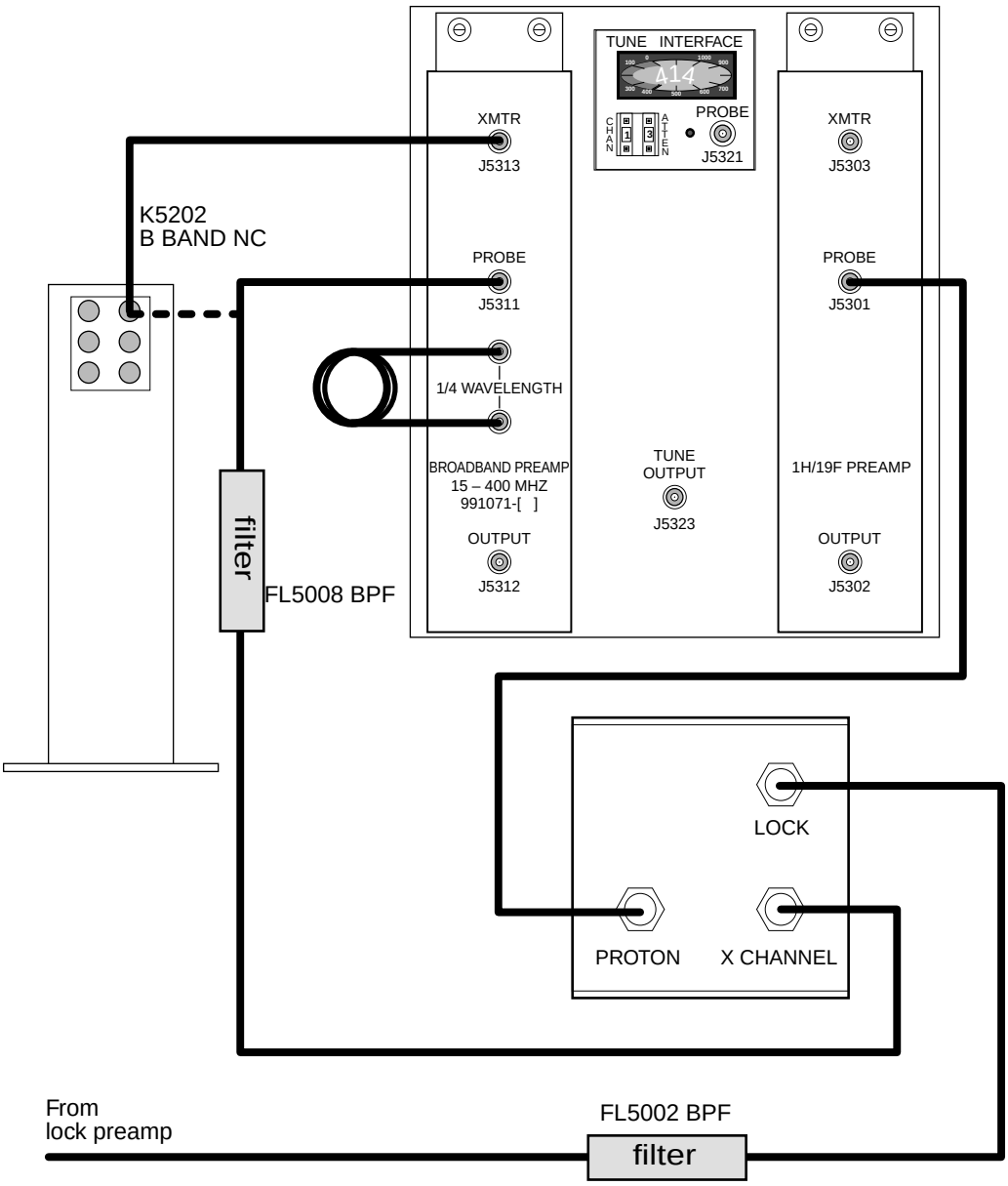


Figure 1. Probe Connection Diagram

3. Connect the nitrogen VT air line to the glass dewar projecting to the side at the base of the probe.
4. Connect the body-cooling air line to the hose bard from the bottom of the probe.
5. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.

Connecting to UNITY-Series

Install the probe as described in Installing the Probe, page 3. Then, connect the PFG Indirect probe to a UNITY-series systems as follows:

1. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided.
   a. Push the probe up until contact is made with the upper barrel.
   b. While continuing to apply pressure, rotate the probe until the probe shield key engages; this aligns the PFG pins with the sockets in the upper barrel.
   c. Continue to push the probe up until the pins engage and the upper barrel rises slightly (this ensures a good seal between the upper barrel and probe).
   d. Secure the probe flange to the magnet with the thumbscrews.
   e. Press the upper barrel down until it seats into the room-temperature shim coil.
   f. Tighten the three set screws in the probe flange against th probe shield. Overtightening the set screws will dent the probe shield.
2. In the lock channel line, install the appropriate ^{2}H bandpass filter listed in the following table. Expect the lock phase to change upon addition of the filter. Note that the ^{2}H bandpass filter can be left in the system at all times; however, it causes about 20% loss in lock sensitivity.

Magnet (MHz)	*^{2}H Bandpass Filter*
300	BE46-4-6BB
400	BE61-10-8BB
500	BE77-3.8-8BB
600	BE95-12-8BB

   Filter model numbers describe the filter's function. For example, for model number BE77-3.8-8BB, the 77 indicates 77 MHz and "B" indicates bandpass—an "L" instead would indicate low-pass.
3. Connect the low-band decoupler cable according to the appropriate procedure below. Choose the appropriate bandpass filters from the following table:

Magnet (MHz)	*^{13}C Bandpass Filter*	*^{15}N Bandpass filter*
300	BE75-15-8BB	BE30.4-7.6-9BB
400	BE109-22-8BB	BE40-14-9BB
500	BE135-35-8BB	BE53-15-8BB
600	BE154-40-8BB	BE61-10-8BB

   - *300-MHz systems* – Connect a cable from the magnet leg B-Band Probe port (UNITY*INOVA* or UNITY*plus*), B-Band port (UNITY), or the Probe NORMAL port (VXR) to the appropriate bandpass filter. Then, attach a cable from the bandpass filter to the port labeled X CHANNEL.
   - *400-MHz systems* – Connect a cable from the B Band port, or the Probe NORMAL port on the magnet leg, to the appropriate bandpass filter. Then attach a cable from the filter to the probe port labeled X CHANNEL.

- *500-MHz systems with a $^{1}H/^{19}F$ OBS preamplifier* – Connect a cable from the Decplr or NORMAL port to the appropriate bandpass filter. Then attach a cable from the filter to the probe port labeled X CHANNEL.
- *500-MHz systems with a dual preamplifier* – Connect a cable from the B Band CM port to the appropriate bandpass filter. Then connect a cable from the filter to the probe port labeled X CHANNEL.
- *600-MHz systems* – Connect the appropriate bandpass filter to the cable labeled "Indirect Detection" or "L Band Decpl." Then connect a cable from the filter to the probe port labeled X CHANNEL

4. Connect the ^{1}H channel according to the appropriate step below:
   - *300-MHz systems* – Connect a 1H high-pass filter to the magnet leg H-Band Probe port (UNITY*INOVA* or UNITY*plus*), to the H-Band port (UNITY), or to the 1H/OBS/DEC port (VXR). Then connect a cable from the filter to the PROTON port on the probe.
   - *400-MHz systems* – Connect a ^{1}H high-pass filter to the H Band port or to the 1H/OBS/DEC port. Then connect a cable from the filter to the PROTON port on the probe.
   - *500-MHz systems with the $^{1}H/^{19}F$ OBS preamplifier* – Connect a ^{1}H high-pass filter to the PROBE port (J5301) on the preamplifier. Then connect the a cable from the filter to the PROTON port on the probe.
   - *500- and 600-MHz probes with the dual preamplifier* – Connect a ^{1}H high-pass filter to the H BAND CM or the PROBE port (J5301) on the preamplifier. Then connect a cable from the filter to the PROTON port on the probe.
5. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.
6. Connect the nitrogen VT gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe.
7. Connect the body-cooling air hose to the hose barb protruding from the bottom of the probe.

Connecting to *MERCURY*-Series

Install the probe as described in Installing the Probe, page 3. Then connect the cables as described below.

Connecting the Cables for High-Band (^{1}H) Operation

1. In the lock channel line, install the ^{2}H bandpass filter; expect the lock phase to change upon addition of the filter. (The ^{2}H bandpass filter *can* be left in the system at all times; it causes, however, about 20 percent loss in lock sensitivity.)
2. The observe cable now carries X nucleus signals. Accordingly, attach the observe cable to the ^{13}C connector (J5302), insert an X-nucleus bandpass filter to the other end, and then connect the filter to the probe connector marked {x} DECOUPLE.

3. Install the appropriate quarter-wavelength cable on the broadband preamplifier. Connect the proper cable for the observe frequency, as indicated in the following table, to J5304 and J5305 on the left magnet leg.

Frequency (MHz)	*Cable (Color)*
44–178	Yellow
37–118	Black
11–45	Blue

4. Connect the Probe connector of the proton preamplifier to a ^{1}H high-pass filter and connect the filter to the connector of the probe marked 1H OBSERVE.
5. If the VT option is present, connect the cable from VT controller to the 9-pin connector (VT Control) on the probe, and connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe.
6. Connect the body-cooling air hose to the hose barb on the bottom of the probe and adjust the air flow to optimize sample insertion, ejection, and spinning.

Connecting the Cables for Low-Band (Broadband) Operation

The indirect detection probe is also capable of being used as a conventional broadband probe with X-nucleus observe and ^{1}H decoupling. Connecting the cable for low band should be the same as the high-band operation cable connections described above—the observe signal should be connected to the probe connector marked {x} DECOUPLE and the decoupler signal should be connected to the probe connector marked 1H OBSERVE.

Tuning the Probe

The Indirect Detection probe performs best when the sample to be analyzed is placed in the probe during the tuning operation. For best probe performance, follow the ^{1}H channel and X channel tuning procedures below every time you change samples.

