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A monthly collection of informal private letters from Laboratories of NMR. Information contained herein is solely for the use of the reader. Quotation is not permitted, except by direct arrangement with the author of the letter, and the material quoted must be referred to as a "Private Communication". Reference to the TAMU NMR Newsletter by name in the open literature is strictly forbidden. These restrictions apply equally to both the actual Newsletter participant-recipients and to all others who are allowed open access to the Newsletter issues. Strict adherence to this policy is considered essential to the successful continuation of the Newsletter as an informal medium of exchange of NMR information.
## TABLE 1 DEUTERATED SOLVENTS

<table>
<thead>
<tr>
<th>Cat. No.</th>
<th>Description</th>
<th>Formula</th>
<th>Min. (g/mL)</th>
<th>Dens (g/mL)</th>
<th>MP (°C)</th>
<th>BP (°C)</th>
<th>$\chi_x \times 10^6$ @ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>D-11</td>
<td>Acetone-d_6</td>
<td>C_6H_12O_6D_6</td>
<td>0.64</td>
<td>0.97</td>
<td>17</td>
<td>64</td>
<td>0.551 (32)</td>
</tr>
<tr>
<td>D-129</td>
<td>Acetone-d_6 + 1% TMS</td>
<td>C_6H_12O_6D_6</td>
<td>99.8%</td>
<td>0.97</td>
<td>17</td>
<td>64</td>
<td>0.460 (20)</td>
</tr>
<tr>
<td>D-14</td>
<td>Tetrachloroethane-d_2</td>
<td>C_2H_4Cl_2D_2</td>
<td>0.543 (20)</td>
<td>0.611</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D-28</td>
<td>Chloroform-d</td>
<td>CDCl_3</td>
<td>99.8%</td>
<td>1.50</td>
<td>-64</td>
<td>62</td>
<td>0.440 (20)</td>
</tr>
<tr>
<td>D-31</td>
<td>Chloroform-d + 1% TRAC</td>
<td>CDCl_3</td>
<td>99.8%</td>
<td>1.50</td>
<td>-64</td>
<td>62</td>
<td>0.440 (20)</td>
</tr>
</tbody>
</table>

Cost-conscious quality NMR solvents offered by Wilmad, such as CDCl₃, are frequently priced lower than more traditional sources. Included in this offering are the most common solvents, like Acetone-d₆, Benzene-d₆, D₂O, and DMSO-d₆, as well as some of the most unusual solvents for specialty applications, like 1,1,2,2-Tetrachloroethane-d₂, Octane-d₈, and Trifluoroacetic Acid-d.

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<table>
<thead>
<tr>
<th>Cat. No.</th>
<th>Size</th>
<th>Instrument/Model</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>WCV-100 (G-100X)</td>
<td>11&quot; X 26&quot;</td>
<td>H-100, HA-100, HA-100D</td>
<td>Gridded-2 Color</td>
</tr>
<tr>
<td>WCV-60 (G-60X)</td>
<td>11&quot; X 26&quot;</td>
<td>A-60, -60X, -60A</td>
<td>Gridded-2 Color</td>
</tr>
<tr>
<td>WCV-XLFT-60</td>
<td>11&quot; X 26&quot;</td>
<td>XL-600</td>
<td>Gridded-2 Color</td>
</tr>
<tr>
<td>WCV-XLFT-300</td>
<td>11&quot; X 26&quot;</td>
<td>XL-300, XL-200, XL-400</td>
<td>Gridded-2 Color</td>
</tr>
<tr>
<td>WCV-20 (CFT-20)</td>
<td>11&quot; X 16 3/4&quot;</td>
<td>CFT-20, FT-80, FT-80A</td>
<td>Gridded Two Color</td>
</tr>
<tr>
<td>WCV-20 (W-20)</td>
<td>11&quot; X 17&quot;</td>
<td>All Models</td>
<td>Blank</td>
</tr>
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</table>

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FORTHCOMING NMR MEETINGS

9th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, July 10-14, 1989; University of Warwick, Coventry, England; Contact: Dr. John F. Gibson; (01) 437-8656; See Newsletter 364, 72.

