TEXAS A&M UNIVERSITY
NMR
NEWSLETTER

No. 434

November 1994

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Iso. (%D)	Density	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	[illegible]	[illegible]	[illegible]	1.12	[illegible]	[illegible]	0.551 (32)
D-120	[illegible]	[illegible]	[illegible]				
D-13	Acetone-d_6	CD_3COCD_3	99.[illegible]	0.87	[illegible]	[illegible]	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14	[illegible]	[illegible]	[illegible]				[illegible]43 (20)
D-21	[illegible]	[illegible]	[illegible]				[illegible] 611
D-122							
D-130							
D-28	Chloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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Cat. No.	Size	Instrument Model	Type
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WCV-60 ([illegible]-60C)	[illegible]1" X 26"	[illegible]A-60, [illegible]-60, [illegible]A-60D	Gridded-Two Color
WCV-XL ([illegible])	[illegible]	[illegible]	[illegible]
WCV-XLFT-[illegible]	[illegible]	[illegible] and FT)	[illegible]
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
[illegible]	[illegible]	[illegible]	[illegible]
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WC-11[illegible]	X 17"	All Models [illegible] 16 1/2" or 16	Blank

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FORTHCOMING NMR MEETINGS

36th ENC (Experimental NMR Conference), Boston, MA, **March 26 - 30, 1995**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.

Keystone Symposia on Molecular and Cellular Biology, Frontiers of NMR in Molecular Biology - IV, Keystone, Colorado, **April 3 - 9, 1995**; Organizers: S. W. Fesik, T. L. James, and G. Wagner; Contact: Keystone Symposia, Drawer 1630, Silverthorne, CO 80498; Phone: (303) 262-1230; Fax.: (303) 262-1525.

International School of Biological Magnetic Resonance, 2nd Course: Dynamics and the Problem of Recognition in Biological Macromolecules, Erice, Trapani, Sicily, Italy, **May 22 - 30, 1995**; Contact: Prof. O. Jardetzky, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, CA 94305-5055; Phone: (415)723-6270; Fax: (415) 723-2253; or, Prof. J.-L. Lefèvre, ESBS, CNRS-UPR9003, Univ. Louis Pasteur, Blvd. Sébastien Brant, F67400 Illkirch Graffenstaden, France; Phone: (+33) 88-655269; Fax.: (+33) 88-655343- See TAMU NMR Newsletter 432, 38.

12th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, Manchester, England, **July 2 - 7, 1995**; Contact: Dr. J. F. Gibson or Ms. G. B. Howlett - See TAMU NMR Newsletter 415, 5; Phone: (44-71) 437-8656; Fax: (44-71) 437-8883.

ISMAR 1995, Sydney, NSW, Australia, **July 16-21, 1995**; Contact: Dr. Les. Field, Dept. of Organic Chemistry, Univ. of Sydney, Sydney, NSW 2006, Australia. Phone: +61-2-692-2060; Fax: +61-2-692-3329; Email: ismar-95@biochem.su.oz.au Also, see TAMU NMR Newsletter 419, 26.

NMR Symposium at the 37th Rocky Mountain Conference on Analytical Chemistry, Denver Colorado, **July 24-27, 1995**; Contact: Dr. Alexander J. Vega, DuPont Central Research and Development, P.O. Box 80356, Wilmington, DE 19880-0356; Tel. (302) 695-2404; Fax: (302) 695-1664; e-mail: vego@esvax.dnet.dupont.com. See TAMU NMR Newsletter 432, 34.

37th ENC (Experimental NMR Conference), Asilomar Cnference Center, Pacific Grove, California, **March 17 - 22, 1996***[sic]*; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997***[sic]*; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.

Additional listings of meetings, etc., are invited.

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Dr. Bernard Shapiro
TAMU NMR Newsletter
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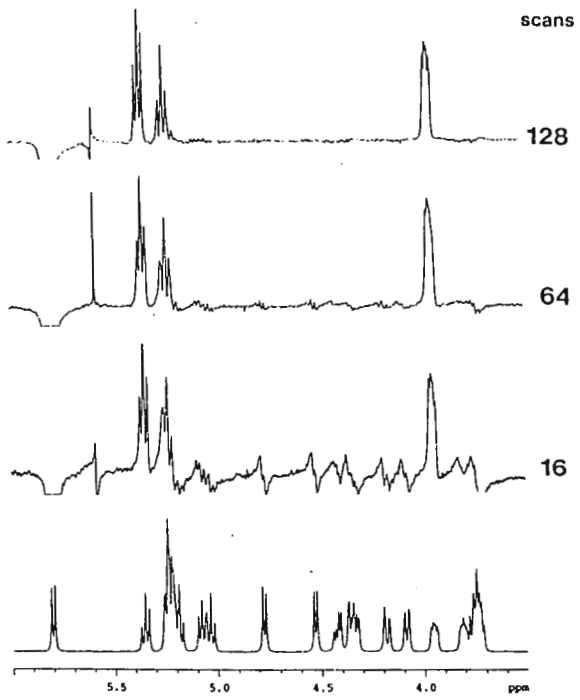
Palo Alto, CA 943030 USA

September 20th 1994
(received 9/24/94)

FIRST STEPS ON BRANDNEW BRUKER DRX-SPECTROMETERS

Dear Dr. Shapiro

equipped with two brandnew BRUKER DRX-spectrometers we have made our first adventurous steps from our old AM 400 into the fascinating world of modern NMR. Among the various sophisticated techniques the very simple and "old-fashioned" 1D NOE experiment is still of some interest - at least here in Berne - to solve stereochemical problems, i. e. to measure and study the molecules 1H-NOE-response after irradiation of a few carefully selecetd target spins. The quality of NOE-difference spectra is of utmost importance especially to prove the presence/absence of the most meaningful but in most cases rather weak long-range NOEs. The series of NOE-difference spectra of a peracetylated trisaccharide (indeed a typical ROESY-case) with rather weak but still positive NOEs proves the excellent stability of our DRX-500 spectrometer. The spectrometer is equipped with the standard B-VT 2000 temperature controller and a set of 3 vibrations dampers. The sample was measured on a TBI-probehead (triple/1H,13C, BB/inverse). The series demonstrates that reliable NOEs may be obtained with the accumulation of only a few scans (64). This is a real advantage compared to our former AM-times and improves the sample throughput dramatically.



Yours sincerely

Dr. Peter Bigler

BRUKER 8mm LARGE VOLUME PROBE

Large volume probes for biomolecular research have always been preferred for NMR analysis. The low solubility of bio-molecules makes it necessary to use larger sample volumes to increase the measured sensitivity. By expanding the coil diameter, an increased number of molecules can reside in the coil region thus resulting in higher signal-to-noise performance. This allows extension of the NMR analysis to the micro molar concentrations.

Yet, as the diameter of the coil increases so does the difficulty in achieving excellent homogeneity over the sample volume (i.e. probe lineshape) and short $\pi/2$ pulse widths. These are important parameters since the lineshape directly impacts the probe's water suppression capabilities, and a short $\pi/2$ pulse width increases the effective bandwidth covered by the pulse.

Now with Bruker's latest generation 8mm probes, excellent lineshape and pulse widths are assured with the larger diameter samples.

Lineshapes typical of 5mm probes can now routinely be achieved on 8mm samples!!! In conjunction with Bruker's high performance BOSS™ 2 shim system and the BSMS™ with Digital Lock, the 8mm probe can meet the highest performance standards for optimum water suppression. Figure 1 demonstrates the lineshape performance on an 8mm triple resonance inverse (TXI) at 750 MHz.

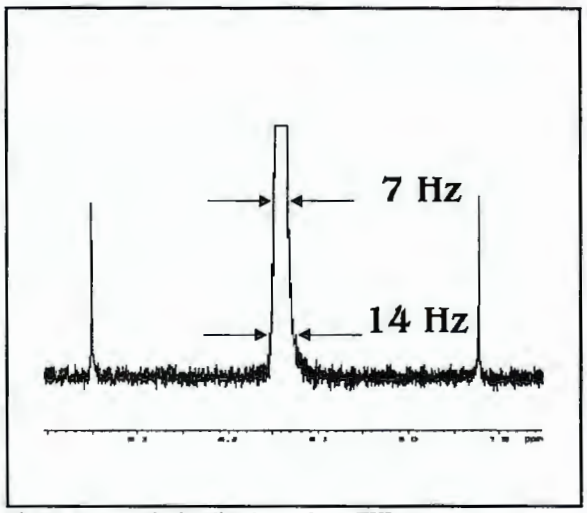
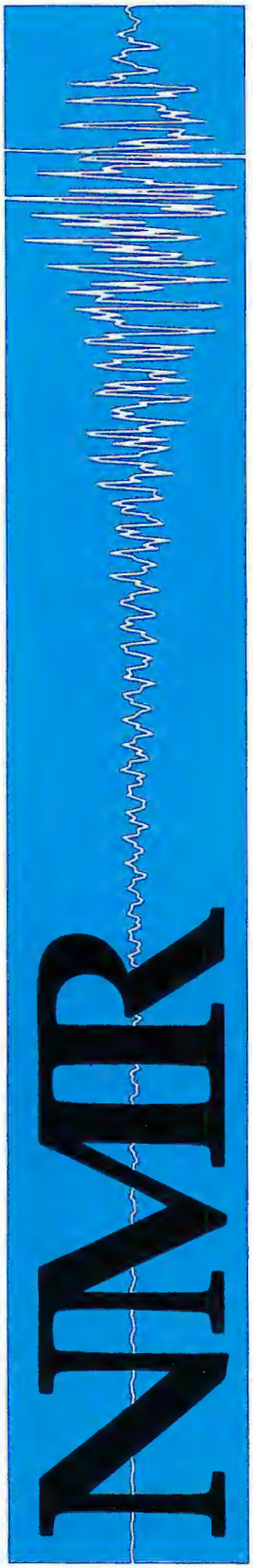


Figure 1: non-spinning lineshape, 8mm TXI probe

Additionally, typical $\pi/2$ pulse widths of ≤ 12 µsec ensure complete coverage of the entire ^{1}H bandwidth.



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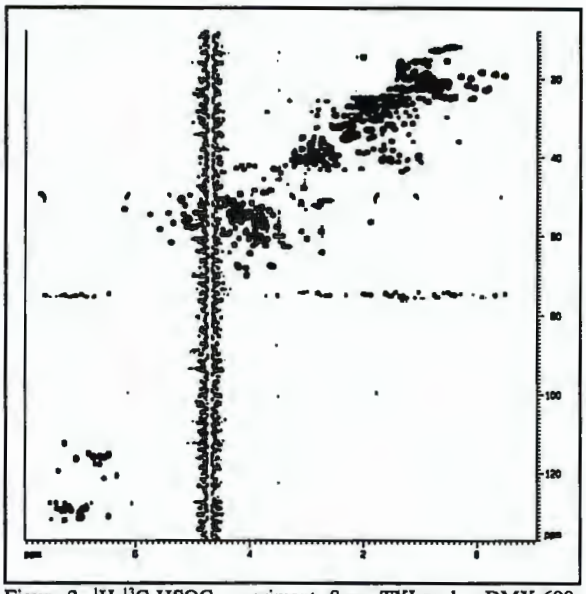

Figure 2: ^{1}H-^{13}C HSQC experiment, 8mm TXI probe, DMX 600

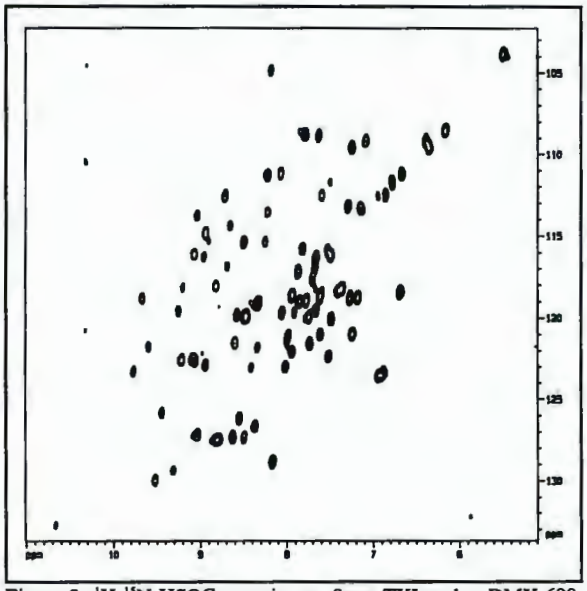

Figure 3: ^{1}H-^{15}N HSQC experiment, 8mm TXI probe, DMX 600

The larger coil diameter allows consideration of concentrations previously unimaginable. An example is shown in Figure 2. A ^{1}H-^{13}C HSQC experiment was acquired on a doubly enriched sample of 170μM SH2 domain in 90% H_2O/10% D_2O with 350 NaCl *(courtesy of Glaxo Research Institute)*. A total of 512 increments with 16 scans each were acquired in only 1 hour and 20 minutes on an *AVANCE™* DMX 600 using an 8mm triple resonance inverse probe!!!

Note, water suppression for this experiment consisted of presaturation instead of gradient solvent suppression since presaturation of the H_2O signal is much more rigorous test of the coil design.

Figure 3 demonstrates a ^{1}H-^{15}N HSQC on the same SH2 domain sample run on the *AVANCE™* DMX 600. Again, total acquisition time was 1 hour and 20 minutes!!!

Available for all field strengths, the 8mm family of probes can be produced in several configurations including:

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Prof. B.L. Shapiro
TAMU NMR Newsletter
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USA

Basel, September 21, 1994
(received 9/26/94)

Properties of deformation corrections (solvent effects on the structure of molecules determined by NMR in liquid crystals)

Dear Barry,

Some years ago I wrote a short paper on the detection and localisation of solvent effects on the structure of molecules derived from NMR-spectra in anisotropic solvents (Mag.Res. in Chemistry 24, 667 (1986). In this paper I suggested to use three different liquid crystal solvents (LC^A, LC^B, LC^C) for molecules with one degree of order and to construct the 2-dimensional diagram of ratios of direct couplings $D_\nu(LC^B)/D_\nu(LC^A)$ versus $D_\nu(LC^C)/D_\nu(LC^A)$. In such a diagram there should be a single point independent of ν if there are no solvent effects. On the other hand the deviations from the single point corresponding to the equilibrium structure indicate the size of solvent effects (See the illustration).