The Indirect probe, pictured in Figure 2, incorporates a numerical counter system and probe label on the broadband channel that corresponds to the tuning of the nuclei to be decoupled/observed. Counter values are established for each probe, without a sample. The knurled tune knob changes the values on the numerical counter, while the smooth knob operates the matching capacitor. The lower the frequency of the nucleus, the more the smooth match knob needs to be turned clockwise.

Tuning the ^{1}H Channel

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the red PROTON inset label turns the tuning capacitors, while the smooth lower knob turns the match capacitor (see Figure 2).

Tuning the X Channel

Turn the knurled X CHANNEL knob, shown in Figure 2, to a numerical counter value that corresponds to the nucleus to be observed. The label on your probe shows four popular

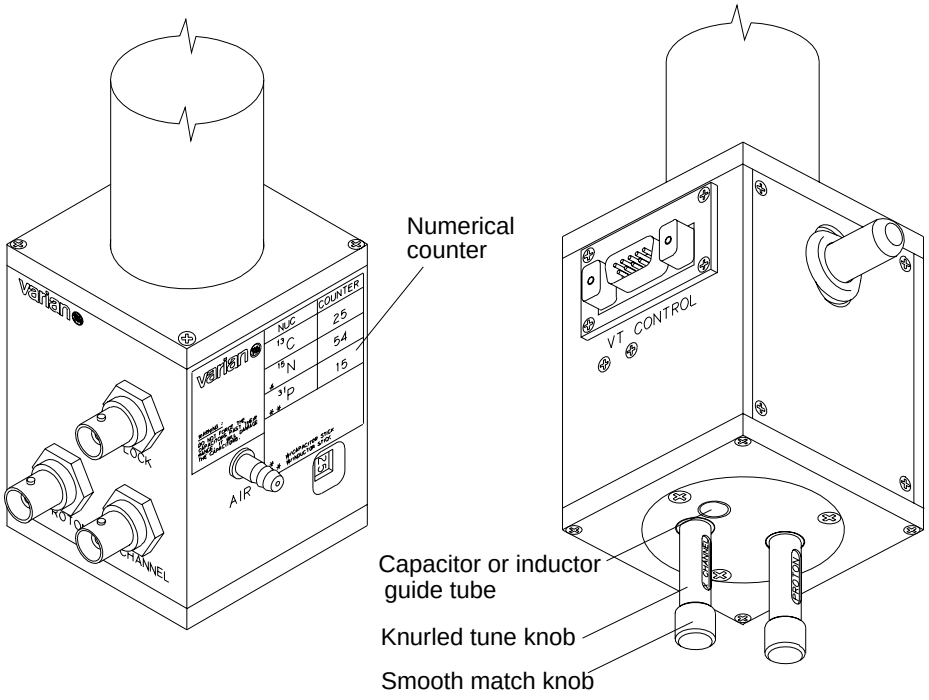


Figure 2. Indirect Detection Probe Tuning Controls

nuclei. Then, to fine tune, alternately adjust the smooth and knurled X CHANNEL match and tune knobs to find the minimum reflected power on the tune meter.

When turning the X channel on 300-MHz PFG Indirect probes, the familiar dip behavior for minimum reflected power may not be achievable on the tune meter for ^{15}N and ^{31}P. For these cases, tune the probe to the lowest meter value without forcing the matching capacitor (smooth knob) beyond its range. Adjustment to this meter value provides the maximum rated rf performance at these frequencies.

Returning to Normal Operation

After tuning the probe, enter **`dm='n' su`** to make sure the decoupler is not inadvertently left on. Check ^{1}H channel tuning after X-channel tuning as well as after a sample change.

Tuning Ranges for Indirect Probes

Use the following tables to select a capacitor or inductor stick for each tuning range (note that some sticks are optional equipment).

Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Tuning Ranges for 300-MHz PFG Indirect Detection Probes

Capacitor Stick (pF)	*Range (MHz)*	*Inductor Stick (Turns)*	*Range (MHz)*
none	60–78 (^{13}C, ^{29}Si)	12	95–128 (^{31}P)
3	51.9–67		
10	44.5–52		
14.3	42.8–48		
21	37.8–41		
26	35–37.8		
30	33.3–35		
33	32.8–34.8		
43	30.2–32		

Tuning Ranges for 400-MHz PFG Indirect Detection Probes

Capacitor Stick (pF)	*Range (MHz)*	*Inductor Stick (Turns)*	*Range (MHz)*
none	75–102 (^{13}C)	7	110–190 (^{31}P)
9	60–74		
14	54–61		
17	54–56		
23	50–55		
27	47–54		
30	45–49		
36	42–46		
43	40.4–42.4 (^{15}N)		

Tuning Ranges for 500-MHz PFG Indirect Detection Probes

Capacitor Stick (pF)	*Range (MHz)*	*Inductor Stick (turns)*	*Range (MHz)*
none	78–133 (^{13}C)	8	130–210 (^{31}P)
9	70–87		
10	65–79		
14	60–70		
17	55–62		
23	53–57		
27	47–55 (^{15}N)		

Tuning Ranges for 600-MHz PFG Indirect Detection Probes

Capacitor Stick (pF)	*Range (MHz)*	*Inductor Stick (turns)*	*Range (MHz)*[a]
none	100–153 (^{13}C)	5	190–245 (^{31}P)
5	80–104	7	155–190
10	70–83		
14	63–73		
19	57–63 (^{15}N)		

Testing Indirect Detection Probes on UNITYINOVA

This section contains test and calibration procedures for the indirect detection probes:

- Table 1 lists the tests appropriate for each probe type.
- Table 2 lists the test samples.
- Specifications for Indirect Detection Probes, page 31, lists the test specifications.

Table 1. UNITY*INOVA* Probe Test Order and Indirect Detection Probe Types

Test	*300 PFG $^{1}H\{^{15}N–^{31}P\}$*	*400, 500, 600 PFG $^{1}H\{^{15}N–^{31}P\}$*
^{1}H Observe PW90 and RF Homogeneity, page 12	✔	✔
^{1}H Spinning Resolution & Lineshape, page 13	✔	✔
^{1}H Spinning Sidebands, page 14	✔	✔
^{1}H Nonspin Resolution & Lineshape, page 14	—	✔
Performa I or II PFG Profile and Recovery, page 14	✔	✔
^{1}H Sensitivity, page 16	✔	✔
^{13}C pwx90 Pulse Width and RF Homogeneity, page 17	✔	✔
^{15}N pwx90 Pulse Width and RF Homogeneity, page 18	—	✔

Table 2. Indirect Detection Probe Acceptance Tests and Samples

NMR Test	*Sample*	*Part No.*
^{1}H PW90, ^{1}H sensitivity	0.1% ethylbenzene, 0.01% TMS 99.89% deuterochloroform ($CDCl_3$)	00-968120-70
^{1}H spinning res. & lineshape, ^{1}H spinning sidebands, ^{1}H non-spin res. & lineshape	1% chloroform in acetone–d_6	00-968120-89
PFG (200- to 400-MHz)	2-Hz 20% H_2O, 80% D_2O	01-901855-02
PFG (500-MHz and higher)	2-Hz 1% H_2O, 99% D_2O	01-901855-01
^{13}C PWX90	1% iodomethane–^{13}C, 1% trimethylphosphite, 0.2% Cr(acac) in $CDCl_3$	00-968120-96
^{15}N PWX90	2% benzamide-^{15}N, 0.2% Cr(acac) in DMSO–d_6	00-968120-97

^{1}H Observe PW90 and RF Homogeneity

Use this procedure to determine the ^{1}H observe 90° pulse width and rf homogeneity.

1. On 200-, 300-, and 400-MHz systems, make sure the ^{1}H quarter-wavelength cable is installed.
2. Tune the probe using a doped 2-Hz D_2O sample listed above.
3. Enter **`rtp('/vnmr/tests/shmd2o')`** to retrieve the test parameter set to the current experiment. Enter **`at=2 sw=2000 d1=2 nt=1 lb='n' spin='n'`**.
4. Acquire a normal spectrum and shim the water signal to less than or equal to 3-Hz linewidth at 50%.
5. The water signal should be on-resonance. Place the cursor on the water signal and enter **`nl movetof`** to move the transmitter offset on the water peak.

   The transmitter must be on resonance for the test to be accurate.
6. Determine the correct tpwr as follows:
   a. Enter **`tpwr=50 pw=7 ga`**.
   b. After the acquisition is complete, enter **`aph`** to phase the spectrum.
   c. Set the **`pw`** to twice the pw90 specification for the probe and array **`tpwr`** from 48 to 58.

      Observe the spectra as they are being collected. When the peak of interest passes through the null and goes negative, the acquisition can be halted by entering `aa`. The `tpwr` value corresponding to the first negative spectrum should then be used for pw90 calibrations.
7. Expand the spectrum so that the quartet is centered on the screen with **`wp=250`**. Enter **`ai`**. Set **`vs`** so that the signal occupies about half the vertical size of the screen. Enter **`vp=50`**.
8. Setup the following array to determine the pw90 for a probe:
   a. Enter **`r1=`*`xx`*`/5`**, where *xx* is the specified pw90 specification for the probe.
   b. Enter **`array('pw',20,r1,r1)`** to create a pw array covering approximately 360 degrees of nutation.
9. Set up the following array to determine rf homogeneity:
   a. Enter **`r1=`*`xx`*`/10`**, where *xx* is the specified pw90 specification for the probe.
   b. Enter **`array('pw',100,r1,r1)`** to create a pw array covering approximately 900 degrees of nutation.
10. Enter **`ga`** to start the acquisition. After the last spectrum finishes, enter **`ds(1)`** to display the first spectrum. Enter **`aph0`** to phase the spectrum, then enter **`ai dssh`** to display the arrayed spectra.
11. Enter **`ds(10)`** (or whatever the spectrum number corresponds to the first maximum spectrum) **`full`** to display only the single spectrum. Determine the amplitude by entering **`peak`** to display the height of the peak (in mm) and the frequency of the peak (in Hz). Record the peak height (in mm) corresponding to the 90° pulse width spectrum.

