

## A Brief Overview of NMR probes in the Department of Chemistry at the University of Toronto

By Dr Darcy Burns, NMR Laboratory, Department of Chemistry

[Note by Bryan Jones: The Department's first NMR was a Varian A60 60MHz permanent magnet instrument which was acquired *circa* 1961].

1. Probe: Varian V-4333  
Spectrometer: HA-100

Details: The V-4333 NMR probe (IMG 4470 and 4473) was manufactured by Varian Associates and was designed for use with a Varian HA-100 NMR console and its associated 23.5 kG permanent magnet. The spectrometer operated in swept, or continuous wave (CW), fashion and probed the H1 nucleus at a frequency 100 MHz.

Provenance: The V-4333 NMR probe was included as part of the 1965/1966 purchase of a Varian HA-100 spectrometer. Prior to this purchase the Department owned both a Varian A60 NMR spectrometer along with a Varian DP60 spectrometer. Both spectrometers excited the H1 nucleus at frequencies of 60 MHz, however, the DP60 spectrometer was outdated and not in general use, while the A60 was approaching end-of life.\* Thus, the department was in need of a new spectrometer that could be broadly used by organic faculty. In 1965, Prof. William Reynolds and Prof. Peter Yates submitted a grant to update the aging A60 NMR spectrometer to the newer HA-100 model, with a vendor buy-back option on the (valuable) DP60 electromagnet. The V-4333 probe and HA-100 spectrometer were installed in 1966 and remained in service until 1975, when it was replaced with an XL-100 NMR spectrometer operating at 100 MHz. The XL-100 spectrometer was the Department's first pulsed Fourier transform (FT) NMR spectrometer. \*The A60 was no longer in production by 1972.

Points of interest:

- a) In 1968, shortly after the HA-100 purchase, the department was successful in its joint application for a Varian high resolution HR-220 NMR spectrometer equipped with a superconducting cryomagnet. The strategic grant was jointly submitted by the University of Toronto and MacMaster University in an effort to secure Canada's first superconducting NMR magnet, operating at 220 MHz. The Varian HR-220 was sited at the Ontario Research Foundation, which was located midway between the two campuses at Sheridan Park. The facility shut its doors permanently in 1972-1973 and the HR-220 NMR spectrometer was decommissioned as part of the closure. The HR-220 was never recommissioned, in part due to the high running costs associated with helium fills for the superconducting magnet.
- b) A typical NMR spectrum, as acquired on the HA-100 NMR spectrometer (IMG 2347).

***For further information on historical aspects see*** Historical Distillates: Chemistry at the University of Toronto since 1843 (Brook, A.)

2. Probes: Varian 5mm 4 nucleus probe, operating at 300 MHz

Spectrometer: Varian Gemini 300

Details: The Varian 5mm 4-nucleus probe (Model number 4N/0190211700, (IMG 4465a and b), operating at 300MHz, was designed for use with a Varian Gemini 300 console and its associated 47.58 kG superconducting Oxford 'R2D2' magnet. The spectrometer operated using pulsed FT technology and, in this case, probed the H1 nucleus at a frequency of 300 MHz. The probe was of direct-detect design, while the '4N' in the model number indicates that the probe was capable of exciting four nuclei: H1 or F19, C13, and P31. Specifically, this probe was simultaneously tuned to 1H and F19 as well as C13 and P31.

Provenance: The Department was in a mode of continual upgrade in the two decades following the purchase of its XL-100 NMR spectrometer. Briefly; an A5660 was acquired by Prof. Jim Thompson in 1967 for F19/H1 experimentation; two T60 NMR spectrometers for routine use in the department, a CFT-20 was acquired by Prof. Adrian Brook in 1973 for 13C-detection with H1 decoupling (the instrument was too unstable for H1-detection); a Bruker 'workhorse' WH400 was acquired in 1978 as part of a joint grant submitted by a consortium of Universities<sup>\*\*</sup>; a Varian XL-200 was purchased in 1980 and included the Department's first superconducting magnet; a Varian XL-400 was acquired by Professor William (Bill) F. Reynolds in 1984; Canada's first Gemini 200 console was acquired by Prof. Reynolds in 1987<sup>\*\*\*</sup>; a Varian Gemini 300, along with a second Gemini 200, were acquired c. 1991. The chief advantage of the Gemini spectrometers was their low cost combined with ease of use. The 5mm Varian switchable probe, operating at 300 MHz for H1, was included in the Gemini 300 grant and was subsequently utilized as a general purpose probe for the Department.

