

System Overview

MERCURYplus NMR Spectrometer Systems

Pub No. 01-999184-00, Rev. B1002

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Agilent Technologies

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Introduction

The *MERCURYplus* NMR spectrometer system operates at proton frequencies of 200, 300, and 400 MHz, depending on the model. The 200 and 300 systems are also each available in a lower power, 4-nuclei version. A superconducting magnet bore of 54 mm is available, with standard liquids probes accepting sample tube diameters of 5 and 10 mm. The spectrometer was designed with built-in modularity, to permit easy selection of configurations for the experimental requirements of a specific laboratory.

This manual provides both a general and a functional description of the spectrometer's major systems and subsystems. Throughout the manual, systems are identified by frequency and bore, shown as a fraction. For example, the notation "400/54" refers to a system with a 400-MHz proton frequency and a 54-mm magnet bore.

System Components

A *MERCURYplus* system intended for liquids operation has three major groups of components:

- *Host computer system* – Includes a Sun Microsystems workstation with networking capabilities, keyboard, mouse, color monitor, hard disk drives, optional plotters and printers, CD-ROM drive, floppy drive, and optional tape units for data storage.
- *Magnet and magnet console interface* – Includes the probe, upper barrel, preamplifiers and related electronics, and air supply controls.
- *NMR console* – Includes rf and digital cardcages, rf amplifiers, and power supplies.

The magnet is a superconducting solenoid contained within a dewar system. Without refilling helium, the 200/54 and 300/54 magnets are available in 80-day, 235-day, and 365-day temperature hold-time periods. The 400/54 magnet is available with 183- or 365-day temperature-hold capabilities.

The NMR probe and its matching upper barrel, as well as the room temperature shim coils, are located in the ambient temperature bore of the magnet. The upper barrel typically contains the sample insertion, spinning, and ejection systems and a circuit that senses the sample spinning rate.

The NMR probe contains rf circuits that apply the observe, lock, and decouple transmitter frequencies to the sample and detect the resulting NMR signals. Most probes can be tuned to detect NMR signals at several frequencies. Many probes also contain provisions for controlling the sample temperature or applying pulsed field gradients. The preamplifiers that receive the NMR signals from the probe amplify the input and send the resulting signal to the Receiver board and processing circuits in the console. The preamplifiers and sample spinning controls are located near the magnet in the Magnet Interface box (MIB).

The NMR console contains circuits that generate the rf energy to irradiate the sample, detect the NMR signal from the sample, and digitize that signal for processing by the host computer system. Support functions in the NMR console include power supplies, magnetic

field shimming current sources, and optional modules, including the variable temperature controller and the pulsed field gradient amplifier.

The spectrometer operator runs experiments using VNMR (the Varian NMR software package that runs on Sun workstations), Solaris, and CDE (Common Desktop Environment) software.

The following sections describe in general terms each major unit in the *MERCURYplus* spectrometer. Sections later in this manual provide a more detailed look at the units that make up each group of components.

Refer to the manual *MERCURYplus Technical Reference* for detailed specifications and theory of operation for each spectrometer board and module. The schematics listed in that publication are available in the publication *MERCURYplus System Schematics*.

Host Computer

The *MERCURYplus* host computer system is the user interface to the spectrometer. **Figure 1** shows a typical host computer system, consisting of a Sun workstation with a CPU, monitor, keyboard, hard drive, mouse, CD-ROM, optional tape, and hard drives.