    Repeat the steps for the spectra numbers corresponding to the 450° pulse width and the 810° pulse width spectra, respectively. Determine the peak amplitudes for the 450° pulse width and the 810° pulse width spectra. Record the peak heights (in mm) corresponding to the 450° pulse width and the 810° pulse width spectra.

12. Calculate the amplitude ratios as a percentage (e.g., (450°/90° ratio)(100) is the rf homogeneity specification) for 450°/90° and 810°/90°. Plot the arrayed spectra. Write down the probe type, probe serial number, and the 450°/90° and 810°/90° percentages on the plot in the form Specifications for Indirect Detection Probes, page 31.

^{1}H Spinning Resolution & Lineshape

This test measures the 50%, 0.55%, and 0.11% levels of the $CHCl_3$ sample. This test and the ^{1}H spinning sidebands test (the next test) *must be passed simultaneously* and both tests plotted together.

1. Put the appropriate $CHCl_3$ sample in the probe and spin it.
2. Enter **`rtp('/vnmr/tests/h1lshp')`**. Enter **`nt=1 vs=1000`**. Enter **`su`** to set up the system hardware.
3. Enter a value for `pw` appropriate for your sample:

Sample (Part No.)	*Value for* `pw`
1% $CHCl_3$ (00-968120-89)	90° pulse
5% $CHCl_3$ (00-968120-99)	90° pulse
20% $CHCl_3$ (00-968120-76)	20° pulse

4. Enter **`ga`** to acquire the spectrum. Phase the spectrum, then plot it using **`wp=250`**. Enter **`vs=10k`** and plot the expanded spectrum. Make sure the spectrum is normalized.

   If floor vibration results in excessive noise around the base of the peak, **`nt`** can be changed to a larger value (e.g., `nt=4` or `nt=16`); however, if extreme vibrations are present, it may be impossible to measure the lineshape accurately.
5. Measure lineshape as the linewidth of the $CHCl_3$ peak at 50%, 0.55%, and 0.11% of the main peak amplitude. Refer to Figure 3 for the measurement points.

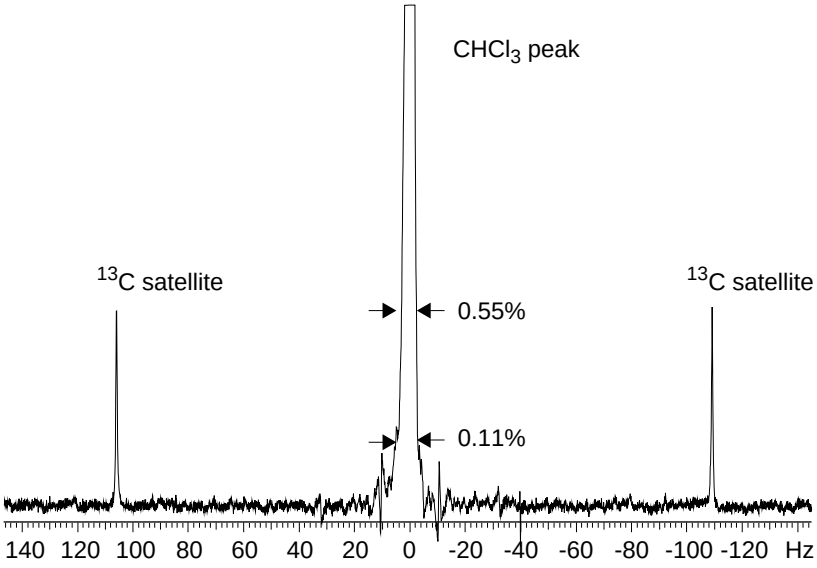


Figure 3. ^{1}H Lineshape Spinning Measurement

6. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.

^{1}H Spinning Sidebands

This test and the ^{1}H spinning resolution and lineshape test (the previous test) *must be passed simultaneously* and both tests plotted together.

1. Using the appropriate $CHCl_3$ sample, measure the ^{1}H spinning sidebands on the same spectrum as ^{1}H lineshape.
2. Use the spectra and parameter set from the ^{1}H lineshape test. Plot the spectrum again using a large enough value of `wp` to show all the spinning sidebands.
3. Measure spinning sideband amplitudes as a percentage of the main peak.

   Spinning sidebands occur at frequency intervals on either side of the central peak equal to the spinning rate. The sidebands may not be split.

   The standard test requires `nt=1` or `nt=4`. If sidebands meet specifications at `nt=4`, repeating the test at `nt=16` is unnecessary.
4. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.

^{1}H Nonspin Resolution & Lineshape

This test measures the 50%, 0.55%, and 0.11% levels of the $CHCl_3$ sample.

1. Use the appropriate $CHCl_3$ sample
2. Enter **`rtp('/vnmr/tests/h1lshp')`**. Enter **`nt=1 vs=1000`**. Enter **`su`** to set up the system hardware.
3. Turn the spinner off and set the spin rate to 0.
4. Enter **`ga`** to acquire the spectrum. Phase the spectrum, then plot it using **`wp=250`**. Enter **`vs=10k`** and plot the expanded spectrum.

   If floor vibration results in excessive noise around the base of the peak, **`nt`** can be changed to a larger value (e.g., `nt=4` or `nt=16`); however, if extreme vibrations are present, it may be impossible to measure the lineshape accurately.
5. Measure lineshape as the linewidth of the $CHCl_3$ peak at 50%, 0.55%, and 0.11% of the main peak amplitude. Refer to Figure 3 for the measurement points.
6. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.

Performa I or II PFG Profile and Recovery

PFG Profile

It is useful to translate the gradient control in DAC units to G/cm by a constant that represents G/cm-DAC units. This constant, `gcal`, is a user global real-valued parameter.

1. Use **`mp`** to move the doped D_2O parameters used in the rf homogeneity test to a new experiment. Join (**`jexp`**) the experiment.
2. Enter **`profile`**.
3. Enter the appropriate value for gzlvl1:
   - Performa I: **`gzlvl1=250`**
   - Performa II: **`gzlvl1=4000`**
4. Enter **`ga`** to acquire a spectrum. When acquisition finishes, enter **`f full dc vsadj`**.

You should see a very wide peak (about 16 kHz for Performa I or about 50 kHz for Performa II), similar to Figure 4.

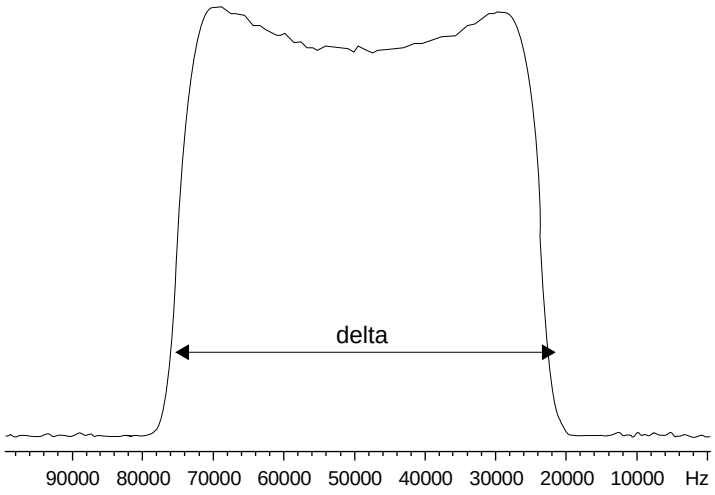


Figure 4. Z-Gradient Profile Measurement Using 1% Doped D_2O

5. Use the cursors to find the width of this peak at 20% of the height.
6. Enter **`setgcal`** to convert the width of this plateau (`delta`) and the gradient strength (`gzlvl1`) to a gradient strength per DAC value (`gcal`).

   The `setgcal` macro needs the physical extent of the rf sensitivity region (1.6 cm), as well as the gradient strength (`gzlvl1`) and the resulting plateau width (`delta`).

   When the system asks “Set this value?” respond with **`y`**.

   The value of `gcal` times the appropriate value below gives the maximum achievable gradient strength.

   - Performa I: `gcal` × `2048`.
   - Performa II: `gcal` × `32767`.
7. Repeat the experiment with `gzlvl1` set to one of the following. The `delta` should be equal to that previously measured.
   - Performa I: **`gzlvl1=-250`**
   - Performa II: **`gzlvl1=-4000`**

PFG Recovery

1. Using **`mp`**, move the doped D_2O parameters to a new experiment. Join (**`jexp`**) the experiment.
2. Enter **`grecovery`**.
3. For Performa I, enter **`at=1 in='n'`**. Set `tpwr` and `pw` for the 90° pulse width.
4. Set the appropriate recovery array (refer to Specifications for Indirect Detection Probes, page 31 for recovery specifications):
   - Probes with 1 ms recovery specification: enter **`array('d2',30,.001,.001)`**
   - Probes with 50 µs recovery specification: enter **`array('d2',30,.00005,.00005)`**
5. Enter **`d2=d2,1`** to add a 1-second reference spectrum.

6. Enter the appropriate value of `gzlvl1`:
   - Performa I: **`gzlvl1=10/gcal`** to change `gzlvl1` to a value that equals 10 G/cm.
   - Performa II: **`gzlvl1=30/gcal`** to change `gzlvl1` to a value that equals 30 G/cm.
7. Enter **`ga`** to start the acquisition.
8. Enter **`wft ds(arraydim)`**.
9. Phase the final spectrum, adjust its vertical scale, and enter **`dssh`**.

   The resulting profile should show a smooth and monotonic recovery pattern, similar to Figure 5.

