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## *Introduction*

Spectrostat$^{NMR}$ cryostats are purpose designed for Nuclear Magnetic Resonance measurements. The cryostats have been optimised to provide a variable temperature sample environment over the temperature range from 1.6 to 500 K.

Benefits include:

- Constructed from non-magnetic materials and components for use in sensitive NMR measurements
- Sample in exchange gas allowing fast change times for high sample throughput
- Optimised thermal design providing excellent control and stability of the sample temperature
- Cryostat outer diameters tailored to industry standard superconducting magnet bores; sample space diameters are maximised for experimental flexibility
- Data acquisition software to automate experiments

# *Spectrostat* NMR

## NMR measurements

The Spectrostat$^{NMR}$ range has been purpose designed for NMR measurements. In solid state NMR, variable temperatures may be used to study intramolecular rearrangements or to slow down chemical processes to study reactive intermediates.

## Spectrostat $^{NMR}$

The Spectrostat$^{NMR}$ is a top loading, exchange gas continuous flow cryostat. The sample is cooled by the circulating, temperature stabilised cryogen which flows into the exchange gas space.

The cryostat may be ordered in a configuration which extends the continuous operating range to 1.6 K. An adjustable needle valve in the side arm is used to control the temperature below 4.2 K. This innovative design allows continuous sample cooling at temperatures below the lambda point in a compact cryostat.

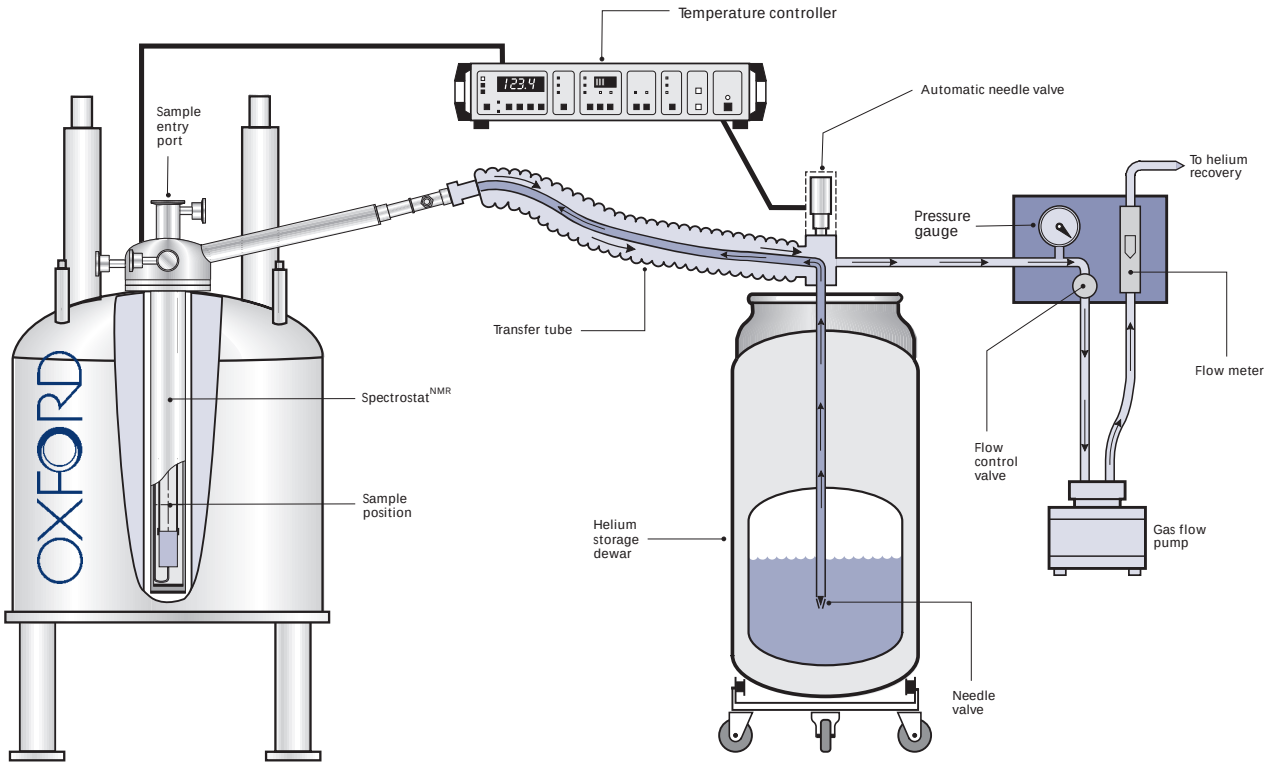
The high temperature limit of the cryostat may be extended to 500 K with the substitution of a high temperature thermocouple temperature sensor.