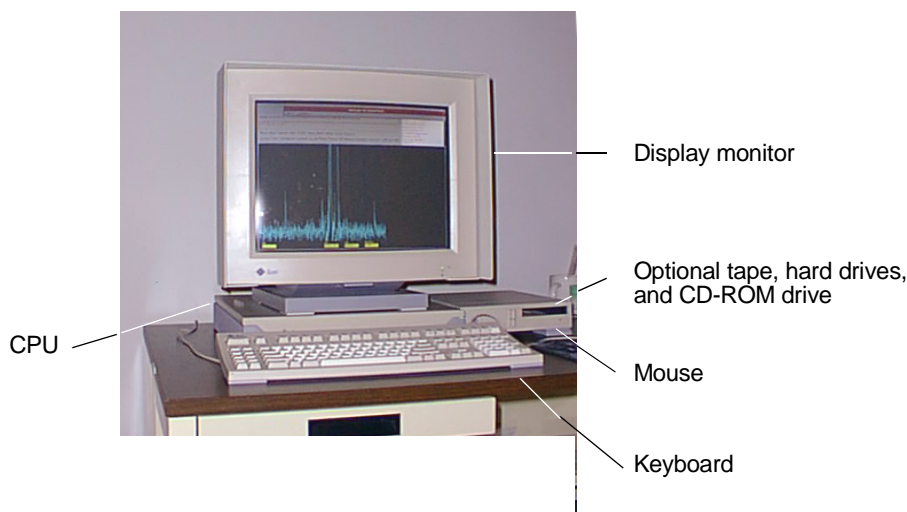


Figure 1. Typical Host Computer System

The VNMR software and the Sun Solaris operating system, with CDE desktop software installed, are resident in the CPU. Developed by Sun, IBM, HP, and Novell, CDE integrates the VNMR software with the UNIX operating system and creates an environment in which the software can be tailored to the user's needs and preferences. CDE includes a tool bar, file manager, icons, and point-and-click capability.

The display monitor shows FIDs, spectra, system configuration, and other information. A CD-ROM drive is standard on Sun workstations. Additional hard drives as well as 1/4-inch, 8-mm, and 4-mm tape backup drives, are also available. This additional storage is especially necessary when large quantities of data are processed.

Many versions and configurations of Sun hardware are available. The amount of RAM, size of the hard drives, and types of accessories needed depend on the scope of the spectrometer experiments.

When new applications are planned for an existing spectrometer, Varian product support personnel can assist in determining the correct configuration and in suggesting any other updates required to expand the spectrometer's capability.

Magnet

The magnet assembly consists of a superconducting, gradient-compensated solenoid enclosed in a low-loss liquid nitrogen and liquid helium dewar. **Figure 2** shows a 300-MHz magnet assembly.

The ambient-temperature bore tube through the solenoid axis is provided for insertion of the room temperature shim coils and probe assembly.



Figure 2. 300- MHz Magnet Assembly

Probe and Upper Barrel

The probe assembly for the magnet consists of two parts: a unit that extends through the top of the dewar, called the upper barrel, and a lower unit, called the probe. **Figure 3** shows both parts.

These two units are mounted within the vertical axis of the magnet and are tightly mated, with the probe inserted from the bottom of the magnet and the upper barrel mounted from the top.

The probe contains tuned rf circuits that transmit energy to the sample and receive the resulting NMR signal. For NMR observations at different frequencies, most probes have circuits that are tunable through controls located on the probe.

Systems equipped with the variable temperature (VT) accessory have probes incorporating heating and sensing elements. Good thermal isolation results because these elements are located within a dewar jacket inside of the VT probe. The optional PFG (pulsed field gradient) probes have an additional coil that applies a magnetic field gradient for coherence selection or solvent suppression.

The upper barrel contains the liquid sample spinning mechanism, tachometer (to sense rotational speed), and pneumatic sample insertion and ejection system. The liquid sample is placed into the upper barrel and conveyed by a cushion of air to the air bearing, which



Figure 3. Probe Assembly

allows the turbine and sample to rotate at a controlled rate. The PFG probe may require a PFG upper barrel.

An assortment of probes are available, including $^1\text{H}/^{13}\text{C}$ computer-switchable, broadband, Autoswitchable (4-nucleus), indirect detection, and Nano probes.

Magnet Interface box

The magnet is mounted on a magnet stand, and the preamplifiers and pneumatics are installed in the Magnet Interface Box (MIB) (see [Figure 4](#)), which is placed adjacent to the magnet.

Lock and observe channel preamplifiers, rf tune interface, sample handling pneumatics, and the Automatic Liquid/Solvent (Auto Liq/Sol) Spinner board are contained in the Magnet Interface box.

The Auto Liq/Sol Spinner board has an embedded controller and is used to regulate liquid and solid sample spinning. This board also has a tune detector for probe tuning, an analog meter driver to display spin speed and probe tune level, and a register to track the probe identification. The spinning valves are located in the Magnet Interface box.

A proprietary unit called the Q-Tune detector provides a simple way to quickly tune the various high- and low-band probes. The unit contains a directional coupler, rf diode detector, software feedback, and a reflected rf signal output.