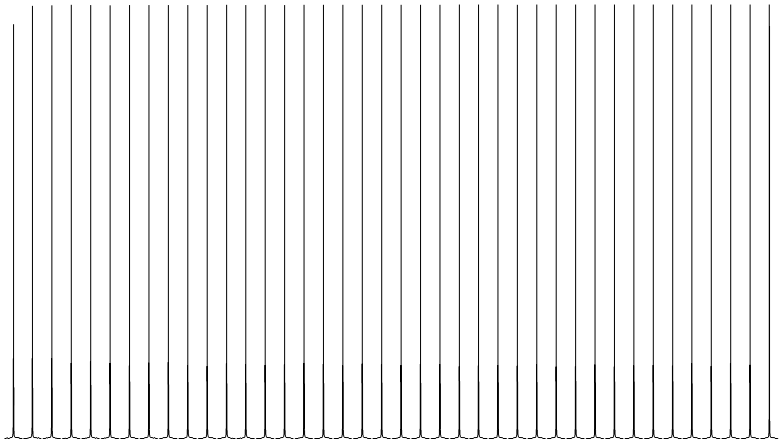


Figure 5. Gradient Recovery Pattern Profile

Note: For Performa III amplifiers if recovery specifications are not met, the slewrate of the PFG amplifier might have to be adjusted. To adjust the slewrate, enter **`d2=0.00006 nt=1e5 ga`**. Adjust the potentiometer (slew20) until maximum amplitude of the water peak is achieved.

^{1}H Sensitivity

1. Enter **`rtp('/vnmr/tests/h1sn') su`**.
2. Tune the probe with the appropriate sample inserted.
3. Determined the pw90 on this sample using the procedure in ^{1}H Observe PW90 and RF Homogeneity, page 12.

   You can also determine the pw90 from the pw360 and dividing that value by 4.
4. Enter **`ga`** to acquire the spectrum.
5. When the spectrum is displayed, phase it and display the quartet, using the region from the right of the aromatic peak to the right of the quartet.
6. Use the cursors to locate the best 200-Hz noise region to the left (downfield) of the quartet (see Figure 6).
7. Enter **`dsnmax(200)`**. The computer calculates the S/N ratio. At the prompt, answer **`y`** to plot the results.

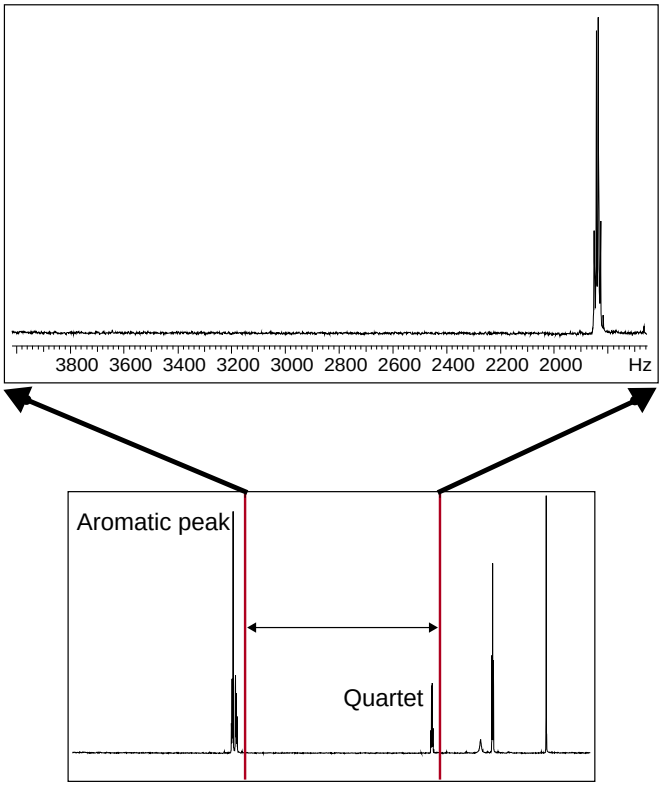


Figure 6. ^{1}H Sensitivity Measurement

8. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31

^{13}C pwx90 Pulse Width and RF Homogeneity

1. Check that the ^{1}H quarter-wavelength cable is installed (200-, 300-, and 400- MHz systems), the appropriate filters for ^{1}H and ^{13}C installed, and the probe is tuned for ^{1}H observe on channel 1 and ^{13}C on channel 2 using the appropriate sample listed above.
2. Enter **`rtp('/vnmr/tests/C13pwcal')`** to retrieve the test parameter set to the current experiment.
3. Change **`pw`** to the ^{1}H 90° pulse width (determined from the previous section). Change **`tpwr`** to a `tpwr` level to give the ^{1}H 90° pulse width. Acquire a normal ^{1}H spectrum of the 1% iodomethane-^{13}C, 1% trimethyl phosphite, 0.2% Cr(acac) in $CDCl_3$ sample.

   The large three-line pattern (1:1:1 should be centered approximately in the spectrum) in the spectrum at about 2.2 ppm are the peaks of interest. Place the cursor on the center peak of the three-line pattern, and enter **`nl movetof`**.
4. Enter **`pwxcal`** to set up the parameters for the ^{13}C rf homogeneity test.

   The `pwxcal` pulse sequence allows for the indirect observation of the X nuclei of interest. To use channel 2 (decoupler 1) for ^{13}C, answer the questions as follows:

```
Use decoupler 1, 2, or 3? 1
Calibrate C13, N15, or P31? C13
```

5. Enter the appropriate **dof** and **j** values, as listed in the following table:

Parameter	*^{1}H frequency (MHz)*	*^{13}C (Hz)*	*^{15}N (Hz)*	*^{31}P (Hz)*
dof	800	-22600	1200	18500
dof	750	-21000	1100	17500
dof	600	–17000	700	14000
dof	500	–14500	0	12000
dof	400	–11000	–12000	9000
dof	300	–9000	–9200	7000
j	all	151	86	20

6. Determine the X-nucleus 90° pulse width and rf homogeneity by arraying **pwx1**:

   a. Enter **r1=*xx*/10**, where *xx* is the specified pwx90 value for the probe.

   b. Enter **array('pwx1',85,0,r1)** to create a pwx1 array covering approximately 720 degrees of nutation.

   For example, if a probe has a pwx90 specification of 15 µs, following the above protocol sets up the experiment with an array if pwx1 from 0 µs to 150 µs, in 1.5-µs increments. The maximum positive peaks occur at 0°, 360°, and 720° pulses. A 90° pulse gives the first null in the pwx1 array of spectra. If the 90° pulse width is 15 µs, the 360° pulse width corresponds to pwx1= 60 µs (4*pwx1 90). The 720° pulse width corresponds to pwx1=120.4 µs (8*pwx1 90).

7. Enter **ga** to start the acquisition.

8. After the last spectrum finishes, enter **ds(1)** to display the first spectrum.

9. Enter **aph0** to phase the spectrum, and then enter **vsadj ai dssh** to display the arrayed spectra.

10. Enter **ds(1) full** to display only the single spectrum. Move the cursor to one of the ^{13}C enriched peaks of the iodomethane (one of the outer peaks of the three-peak pattern). Determine the amplitude by entering **nl** and mark to display the height of the peak (in mm) and the frequency of the peak (in Hz).

    Record the peak height (in mm) corresponding to the 360° pulse width spectrum. Repeat the steps for the spectra corresponding to 360° and the 720° pulse width spectra, respectively. Determine the peak amplitudes for the 360° pulse width and the 720° pulse width spectra. Record the peak heights (in mm) corresponding to the 360° pulse width and the 720° pulse width spectra.

11. Calculate the amplitude ratios as a percentage (e.g., (360°/0° ratio)(100) equals the rf homogeneity specification) for the 360°/0° and the 720°/0°. Plot the arrayed spectra. Record the probe type, probe serial number, and the 360°/0° and 720°/0° percentages on the plot in the form Specifications for Indirect Detection Probes, page 31.

^{15}N pwx90 Pulse Width and RF Homogeneity

1. Check that the ^{1}H quarter-wavelength cable is installed, the appropriate filters for ^{1}H and ^{15}N installed, and the probe is tuned for ^{1}H observe on channel 1 and ^{15}N on channel 2 (or channel 3 if applicable) using the appropriate sample listed above.

2. Enter **`rtp('/vnmr/tests/n15pwxcal')`** to retrieve the test parameter set to the current experiment.
3. Change **`pw`** to the ^{1}H 90° pulse width (determined from the ^{1}H rf homogeneity section). Change **`tpwr`** to the `tpwr` level to give the ^{1}H 90° pulse width.
4. Acquire a normal ^{1}H spectrum of the 2% benzamide-^{15}N, 0.2% Cr(acac) in DMSO-d_6 sample.

   The peaks of interest are the benzamide multiplet at the far left side of the spectrum.
5. Move the cursor to the center of the multiplet and enter **`movetof`**.
6. Enter **`pwxcal`** to set up the parameters for the ^{15}N rf homogeneity test.

   The `pwxcal` pulse sequence allows indirect observation of the X nuclei of interest.

   To use channel 2 (decoupler 1) for ^{15}N, answer the questions as follows:

   ```
   Use decoupler 1, 2, or 3? 1
   Calibrate C13, N15, or P31? N15
   ```

   After the `pwxcal` for ^{31}P has been run using `dec1`, the pwx90 can be determined using the remaining channels by adjusting the parameters as shown:

   - If the system has third channel rf, channel 3 (decoupler 2) can be used by entering `2` in response to the first question.
   - If decoupler 2 is used for ^{15}N, use the appropriate parameters (`dpwr2`, `dn2`, `dof2`, etc.) for decoupler 2.
7. Enter the appropriate values of **`dof`** (or `dof2`—for decoupler 2) and **`j`**, as listed in the following table:

Parameter	*^{1}H frequency (MHz)*	*^{13}C (Hz)*	*^{15}N (Hz)*	*^{31}P (Hz)*
`dof`	800	-22600	1200	18500
`dof`	750	-21000	1100	17500
`dof`	600	–17000	700	14000
`dof`	500	–14500	0	12000
`dof`	400	–11000	–12000	9000
`dof`	300	–9000	–9200	7000
`j`	all	151	86	20

8. Determine the X-nucleus 90° pulse width and rf homogeneity by arraying **`pwx1`**:
   a. Enter **`r1=xx/10`**, where *xx* is the specified `pwx90` value for the probe.
   b. Enter **`array('pwx1',85,0,r1)`** to create a `pwx1` array covering approximately 720 degrees of nutation.