Lately I have noted that in such diagrams all the points corresponding to the various couplings ν are on a straight line which passes through the equilibrium point. It can be shown that this is fulfilled as long as the deformation corrections are small. For the angle α we find the relation:

$$I: \qquad tg\alpha = (S^B/S^C)[(p_\nu^B - p_\nu^A)/(p_\nu^C - p_\nu^A)]$$

where the p_ν's are defined by

$$D_\nu^A = D_\nu^e(1 + p_\nu^h + p_\nu^A)$$

with:

p_ν^h = harmonic vibration correction

p_ν^A = percentage deformation correction for LC^A

D_ν^e = equilibrium distance direct coupling

D_ν^A = measured direct coupling in LC^A

As relation I is fulfilled independently of ν, there must exist relations between the various p_ν's which can only depend upon the molecular geometry. For small p_ν's and three different internuclear equilibrium distances r_1, r_2 and r_3 in molecules with one degree of order we find:

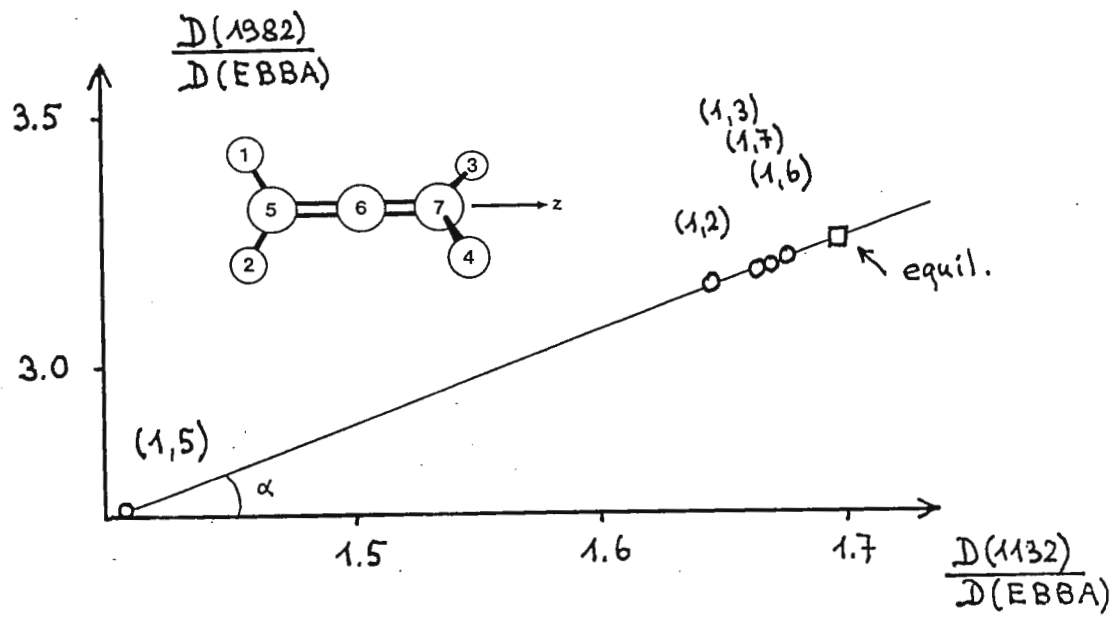
$$II: \quad (p_2^B - p_2^A)(\frac{r_1}{r_2})^3[1-(\frac{r_1}{r_3})^3] = (p_3^B - p_3^A)(\frac{r_1}{r_3})^3[1-(\frac{r_1}{r_2})^3] + (p_1^B - p_1^A)[(\frac{r_1}{r_2})^3 - (\frac{r_1}{r_3})^3]$$

Of course the relation is also valid if the data of LC^C are substituted for the ones of LC^B.

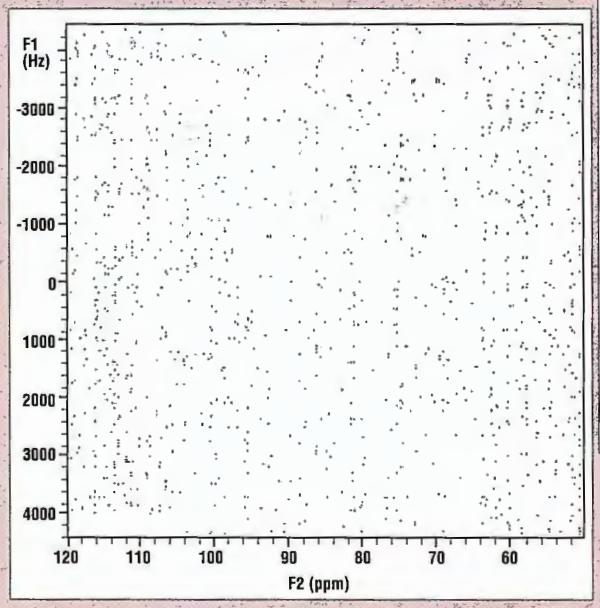
This simple relation is quite surprising if we think of the very complex computer calculations for the determination of the p_ν's.

With best regards

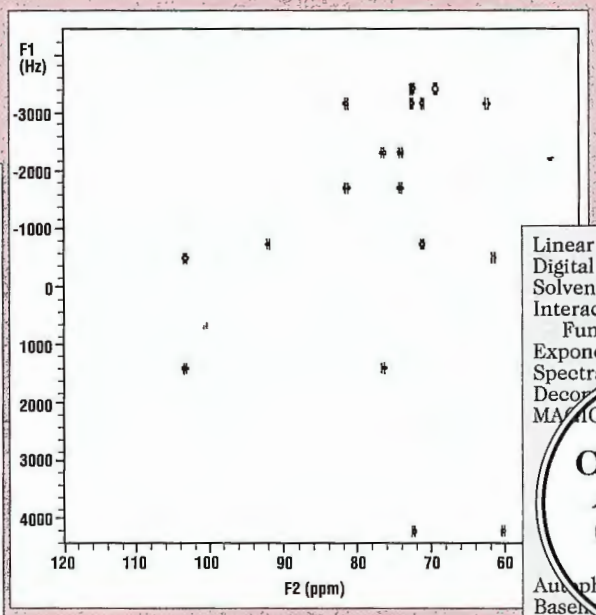
P. Diehl



Can You Find All of the ^{13}C-^{13}C Bonds in this Spectrum?



Without FRED



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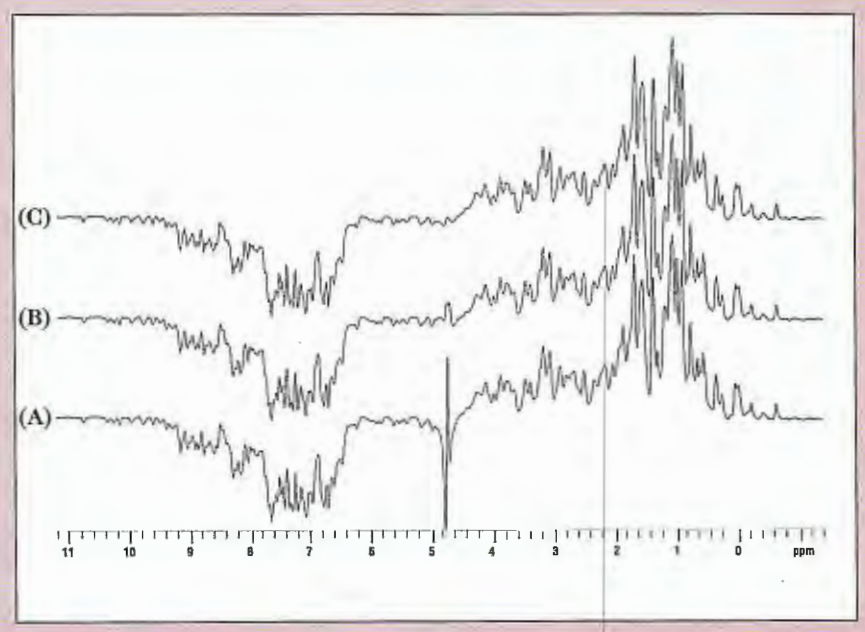
Featured at This Year's ICMRBS*

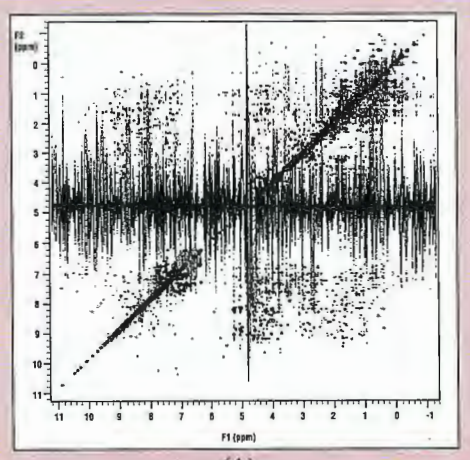
*International Conference on Magnetic Resonance in Biological Systems

Get the most out of your data by removing solvent peaks with time domain digital filtering

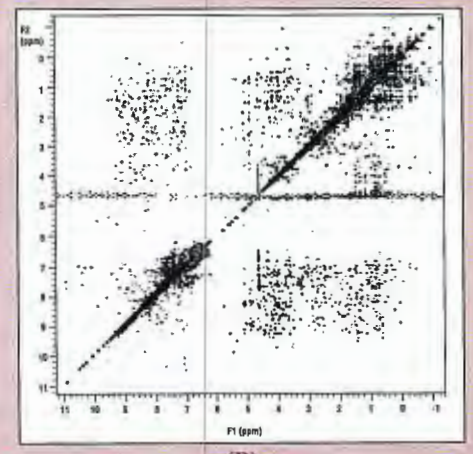
The residual water signal in a first increment SSNOESY experiment, shown after (A) Simple FT (B) Low-frequency subtraction (LFS) and (C) Zero-frequency subtraction (ZFS).



A non-excitation 2D sequence (SSNOESY) on 1 mm lysozyme, dramatically improved in (B) by Zero-frequency subtraction processing in t2. Note the peaks extremely close to the water in F2.



(A)



(B)

The University of Akron

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October 13, 1994 (received 10/20/94)

Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

Dear Barry,

Subject: $^{1}H/^{19}F/^{13}C$ Triple Resonance NMR of Fluoropolymers.

Since equipping our Unityplus-600 NMR with a third Rf channel having ^{1}H to ^{15}N irradiation capabilities this past summer, we have had tremendous success using $^{1}H/^{19}F/^{13}C$ triple resonance experiments to characterize fluoropolymers. $^{19}F/^{13}C$ HMQC and HMBC experiments (with continuous ^{1}H decoupling) have been among the simplest experiments we have used.

Figure 1 shows $^{19}F/^{13}C$ HMQC (Figure 1a-d) and $^{19}F/^{13}C$ HMBC (Figure 1e-g) spectra of 1-fluorohexane as an example. Owing to the high abundance and high magnetogyric ratio of ^{19}F, there is no disadvantage to performing these experiments compared to the often used $^{1}H/^{13}C$ indirect detections experiments. They do offer some significant advantages; the most important being the large ^{19}F chemical shift range and its sensitivity to minor changes in electronic environment.

Other advantages of $^{19}F/^{13}C$ over $^{1}H/^{13}C$ indirect detection are related to range of couplings with ^{13}C. The two- and three-bond C-H couplings are relatively small and have overlapping ranges. It is often difficult to distinguish between two- and three-bond C-H couplings in HMBC experiments. Furthermore, longer range C-H couplings are not usually resolved. C-F couplings on the other hand are relatively large and there is little overlap of the ranges for two-bond (20-40 Hz), three-bond (5-20 Hz), and four-bond (0-5 Hz) C-F couplings. This not only leads to unambiguous assignments, but also provides structural information up to four-bonds from fluorine and enables us to detect crosspeaks from high viscosity polymer solutions in instances where $^{1}H/^{13}C$ HMBC experiments do not work.

We have found that it is usually preferable to perform $^{19}F/^{13}C$ HMQC experiments with delays optimized for the longer range couplings rather than using HMBC experiments to identify long range correlations. Figures 1a-d show $^{19}F/^{13}C$ HMQC spectra obtained with delays optimized for one-bond (160 Hz), two-bond (20 Hz), three-bond (5 Hz) and four-bond (1 Hz) C-F coupling, respectively. Figures 1e-g show $^{19}F/^{13}C$ HMBC spectra obtained with delays optimized for two-bond (20 Hz), three-bond (5 Hz) and four-bond (1 Hz) C-F coupling, respectively. By comparing successive HMQC spectra it is possible to identify new peaks in the spectra as longer delays are used. We have also found that we can usually detect crosspeaks from four bond C-F couplings from HMQC spectra (Figure 1e) when cross peaks from similar long range interactions are not evident in the HMBC spectra (Figure 1g). Also note the very large one-bond ^{13}C isotope shift of carbon bound to fluorine (at F1=84 ppm) compared to the small isotope shift of ^{13}C which is three bonds from fluorine (at F2=24 ppm) in Figure 1c.