Figure 4. Magnet Interface box

NMR Console

The NMR console consists of one small, compact cabinet (see [Figure 5](#)). The front door is hinged to swing open and is removable. The optional pulsed field gradient (PFG) amplifier and optional variable temperature accessory are accessible through this door.

When the NMR console is viewed from the rear (see [Figure 6](#)), the left-hand cardcage contains the digital boards, including the acquisition CPU. The right-hand cardcage contains the rf-related boards and the shim/DAC driver.



Figure 5. NMR Console, Front View

The system power supply module contains the power supplies for the rf cardcage and two rf power amplifiers. The power supply for the digital cardcage is inside the console and is attached to the side of the digital cardcage. Access is obtained by removing the console side panel.

System Options

MERCURYplus spectrometers can be equipped with many options, including the following:

- Pulsed field gradient (PFG) amplifier
- Variable temperature (VT) accessory
- NMS Sample changers (9, 50, or 100 samples)
- Nanoprobe Multi Sampler (NMS)
- Versatile Auto Sample Transport (VAST)
- RF Amplifier Options:
 - 35W/35W 4-Nucleus rf configuration
 - 75W/125W Broadband rf configuration
 - 100W/300W CP/MAS rf configuration
- Printers and plotters for color and higher-resolution data output

The following upgrades can be purchased directly from Sun:

- Additional RAM modules
- Floppy drive
- Tape drives
- Additional hard disk drives

Refer to “Optional Modules,” page 17, for more information about options.

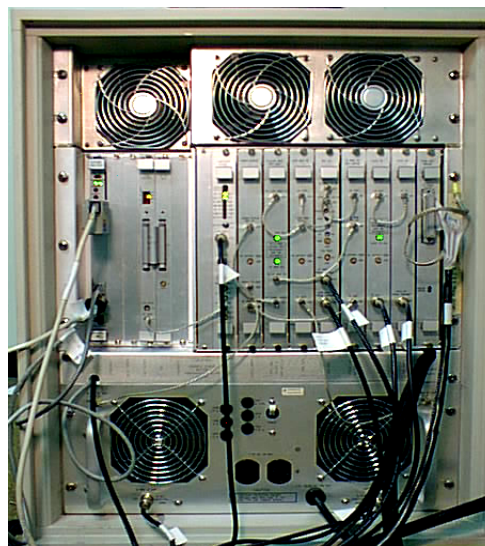


Figure 6. NMR Console, Rear View

Functional Description

This section provides a functional description of the units that make up the hardware of the *MERCURYplus* NMR spectrometer. The system consists of the following units:

- Host computer (Sun workstation) provides the user interface.
- Acquisition system controls the spectrometer boards and modules. This system is connected to the host computer through Ethernet.
- RF system includes receiver- and transmitter-related processes and shim/DAC drivers.
- Magnet and probe holds the sample in a homogeneous field for irradiation.
- Power supplies provide stable regulated power for use throughout the NMR console.

Figure 7 shows the system block diagram.