   For example, if a probe has a pwx90 specification of 15 µs, following the above protocol sets up the experiment with an array if `pwx1` from 0 µs to 150 µs, in 1.5-µs increments. The maximum positive peaks occur at 0°, 360°, and 720° pulses. A 90° pulse gives the first null in the `pwx1` array of spectra. If the 90° pulse width is 15 µs, the 360° pulse width corresponds to `pwx1`= 60 µs (`4*pwx1 90`). The 720° pulse width corresponds to `pwx1=120.4` µs (`8*pwx1 90`).
9. Enter **`ga`** to start the acquisition.
10. After the last spectrum finishes, enter **`ds(1)`** to display the first spectrum.

11. Enter **aph0** to phase the spectrum, and then enter **vsadj ai dssh** to display the arrayed spectra.
12. Enter **ds(1) full** to display only the single spectrum. Move the cursor to one of the ^{13}C enriched peaks of the iodomethane (one of the outer peaks of the three-peak pattern). Determine the amplitude by entering **nl** and mark to display the height of the peak (in mm) and the frequency of the peak (in Hz).

    Record the peak height (in mm) corresponding to the 360° pulse width spectrum. Repeat the steps for the spectra corresponding to 360° and the 720° pulse width spectra, respectively. Determine the peak amplitudes for the 360° pulse width and the 720° pulse width spectra. Record the peak heights (in mm) corresponding to the 360° pulse width and the 720° pulse width spectra.
13. Calculate the amplitude ratios as a percentage (e.g., (360°/0° ratio)(100) equals the rf homogeneity specification) for the 360°/0° and the 720°/0°. Plot the arrayed spectra. Record the probe type, probe serial number, and the 360°/0° and 720°/0° percentages on the plot in the form Specifications for Indirect Detection Probes, page 31.

Testing Indirect Detection Probes on *MERCURY-Series*

This section contains test and calibration procedures for the indirect detection probe on *MERCURY*-series systems:

- How to Test a Probe, page 20, lists the general steps for testing a probe.
- Table 2, page 11, lists the test samples.
- Specifications for Indirect Detection Probes, page 31, contains the test specifications.

How to Test a Probe

This section provides the basic steps for testing a probe.

1. Create a probe calibration file as described in Probe Calibration Files, page 20.
2. Follow the test procedures listed in Table 3. If you wish, you can check off each test as it is finished.
3. After the appropriate tests are completed for the probe, print the corresponding probe calibration file by entering **ACreport** in the VNMR input window.
4. Follow the instructions in Setting Up the Solvent-Based Shim Files, page 29 for configuring solvent-based shims and the setlk macro for proper *GLIDE* and automation operation.

Probe Calibration Files

Probe calibration files (in /vnmr/probes) contain pw90, tpwr, dmf, dpwr, and pwx settings for each probe used with the system. *GLIDE*, automation, and manual operation using macros—including h1, hcosy, and dept—retrieve appropriate power levels, the pw90 value, and the dmf value from the probe calibration file, instead of using preset values in the parameters files. *Successful operation depends on the existence of a probe calibration file for each probe that is used with the system.*

When the *MERCURYplus* NMR spectrometer is installed, the proper probe calibration files are set up, so that the system is ready to use when the installer leaves the site. *Customers must update these files regularly to maintain optimal system performance.*

Table 3. *MERCURY* Probe Test Order and Indirect Detection Probe Types

Tests	*300 PFG* $^{1}H\{^{15}N–^{31}P\}$	*400 PFG* $^{1}H\{^{15}N–^{31}P\}$
Probe Calibration Files, page 20	✔	✔
^{1}H Spinning Resolution & Lineshape, page 22	✔	✔
^{1}H Spinning Sidebands, page 23	✔	✔
^{1}H Observe 90° Pulse Width, page 23	✔	✔
^{13}C pwx90 Pulse Width, page 24	✔	✔
^{13}C Decoupling Calibration—Measuring γH_2 for Indirect Detection, page 26	✔	✔
^{1}H Sensitivity, page 27	✔	✔
Pulsed Field Gradient Profile and Recovery, page 28	✔	✔
Setting Up the Solvent-Based Shim Files, page 29	✔	✔
Configuring the setlk Macro, page 30	✔	✔

The probe calibration files must be created when the system is installed or the first time a probe is used. Create or change the probe calibration file as follows:

To Create a Probe Calibration File

Perform these steps for each probe that is tested.

1. Log in as `vnmr1`.
2. Enter the following command in the VNMR input window:
   `addprobe(`*`probe`*`<,'system'>)`
   where *`probe`* is a descriptive name for the probe (e.g., `addprobe('id5mm')` for an 5-mm indirect detection probe).
   The `'system'` keyword (e.g., `addprobe('id500','system')` makes the probe calibration files for this probe available to all users by placing the files in `/vnmr/probes`.Without the keyword, the files are placed in the `vnmrsys` directory of the user entering the command (e.g., `~vnmr1/vnmrsys/probes`) and are only available to that user.
   The values for power, `pw90`, `dmf`, and `pp` are initially set to 0 and filled in later by running the calibration procedures for the appropriate nuclei using *GLIDE*.

To Change to a Different Probe Calibration File

After the probes are tested and calibrated, use this procedure to switch to the appropriate calibration file when a different probe is installed.

1. Log in as `vnmr1`.
2. Enter the following command in the VNMR input window:
   `probe=`*`probe_dir`*
   Where *probe_dir* the name of the existing probe directory in `/vnmr/probes` named for the new probe (e.g., `probe='sw5mm'`).

^{1}H Spinning Resolution & Lineshape

This test measures the 50%, 0.55%, and 0.11% levels of the $CHCl_3$ sample. This test and the ^{1}H spinning sidebands test (the next test) *must be passed simultaneously* and both tests plotted together.

1. Insert the appropriate $CHCl_3$ sample in the magnet and spin it.
2. Enter **`rtp('/vnmr/tests/H1lshp')`** **`nm`** and set **`nt=1 vs=100`**. Enter **`su`** to set up the system hardware.
3. Tune the probe.
4. Enter a value for `pw` appropriate for your sample:

If your sample is:	*Then use the value for a:*
1% $CHCl_3$, (00-968120-89)	90° pulse
5% $CHCl_3$, (00-968120-99)	90° pulse
20% $CHCl_3$, (00-968120-76)	20° pulse

5. Enter **`ga`** to acquire the spectrum. Phase the spectrum, set **`wp=250`**, and plot using **`pl`**. Increase **`vs`** by a factor of 100 and plot the expanded spectrum using **`pl pscale page`**.

   If floor vibration results in excessive noise around the base of the peak, `nt` can be set to a larger value (e.g., `nt=4` or `nt=16`); however, if extreme vibrations are present, it may be impossible to measure the lineshape accurately.
6. Measure lineshape as the linewidth of the $CHCl_3$ peak at 50%, 0.55% and 0.11% of the main peak amplitude. Refer to Figure 3 and use the substeps below to determine the lineshape:

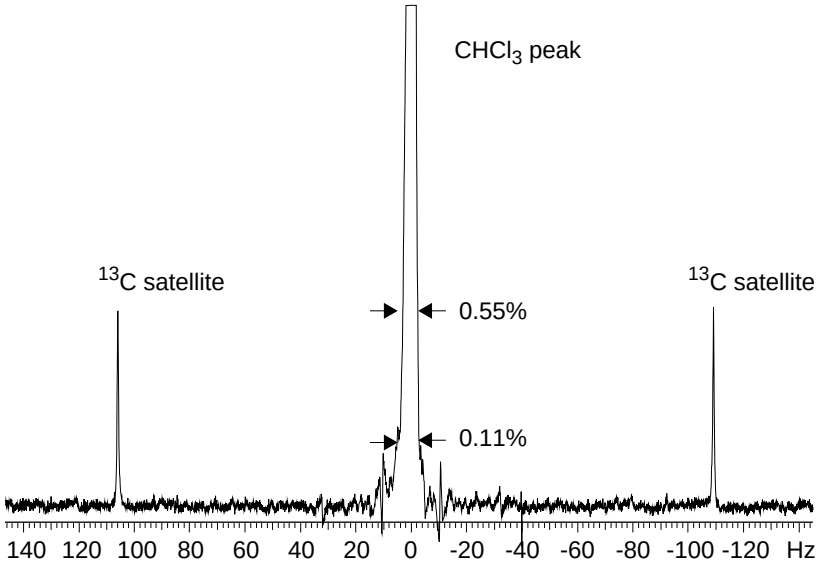


Figure 7. ^{1}H Lineshape Spinning Measurement

   a. Display and expand the desired peak.
   b. Enter **`nm`**, then **`dc`** for drift correction to ensure a flat baseline. Set **`vs=10000`**. Place a cursor on the chloroform line and enter **`nl dres`** (the 50% level).

c. Enter **nm**, then **dc** for drift correction to ensure a flat baseline. Set **vs=10000**. Click the VNMR menu button labeled Th to display the horizontal threshold cursor. Set **th=55** (the 0.55% level).

d. Click the first menu button labeled Cursor or Box to display two vertical cursors, and align them on the intersections of the horizontal cursor and the peak. Enter **delta?** to see the difference in Hz between the cursors.

e. Set **th=11** (the 0.11% level) and repeat.

7. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.
8. After the specifications are met, do the following:
   - Write down the values for `z0`, `lockpower`, and `lockgain` as they appear in the `Acqi` window. Use the forms provided in Specifications for Indirect Detection Probes, page 31.
   - Save the shims by entering in the VNMR input window:
     svs('acetone')

^{1}H Spinning Sidebands

This test and the ^{1}H lineshape test (the previous test) *must be passed simultaneously* and both tests plotted together.

1. Using the appropriate $CHCl_3$ sample, measure the ^{1}H spinning sidebands on the same spectrum as the ^{1}H lineshape test.
2. Use the spectra and parameter set from the lineshape test. Plot the spectrum again using a large enough value of `wp` to show all the spinning sidebands.
3. Measure spinning sideband amplitudes as a percentage of the main peak.

   Spinning sidebands occur at frequency intervals on either side of the central peak equal to the spinning rate. The sidebands might not be split.
4. The standard test requires **nt=4**. If the sidebands meet specifications at `nt=4`, repeating the test at `nt=16` is not necessary.
5. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.