Best Regards,

Peter L. Rinaldi
Professor of Chemistry

Dale G. Ray III
Res. Assistant Professor

Lan Li
Research Assistant

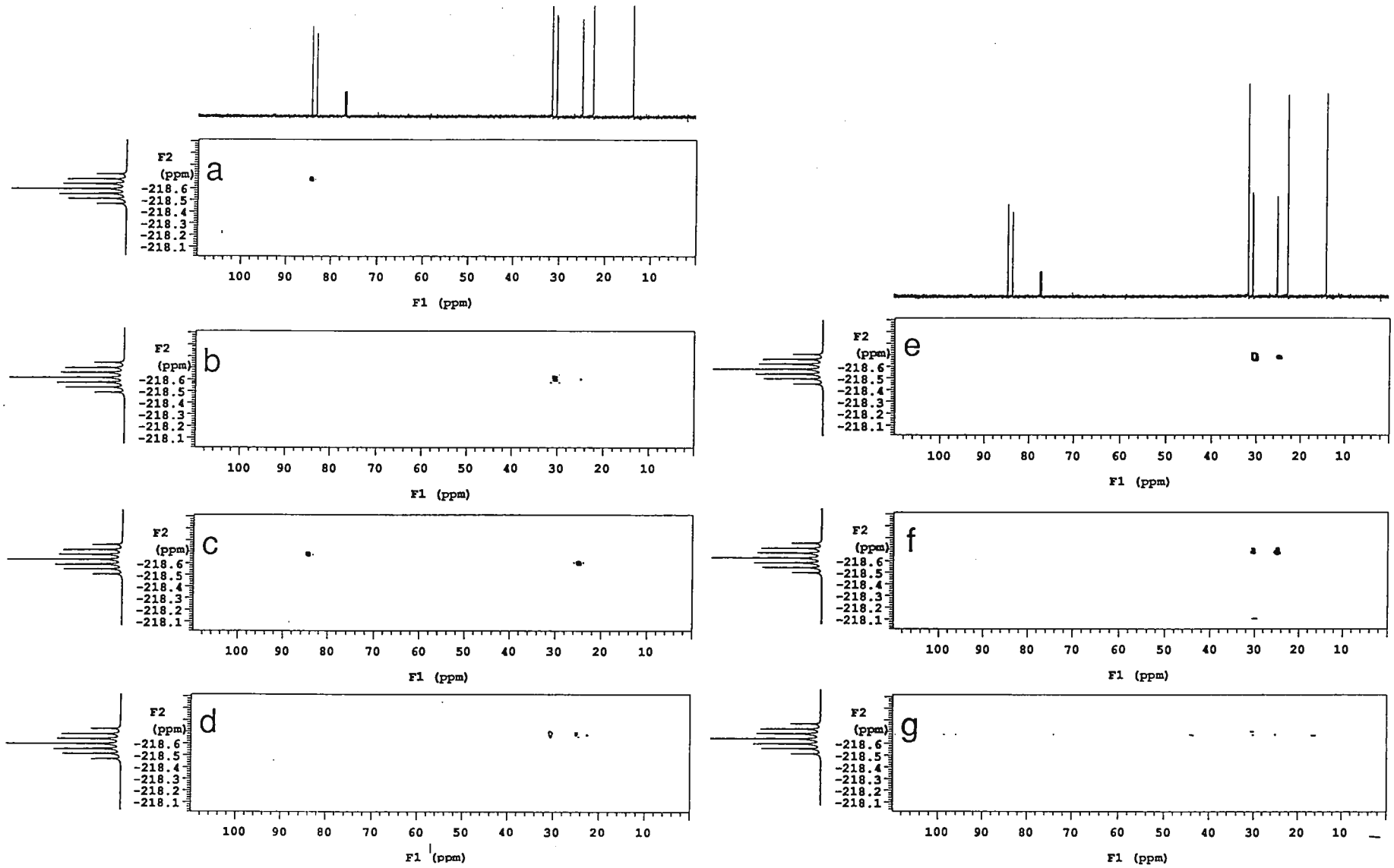

Figure 1. $^{19}F/^{13}C$ HMQC spectra with delays optimized for: a) $^{1}J_{CF}$ =160 Hz; b) $^{2}J_{CF}$ = 20Hz, c) $^{3}J_{CF}$ 5Hz, d) $^{4}J_{CF}$ = 1Hz; and $^{19}F/^{13}C$ HMBC spectra with delays optimized for e) $^{2}J_{CF}$ = 20Hz, f) $^{3}J_{CF}$ = 5Hz, and $^{4}J_{CF}$ = 1Hz.

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But it does not stop there; supporting our customers day to day, and around the world, is a team of engineers and technical specialists. Always on hand, they keep Oxford products fully functional and equipped with the latest refinements to keep our customers at the leading edge.

New products such as the Oxford NMR[750] are practical examples of our innovation so you can be sure of Oxford's commitment to providing the very best in people and products for many years to come.

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Magnetic field Strength (^{1}H-MHz)	Room Temperature Bore Diameter (mm)	Field Stability (^{1}H-Hz/Hour)	Maximum Helium Refill Interval (Days)	Minimum Operational Ceiling Height (m)
750	51	15	60	3.8
600	51	10	120	3.4
500	51	10	150	3.2
400	54	8	365	2.8
360	54	8	365	2.8
300	54	3	365	2.8
270	54	2.7	365	2.8
200	54	2	365	2.8
100	54	1	365	2.8
500	89	15	120	3.4
400	89	10	180	2.8
360	89	10	365	2.8
300	89	3	365	2.8
270	89	2.7	365	2.8
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17 October 1994
(received 10/18/94)

Dr. B.L. Shapiro
Editor
TAMU Newsletter
966 Elsinore Court
Palo Alto, CA 94303

900 G/cm self-diffusion measurements in a narrow bore 500 MHz spectrometer

Dear Dr. Shapiro,

Recently the NMR group within Eastman Kodak Company (Rochester, NY) took delivery of a 5 mm, $^1H/^{19}F$, air cooled, z-gradient probe built by Doty Scientific, Inc (Columbia, SC). The scientists at Varian Associates Inc. (Palo Alto, CA) have successfully integrated the probe with our Varian Unity 500, narrow bore (51 mm) spectrometer. A Highland DC amplifier enhanced to provide 20 amps (the 10 amp version is standard for Varian's PFG accessory) combined with a Sorensen VT power supply enable the probe to reach up to 900 Gauss/cm through a temperature range of -100 to +240 °C. Varian has also provided a comprehensive software package for fully automating the experiments, enabling ease of setup, processing and plotting. We use this system for pulsed gradient spin echo (PGSE) NMR experiments in order to measure self-diffusion coefficients (D) within a variety of chemical systems.

This diffusion system offers excellent measurement accuracy and precision. The following lists some standards which we have used for calibrating the instrument:

Standard	D† (x 10^{-7} cm^2/s)	literature reference and value
50% w/w D_2O in H_2O	205 ± 1.8 (0.9%)	1) 208
10% w/w D_2O in H_2O	215 ± 2.5 (1.2%)	1) 223
99.9% D_2O	188 ± 2.1 (1.1%)	1) 190
neat n-dodecanol	5.74 ± 0.10 (1.7%)	2) 6.3
10% w/v poly(styrene)‡ in CCl_4	0.144	3) 0.150
25% w/v poly(styrene)‡ in CCl_4	0.0111	4) 0.0066

† D ± σ for 14 measurements @ 25 °C
‡ D @ 30 °C for 212k MW monodisperse poly(stryrene)

The eddy current problems as well as amplifier overdrive have been solved using ramped (trapezoidal shaped) gradients.[5] For a ramp time of 200 μs the eddy ringdown times for a 900 G/cm gradient pulse on for 1 ms and 10 ms are less than 8 ms and 30 ms, respectively. Primarily, we employ the standard Stejskal-Tanner spin-echo pulse sequence with a three-gradient pulse train prior to the 90° pulse. The pulse train helps to provide a steady-state condition for the gradients so that the second gradient provides the same strength as the first.[6] If a pulse train is not used, the eddy currents from the first gradient will superimpose upon the second gradient and lessen its strength by a slight amount. The result will be a slight phase difference among each of the acquired spectra. When large and long gradients are required, we employ the 5-pulse stimulated-echo pulse sequence with the long eddy current delay (LED).[6] Another approach that should further reduce eddy current effects is multi-exponential compensation,[7] although we have not attempted to implement this technique.

Figure 1 shows an example of a 25% w/v, 212k MW, monodisperse poly(styrene) in CCl_4. I will add that we share time on the instrument with the standard PFG probe for more general organic characterization problems. Despite the fact that each probe requires its own settings on the Oxford shim power supply in the cabinet, it only takes us about 45 minutes to successfully change probes in either direction (this includes shimming to linewidth specs! but not a trip for coffee).

Sincerely,

Eastman Kodak Company: Brian Antalek, Michael Hewitt, Tony Williams

Varian Associates, Inc.: Laima Baltusis, John Kilpatrick

Doty Scientific, Inc.: David Doty, James Wilcher

1) Longsworth, L.G. *J. Phys. Chem.*, **61**, 1914 (1960)
2) Stejskal, E.O.; Tanner, J.E. *J. Chem. Phys.*, **49**, 1768 (1968)
3) Callaghan, P.T.; Coy, A. *Phys. Rev. Lett.*, **86**, 3176 (1992); MW = 200k, T = 30 °C
4) Callaghan, P.T; Pinder, D.N. *Macromolecules*, **17**, 431 (1984); The predicted value for MW = 233k and T = 28 °C is given. The measured value is 0.0052 ± 0.002.
5) Price, W.S.; Kuchel, P.W. *J. Magn. Reson.* **94**, 133 (1991).
6) Gibbs, S.J.; Johnson, C.S., Jr. *J. Magn. Reson.* **93**, 395 (1991).
7) Van Vaals, J.J.; Bergman, A.H. *J. Magn. Reson.* **90**, 52 (1990).

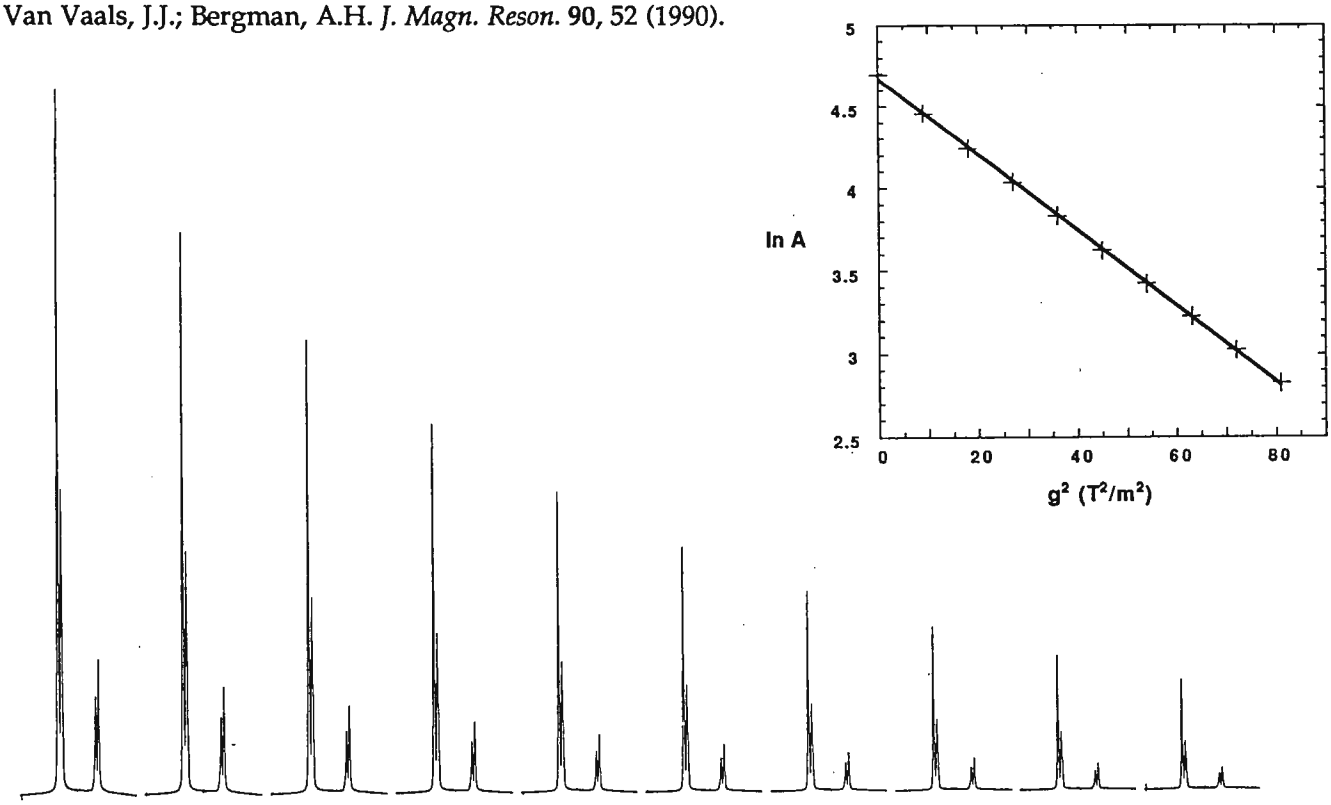

Figure 1. Attenuation plot and spectra for 25% w/v monodisperse 212K MW poly(styrene) in CCl_4. The stimulated echo pulse sequence with LED[6] was used with a diffusion time (Δ) of 82.1 ms, a gradient width (δ) of 6 ms (ramped gradients were used where the ramp time was 200 μs), and a gradient strength (g) which varied between 5 and 900 Gauss/cm. The integral for the aromatic region is plotted. The slope is -0.0229 and hence the diffusion coefficient is 1.11×10^{-9} cm^2/s.

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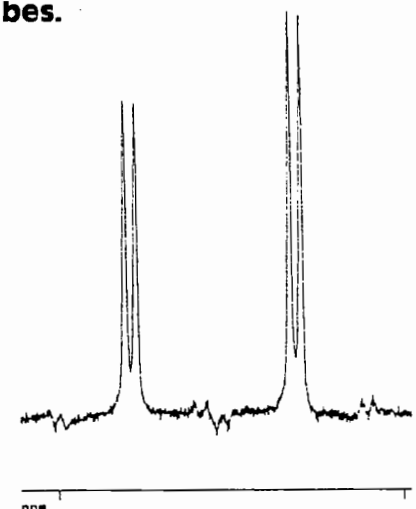
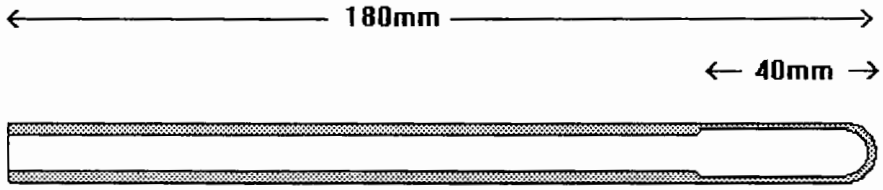
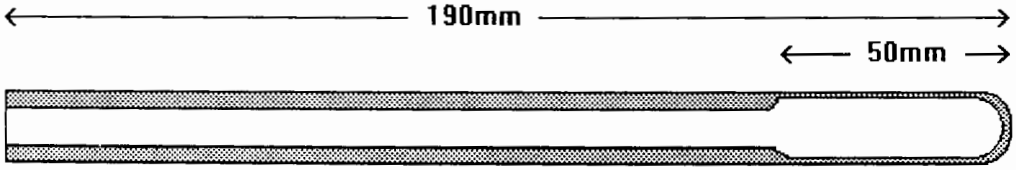


Fig.1a Fig.1b

The spectra of 20mm sucrose in D_2O were obtained with a single scan without apodization prior to Fourier transformation on a Bruker AMX-600 spectrometer at 298 K. By using Shigemi high quality 5mm standard tube (Fig.1a) and the Shigemi highly sensitive thin wall 5mm tube (Fig.1b), the spectra confirms a sensitivity enhancement of about 10%.