NMR Spectroscopy In Vivo (Clinical Applications), July 10-12, 1989; Lyon France; Contact Prof. M. Amiel - see Newsletter 364, 73.

10th ISMAR Conference, July 16-21, 1989; Morzine (Haute-Savoie), France. (Note the newly announced location.); Contact: P. Servoz-Gavin, Department de Recherche Fondamentale, Centre d'Etudes Nucleaires de Grenoble, B.P. 85X, 38041 Grenoble Cedex, France.

Fifth International Symposium on Magnetic Resonance in Colloid and Interface Science, August 7-11, 1989; See Newsletter 367, 57.

The Society of Magnetic Resonance in Medicine - Eighth Annual Scientific Meeting and Exhibition, August 12-19, 1989; Amsterdam, The Netherlands; Contact: The S.M.R.M. Business Office, 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415)841-1899, FAX (415)841-2340.

Eastern Analytical Symposium, September 24 - 29, 1989; New York City; Contact: EAS, P. O. Box 633, Monchannin, DE 19710-6633; (302) 453-0785.

International Symposium NMR Spectroscopy: Structure and Dynamics of Polymeric Materials in the Solid State, Sponsored by the ACS Division of Polymer Chemistry, December 5-8, 1989; Keystone, Colorado; Contact: Mrs. Betty J. Schreiner, E.I. Du Pont de Nemours & Co., Experimental Station, Wilmington, DE 19880-0356; (302) 695-4817.

Spatially Determined NMR, Sponsored by the British Radiofrequency Spectroscopy Group; December 17-20, 1989; Cambridge University, U.K.; Contact: Prof. L. D. Hall, Level 4 RTC, Addenbrookes Hospital, Hills Road, Cambridge CB2 2QO, England: (44) (223) 356605.

Additional listings of meetings, etc., are invited.  

DEADLINE DATES

No. 371 (August) —— 21 July 1989
No. 372 (September) —— 18 August 1989
No. 373 (October) —— 22 September 1989
No. 374 (November) —— 20 October 1989
Dear Barry:

Ordinarily NMR is an unsuitable technique for studying ion-pairing, (a favorite subject here) since the exchange rate of ions among different ion pairs is fast enough to average out shifts among the different species present. Lately we ran into an interesting example of a system in which not only is the interspecie ion exchange rate slow but so also is rotation of one ion with respect to the counter ion.

The compound is 1,3-bis(trimethylsilyl)allyllithium, 1. However we make it or whatever we dissolve it in we never see more than one species, with vicinal coupling \( C_1 \), \( C_2 \) Hz of 15.5 Hz showing the system to exist in the exo-exo configuration. Note the most 1-alkyl-allyllithiums form mixtures of endo and exo isomers with endo slightly favored.

Carbon-13 NMR of 1 complexed to N,N,N',N'-tetramethylethlenediamine (TMEDA) at 155 K in diethyl ether-\( D_2 \) exhibits shifts at 1.78 (CH\(_3\)Si) 153.1 (C\(_2\)) and unexpectedly two peaks of equal intensity 67.75 and 67.25 for \( C_1 \) and \( C_3 \) as well as two for CH\(_3\)N in complexed TMEDA at 44.44 and 49.29, all \( s \) units. Above 150 K with increasing temperature, these two doublets progressively signal average to single lines at their respective centers. Line shape analysis of these collapsing doublets indicates that the same process must be responsible for both sets of line-shapes since the derived rates come out the same within experimental error; \( 1/\tau \) at 160 K is 130 s\(^{-1}\).