^{1}H Observe 90° Pulse Width

This procedure calibrates `tpwr` and the 90° pulse width for ^{1}H.

1. Open *GLIDE* by clicking the **GLIDE** button in the VNMR menu.
2. Click on the **Setup** icon.
3. Insert the sample in the magnet using the **Insert** button in the Setup window.
4. Select **Calibrate Proton** from the Experiment drop-down menu. You do not need to select a solvent.
5. If you have already locked and shimmed the sample, turn off autolock and autoshim.
6. Click the **Setup** button at the bottom of the Setup window.
7. Tune the probe.
8. Click on the **Acquire** button under Custom.
9. Enter an appropriate value for **pwmax** (listed in Specifications for Indirect Detection Probes, page 31).

10. Click on **Close** at the bottom of the Acquire window.
11. Click the **Go** icon in the *GLIDE* window.

    You should get a plot of arrayed spectra.
12. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.

^{13}C pwx90 Pulse Width

To perform the ^{13}C or X-nucleus 90° pulse with calibration in the indirect mode, use the HMQC experiment.

1. Make sure the appropriate quarter-wavelength cable is installed.
2. Insert the 1% iodomethane–^{13}C sample (Part No. 00-968120-96). Tune the probe on this sample.
3. Enter **`rtp('/vnmr/tests/H1sn')`** to retrieve the parameters from the ^{1}H sensitivity measurement.
4. Set **`pw`** and **`tpwr`** to the values determined for the ^{1}H sensitivity procedure; you can get these values from the probe calibration file by entering:

   `getparam('tpwr'):tpwr`
   `getparam('pw90'):pw`
5. Set **`gain=10 dn='C13' d1=5`** and enter **`ga`**.
6. Place two cursors around the region with the peaks. Enter **`movesw`** to narrow the spectral width.
7. Determine the 90° pulse width by arraying **`pw`** to ≥360° pulse width. Enter the macro **`array`** to set up the array. As the macro displays the following prompts, type in the response shown in bold:

   ```
   parameter to be arrayed:        pw
   number of steps in the array:   25
   starting increment:             2
   array increment:                2
   ```

   In the above example, the experiment is set up to array the `pw` parameter from 2 µs to 52 µs, in 2 µs increments. If the 90° pulse width is 10 µs, the 450° pulse width corresponds to `pw=50` µs (5 * `pw90`).
8. Enter **`ga`** to start the acquisition. After the last spectrum is finished, enter **`ds(5) vp=50`** to display the fifth spectrum. Enter **`aph`** to phase the spectrum, then enter **`ai dssh`** to display the arrayed spectra.
9. Set **`pw`** equal to the 90° pulse width you have determined. Ether **`ga`** to acquire the spectrum. Enter **`f ds`** to display the spectrum.

   The spectrum should appear similar to the example shown in Figure 8. The large, three-line pattern at about 2.2 ppm is the signal of interest.
10. To perform the ^{13}C X-nucleus 90° pulse width calibration in the indirect mode using HMQC, enter **`hmqc`** in the same experiment or move the parameters to a different experiment by entering **`mp(x,y)`**, where *x* is the current experiment and *y* is the experiment to which the parameter set is to be moved.
11. Set **`fn=8192 ni=1 phase=1 nt=1 ss=0 spin=0 d1=5`**. Set **`pw`** to the ^{1}H 90° pulse width (determined from the previous steps). Set **`tpwr`** to the `tpwr` level to give the ^{1}H 90° pulse width (determined from the previous steps). Set **`null=0 j=151`**. Set **`tof`** to the `tof` determined from the ^{1}H spectrum of the 1%

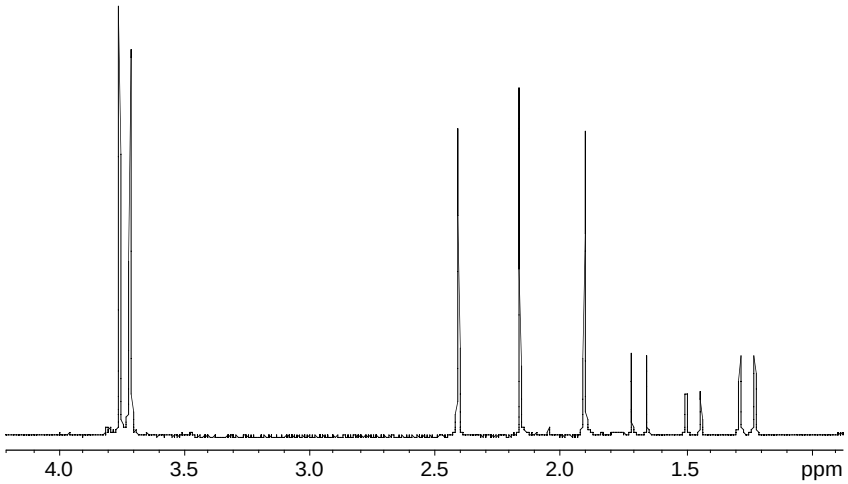


Figure 8. Normal ^{1}H Spectrum of Test Sample

iodomethane-^{13}C in step 6 above. Set **`dn='C13' dm='nnn'`**. Set **`dof`** from the following table:

^{1}H frequency (MHz)	*^{13}C (Hz)*	*^{15}N (Hz)*	*^{31}P (Hz)*
400	–11965	–12000	9000
300	–9000	–9200	7000

12. To determine the ^{13}C X-nucleus 90° pulse width and rf homogeneity using the HMQC pulse sequence, `pwx` is arrayed for a particular `pwxlvl`. The spectrum corresponding to the ^{13}C X-nucleus 90° pulse width using the HMQC pulse sequence is the maximum amplitude spectrum.

    An array of spectra appears as shown in Figure 9. The array shows only the response of the outer line (at about 2.4 ppm) of the three-line pattern. In HMQC, `null` occurs at 45, 135 maximum at 90° and negative at 180°.

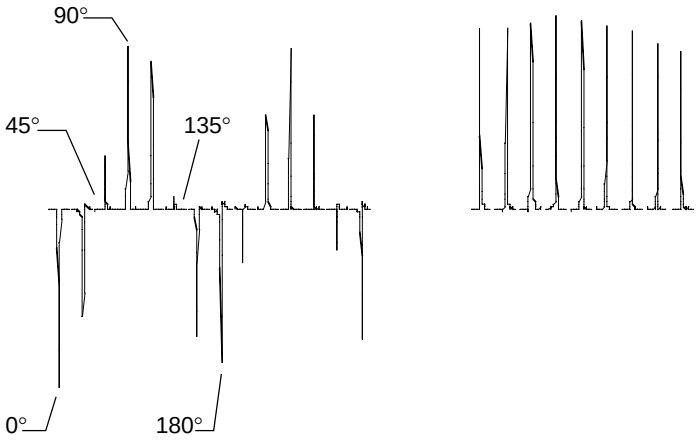


Figure 9. `pwx` Calibration, Coarse (Left) and Fine (Right)

Set **pwxlvl** to the tpwr value for normal ^{13}C observe (to get this value you can enter **getparam('tpwr','C13'):pwxlvl**). Enter **array** to array pwx. As array displays the following prompts, enter in the responses shown in bold:

```
parameter to be arrayed:          pwx
number of steps in the array:     20
starting increment:               2
array increment:                  1
```

In this example, the experiment is set up to array pw from 2 µs to 22 µs, in 1 µs increments.

13. Enter **ga** to start the acquisition. After the last spectrum is finished, enter **ds(1)** to display the first spectrum. Enter **aph** to phase the spectrum, then enter **ai dssh** to display the arrayed spectra.
14. Determine **pwx90** from the first maximum. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.
15. To store the values in the probe calibrations file, enter the following commands:

    ```
    setparams('pwxlvl','61.0','C13')
    setparams('pwx','14.2','C13')
    ```

    Be sure to enclose the values in single quotes because they are treated as strings.

^{13}C Decoupling Calibration—Measuring γH_2 for Indirect Detection

This procedure describes how to perform ^{13}C decoupling calibration as well as how to measure γH_2 for indirect detection.

1. Set **pwx** equal to the value determined in step 14 in the previous procedure.
2. Enter **ga** to acquire a spectrum.

   The spectrum should look like the second spectrum in Figure 10, with the outer two peaks of the three line pattern up and the center peak down.

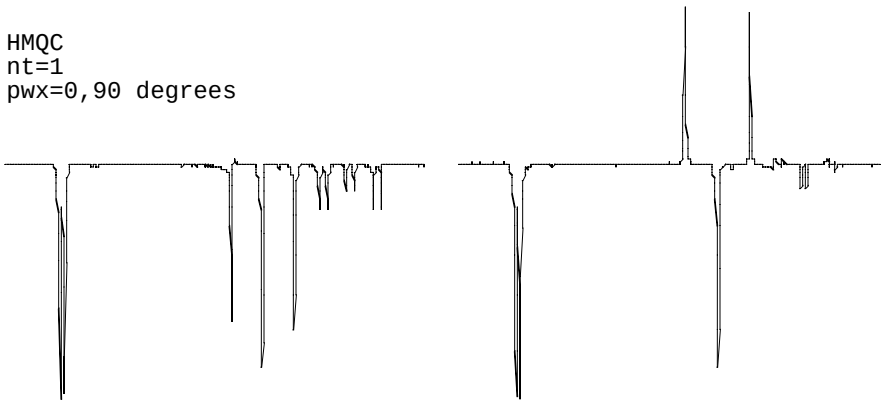


Figure 10. HMQC With and Without X-Nucleus Pulses

3. Set **dmm='ccc' dm='nny' dpwr=30 dof=dof+2000,dof-2000**. Enter **ga**.
4. Enter **ds(1)**, place a cursor on each of the positive peaks, and write down the delta.
5. Enter **ds(2)**, place a cursor on each of the positive peaks, and write down the delta.
6. Enter **h2cal** and enter the delta values for the high field and low field coupling.

7. When prompted, enter **151** for the coupling constant.

   A γH_2 of 5000 is necessary for decoupling in indirect detection. If the value is not 5000, increase `dpwr` by 3 dB until γH_2 is 5000.