PST-001 and PST-002



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10	ST10-001	0.25	40/ 8	9.98 + 0.00 - 0.01	9.52 ± 0.01	$36.00	$32.00
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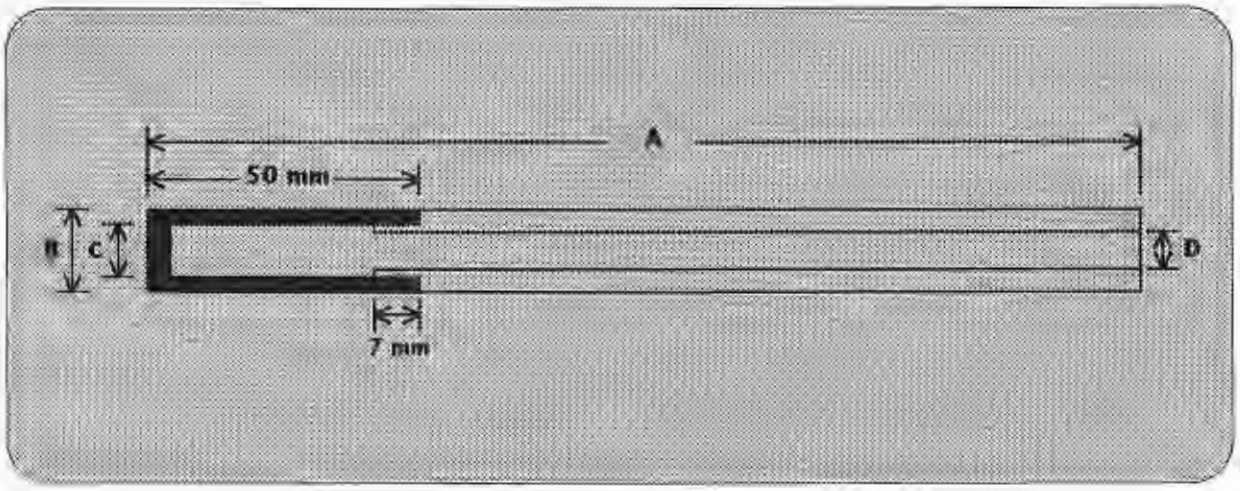

Type	A Length (mm)	B OD (mm)	C ID (mm)	D OD (mm)	Camber (μ)
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Dr. Bernard L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303

October 6, 1994
(received 10/11/94)

What is the smallest volume one can measure ?

Dear Barry,

Early in 1992 Bruker reintroduced their line of **2.5 mm microsample probes**, which had been around for quite some time, but advances in probe technology now permitted a design, which gave comparable resolution and lineshape to a regular 5 mm probe. Using a standard 2.5 mm sample tube requires a volume of at least 70 microliters. The spectroscopist working with very small amounts wants to avoid any extra microliter of solvent, which would unnecessarily dilute the sample. Restricting the volume is a technique which has been applied in a variety of ways (vortex plugs, spherical and cylindrical samples or tubes with very thick walls). Shigemi now introduces the **BMS-0025** sample tube system which, just like the well known 5,8 and 10 mm tubes, is a NMR sample tube with a thick bottom and a tight fitting insert (Fig 1). The sample tube is 100 mm in length. It can be used in a standard 5 mm spinner, if it is held in an inverted 5mm tube by a teflon spacer. The sample height best matching the Rf-coil for the 2.5 mm microsample probes is 12 mm and with the inner diameter of 1.9 mm, this results in a volume of 34 mm^3 or 34 microliters. We prepared a sample of 2 mM Lysozyme in 90%H_2O/10%D_2O and transferred this solution to the BMS-0025 sample tube. All experiments were run on a Bruker **AVANCE DMX-600** spectrometer equipped with a 2.5 mm ^1H - $^{13}C/^{15}N$ triple resonance probe. The experiments shown in Fig 2 and 3 were run with a 10 mm sample height, corresponding to 28.35 microliters, containing 790 µg Lysozyme. Fig 2 is a 1D proton spectrum with presaturation for water suppression. Fig 3 is a 1H-13C HMQC spectrum. Experimental details are given in the figure captions. The insert was then lowered to a 3.5mm sample height, corresponding to 10 microliters, containing 280 micrograms of Lysozyme. Fig 4 shows a 1D spectrum and Fig. 5 is a 2D NOESY with presaturation recorded at this volume. Finally the insert ended up only 0.7 mm above the sample bottom, resulting in the incredibly small volume of **2 (two)**

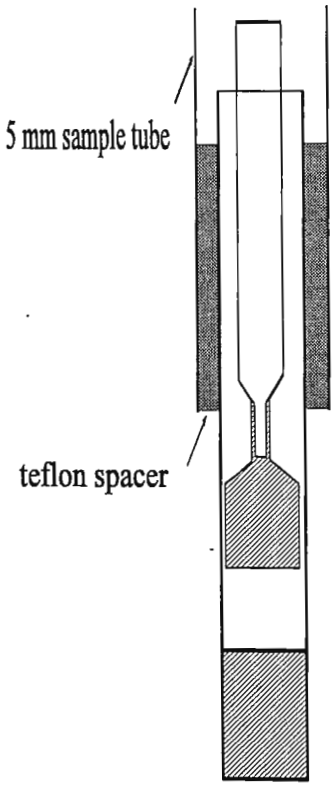

Figure 1

microliters which contain only 56 micrograms of Lysozyme. Fig 6 shows the 1D proton spectrum obtained at this volume. Comparison of the different volumes shows that, although the resolution suffers somewhat, the quality of water suppression can be maintained over the entire range of volumes.

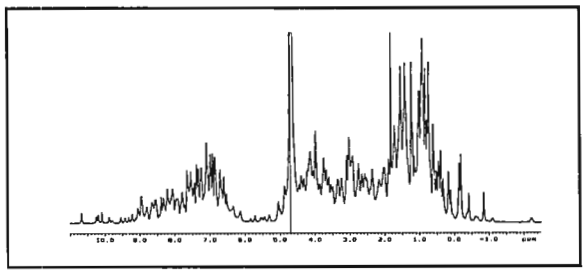

Figure 2: 28.35 microliter volume: 1D spectrum with 2 sec presaturation, 64 scans, 2 min experiment time

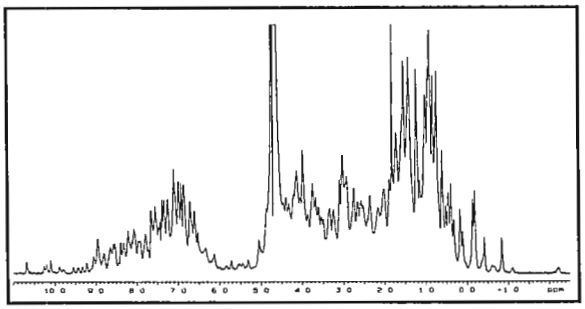

Figure 4: 10 microliter volume:1D spectrum with presaturation, 64 scans, 2 min experiment time

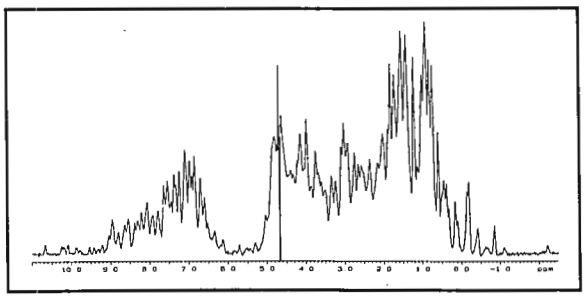

Figure 6:**2 microliter volume**:1D spectrum with presaturation, 1024 scans, 22 minutes experiment time

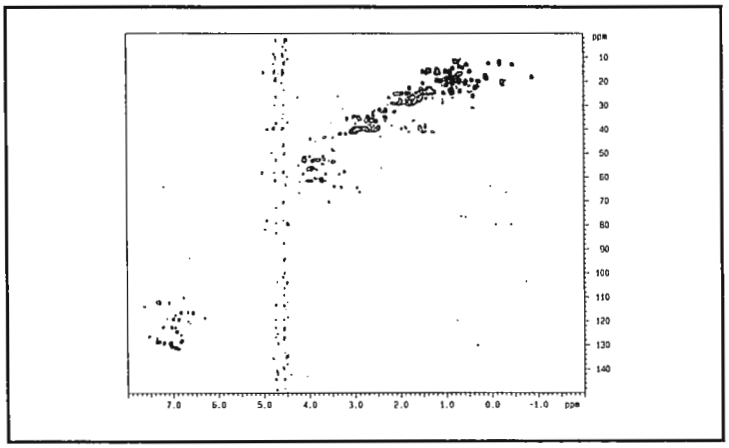

Figure 3: 28.35 microliter volume: 2D HMQC with presaturation, 64 scans , 256 increments, 4 h 55 min experiment time

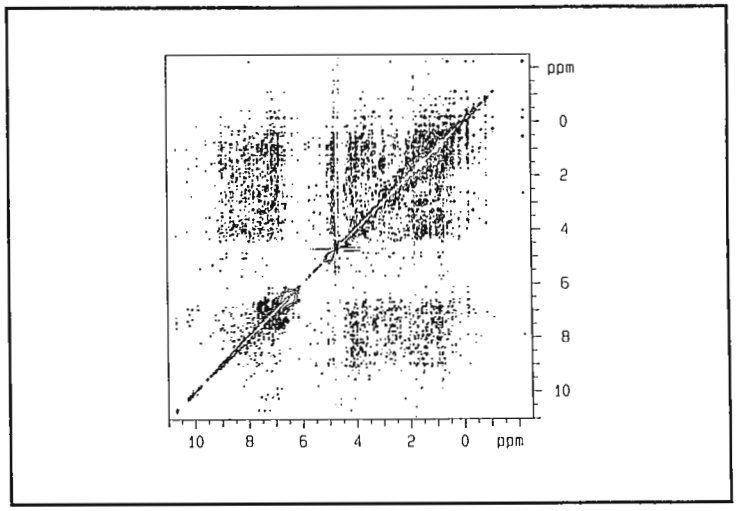

Figure 5: 10 microliter volume: 2D NOESY with presaturation, 150 msec mixing time, 64 scans, 512 increments, 11 hours experiment time

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6th October 1994
(received 10/11/94)

Dr B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
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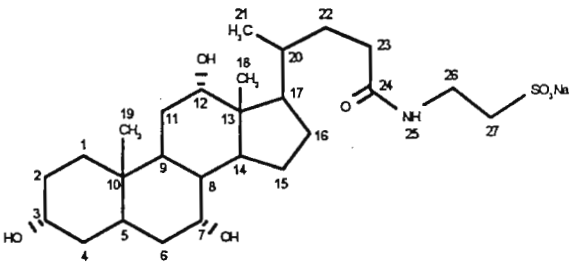
Dear Barry,

MAXY-NMR

As in protein NMR spectroscopy, so for complex biofluid mixtures - the resonance assignment stage must come first, but usually without the benefit of stable isotope labelling. The molecular diversity and concentration range in biofluids is enormous and so we have been developing methods to help the assignment process and as these approaches are of general use we thought that we would share them with the TAMUNMR readership.

Using the principle of a maximum quantum filter which is different for $^{13}CH_3$, $^{13}CH_2$ and ^{13}CH groups, we have come up with maximum quantum spectroscopy (MAXY) for the separate detection of the ^{1}H spectra of CH_3, CH_2 and CH groups. The approach can be used in 1-dimensional NMR and in n-dimensional NMR to produce pulse sequences for MAXY-TOCSY and MAXY-NOESY for example which show scalar and dipolar coupling connectivity from only the selected group of resonances (CH_3, CH_2 or CH). In the multi-dimensional version, data on all protons are collected simultaneously in one experiment, but the resonances of the CH_3, CH_2 or CH groups can be editted into different regions of the contour plot. Details of the pulse sequences, in which pulsed field gradients can also be used, will appear in three papers accepted for the Journal of Magnetic Resonance but a couple of examples are given here, where the first is of the MAXY-J-resolved spectrum of the bile salt sodium taurocholate in dmso-d_6. This shows a small part of the 600 MHz proton NMR spectrum and the selective detection of CH and CH_2 JRES spectra in a crowded region.



The method has also been applied to heteronuclear correlation experiments using inverse detection as in MAXY-HMQC giving results similar to DEPT-HMQC (Kessler *et al.*, J. Magn. Reson., 85, 400 (1989)) and for direct detection, MAXY-HETCOR, for instruments without inverse geometry probes. The second figure shows the MAXY-HETCOR result for sodium taurocholate with assignment of all of the protonated carbons.