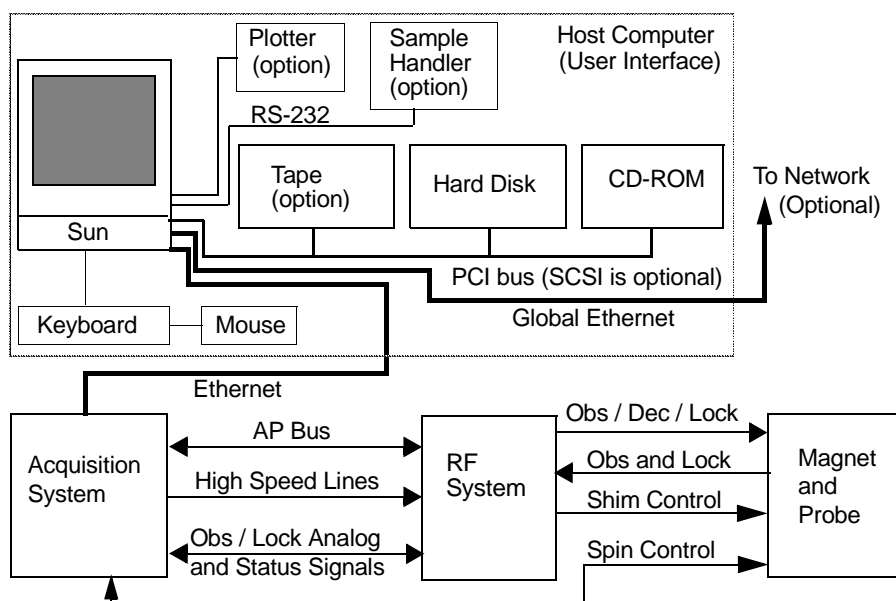


Figure 7. System Block Diagram

Host Computer

The minimum configuration for a host computer consists of a Sun workstation with CPU, 8-gigabyte hard drive, 256-megabytes RAM, CD-ROM drive, color monitor, keyboard, and mouse. Options include streaming magnetic tape, 4- or 8-mm tape, more RAM, plotters and printers, and a larger or second hard drive.

A larger or second hard drive is recommended if data is to be archived on the host computer.

The spectrometer software package includes the Sun Solaris operating system, CDE (Common Desktop Environment) software, and Varian VNMR software. CDE provides the tool bar and other useful interface elements, and VNMR manages the experiments. VNMR is integrated with UNIX features, such as the CDE file manager.

Acquisition System

The acquisition system uses a stand-alone processor that digitally controls the spectrometer, acquires digital data and responds to signals from the spectrometer. The acquisition system is located in the digital cardcage of the NMR console and uses the VME bus on its backplane. **Figure 8** shows the digital cardcage.

The acquisition system is composed of the following boards:

- Acquisition CPU
- Sum-to-Memory (STM)/Output
- Analog-to-Digital Conversion (ADC)

Figure 9 shows a system block diagram for the acquisition system. Each board in the system is described separately below.

Acquisition CPU Board

The Acquisition CPU board is a VME bus-compatible, single-board microcomputer, based on a Motorola 68040-series microprocessor with 4 serial ports and an Ethernet port. The board uses Wind River Systems VxWorks, a real-time operating system, to provide an Ethernet protocol for communications with the host. The board includes internal jumpers to establish the correct connections for the spectrometer.

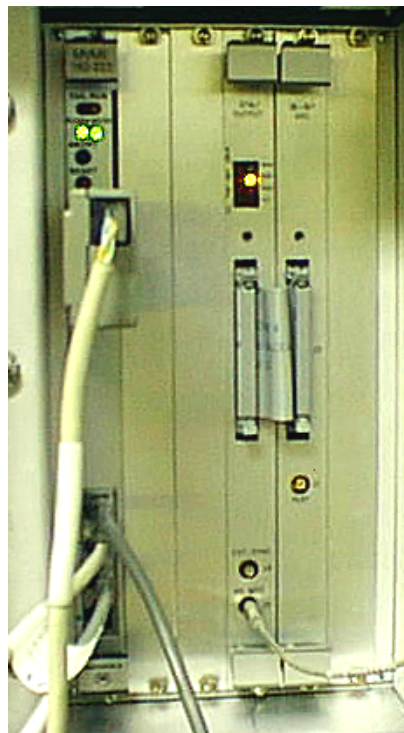


Figure 8. Digital Cardcage

STM/Output Board

The Sum-to-Memory (STM) and Output board is a data acquisition controller and a precision timing generator used to control the rf system via the high-speed (HS) bus and the analog port (AP) bus. The acquisition CPU programs the STM/Output board with event