8. To store the values in the probe calibrations file, enter the following commands:

   ```
   setparams('dpwr','43','C13')
   setparams('dmf','20000','C13')
   ```

   Be sure to enclose the values in single quotes because they are treated as strings.

^{1}H Sensitivity

1. If you just finished the 90° pulse width calibration, enter **pw=pw90 f full nm** and skip to step 5.
2. Enter **rtp('/vnmr/tests/H1sn') su**.
3. If you completed the 90° pulse width calibration using *GLIDE*, enter the following:

   ```
   getparam('tpwr'):tpwr
   getparam('pw90'):pw
   ```

4. Tune the probe.
5. Enter **nt=1 ga** to acquire the spectrum.
6. When the spectrum is displayed, phase it and enter **wp=500 vs=0** to display the quartet.
7. Use the cursors to locate a 200-Hz noise region to the left (downfield) of the quartet, as shown in Figure 11.

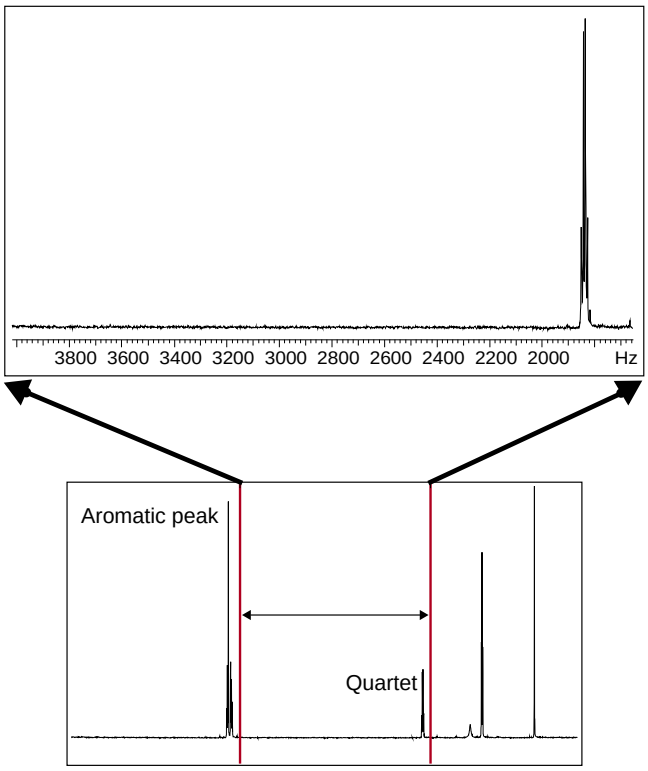


Figure 11. ^{1}H Sensitivity Measurement

8. Enter **dsn**. The computer calculates the signal-to-noise ratio.
9. Write the results for each probe in the forms provided in Specifications for Indirect Detection Probes, page 31.
10. After the specifications are met, do the following:
    - Write down the values for `z0`, `lockpower`, and `lockgain` as they appear in the Acqi window. Use the forms provided in Specifications for Indirect Detection Probes, page 31.
    - Save the shims by entering the following VNMR command:

      svs('cdcl3')

Pulsed Field Gradient Profile and Recovery

It is useful to translate the gradient control in DAC units to G/cm by a constant that represents G/cm-DAC units. This constant is called `gcal` and is a user global real-valued parameter.

1. Perform the PFG profile calibration as follows:
    a. Enter **rt('/vnmr/tests/shmd2o')**.
    b. Enter **profile**.
    c. Enter **gzlvl1=250**.
    d. Enter **ga** to acquire a spectrum. When the acquisition is finished, enter **f full dc vsadj**.

       You should see a wide peak (about 16 kHz), similar to Figure 12. Use the cursors to find the width of this peak at 20% of the height, enter **nm vs=100 th=20**.

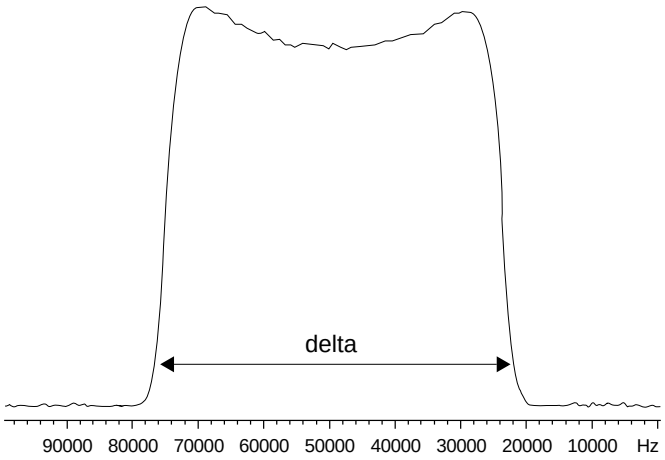


Figure 12. Z-Gradient Profile Measurement Using 1% Doped D_2O

    e. Enter **setgcal** to convert the width of this plateau (`delta`) and the gradient strength (`gzlvl1`) to a gradient strength per DAC value (`gcal`).

       The `setgcal` macro needs the physical extent of the rf sensitivity region (1.5 cm), as well as the gradient strength (`gzlvl1`) and the resulting plateau width (`delta`).

       When the system asks `Set this value?` respond with **y**.

The value of `gcal` times 2048 gives the maximum achievable gradient strength.

f. Repeat the experiment with **`gzlvl1=-250`**. The `delta` should be equal to that previously measured.

2. Perform the PFG recovery calibration as follows:

   a. Enter **`rt('/vnmr/tests/shmd2o')`**.

   b. Enter **`grecovery`**.

   c. Enter **`at=1 in='n'`**. Set **`tpwr`** and **`pw`** for the 90° pulse width.

   d. Change **`gzlvl1`** to a value that equals 18 G/cm (2047)

   e. Enter **`ga`** to start the acquisition.

   f. Phase the final spectrum, adjust its vertical scale, and enter **`dssh`**.

   The resulting profile should show a smooth and monotonic recovery pattern, similar to Figure 13.

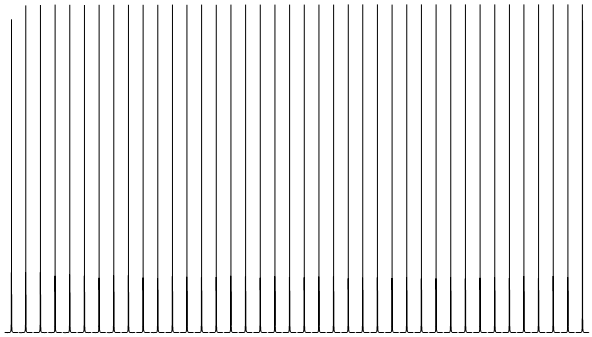


Figure 13. Gradient Recovery Pattern Profile

Setting Up the Solvent-Based Shim Files

During the probe acceptance test procedures (ATP), at least three solvent-based shim files were saved: `acetone`, `cdcl3`, and `c6d6` (the file `dmso` would have been saved for a 400-MHz system or with a probe with ^{15}N). Other solvents that *GLIDE* supports by default are D_2O and CD_3OD. The steps below describe how to save other solvent-based shims and move the shim files to the `/vnmr/shims` directory. You must still be logged in as `vnmr1`. Note that starting with VNMR 6.1, the names for shim files should be all lower case.

1. If you primarily use only the solvents used in the ATP (acetone, $CDCl_3$, and C_6D_6), skip to step 2 below. If you normally use D_2O or CD_3OD (or DMSO and it wasn't used in the ATP) in addition to the solvents used in the ATP, set up the shim files for these solvents as follows:

   a. Lock and shim on each solvent.

   b. Write down the values for `z0`, `lockpower`, and `lockgain` for each solvent. These values are displayed in the Acqi window.

   c. Save the shims with the **`svs`** command. Use the following commands in the VNMR input window:

   - **`svs('d2o')`** if the solvent is D_2O
   - **`svs('cd3od')`** if the solvent is CD_3OD
   - **`svs('dmso')`** if the solvent is DMSO.

2. In a shell tool or a Terminal window (still logged in as `vnmr1`), move the shim files from `~vnmrsys/shims` to `/vnmr/shims` by changing to the `/vnmr/shims` directory and using the `mv` command. Note the dot at the end of each `mv` command.

   ```
   cd /vnmr/shims
   mv ~/vnmrsys/shims/acetone .
   mv ~/vnmrsys/shims/cdcl3 .
   mv ~/vnmrsys/shims/c6d6 .
   ```

   And if present,

   ```
   mv ~/vnmrsys/shims/dmso .
   mv ~/vnmrsys/shims/d2o .
   mv ~/vnmrsys/CD3OD .
   ```

Go to the next section to configure the `setlk` macro.

Configuring the setlk Macro

A properly configured `setlk` macro is critical to Autolock and Autoshim performance and *GLIDE* performance. Once properly configured, `setlk` allows the system to perform the following adjustments for each system:

- Explicitly set `z0`, `lockpower`, and `lockgain`.
- Retrieve solvent-based shim sets.

Explicitly setting `z0`, `lockpower`, and `lockgain` for solvents typically used ensures the best possible results when using Autolock. The reliability of Autolock is especially important for the first sample of an automation run—if lock power has been low, the first sample may not be run because of an Autolock failure.

Use the following steps to configure the `setlk` macro.

1. Using a text editor such as **`vi`**, open the file `/vnmr/maclib/setlk`.
2. To allow the system to automatically retrieve a set of shims for each solvent, remove the double quotes around the following lines:

   ```
   if ($e>0.5)or($e1>0.5 then
     rts($solv)
   else
     exists(userdir+'/shims/cdcl3','file'):$e
     exists(systemdir+'/shims/cdcl3','file'):$e1
     if (($e>0.5)or($e1>0.5) then
       rts('cdcl3')
     endif
   endif
   ```

3. Remove the quotes from the lines with the solvents you plan to use. Replace the appropriate values of `z0`, `lockpower`, `lockgain` using the values you wrote down during the ATP or when you locked and shimmed on other solvents.

   The example below shows some of the entries with the Z0 value entered. The Z0 value represents the value of Z0 shown in the lock display in `acqi`.