Yours sincerely,

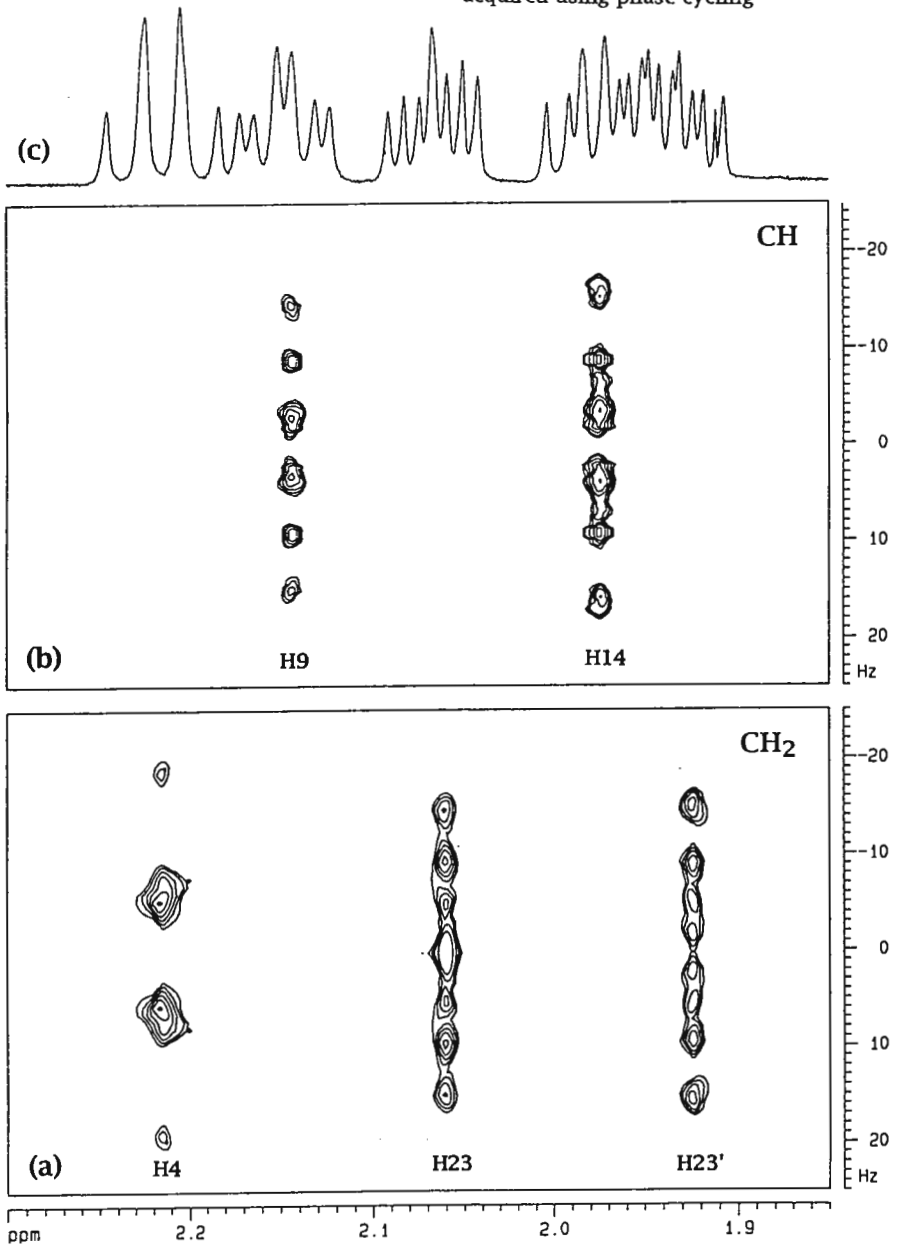
刘买利 约翰 林敦 杰里米 尼可尔逊

Maili Liu
Physical Sciences

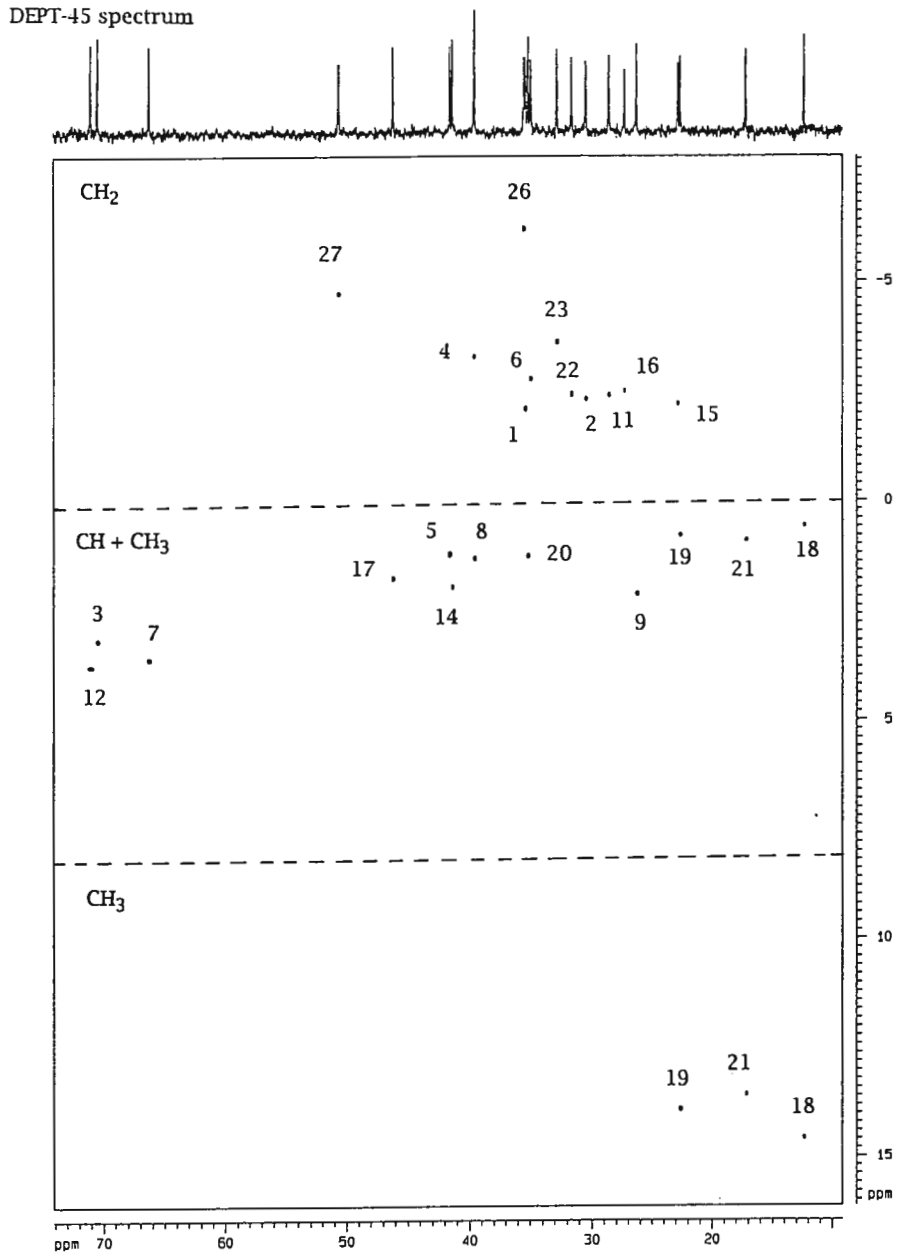
John Lindon
Physical Sciences

Jeremy Nicholson
Birkbeck College

600 MHz ^{1}H MAXY JRES NMR spectrum of sodiumtaurocholate in dmso-d_6 solution acquired using phase cycling



600 MHz MAXY-HETCOR spectrum of sodium taurocholate in dmso-d_6



THE ROYAL INSTITUTE OF TECHNOLOGY

Department of Chemistry, Physical Chemistry

Professor Peter Stilbs

Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto
CA 94303
USA

October 18, 1994
(received 10/19/94)

Re: High field gradient calibration in FT-PGSE, equipment for sale

Dear Barry,

Thank you for your fluorescent Ultimatum. The field of self-diffusion measurements by FT-PGSE techniques continues to evolve at a rapid pace, and we believe that many more will start to use these methods, now that field gradient coils have become a standard feature of modern NMR probes - as a result of the booming interest in "pulsed field gradient NMR" among "multi-dimensional NMR spectroscopists". I still remember trying to persuade the very reluctant major Spectrometer manufacturers to provide a high resolution field gradient probe back in 1984; finally Varian agreed... By the way, Olle Söderman and myself have recently written a review on "NMR studies of Complex Surfactant Systems" for Progr. NMR Spectrosc., which we proofread some time ago, so it should be in print this autumn, we believe. For those interested, there is a fairly long section on the many diverse developments in PGSE techniques for self-diffusion measurements since 1985.

Going through the literature, one can note several reinventions of the PGSE wheel also among the new big majority of pulsed field gradient NMR spectroscopists (like previously in the field of medical NMR). Papers like those of Stejskal and Tanner are rarely cited. Starting with P.C.M. van Zilj and C.T.W. Moonen (JMR 87 (1990) 18-25), solvent suppression schemes based on the differences in diffusional rates of water and larger molecules have recently been presented. The basic effects, of course are a well-known phenomenon among those interested in NMR-based self-diffusion monitoring as such, and were discussed in some detail in the context of solvent suppression as well in my own review in Progr. NMR Spectrosc. 19 (1987) 1, section 7.1.1.2.

I would like to provide the readers with a procedure we sometimes use to check the calibration of field gradient strengths i PGSE. Basic calibration is commonly done in a relative way, on protons in water - using literature data for their self-diffusion (of the order of $2 \cdot 10^{-9}$ m^2 s^{-1}

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at room temperature. Necessarily, the gradient strengths then have to be quite small. Can one extrapolate, and trust the linearity at very much higher gradient values or longer gradient pulse lengths ? This will depend on the performance of the gradient driver, and the rest of the hardware used. If one is unsure, one would like to recalibrate with a substance with a diffusion rate more similar to that of the one under investigation. However, there do not really exist any established low-diffusion rate standards, except glycerol, which is a very poor choice, for reasons summarized in section 6.5.9.4 of my 1987 review. The last paragraph of that section also describes a multistep relative calibration procedure, which works fine, provided one has good secondary and tertiary standards. We have found that micellar poly(ethyleneoxide) alkyl ethers (C12E6, C12E8 etc.) in heavy water probably may be the best choice. The residual water peak provides a "water calibration signal", and the EO peak of the surfactant has a long transverse relaxation rate and is essentially singlet-like, at least at low fields. Note that due to fast intermicellar exchange and a very low CMC the CxEy diffusion rate will be monomodal, unlike that found for even highly fractionated PEO standards. We hope that this calibration procedure will be found useful by others in the PGSE self-diffusion area, and that this letter arrives before the dreaded deadline date.

- A friend of mine wants the world to know that he has some equipment for sale to those that need to repair or exchange their Varian CFT20 system: If you need spare parts to the CFT20; probes, boards, anything, send a fax to ASF, Sweden +46-87 32 93 88.

Yours Sincerely

Peter Stilbs

Postdoctoral Position. Available immediately for solid-state NMR studies of environmental science problems in the area of solidification/stabilization of hazardous waste in cement and other matrices. Requires Ph.D. in chemistry or physics with experience in solid-state NMR. The research will span across common solid-state NMR methods, field-swept NMR with an 18 T magnet, stray-field imaging, and synchrotron x-ray tomography. Please send a curriculum vitae, three letters of reference, and copies of pertinent publications to: Prof. Les Butler, Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804.

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Faculteit der Scheikundige Technologie en der Materiaalkunde

Dr BL Shapiro
Editor TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

22 September 1994

(received 9/29/94)

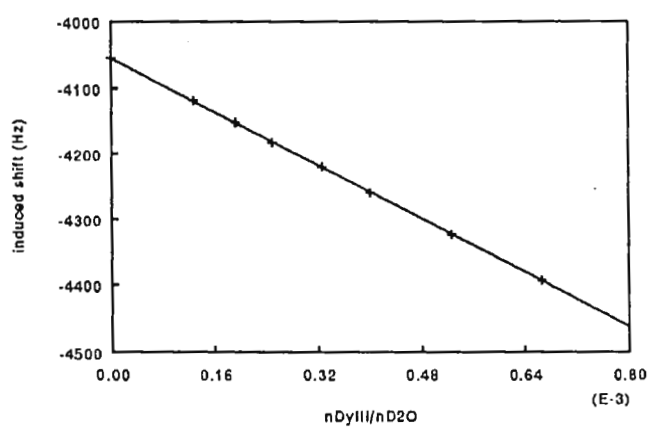
^{17}O Experiments performed on complexes of Dy(III) and oxidised carbohydrates

Dear Dr Shapiro

We routinely carry out multinuclear NMR experiments using Ln(III) ions as model ions for Ca(II) in order to study the manner of binding in ligand-metal complexes. We were able to investigate with ^{17}O NMR, the complexation behaviour of an oxidised carbohydrate oligomer, dicarboxy inulin (1) and that of its model compound, dicarboxy methyl α-fructofuranoside (2) by observing the induced ^{17}O shifts of D_2O a consequence of adding successive increments of Dysprosium(III) chloride to aqueous solutions of these ligands.

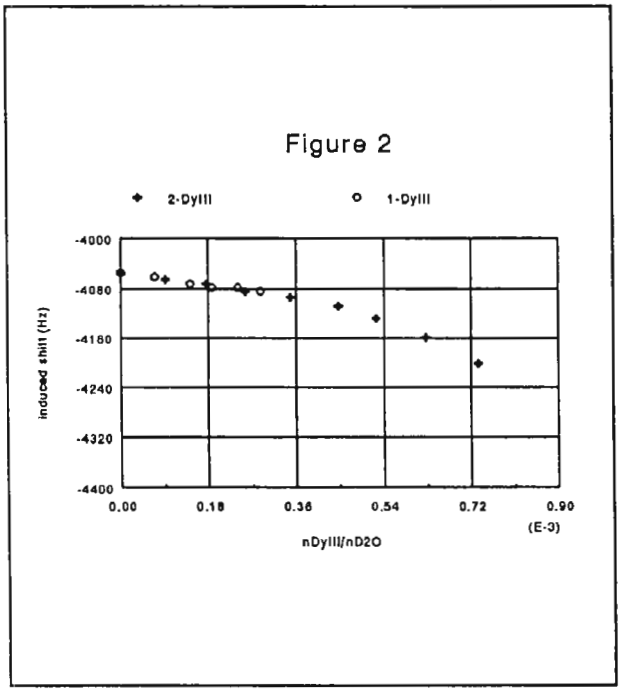
An ^{17}O nucleus bound to a Lanthanide cation exhibits an induced shift for which only the contact contribution, ie. the shift of the ^{17}O nucleus coordinating to the DyIII ion is relevant. In addition to this, the exchange between bound and free D_2O is fast on the NMR time scale. The induced shift is independent of the nature of the coordinating ligand and knowing the hydration number of a Dy(III) cation to be 8-9, we are able to determine the number of bound D_2O molecules. Measurement of the DyIII induced ^{17}O shifts as a function of DyIII concentration allows us to calculate the average shift contribution per D_2O molecule which is illustrated in figure 1, a plot of induced shifts as a function of molar DyIII concentration.