We would like to propose that 1 exists as a tight ion pair, locked into one conformation in which a symmetrically solvated Li\(^+\) renders \( C_1 \), \( C_3 \) and the two (CH\(_3\)Si)\(_2\)N groups magnetically non-equivalent. The averaging of the shifts must be due to rotation \( \Delta H^\circ = 7 \) kcal, of the solvated Li\(^+\) entity with respect to the allyl moiety, 2.

All the best.

Gideon Fraenkel
Professor of Chemistry

William R. Winchester
Associate

Albert Chow
Associate

---

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2. Present address, Union Carbide Corp. NJ
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Now Available: NMR Microscopy probes with perfusing capability for horizontal magnets. VT range -40 °C to +160 °C.
Two-Dimensional J-Resolved Proton Spectra with Complete Attenuation of the Water Resonance in the Presence of Relaxation Agents

Ashok Krishnaswami, Ernest W. Robb and Ajay K. Bose
Dept. of Chemistry, Stevens Institute of Technology, Hoboken, NJ 07030

Application of $^1$H NMR for the study of dilute aqueous samples is hindered by the strong resonance due to the water resonance. Not only does the water resonance mask a considerable part of the spectra, signals due to weaker solute resonances are not observed because of the limited dynamic range of A/D convertors available.

A number of techniques are presently available for the attenuation of the water resonance in one dimensional NMR. The most widely used are presaturation and selective excitation pulse sequences (1). Extension of these techniques to the measurement of two dimensional spectra leads to the problem observed in 1-D, namely the attenuation of solute resonances in the area of the water resonance.

In 1985 Rabenstein and coworkers (2), using chemical relaxation agents to shorten the $T_2$ relaxation period of the water protons, successfully achieved attenuation of the water resonance by applying the Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence. Use of the CPMG pulse sequence in combination with the COSY pulse sequence produced two dimensional correlated spectra with attenuation of the water resonance (2).

In 1987 Rabenstein extended the use of chemical relaxation agents to obtain two dimensional J-resolved spectra (3). The sequence employed was:

$$D_1 - 90^\circ (\theta) - (\tau - 180^\circ (\beta) - \tau)_n - t_{1/2} - 180^\circ (\phi) - t_{1/2} - ACQ$$

As can be observed the sequence employs a combination of CPMG and spin echo.

The modification presented in this communication is an extremely simple step. The CPMG section $(s-180-s)_n$ can be replaced by a delay period $D_2$.

$$D_1 - 90^\circ (\theta) - D_2 - t_{1/2} - 180^\circ (\phi) - t_{1/2} - ACQ$$

Apparently, dephasing does not take place to an appreciable extent, while the delay is long enough to attenuate the water resonance (0.2 s).

This sequence was used to obtain the spectra of norephedrine HCl and Xylocaine HCl (Figures 1 and 2). The relaxation agent appropriate to the pH of the solution was used.

The advantage of this sequence over the CPMG/spin-echo is that by avoiding the CPMG portion, reflected power on the observe channel is avoided.

(received 5/5/89)
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- Range of computer peripherals and enhancements
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<table>
<thead>
<tr>
<th>Magnet</th>
<th>Center Field</th>
<th>Magnet Bore</th>
<th>Clear Bore</th>
<th>Maximum Gradient</th>
<th>Plotted Homogeneity</th>
<th>HHLW Resolution</th>
<th>5 Gauss On-Axis</th>
<th>5 Gauss On-Radius</th>
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</thead>
<tbody>
<tr>
<td>300/180</td>
<td>7.05T</td>
<td>183 mm</td>
<td>125 mm</td>
<td>4.0 G/cm</td>
<td>80 mm DSV ± 6 ppm</td>
<td>35 mm DSV 0.1 ppm</td>
<td>5.60 m</td>
<td>4.45 m</td>
</tr>
<tr>
<td>200/330</td>
<td>4.7T</td>
<td>330 mm</td>
<td>254 mm</td>
<td>2.3 G/cm</td>
<td>140 mm DSV ± 5 ppm</td>
<td>70 mm DSV 0.1 ppm</td>
<td>6.95 m</td>
<td>5.60 m</td>
</tr>
<tr>
<td>200/400</td>
<td>4.7T</td>
<td>400 mm</td>
<td>324 mm</td>
<td>1.8 G/cm</td>
<td>140 mm DSV ± 4 ppm</td>
<td>80 mm DSV 0.1 ppm</td>
<td>8.50 m</td>
<td>6.75 m</td>
</tr>
<tr>
<td>85/310</td>
<td>2.0T</td>
<td>310 mm</td>
<td>225 mm</td>
<td>3.0 G/cm</td>
<td>100 mm DSV ± 5 ppm</td>
<td>70 mm DSV 0.1 ppm</td>
<td>4.50 m</td>
<td>3.93 m</td>
</tr>
</tbody>
</table>