   ```
   if $solv='cdcl3' then z0=512 lockpower=37 lockgain=41 else
   if $solv='d2o' then z0=521 lockpower=37 lockgain=48 else
   if $solv='acetone' then z0=512 lockpower=21 lockgain=39 else
   if $solv='dmso' then lockpower=33 lockgain=36 else
   if $solv='c6d6' then lockpower=20 lockgain=39 else
   if $solv='cd3od' then lockpower=25 lockgain=35 else
   lockpower=30 lockgain=40 endif endif endif endif endif endif
   ```

Note that you should have one `endif` for each `if` in the entries.

4. Save the file and exit the text editor. Clean up the VNMR file system as necessary.

Specifications for Indirect Detection Probes

- 300-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe, page 32
- 400-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe, page 33
- 500-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe, page 34
- 600-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe, page 35

All specifications are subject to change without notice. The specifications published in this document shall prevail unless customer contract determines otherwise. *Request of any additional specifications beyond those listed in this document must be agreed upon in writing as part of the customer contract.*

Specifications are for probes installed with UNITY*INOVA* or *MERCURYplus* NMR spectrometer systems. For probes purchased for use in systems other than UNITY*INOVA* or *MERCURYplus*, no guarantee is given that these probes meet current specifications.

Tests are performed with the following sample tubes:

- 5-mm probes: 5-mm tubes with 4.2-mm I.D. (Wilmad 528-PP, or equivalent).
- *Using sample tubes with thinner wall thickness (e.g., Wilmad 5-mm 545-PPT, or equivalent) increases signal-to-noise.*

300-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe

Probe Serial No. _____ Customer Initials _____

- 90° Pulse Width

Nucleus	*INOVA (µs)*	*MERCURY BB (µs)*	*Results*	*tpwr/dpwr/Notes*
^{1}H	10	10		
^{13}C pwx	15	18		

- RF Homogeneity (INOVA only)

Nucleus	*Specification (%)*	*Results/Notes*
^{1}H (450/90)	80	
^{1}H (810/90)	70	
^{13}C (360/0)	70	
^{13}C (720/0)	55	

- Resolution and Lineshape (50%/0.55%/0.11%)

Shim Set	*^{1}H Spinning ($CHCl_3$)*	*Results*
MERCURY BB	0.4/6.0/12.0	
INOVA 14-, 18-shim	0.4/6.0/12.0	

- ^{1}H Spinning Sidebands (^{UNITY}*INOVA* and *MERCURYplus*)

Nucleus	*Specification (%)*	*Results*	*Notes*
^{1}H	1		

- Sensitivity (^{UNITY}*INOVA* and *MERCURYplus*)

Nucleus	*(S:N)*	*Results*	*Notes*
^{1}H	220:1		

- PFG Profile and Recovery

PFG System	*Profile*	*Recovery*	*Results*
Performa I	>18	≤ 1ms @ 10 gauss/cm	
Performa II	>65	≤ 1ms @ 30 gauss/cm	

- Variable Temperature Range: –20 to +80°C

400-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe

Probe Serial No. _____ Customer Initials _____

- 90° Pulse Width

Nucleus	*INOVA (µs)*	*MERCURY BB (µs)*	*Results*	*tpwr/dpwr/Notes*
^{1}H	10	10		
^{13}C pwx	12	18		
^{15}N pwx	25	—		

- RF Homogeneity (UNITY*INOVA* only)

Nucleus	*Specification (%)*	*Results/Notes*
^{1}H (450/90)	80	
^{1}H (810/90)	70	
^{13}C (360/0)	70	
^{13}C (720/0)	55	
^{15}N (360/0)	70	
^{15}N (720/0)	50	

- Resolution and Lineshape (50%/0.55%/0.11%)

Shim Set	*^{1}H Spinning ($CHCl_3$)*	*^{1}H Non-spinning ($CHCl_3$)*	*Results*
MERCURY BB	0.45/6.0/12.0	—	
INOVA 14-, 18-shim	0.45/6.0/12.0	—	
INOVA 23-shim	0.45/5.0/10.0	0.65/10.0/20.0	

- ^{1}H Spinning Sidebands (UNITY*INOVA* and *MERCURYplus*)

Nucleus	*Specification (%)*	*Results*	*Notes*
^{1}H	1		

- Sensitivity (UNITY*INOVA* and *MERCURYplus*)

Nucleus	*(S:N)*	*Results*	*Notes*
^{1}H	500:1		

- PFG Profile and Recovery

PFG System	*Profile*	*Recovery*	*Results*
Performa I	>18	≤ 1 ms @ 10 gauss/cm	
Performa II	>65	≤ 50 µs @ 30 gauss/cm	
Performa III	Available by request		

- Variable Temperature Range: –20 to +80°C

500-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe

Probe Serial No. _____ Customer Initials _____

- 90° Pulse Width

Nucleus	*INOVA (µs)*	*Results*	*tpwr/dpwr/Notes*
^{1}H	10		
^{13}C pwx	12		
^{15}N pwx	25		

- RF Homogeneity

Nucleus	*Specification (%)*	*Results/Notes*
^{1}H (450/90)	80	
^{1}H (810/90)	70	
^{13}C (360/0)	70	
^{13}C (720/0)	55	
^{15}N (360/0)	70	
^{15}N (720/0)	50	

- Resolution and Lineshape (50%/0.55%/0.11%)

Shim Set	*^{1}H Spinning ($CHCl_3$)*	*^{1}H Non-spinning ($CHCl_3$)*	*Results*
23-shim	0.45/6.0/12.0	0.65/10.0/20.0	
28-shim	0.45/5.0/10.0	0.65/8.0/16.0	
Ultra-shim	0.45/5.0/10.0	0.65/6.0/12.0	

- ^{1}H Spinning Sidebands

Nucleus	*Specification (%)*	*Results*	*Notes*
^{1}H	1		

- Sensitivity

Nucleus	*(S:N)*	*Results*	*Notes*
^{1}H	800:1		

- PFG Profile and Recovery

PFG System	*Profile*	*Recovery*	*Results*
Performa I	>18	≤ 1 ms @ 10 gauss/cm	
Performa II	>65	≤ 50 µs @ 30 gauss/cm	
Performa III	Available by request		

- Variable Temperature Range: –20 to +80°C

600-MHz $^{1}H\{^{15}N–^{31}P\}$ Indirect Detection Probe

Probe Serial No. _____ Customer Initials _____

- 90° Pulse Width

Nucleus	*INOVA (µs)*	*Results*	*tpwr/dpwr/Notes*
^{1}H	10		
^{13}C pwx	14		
^{15}N pwx	30		

- RF Homogeneity

Nucleus	*Specification (%)*	*Results/Notes*
^{1}H (450/90)	80	
^{1}H (810/90)	70	
^{13}C (360/0)	70	
^{13}C (720/0)	55	
^{15}N (360/0)	70	
^{15}N (720/0)	50	

- Resolution and Lineshape (50%/0.55%/0.11%)

Shim Set	*^{1}H Spinning ($CHCl_3$)*	*^{1}H Non-spinning ($CHCl_3$)*	*Results*
23--shim	0.45/6.0/12.0	0.65/10.0/20.0	
28-shim	0.45/5.0/10.0	0.65/8.0/16.0	
Ultra-shim	0.45/5.0/10.0	0.65/6.0/12.0	

- ^{1}H Spinning Sidebands

Nucleus	*Specification (%)*	*Results*	*Notes*
^{1}H	1		

- Sensitivity

Nucleus	*(S:N)*	*Results*	*Notes*
^{1}H	1000:1		

- PFG Profile and Recovery

PFG System	*Profile*	*Recovery*	*Results*
Performa I	>18	≤ 1 ms @ 10 gauss/cm	
Performa II	>65	≤ 50 µs @ 30 gauss/cm	
Performa III	Available by request		

- Variable Temperature Range: –20 to +80°C

Indirect Detection NMR Probes
Pub. No. 01-999123-00, Rev. E0501

Technical contributor: Rob Rice, Joe Murphy, Bao Nguyen, Laima Baltusis, Everett Schreiber
Technical writer: Dan Steele
Technical editor: Dan Steele

Revision history:
A0599 – Initial release (E.R. 2750)
B0799 – Update specification (ECO# 8621, 7/16/99)
C1199 – Update PFG test/spec and S/N spec (ECO# 8734, 11/10/99)
C0300 – Update Table 3 for MERCURY tests
D0900 – Update installation steps for 300 & 400-MHz INOVA (ECO# 8953, 8/23/00)
E0501 – Update *MERCURYplus* ^{1}H sensitivity specifications, ECO 9140

Copyright © 2001, Varian, Inc.
3120 Hansen Way, Palo Alto, CA 94304-1030
http://www.varianinc.com
All rights reserved. Printed in the United States

The information in this document has been carefully checked and is believed to be entirely reliable. However, no responsibility is assumed for inaccuracies. Statements in this document are not intended to create any warranty, expressed or implied. Specifications and performance characteristics of software and hardware described in this manual may be changed at any time without notice. Varian reserves the right to make changes in any products herein to improve reliability, function, or design. Varian does not assume any liability arising out of the application or use of any product or circuit described herein; neither does it convey any license under its patent rights nor the rights of others. Inclusion in this document does not imply that any particular feature is standard on the instrument.

UNITY*INOVA* and *MERCURYplus* are registered trademarks or trademarks of Varian, Inc. Other product names are trademarks of their respective holders.

Artisan Technology Group is an independent supplier of quality pre-owned equipment

Gold-standard solutions

Extend the life of your critical industrial, commercial, and military systems with our superior service and support.

We buy equipment

Planning to upgrade your current equipment? Have surplus equipment taking up shelf space? We'll give it a new home.

Learn more!

Visit us at **artisantg.com** for more info on price quotes, drivers, technical specifications, manuals, and documentation.

Artisan Scientific Corporation dba Artisan Technology Group is not an affiliate, representative, or authorized distributor for any manufacturer listed herein.

We're here to make your life easier. How can we help you today?

(217) 352-9330 | sales@artisantg.com | artisantg.com

ARTISAN® TECHNOLOGY GROUP