<sup>\*\*</sup> Reminiscent of the Bruker HR-220 siting, the Bruker WH-400 was housed on neutral ground at an Ontario Regional Research Centre located at Guelph University.

<sup>\*\*\*</sup> Professor Bill Reynolds had the opportunity to observe a newly manufactured Gemini 200 during a product demonstration and subsequently applied for the console before anyone in Canada knew of its existence.

3. Probe: Varian 5mm 1H/19F, operating at 400 MHz

Spectrometer: XL-400, VXR-400

(At present, it is unclear if this is the 1H19F/13C31P, Model 4N/0190211700, or is the same model as the previous probe).

Provenance: 1992 saw the installation of a new departmental Varian Unity Spectrometer, operating at 500 MHz. The associated grant included provision for an upgrade to the Varian

XL-400 spectrometer, which had been in use for eight years and was no longer in production. As a result, the XL-400 was converted to a Varian VXR-400 and its accompanying selection of probes were used for routine NMR acquisition. One of these probes was the 5mm Varian 1H/19F probe (IMG 4465c), operating at 400 MHz for H1 or 37.5 MHz for F19.

4. Probes: 300 MHz Autonmr, 300 MHz 4 nucleus 13C/31P 1H/19F, 300 MHz SW broadband, 300 MHz 10mm broadband

Spectrometer: Mercury Series

Details: Each of the probes listed above was capable of acquiring H1 NMR data at 300 MHz. The Autonmr probe was tunable on the high band and low band channels, although most likely was set up to acquire H1 and C13 NMR spectra (IMG 4464a). The 4N ('four nucleus') band probe offered the same capabilities as, but was an updated version of, the probe listed in Section 2 (IMG 4464b). The 300 MHz SW (switchable) broadband probe (IMG 4464c) was tunable on the broadband channel and was therefore capable of acquiring NMR data for the range of nuclei that spanned 121 MHz (P31) to 30 MHz (N15). The Varian 10mm broadband probe (IMG 4464d) was capable of acquiring NMR data for nuclei in the frequency ranges of N15 to P31 and was designed for use with 10mm-diameter tubes. The larger 10mm tube size permitted additional analyte to be positioned within the coils of the probe, thereby boosting the signal-to-noise ratio for cases where analyte was readily available or poorly soluble.

Provenance: The department further improved upon its NMR holdings via the purchase of Mercury series spectrometer between 1995 - 1997. Each of the probes listed above were procured over time, beginning after the purchase of the Gemini 300 and spanning through to and beyond the purchase of the Mercury 300 spectrometer. These probes (the 10mm broadband probe notwithstanding), offered the department an opportunity to acquire simple 1D NMR spectra reliably and with reasonable ease of use.

5. Probe: Bruker 200 MHz Triple Resonance Magic Angle Spinning Wide Bore probe with CP and Variable Temperature capability (i.e. the MAS..WB.CP.BB.VTN-BL probe).

Spectrometer: DRX

Details: The Bruker Triple Resonance solid state MAS probe, operating at 200 MHz, contained a decoupling (CP) channel and broadband-tunable X-nucleus channel. The probe was designed to perform solid state experiments at variable temperatures ranging from -120°C to +120°C.

Provenance: The Department expanded its scope of NMR capabilities in 1995 with the purchase of a 200 MHz Bruker wide-bore DRX spectrometer via a grant submitted by Prof. Geoff Ozin. This spectrometer was capable of acquiring NMR data on solid state samples, which was of particular importance to the inorganic and materials groups. The probe pictured in IMG 4475 was acquired from Guelph University for use with the Bruker DRX NMR spectrometer, and was tunable to a broad range of NMR active nuclei spanning P31 (81 MHz to N15 (20 MHz).