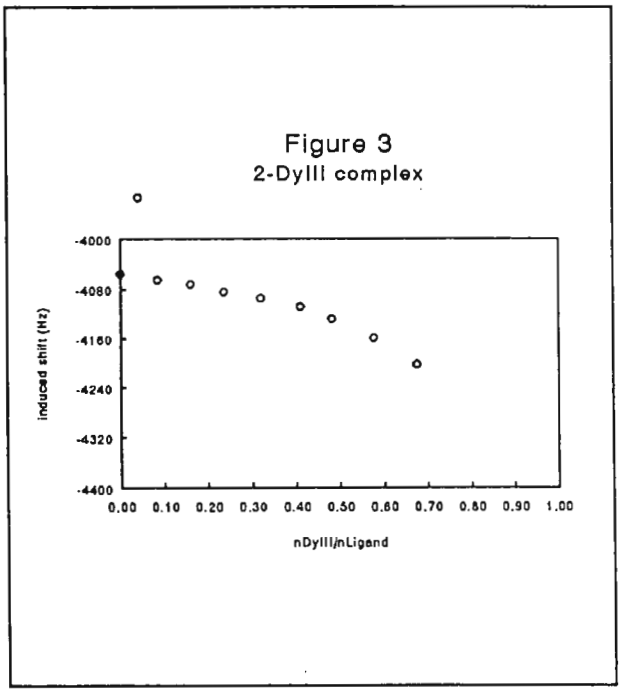
FIGURE 1 Dy(III) induced shift of D2O



When a ligand coordinates to Dy(III) there is a reduction in binding sites available for D_2O molecules. This is illustrated in figure 2 where the initial slope of induced shift versus molar ratio Dy(III)/D_2O corresponds to two bound water

molecules. The slope decreases sharply at molar ratio Dy(III)/D_2O ≈0.45E-3 indicating that significantly more D_2O molecules are coordinating to Dy(III). The complex stoichiometry may be deduced from a plot of induced shift as a function of the molar ratio Dy(III)/ligand as shown in figure 3. Figure 3 illustrates that at a molar DyIII/Ligand ratio of ≈½, there is a change in gradient corresponding to a change in composition of complex from a 1:2 Dy(III):ligand species to one predominantly 1:1 (Dy:ligand) in nature.





Successive amounts of Dysprosium(III) chloride were added to ligand solutions (±0.1M) with the intention of reaching a 1:1 Dy:ligand molar ratio. The latter was however not always possible due to precipitation taking place.
The ^{17}O experiments were carried out at 72 °C and 27.12 MHz using a Nicolet NT-200 WB spectrometer.
Further studies on the complexation behaviour of oxidised carbohydrates are currently in progress and encompass lanthanide-induced ^{13}C relaxation rate enhancement measurements as well as potentiometric experiments.

Yours Sincerely

L Johnson

Dr Louise Johnson

P.S. Please credit this to the account of Dr Joop A Peters.

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USA

(received 10/8/94)

Grinnell College

Magnetic Relaxation in *Quadrupolar*-split Systems: Application to Nb in $LiNbO_2$

Dear Dr. Shapiro:

During our recent study of $LiNbO_2$, we observed highly non-exponential Nb magnetization recovery curves. By scaling the magnetization and time axes of the recovery curves measured at different temperatures, we found it possible to superpose the data. The shape of these curves was clearly significant. Hence, we suspected that the non-exponential character of the observed relaxation was intrinsic to the spin I=9/2 Nb nucleus and not due to sample inhomogeneity. The nearly linear temperature dependence of the Nb relaxation rate T_1^{-1} lead us to conclude that the relaxation mechanism was magnetic (dipole-dipole) interaction with conduction electron spins. As the literature did not contain the solution for magnetic relaxation of I=9/2, we solved this case and found that we could calculate the observed shape of the recovery curves.

So that others may avoid the tedious linear algebra necessary to solve this problem, we present below the recovery curves appropriate for two common experiments. We also note some subtle pitfalls to avoid in conducting such experiments and in analyzing the data.

The solution to the rate equations that govern the population differences for the various observable transitions is straightforward, if somewhat tedious. The solution is the sum of exponential terms, and the decay rates have been known since Andrew and Tunstall. The difficulty lies in determining the weights of the individual exponential terms, which depend on the initial level populations. Hence, different magnetization recovery curves are observed for different preparation pulse sequences.

We present here the expected magnetization recovery curves for two common experiments. In both cases, we assume that the static quadrupole interaction is so large that only the central transition ($+\frac{1}{2} \leftrightarrow -\frac{1}{2}$) is directly perturbed and observed. The first case is "cw" saturation of the central transition, in which a long rf pulse is applied to the central transition. This pulse must be long enough to allow the satellite transitions to re-equilibrate amongst themselves and with the saturated central transition. Alternatively, the same initial spin populations are achieved by using a long train of 90° pulses, if the train is long enough to allow satellite equilibration. Under conditions of "cw" saturation, the magnetization recovery curves are given by:

I=3/2: $M(t)/M(\infty)=1 -0.4 \exp(-2Wt) -0.6 \exp(-12Wt)$

I=5/2: $M(t)/M(\infty)=1 -0.25714 \exp(-2Wt) -0.26667 \exp(-12Wt) -0.47619 \exp(-30Wt)$

I=7/2: $M(t)/M(\infty)=1 -0.19048 \exp(-2Wt) -0.18182 \exp(-12Wt) -0.21978 \exp(-30Wt) -0.40793 \exp(-56Wt)$

I=9/2: $M(t)/M(\infty)=1 -0.15152 \exp(-2Wt) -0.13986 \exp(-12Wt) -0.15385 \exp(-30Wt) -0.19197 \exp(-56Wt) -0.36281 \exp(-90Wt)$

The I=3/2 and I=5/2 results were first given by Andrew and Tunstall, and the I=7/2 result by Narath. We derived the I=9/2 result to compare to our Nb data.

A second common experiment is the application of a single 90° pulse to the central transition. Since the satellite transitions are not allowed to equilibrate with the saturated central before recovery begins, the initial conditions are different. The recovery curves in this case are:

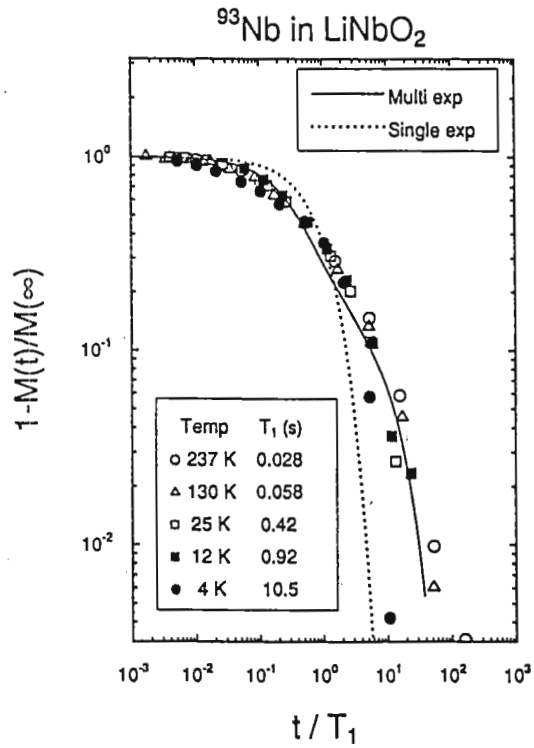
I=3/2: $M(t)/M(\infty)=1 -0.1 \exp(-2Wt) -0.9 \exp(-12Wt)$

I=5/2: $M(t)/M(\infty)=1 -0.02857 \exp(-2Wt) -0.17778 \exp(-12Wt) -0.79365 \exp(-30Wt)$

I=7/2: $M(t)/M(\infty)=1 -0.01191 \exp(-2Wt) -0.06818 \exp(-12Wt) -0.20604 \exp(-30Wt) -0.71387 \exp(-56Wt)$

I=9/2: $M(t)/M(\infty)=1 -0.00607 \exp(-2Wt) -0.03357 \exp(-12Wt) -0.09231 \exp(-30Wt) -0.21501 \exp(-56Wt) -0.65306 \exp(-90Wt)$

For comparisons between the relaxation rate and other measurements (such as the Knight shift when considering conduction electrons and the Korringa relation), the appropriate value for T_1^{-1} is 2W. This is the rate that would be measured in the absence of quadrupole splittings, when only a single exponential would be observed.



$LiNbO_2$ is a semiconductor with a hexagonal, layered structure. It has potential applications in battery technology, stemming from the Li ion mobility and the large cell potentials achieved when $LiNbO_2$ is used as an electrode. The Li ions form planes separated by NbO_2 layers. The Nb atoms lie at the centers of trigonal prisms formed by O atoms. The non-cubic environment of the Nb gives rise to a large quadrupole interaction, $\upsilon_Q \approx 1$ MHz. Despite the large quadrupole interaction, we find that the Nb relaxation is magnetic, not quadrupolar, in nature. The reason is that the conduction electron spins ($LiNbO_2$ is substoichiometric in Li and Li vacancies act as electron acceptors) interact strongly with the Nb nuclei. Hence, we have a quadrupole-split spin system in which the inter-level transitions (i.e. relaxation) are driven by a magnetic mechanism.

The magnetic nature of the Nb relaxation is reflected in the shape of the recovery curve, shown here. Magnetization recovery curves measured at several temperatures are shown. The values of T_1 listed are the 1/e points of the individual recovery curves. The time axis for each temperature has been scaled by the 1/e T_1 at that temperature. The solid line is the multi-exponential function for "cw" saturation. The dotted line is a single-exponential. The experiment consisted of twenty-four 90° pulses separated by 1 ms ("cw" saturation of the central transition) followed by recovery and inspection of the central transition. The data points follow the line calculated from the I=9/2 "cw" saturation equation given above. Deviations of the 4 K data from this line probably result from incomplete satellite equilibration, due to the slow relaxation at 4 K.

We note that there are two major sources of error in applying the above equations. The first source of error is in assuming the incorrect initial spin populations. In particular, if quadrupolar relaxation is important, then the $\Delta m=\pm 2$ transitions change the initial spin populations from those achieved in the magnetic case. Under these circumstances the initial conditions have to be calculated carefully. Recovery curves for quadrupolar relaxation have been calculated by Gordon and Hoch.

A second source of error is experimental, insidious, but easily avoided. An incorrect determination of the fully recovered magnetization $M(\infty)$ can cause the "true" T_1^{-1}, 2W, to be overestimated by an order of magnitude. The experimental recovery curves can look nearly single exponential in cases where the magnetization recovery not is followed to completion. These curves will overemphasize the initial fast decay. Care must be taken to allow for complete magnetization recovery.

Sincerely,

A.F. McDowell DMS MSC REN

Andrew F. McDowell, David M. Snyderman, Mark S. Conradi, R.E. Norberg

A.F. McDowell, *et al.*, to appear in *Phys. Rev. B*, (Dec. 1994).
E.R. Andrew and D.P. Tunstall, *Proc. Phys. Soc.* **78**, 1 (1961).
A. Narath, *Phys. Rev.* **162**, 320 (1967).
M.I. Gordon and M.J.R. Hoch, *J. Phys. C: Solid State Phys.* **11**, 783 (1978).

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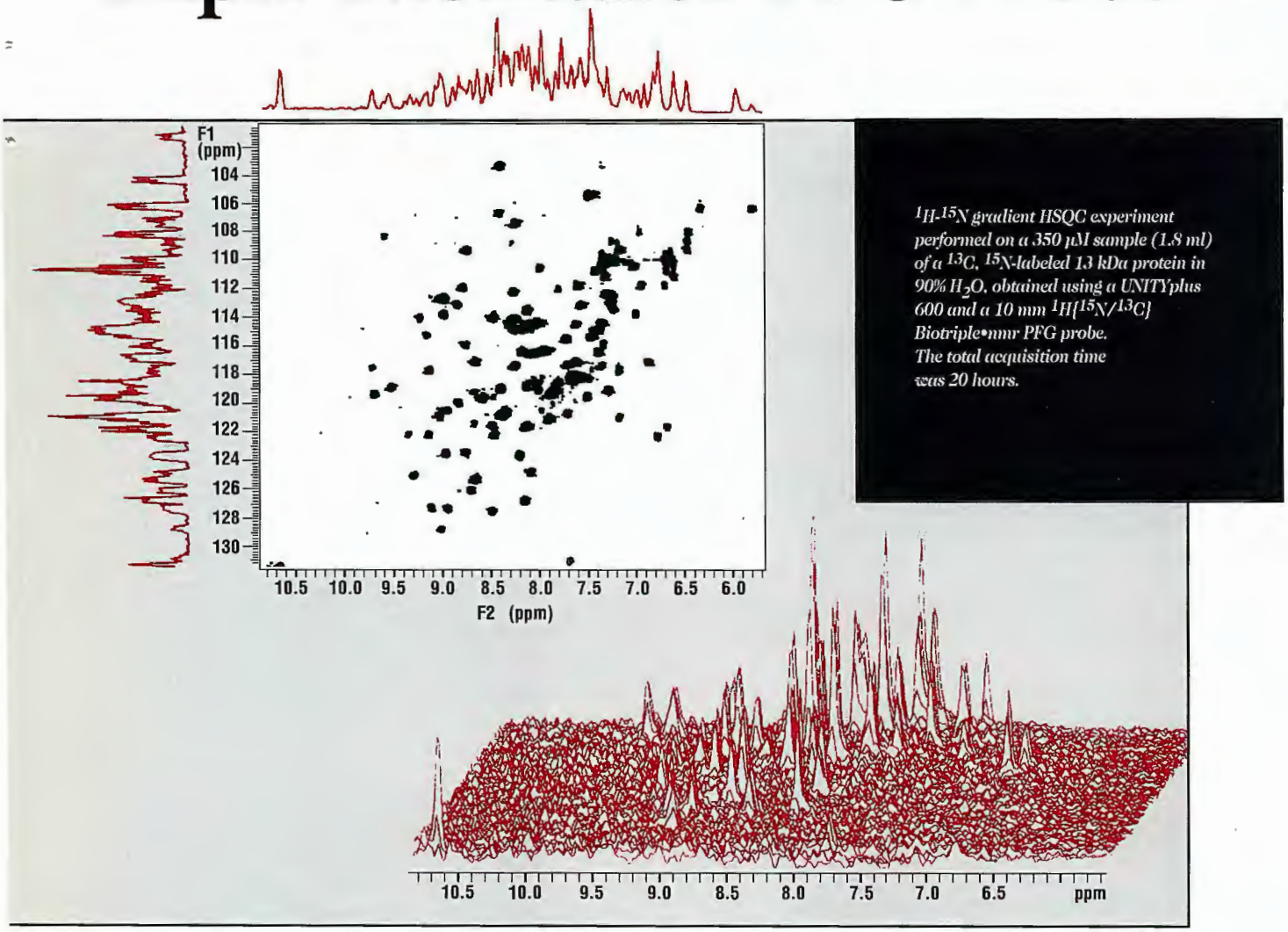

^{1}H-^{15}N gradient HSQC experiment performed on a 350 µM sample (1.8 ml) of a ^{13}C, ^{15}N-labeled 13 kDa protein in 90% H_2O, obtained using a UNITYplus 600 and a 10 mm $^{1}H\{^{15}N/^{13}C\}$ Biotriple•nmr PFG probe. The total acquisition time was 20 hours.