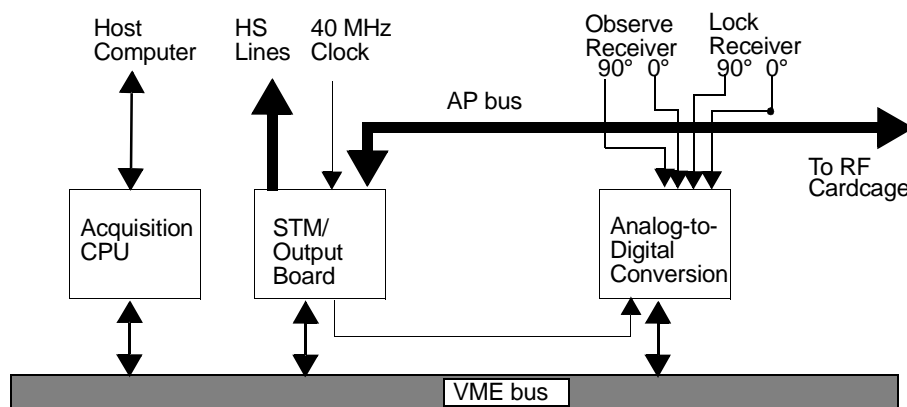


Figure 9. Acquisition System Block Diagram

sequence and timing that pulses the transmitter, turns on the receiver, digitizes the analog data, and finally sums the data to memory for later data manipulation by the host computer.

The output section of the STM/Output board generates the HS bus with a resolution of 100 nanoseconds for gating control. The AP bus, with a 1.2-microsecond resolution, is also generated and is used for configuring various functions on the rf boards and for synchronized communicating with the STM section.

The STM section decodes the AP bus to execute the following tasks: count down the NT and NP counter, increment the source and destination address of the NMR data, access memory to store the data, and control the phase cycling, summing or subtracting of the NMR data as required.

Analog-to-Digital Conversion Board

The 16-bit Analog-to-Digital Conversion (ADC) board receives analog FID signals from the observe and lock receivers. The board samples the 0° and 90° NMR signals at the same time, providing true quadrature detection. A third 14-bit ADC is present for continuous monitoring of the lock level.

RF Generation and Control

The rf cardcage (shown in [Figure 10](#)) of the NMR console contains boards that generate observe and lock rf signals, reference signals, and shim/DAC drive signals. The cardcage also contains the receiver control and processes the rf signals from the probe.

The rf functions are the transmitters, observe receive channel, and lock transmit/receive channel. The shim/DAC board is also in the rf cardcage. Each function is discussed in the following sections.

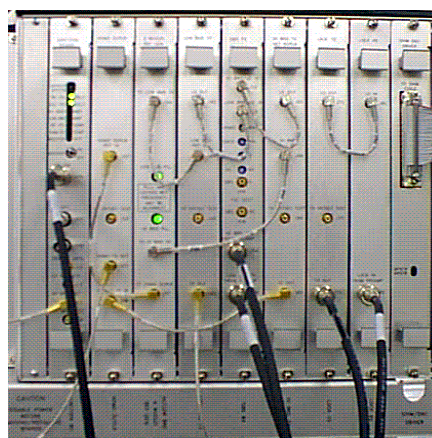


Figure 10. RF Cardcage

Transmitters

The transmitters in the spectrometer provide rf signals to irradiate the sample with the appropriate frequency and power level. [Figure 11](#) shows a block diagram of the observe channel. The transmitters consist of the following hardware:

- The High/Low Band Reference Generator board or 5-Nucleus Reference Generator board provides the local oscillator (L.O.) signals.
- The Junction board generates and distributes the 40-MHz oscillator reference signal used throughout the system and provides the various gating and control signals used by the rf boards.
- The two transmitter boards generate the signals required for high-band (^1H and ^{19}F) and low-band (^{31}P to $^{15}\text{N}/^2\text{H}$ range for broadband, $^{31}\text{P}/^{13}\text{C}$ for the 4-nucleus systems). The range is determined by the reference generator. One transmitter is used for observe and the other can optionally be used as a decoupler. Note that this arrangement allows use as $^{13}\text{C}\{^1\text{H}\}$ as well as $^1\text{H}\{^{13}\text{C}\}$. The software automatically selects the transmitters based on the transmitter nucleus and the decoupler nucleus.

