



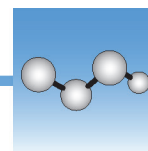
NMR

picoSpin Spectrometer

Frequently Asked Questions

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Frequently Asked Questions

General Questions

Is the Thermo Scientific™ picoSpin™ 45 NMR spectrometer really the world's first compact NMR spectrometer?

Haven't desktop NMR machines been around for years?

In a proton NMR spectrum, lines are typically separated by a few parts per million (ppm). To qualify as a true spectrometer, an NMR system must have resolution well below 1 ppm. The picoSpin 45's resolution is better than 0.06 ppm, or 60 ppb (parts per billion). Before the picoSpin 45, all commercial compact NMR machines had resolution that was too poor for them to qualify as spectrometers. They could be used to analyze NMR relaxation times, but they were not "high resolution" spectrometers in the sense usually meant in NMR. So yes, the picoSpin 45 really is the world's first compact NMR spectrometer.

How does it work?

The picoSpin is, in most respects, a conventional Fourier-transform proton NMR spectrometer. It has all of the usual NMR spectrometer components including a magnet, shim coils, programmable pulse sequencer, RF transmitter, solenoid RF coil, low-noise receiver and digital data acquisition system. What is different is simply that everything is so small. Instead of a large and expensive superconducting magnet, we use a small room-temperature permanent magnet. The electronic circuits are all miniaturized using techniques similar to those used in cell phones. The sample fluid is confined to a small capillary with an inside diameter of about 0.4 mm.

How stable is the picoSpin? Can I use averaging to improve the signal-to-noise ratio?

The system is stabilized by a magnet temperature controller and by software. If the sample itself behaves in a time-independent way, any number of single scans can be averaged together to improve SNR.

Can I look at flowing samples?

Yes, but spectroscopic resolution will suffer unless the sample is stationary during the NMR data acquisition. For most applications, either the inlet port or the outlet port should be closed or sealed during data acquisition, and there should be a small fluid volume between the closed valve or plug and the port. Otherwise, thermal expansion of the fluid can cause flow.

Why does the picoSpin have a capillary cartridge?

For convenient replacement. When reasonable precautions are observed, problems with clogging and contamination of the capillary can be avoided. However, accidents do occur, and when they do, it is a great advantage to be able to replace the capillary in the field. Cartridge changes only take a few minutes. The unit should be reshimmed whenever the cartridge is changed.

What experiments can I do other than 1-D spectroscopy?

The picoSpin contains a general-purpose programmable pulse sequencer with 20 ns time resolution. The sequencer-controlled RF oscillator has 32-bit frequency resolution, 8-bit phase resolution and 8-bit attenuator resolution. The pulse program can contain up to 1024 instructions. With these capabilities, essentially any proton NMR experiment is possible, including spin-echo T_2 measurements and T_1 inversion-recovery. The first released version of the picoSpin software (Version 0.8.*n*) supports 1D spectroscopy only.

Sample Handling

How do I inject a sample?

The spectrometer has an inlet connector and an outlet connector on the front panel. The fluid path between them is a capillary with an ID of about 0.4 mm and a total volume of about 30 μL for the picoSpin 45 and 40 μL for the picoSpin 80. Samples are most often injected into the capillary using a syringe. Kits of parts and accessories for sample handling are available.

What materials are in contact with the sample fluid?

Our standard cartridges use microbore PTFE capillary and a short section of quartz glass capillary at the location of the RF coil. The inlet and outlet front panel fittings are stainless steel. The optional inlet filter has a PEEK body and stainless steel frit filter. PEEK ferrules are used to connect the microbore PTFE capillary to the front panel fittings. Contact us to discuss other materials for special applications.

How do I remove a sample?

Just flush the capillary with either a clean solvent or your next sample.

Chemical Applications

What kind of samples can I measure?

Any proton-containing liquid, not too viscous for injection into a 0.4 mm ID capillary, can be used.

Will the signal I am looking for be strong enough to see?

Good question! It is always a good idea to estimate the signal-to-noise ratio (SNR) before you get started with unfamiliar samples. The exact SNR depends on many things, including the magnet shim and the way data is recorded and analyzed. However, you can usually make an adequate estimate like this: The picoSpin single-scan SNR for pure water is specified at 1000 for the picoSpin 45 and 4000 for the picoSpin 80 (although you can expect your instrument to have a higher value, so please scale accordingly), and the concentration of water molecules in pure water is 55 moles/liter. Since the SNR is directly proportional to concentration, you can scale from this case to estimate your SNR. You should also include a factor for the “weight” of the line relative to the weight of the water line. The weight is the number of protons per molecule contributing to the line times the line’s normalized intensity within a multiplet if it is not a singlet. (For example, a water singlet has a weight of 2 (H₂O), a CH₃ singlet has a weight of 3 and one line in a CH₃ doublet has a weight of 1.5.)

Let’s say your molecule has a concentration of 0.55 moles/liter, and you want to estimate the SNR of one line in a CH₃ doublet. That will be $1000 \times (0.55/55) \times (1.5/2) = 7.5$ for the picoSpin 45. You can increase the single-scan SNR by averaging many scans. The SNR increases as the square root of the number of scans – so if you average 100 scans, you can enhance the SNR by a factor of 10. (We use the spectroscopist’s definition of SNR, which is the peak height of the line divided by the RMS of the baseline height.)

How long does it take to get a spectrum?

This can vary widely. For a high-concentration sample of a simple molecule, you can get very useful information from a single scan acquired in less than a second. On the other hand, if you are looking for a low SNR line, and your sample has a long T₁ relaxation time, you may want to collect data for many minutes or even several hours. See [“Will the signal I am looking for be strong enough to see?”](#) to learn how to estimate the SNR.

Can I detect other nuclei besides hydrogen?

A special version of the picoSpin is available for fluorine. The electronics can be adapted for any frequency, but the SNR will not be adequate for most applications with nuclei other than hydrogen and fluorine. Carbon-13 satellites can be seen on proton spectra in favorable cases.

Do I need to use deuterated solvents?

Not in most cases. Deuterium is not used by the picoSpin to provide a lock signal, and receiver saturation is also not a problem, even with a pure water sample. You might want to use a deuterated solvent if you find that a solvent proton signal overlaps strongly with an important solute signal.

Installation

Where can I install a picoSpin spectrometer?

There are no special facilities or environmental requirements. You can place a picoSpin spectrometer in any indoor location where a person would be comfortable; however, temperature changes must be gradual during the course of a data acquisition. AC magnetic fields generated by nearby utility power that are stronger than 1 mGauss amplitude will cause small side-bands to appear on spectra. In unusual cases where this is a problem, the unit can be oriented so that the AC field vector is perpendicular to the magnet's field.

Will a picoSpin technician install the unit in my lab?

Set up and operation of the picoSpin is far simpler than for conventional NMR spectrometers. Customers with only a little or no knowledge of NMR should have no problem installing the picoSpin system.

Data Analysis

How do I analyze my data?

The picoSpin generates data in the industry-standard JCAMP-DX format. Any NMR data analysis package that can read this format may be used to analyze your data. A one-year license to Mnova™ NMR software from Mestrelab Research is included with each picoSpin spectrometer.

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