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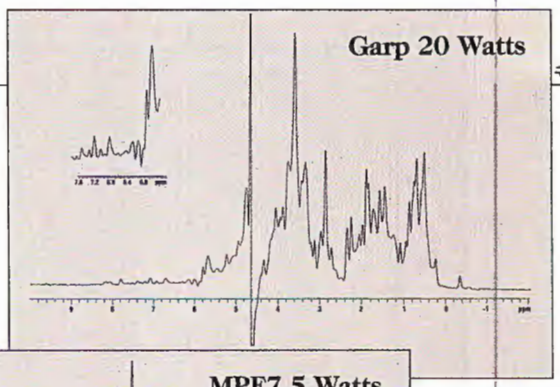

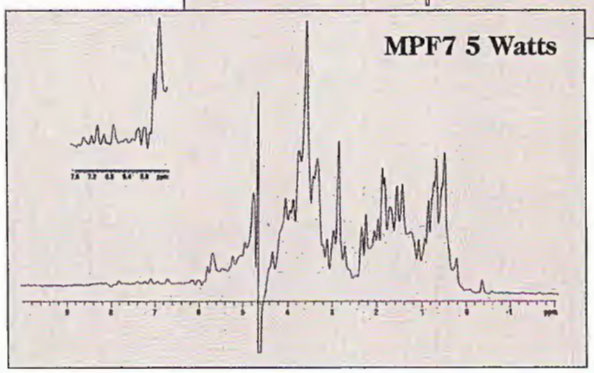

[1] Shaka, A.J., Barker, P.B., Freeman, R., *J Magn. Reson.*, 64, 547 (1985).
[2] Fujiwara, T., Nagayama, K., *J Magn. Reson.*, 77, 53 (1988).

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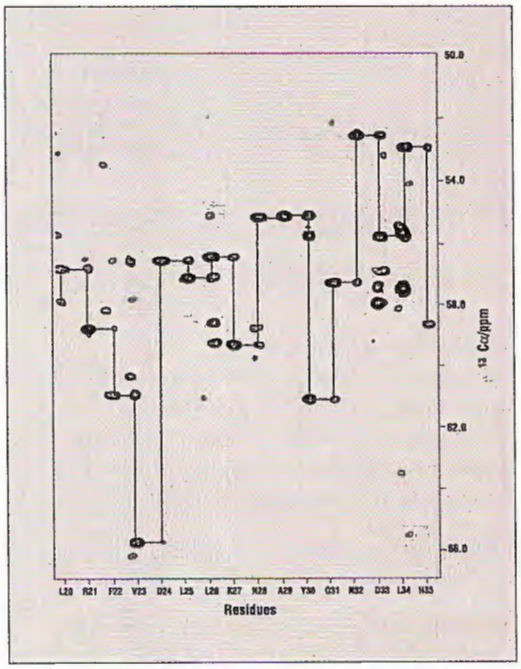
This plot displays the resultant sequential connectivities for helices A and B of the *Trp*-Repressor/DNA Complex. Utilizing the experiment above, L. Kay and co-workers* have obtained 100% of the intra-residue and 94% of the inter-residue correlations for the 37 kDa complex.

Spectrum provided by Toshio Yamazaki, Weon Tae Lee, Matt Revington, Cheryl Arrowsmith and Lewis Kay from the University of Toronto and the Ontario Cancer Institute, Toronto, Canada.

*Yamazaki, T., Lee, W.T., Mattiello, D.L., Dahlquist, F., Revington, M., Arrowsmith, C., and Kay, L.E., "An HNCA Pulse Scheme for the Backbone Assignment of ^{2}H, ^{13}C, ^{15}N-Labeled Proteins: Applications to a 37 kDa *Trp*-Repressor Complex," *J. Amer. Chem. Soc.* (submitted).



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Dr Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Oct 10, 1994
(received 10/17/94)

Don´t swim with this one; 3D structure of a toxic cyclopeptide from water blooms.

Dear Barry,

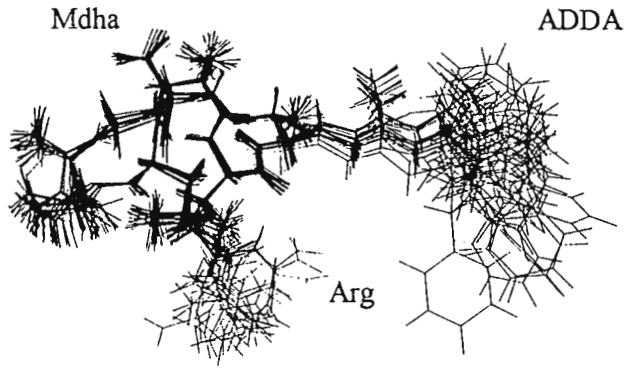
Together with some Finnish groups, we have been interested in the structure-activity relationships of a class of cyclic hepatotoxic heptapeptides formed during cyanobacterial blooms. In addition, these peptides, called microcystin-X,Y´s (MC-X,Y) have been shown to work as potent inhibitors of protein phosphatases 1 and 2A and their promotion of hepatic tumors has recently been verified.

A knowledge of the 3D structure of MC-X,Y is thus of interest to understand the phosphatase inhibition process. Many MC-X,Y´s are known, where the toxicity varies somewhat depending on the choice of the two variable L-amino acids X and Y. Besides unfavorable dynamics, the most tedious work from a modeling point of view has been the parameterization of the more unusual amino acids like the long conjugated ADDA, (2S,3S,8S,9S)-3-amino-9-methoxy-2,6,8-trimethyl-10-phenyldeca-4,6-dienoic acid. Especially important task, since the all-trans configuration of ADDA is essential for the toxicity and that a strongly related cyclic pentapeptide, nodularin, also containing ADDA shows identical binding and toxicity behaviour.

However, working in various aqueous cryoprotective media, we were able to get enough reliable distance (NOESY, MARDIGRAS) and dihedral angle constraints for a single 3D structure to be determined (SA). Contrary to some recent attempts using DMSO as solvent (DG) [1] and giving three conformational families, we found a single saddle-shaped structure where the ADDA "tail" is sticking out from the ring (RMSD < 0.6 Å).

Best regards

Ulf Edlund



1. Rudolph-Böhner et al., FEBS Letters (1994), 319-323

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06 - 10 - 94

(received 10/12/94)

Dear Barry,

Prosthetic devices and magnetic fields

We have recently had an incident that may have been caused by the stray field from our MSL 500 magnet. A student passed about 16 metres away from our MSL 500 magnet and her prosthetic pain killing device that works on the same principle as a heart pacemaker was switched off. The wide bore 500 magnet does have an enormous stray field which is greater in the vertical than the horizontal direction. My estimate based on data supplied by Bruker is that she could only have experienced a maximum field of 1 gauss. On the other hand the device has never switched itself off before, so we are working on the assumption that it was the stray field that caused the incident.

This incident emphasises the importance of visible warning notices and careful instructions to janitorial staff concerning the possibility of such events. We have such notices at the entrances to our building but this did not stop the incident!

Thank goodness it was not a heart pacemaker!!!

Yours sincerely,

Frank

Frank Riddell

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(received 9/26/94)
September 19, 1994
Short Title: Bruker/TCPIP Error Log Files
Dear Dr. Shapiro,

In the past year we have upgraded our three Bruker AM consoles to Bruker AMX consoles with AspectStation computers (MIPS in a box, not SGI, not X-32). These workstations were added to our divisional subnet of the corporate network to transfer data among the spectrometers and among several standalone SGI computers for external data processing. We soon found that the Bruker standard installation procedure and our corporate network standards created very large (60 MByte per week) errorlog files. This letter details the problem and our solution (not unique!), and also documents a security problem with the standard installation procedure.

Our corporate standards require the use of 255.255.255.000 for netmasks on workstations. Unfortunately this allowed packets from corporate subnet routers to reach our workstations; with the above netmask, the routers could not be recognized, and the message "routed: packets from unknown router" was created and placed in the files /usr/adm/console_log.X and /usr/adm/messages.X. These two files grew by about 4.2 MBytes per day for seven days, when a chron file then began to delete the last 24 hours of error messages at midnight, but wasting about 60 MBytes of disk space.

Our solution was short and brutal: we commented out the route daemon startup and killed the existing route daemon. A portion of the file /etc/init.d/netdaemons that is relevant follows

```
#   Check for existence of tcp first.
if [ ! -x /etc/havetcp ] || not /etc/havetcp
then
    exit 0
fi
echo "Internet daemons:\c"
```

```
# The following is commented out to disable "routed"
#if test -x /etc/gated -a -r /etc/gated.conf ; then
#    gated;                echo " gated\c"
#elif test -x /etc/routed ; then
#    if [ -r /etc/routed.conf ]; then
#        routed `head -1 /etc/routed.conf`
#    else
#        routed
#    fi
#    echo " routed\c"
#fi
```

Now, as superuser, identify the existing daemon process ID with "ps", and then kill it with "kill -9 process_ID". Now the only IP addresses that can communicate with the workstation are detailed in /etc/hosts. With this deletion, we now average 2.5KBytes per day and 500 bytes per day in console_log.X and messages.X, respectively.

Since lots of things can go wrong, do not attempt the above unless you are, or have, an experienced UNIX system manager. A service contract with Bruker probably wouldn't hurt, either.

A second, annoying feature of the standard installation was discovered after the above modification. The tftp daemon can be started by an external user, and system files could be transferred to said external user. Since "you should not run the tftp daemon on your system unless it is absolutely required, and then only if it can be run in a secure mode (tftp -s) that restricts tftp access to a specific directory"[1], this is an obvious security no-no. It can be rectified by commenting out the appropriate line in /etc/inetd.conf:

```
#tftp    dgram    udp    wait    root    /usr/etc/tftp    tftpd
```

Unfortunately, a reboot of the system is necessary to (in)activate tftp.

I hope that this information is of use to some subset of TAMUNMR newsletter readers. I must confess that a coworker, Ken Visscher, is our systems manager, and he tracked down and cured these and many other problems over the past year.

Sincerely yours, Paul Fager

[1] Craig Hunt, **TCP/IP Network Administration**, O'Reilly & Associates, 1992, p.308.

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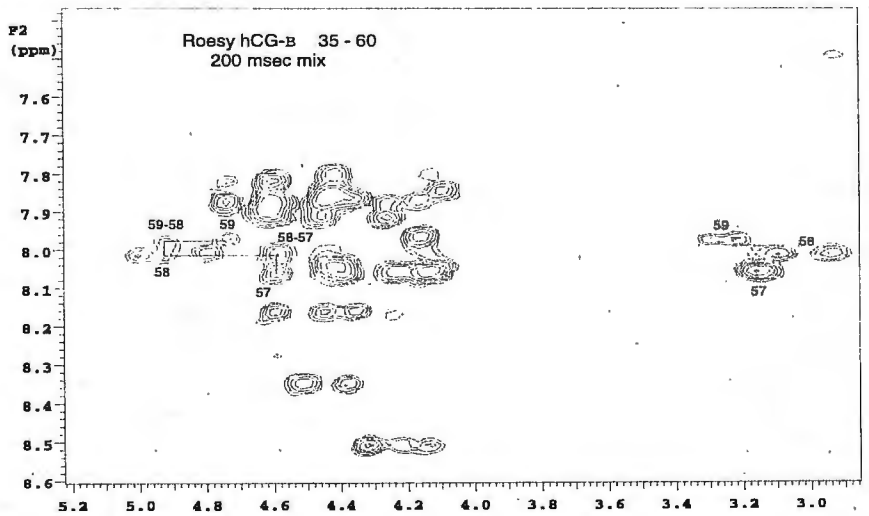
Dr. Bernard Shapiro
TAMU Newsletter
966 Elsinore Court
Palo Alto, CA 94303

October 10, 1994
(received 10/14/94)

Dear Barry,

A recent contribution to TAMU by Phil Bolton taught the utility of using Adobe Illustrator for the MacIntosh to annotate files imported from VNMR. We considered using Silicon Graphic's Irix Showcase for annotation. This program is able to import graphic images captured from the SGI screen and allow annotation. The resultant "screen" resolution, however, was not acceptable for publication. Showcase is capable of using postscript files and retains high resolution but does not allow any alterations other than resizing the image (to our knowledge). We next sought out to use Adobe Illustrator (AI), but using a PC running windows rather than a MacIntosh. As mentioned before, the VNMR plot file must be converted in order to be imported into AI. AI ver. 5.5 for MacIntosh includes the conversion software, Acrobat Distiller, but the latest DOS version (4.02) does not include this feature. Our current protocol is to capture the a VNMR postscript file (earlier Magnetic Moments), bring it over to a PC via ftp and convert to AI format with Transverter Pro (ver. 1.03, Techpool Software, Cleveland, Ohio). The spectral image is then imported into AI and annotated. It can now be printed from AI or imported into a manuscript in a word processor (i.e. WordPerfect). The AI file can also be used to generate slides for presentation. An annotated ROESY spectrum illustrates the results obtained using this approach.



Sincerely yours,

Dave Babcook William Gmeiner

University of Nebraska—Lincoln University of Nebraska Medical Center University of Nebraska at Omaha University of Nebraska at Kearney

Acorn NMR

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ABL - BASIC RESEARCH PROGRAM

Operated for the National Cancer Institute by Advanced BioScience Laboratories, Inc.