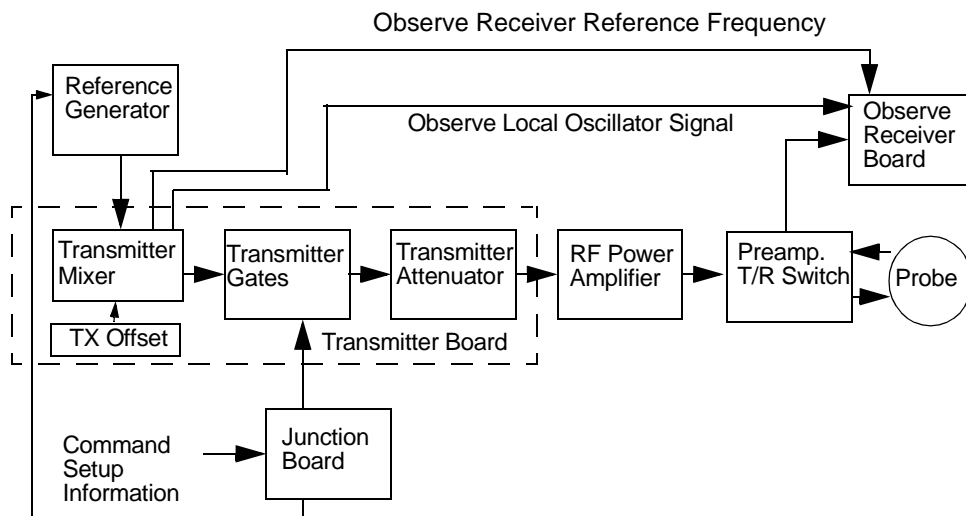


Figure 11. Observe Channel Block Diagram

- The transmitter boards mix the L.O. signal with the intermediate frequency signal to produce the rf signal. The rf signal is then routed through a computer-controlled attenuator section of the transmitter board to set the amplitude level. The signal next goes to the rf power amplifier to raise the level of the signals. The rf power amplifier changes the observe channel signals to a desired level without distortion or undesired phase change.

From the rf power amplifier, the signal goes through the active transmit/receive (T/R) switch of the high-band preamplifier. The low-band preamplifier uses a passive T/R switch. The preamplifier transmitter output is used at the probe to irradiate the sample. The transmitter boards also send the observe receiver reference frequency signal and the observe local oscillator signal to the Observe Receiver board.

The output of the transmitters can be single frequency or broadband. When used as a decoupler, the signal can be phase-modulated to produce a band of frequencies. Waltz, Garp, MLEV16, XY32, square, and fm modulations are available.

Observe Receiver

Figure 12 is a block diagram of the observe receiver system. This system consists of the following hardware:

- The Preamplifier board receives the signal from the probe and amplifies the signal before sending the signal on to the Observe Receiver board.
- The Observe Receiver board is part of a double-conversion receiver system. The function of the receiver is to process the information from the preamplifier into a form suitable for digitalization.

The receiver (100-kHz bandwidth) has three gain stages that amplify the signal to the desired level. After amplification, the signal is then phase-detected into two channels of NMR information, with one channel referenced to 0° and the other referenced to 90° . The NMR signals are then passed through matching audio 4-pole Butterworth filters and output from the receiver to the Analog-to-Digital Conversion board in the acquisition system for digitalization.

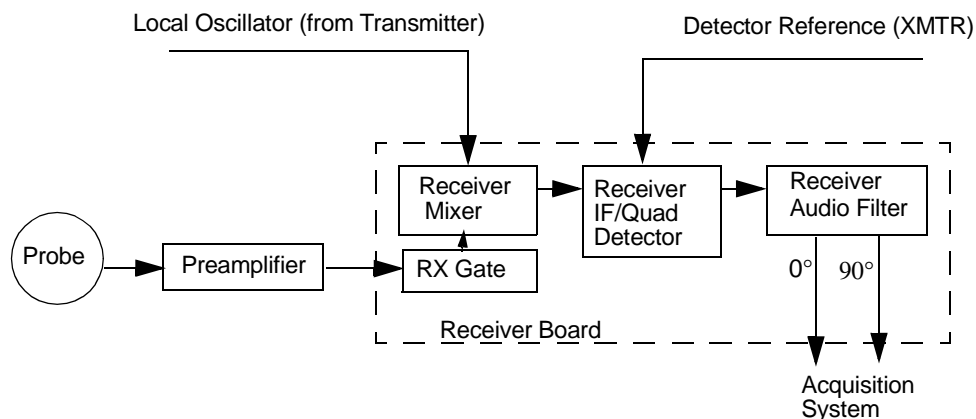


Figure 12. Observe Receiver Channel

Decoupler

Decoupling enables the operator to unlink different nuclei from the observe nucleus. For example, the operator can decouple the proton from the carbon nuclei. The output of the decoupler channel can be a single frequency, pulsed or continuous wave, modulated to produce a band of frequencies. “Transmitters,” page 12, describes decoupling transmitter options.