All materials in the construction of the cryostat have been selected for their compatibility with the extremely sensitive nature of NMR measurements. Only non-magnetic materials, including the cryostat's heater and sensor, are present.

## Continuous flow principle

A continuous flow cryostat comprises a heat exchanger, sample space, radiation shield and vacuum case. Cold liquid/gas from a separate liquid helium or nitrogen storage vessel is drawn through a flexible, vacuum-insulated transfer tube and circulated through the heat exchanger by a gas flow pump operating at room temperature. The exhausting gas cools the radiation shield by conduction, thereby eliminating the need for secondary refrigerants.

The flow is controlled by a needle valve fitted to the transfer tube and a flow controller situated in the gas exhaust line.




*Spectrostat$^{NMR}$ system with an NMR magnet*

Precise temperature control is obtained using a temperature sensor and heater fitted to the heat exchanger, in conjunction with a temperature controller. The relatively small thermal capacity of the heat exchanger ensures that stable temperatures are obtained rapidly.

## Spectrometer compatibility

The Spectrostat$^{NMR}$ range has been designed for use with industry standard superconducting magnets. The following table gives details for appropriate cryostat selection.

The cryostat is interfaced to the magnet with a clamping ring. Rings are available for most commercially available magnets. The cryostat length is customised to suit the individual magnet to ensure that the cryostat's isothermal sample region is aligned with the magnet centerline.

## Automated operation

Automated control of the helium flow in Spectrostat$^{NMR}$ cryostats is possible with the optional automatic needle valve. This, coupled with the advanced features of the ITC$^{503}$ temperature controller, allows fully automated control from 4.2 to 500 K.

Oxford Instruments ObjectBench software included with the ITC$^{500}$ series temperature controllers automates data acquisition. The software, which runs on a PC, takes measurements from any independent instrument (with a computer interface) at different user defined temperatures. Frequently run measurements may be configured for "hands off" operation. A demonstration of this software may be found on the Internet at http://www.oxinst.com/ri/software/obdemo.htm

### Spectrometer compatibility

| Spectrostat type | NMR Magnets |
|---|---|
| Spectrostat$^{NMR}$ 51/30 | Narrow bore: 100 to 600 MHz with a room temperature bore diameter of ≥51 mm |
| Spectrostat$^{NMR}$ 70/49 | Wide bore: 200 to 500 MHz with room temperature shim coils providing 73 mm access |
| Spectrostat$^{NMR}$ 86/62 | Wide bore: 200 to 500 MHz with a room temperature bore of 88 mm |

### System components

| Item | Description |
|---|---|
| GFS600 | Ultra low loss liquid helium/nitrogen transfer tube |
| GF3 | Gas flow pump |
| VC31 | Gas flow controller |
| ITC503 | Temperature controller |

### Optional items

| Item | Description |
|---|---|
| SPECTROLT | Continuous 1.6 K base temperature |
| SPECTROHT | 500 K high temperature |
| GFS650 | Automatic GFS600 (not compatible with SPECTROLT) |
| TTL100 | Low loss cryogen transfer tube |

## *Specifications*

| | Spectrostat$^{NMR}$ 51/30 | Spectrostat$^{NMR}$ 70/49 | Spectrostat$^{NMR}$ 86/62 |
|---|---|---|---|
| Sample space diameter (mm) | 30 | 49 | 62 |
| Tail outer diameter (mm) | 51 | 70 | 86 |
| Temperature range (K) | 3.6–400 | 3.6–400 | 3.6–400 |
| Base temperature with SPECTROLT option (K) | 1.6 | 1.6 | 1.6 |
| High temperature with SPECTROHT option (K) | 500 | 500 | 500 |
| Temperature stability (K) | 0.1 | 0.1 | 0.1 |
| Cooldown time from ambient (min) | 40 | 60 | 90 |
| Liquid helium consumption at 4.2 K (l/hr)<br>at 10 K (l/hr)<br>at 150 K (l/hr) | 1.2<br>1<br><0.5 | 1.2<br>1<br><0.5 | 1.3<br>1<br><0.5 |
| Weight (kg) | 10 | 14 | 17 |

*Oxford Instruments reserves the right to amend specifications without notice.*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