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

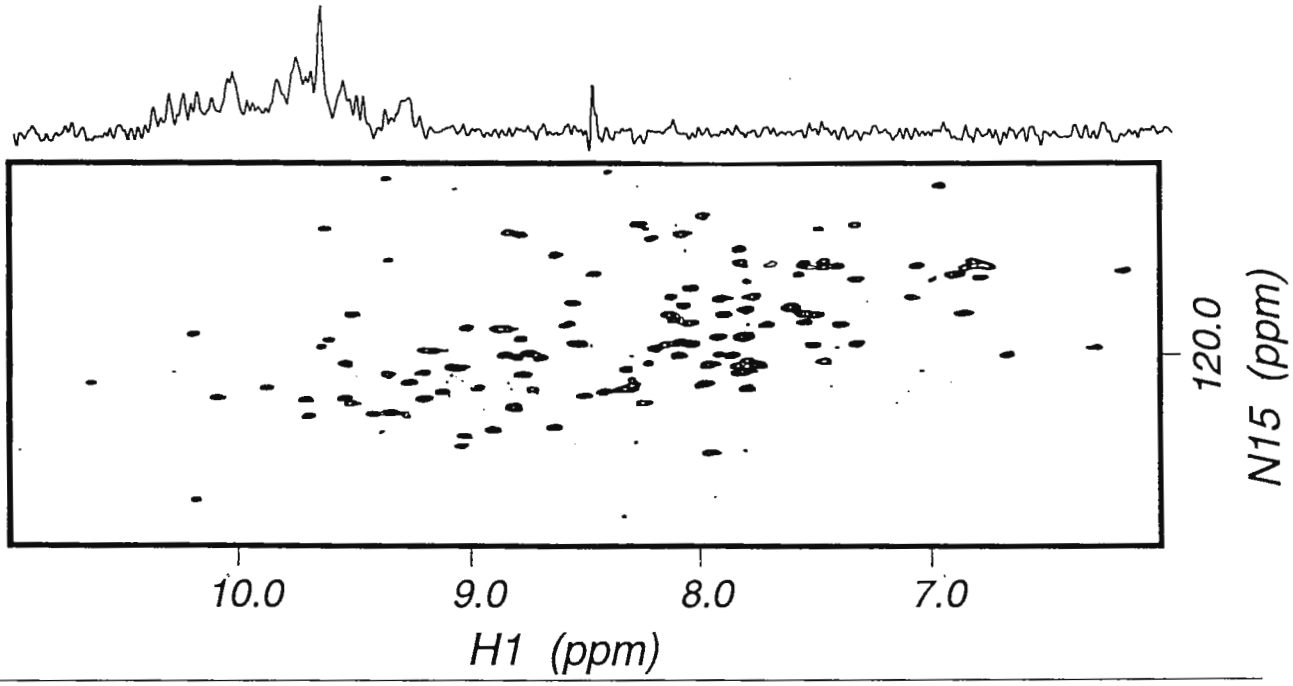
October 4, 1994
(received 10/6/94)

PERFORMANCE OF A NALORAC 8MM PFG TRIPLE RESONANCE PROBE

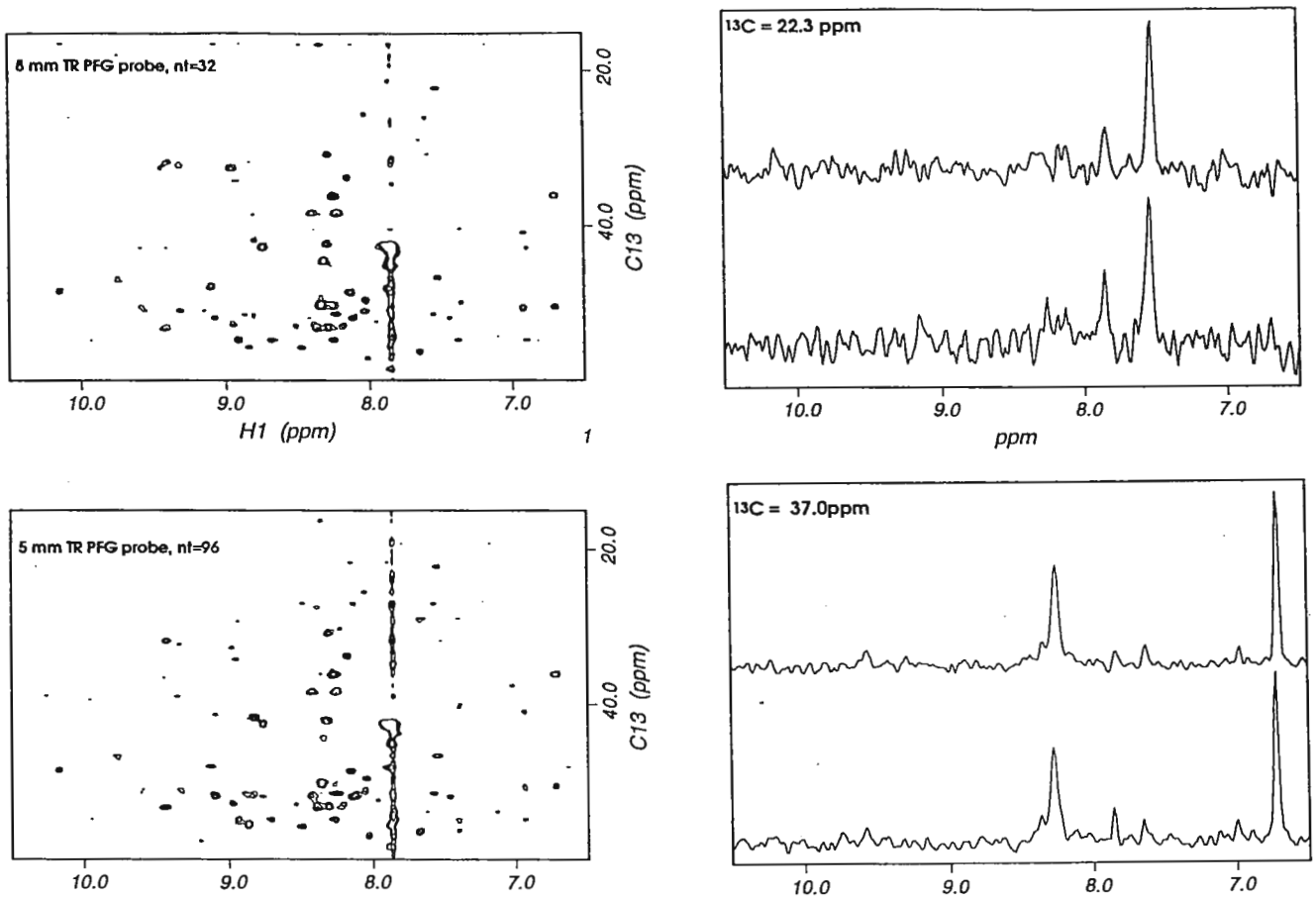
Dear Barry,

We report here some impressive results from a first generation 8mm PFG triple resonance probe provided to us by Toby Zens of Nalorac Cryogenics Corporation. The 8mm probe is an exciting development for the NMR studies of macromolecules since large diameter NMR tubes increase the feasibility of studying biomolecules with poor solubility or with tendencies towards aggregation at typical concentrations of ~ 1mM. The availability of pulsed field gradients in the 8mm probe is a significant advancement since it allows for the acquisition of gradient enhanced pulse sequences and very importantly, the suppression of water using gradient techniques. The improvement in sensitivity available with the 8mm probe can be realized in one of two ways: (1) data can be collected in a shorter period of time than with a 5mm probe, for the same concentration as a 5mm probe sample, or (2) the same sensitivity obtained with a 5mm probe can be realized on a sample of lower concentration. Specifically, the threefold increase in sample volume from a 5mm to an 8mm probe allows for a factor of three reduction in measuring time or a 1.7 reduction in concentration to that of a 5mm sample for comparable sensitivity.

Below shows a 600MHz gradient HSQC spectrum acquired at 37°C on a 123 uM sample of [^{13}C][^{15}N]ΔSNase (Mol.Wt. ~ 18kDa) in 13 minutes. The ^{1}H and ^{15}N 90° pulse widths were 12.7 usec and 55 usec, respectively. The spectrum consists of 128 complex points, 2 scans per point, with an F1 sweep width of 2432 Hz and an acquisition time of 64 msec. The trace at the top is the full spectrum of the first t_1 increment which shows the quality of the water suppression attainable with the gradient hardware. All observable backbone amides are detected under these conditions.



Shown below are the first H-C planes of a gradient enhanced CBCA(CO)NH experiment on a sample of [^{13}C][^{15}N]ΔSNase at 860 uM collected on a 5mm PFG triple resonance probe and on the Nalorac 8mm probe. 90° pulse widths for ^{1}H and ^{15}N were 10.55 usec and 45 usec for the 5mm probe and 14.1 usec and 57 usec for the 8mm probe. For both probes the ^{13}C 90° pulse width was 50 usec. The 8mm sample data set consisted of 50, 32 and 512 complex points in t_1, t_2 and t_3 with acquisition times of 5.4 msec, 13.2 msec and 64 msec, respectively. The 5mm sample data set consisted of the first H-C plane with 50 and 512 complex points in t_1 and t_2. For the 5mm sample, 96 scans were collected for each FID and 32 scans for the 8mm sample. For the full 3D matrix, the total measuring time would be 185 hours with the 5mm probe. In a third of the measuring time, virtually all resonances in the spectrum acquired with the 8mm probe have at least the same intensity as in the 5mm probe spectrum and a majority have more. Two representative F_3 slices from the 8mm (top trace) and 5mm (bottom trace) CBCA(CO)NH data sets are compared.



The ability to use lower concentrations can be crucial if only limited amounts of material are available and may also circumvent problems relating to solubility or states of aggregation. A decrease in the measuring time, enhanced by the PFG hardware, may prove important for biomolecules with limited stability, particularly at 37° C. The clean performance of the PFG hardware for water suppression is invaluable in the implementation of all sequences designed to detect exchangeable protons. For these reasons, the use of the 8mm probe equipped with PFG will allow the examination of more dilute or less stable proteins in water, which is expected to be the norm rather than the exception. Our work was sponsored by the National Cancer Institute, DHHS, under contract no. NO1-CO-74101 with ABL.

Best regards,

Donna Baldisseri R. Andrew Byrd

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(415) 493-5971 - *Please call only between 8:00 am and 10:00 pm, Pacific Coast time.*

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Deadline Dates

No. 436 (Jan.)	16 December 1994
No. 437 (Feb.)	27 January 1995
No. 438 (March)	24 February 1995
No. 439 (April)	24 March 1994

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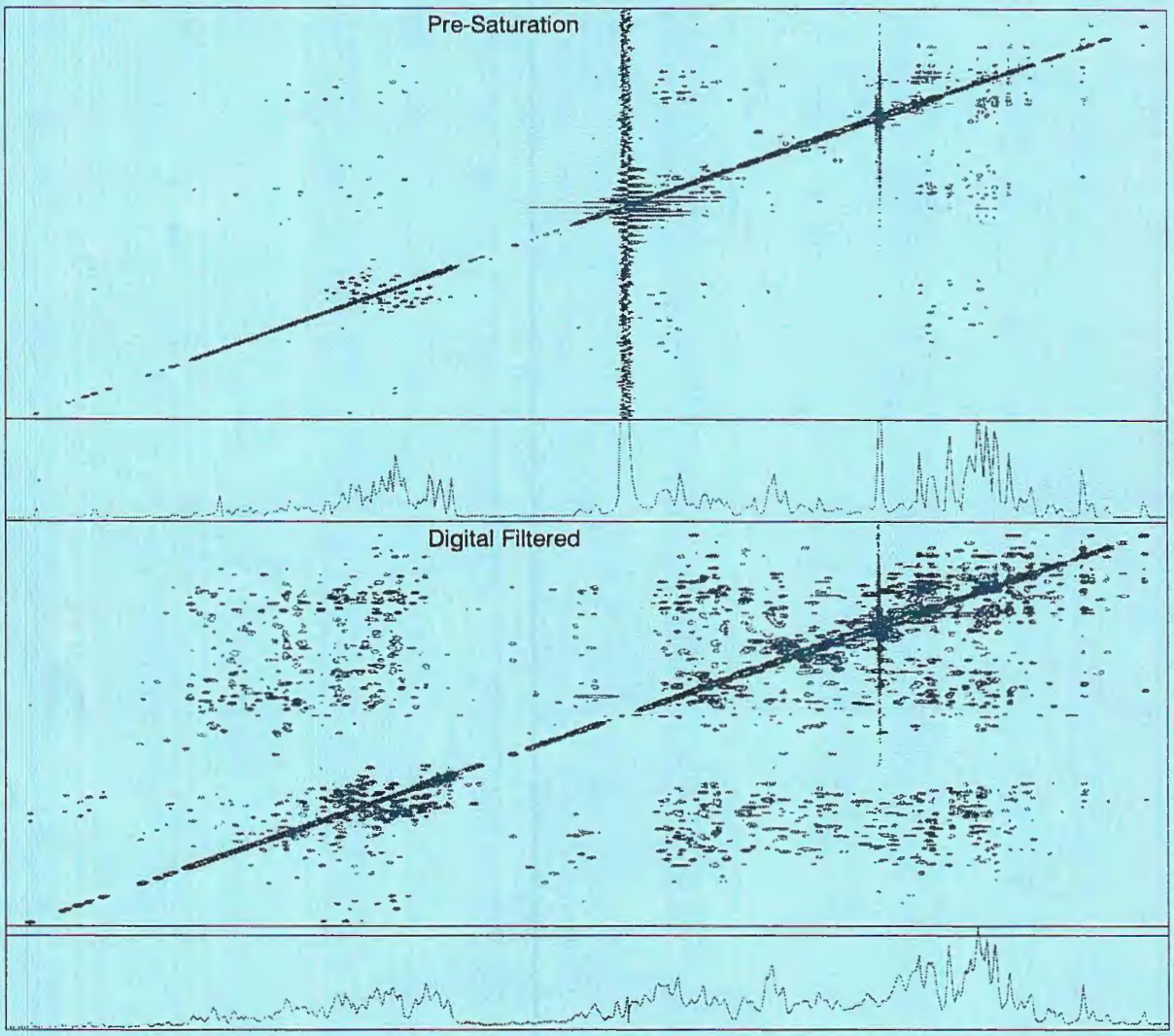
ECLIPSE NMR Advantage: Digital Filtering



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This data shows the digital filtering capability of JEOL USA's ECLIPSE NMR workstation. Eclipse does digital filtering via software after the data is acquired, not via hardware during acquisition. This offers a significant advantage because with software digital filtering the acquisition is completed before you filter the data.

JEOL feels spectrometer time is best spent acquiring new data rather than repeating experiments because conditions were not optimized. It takes more time to write this kind of software, but JEOL took the time. Now you can use the ECLIPSE NMR Advantage to your advantage.

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