Lock Channel

Figure 13 is a block diagram of the lock system. This system compensates for the intrinsic drift of a superconducting magnet field.

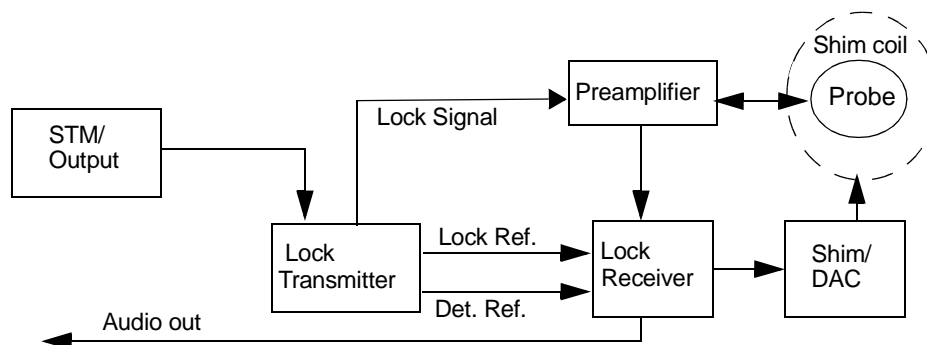


Figure 13. Lock System Block Diagram

While it may take several years for a magnet to drift out of its usable range, even a small amount of drift can adversely affect experiments. The lock system constantly monitors the NMR frequency of the lock solvent, usually deuterium, and when a change in frequency is detected, an error-correction signal is applied to the room temperature shim coil to keep the magnetic field constant.

The lock transmitter generates the lock transmitter frequency at the selected pulse rate and applies the signal to the lock coil in the probe. The signal from the sample is detected by the lock coil in the probe and applied to the lock preamplifier, where it is amplified and routed to the lock receiver.

In the receiver section, the lock signal is amplified, mixed with the local oscillator, phase detected into 0° and 90° signals, and applied to the lock loop circuit to keep the field constant. Outputs from the receiver section also go to the Analog-to-Digital Conversion (ADC) board to be digitized for the lock display, manual shimming, or AutoShim.

Homonuclear Decoupler Board

The Homonuclear Decoupler board generates a gated ^1H homo decoupler signal. This board has three versions, one for each magnet proton frequency (200, 300, or 400 MHz).

Automated Deuterium Gradient Shimming Relay

The Automated Deuterium Gradient Shimming relay switches the low-band preamplifier and transmitter to the ^2H coil in the probe. This allows automated acquisition of a deuterium spectrum for gradient shimming.

Probes

The probe is an electronic device designed to irradiate rf energy into the sample during the transmit cycle. The sample then reradiates low-level NMR rf energy that is picked up by a coil in the probe. The upper barrel holds the sample, which spins at a controlled rate. The design of the upper barrel allows easy insertion and withdrawal of the sample from the magnet. Varian provides a large number of probes that can be used for many types of experiments.

Magnet Interface box

The Magnet Interface box (MIB) contains the following units:

- *High-band preamplifier* – Amplifies $^1\text{H}/^{19}\text{F}$ signals using GaAs FET for optimum signal-to-noise performance, and provides the active transmit/receive (T/R) switch function.
- *Broadband preamplifier* – Provides for low-noise amplification of the probe output. The probe signal received by the broadband preamplifier is amplified and the output is passed to the receiver.
- *Lock preamplifier* – Amplifies the lock signal to a usable level while maintaining a high signal-to-noise ratio.
- *Air valve* – Controls sample insertion and ejection from the magnet.
- *Flow meters* – Measure the rate at which the liquid nitrogen and liquid helium within the magnet dissipate into the atmosphere. Two more flow meters are used to measure gas flow for the variable-temperature (VT) accessory and probe body cooling.
- *Auto Liq/Sol spinner board* – Converts pulses in revolutions per second from the spinner detector for a meter display. The tuning signal is also amplified and fed to the meter.

System Power Supply

The system power supply (see [Figure 14](#)) provides regulated dc voltages to all NMR console units, the rf cardcage, the magnet/console interface, and the two rf power amplifier modules. The digital cardcage has its own power supply.



Figure 14. System Power Supply

The system power supply contains separate power supplies that require 110/120 Vac, 50 or 60 Hz, single-phase power. An external transformer is necessary for voltages other than 110/120. A single 110-Vac, 400-watt duplex outlet for connecting printers and plotters is mounted on the back of the unit. The system power supply provides ac line connection, line filtering, dc output cables, and temperature monitoring.

Optional Modules

The *MERCURYplus* NMR spectrometer has several optional modules: pulsed field gradient (PFG) module, variable temperature accessory, and sample changer. The host computer system also has numerous options available to meet computational needs.

Performa I or Performa II PFG Module

The Performa I and Performa II pulsed field gradient (PFG) modules increase the spectrometer system capabilities in high-resolution liquids experiments. This module can perform many functions, including solvent suppression and coherence pathway selection. The gradient system produces pulses with strengths of up to 18 gauss per centimeter for Performa I and up to 60 gauss per centimeter for Performa II, depending on the probe. The gradient subsystem produces an intense gradient and then returns to spectroscopic conditions quickly.

The PFG amplifiers use transconductance technology to eliminate the need for matching with the gradient coils and provides the most reproducible gradient pulses. A fast rise-time allows gradient to be turned on and off rapidly. The amplifiers contain thermally shunted components for increased stability.

The PFG module requires a PFG amplifier, an optional PFG upper barrel, and PFG probe. **Figure 15** shows the Performa I amplifier in the NMR console. The Perform II is located in a separate housing on top of the console. Each of these components is specified and sold separately as options.

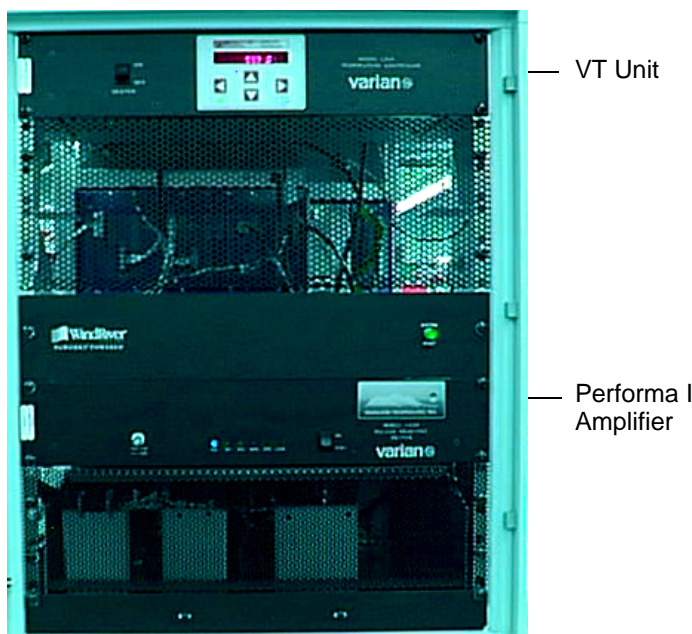


Figure 15. PFG Amplifier in NMR Console

Variable Temperature Accessory

The variable temperature (VT) accessory allows precise computer control of sample temperatures between -150° and 200° C, as set by the user. The temperature is controlled by flowing gas, usually nitrogen, around the sample in the probe.

For temperatures above ambient, gas at room temperature is introduced at the gas inlet on the probe and heated to the desired temperature by a heater element. For temperatures below ambient, gas at room temperature is cooled below the desired temperature by passing

the gas through a heat exchanger immersed in a cryogenic substance, typically liquid nitrogen. The cooled gas then flows to the probe through a vacuum-jacketed transfer tube, and a heater element in the probe warms it to the required temperature. In addition to the basic VT unit, low temperature modules are available.

Automated Sample Management

Sample management systems available as options include the 9-sample carousel autosampler, the 50- or 100-sample management system (SMS), Nanoprobe Multi Sampler (NMS), and Versatile Automatic Sample Transport (VAST) sample management system.

CP/MAS Accessory

This accessory provides the spectrometer with a 100 W high-band and a 300 W low-band amplifier, as well as pneumatics to do either manual or computer controlled high speed spinning. Five mm and seven mm narrow bore probes are available. This accessory gives the user the ability to perform cross polarization/magic angle spinning experiments.

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