**Specifications**

- DSV = Diameter Spherical Volume
- HHLW = Half-Height Line Width
- PPM = Parts Per Million

**Note:** Equipment described is intended for investigational purposes, and is not approved by the FDA for clinical use.
Observation of $^7$Li Quadrupolar Splittings from Oriented LiDNA in Equilibrium with Salt Solution

Dear Dr Shapiro,

We have for quite long time been interested in oriented DNA samples, prepared by Allan Rupprecht using the wet-spinning method (1), and the NMR spectra of the alkali metal counterions (2,3) in such systems. Recently, we found it desirable to study lithium–7 quadrupolar splittings from LiDNA cubes in equilibrium with isotropic salt solution. The problem is that split signal from the oriented sample is broad and very weak compared to the lithium signal from the isotropic solution which makes the determination of the splitting difficult. A representative spectrum is shown in Fig. 1.

Clearly, spin gymnastics of some kind could be helpful. The two signals differ in their relaxation properties and in the fact that the lithium nuclei in the oriented phase can sustain double or triple quantum coherence. We tried to suppress the isotropic phase signal using both these properties and found that the best results could be obtained by combining the inversion–recovery sequence with the delay corresponding to the isotropic signal being approximately nulled and the double–quantum filter. The resulting pulse sequence was thus $180^\circ - \tau_1 - 90^\circ - \tau_2 - 90^\circ - \tau_3 - 90^\circ$. $\tau_1$ is chosen to null the isotropic signal, $\tau_2$ is set to about $1/\Delta\nu$ (where $\Delta\nu$ is the expected splitting) and $\tau_3$ is 10 $\mu$s. The phase of the last pulse is cycled for double–quantum filtering. A spectrum of the same sample as in Fig. 1 is shown in Fig. 2.

Yours sincerely

Jozef Kowalewski

Johan Schultz

Fig. 1.

Fig. 2.
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Apparatus for Drying In-House Compressed Air for Low Temperature NMR Studies

Dear Barry:

Rather than using cylinders of dry nitrogen for lift air and spinner air for NMR experiments at low temperatures, we decided to dry the in-house compressed air for this purpose. The compressed air, at 40 psi pressure, is first filtered through a Monnier #204-1100-2 20 micron, 1/2 pt. capacity air line filter to remove any liquids and solids which may be present. From there, the compressed air enters a Monnier #204-4102-3 oil-coalescing air filter. It then enters the apparatus shown in the diagram at (9) and is immediately cooled by the dry ice/acetone slush. Approximately 5 ml of water will condense in the trap (17) in an overnight run. The dried air then enters a second trap (11) where it may be warmed if needed. (We have not found this to be necessary as the air is at approximately 25°C at this point). The air then enters one of two identical drying tubes, (5) or (6), filled with anhydrous calcium chloride. The ends of the tube are packed with colored Drierite and glass wool. (The second tube is simply a spare to switch to in the event that the first tube becomes moist. In an overnight run, one tube is more than sufficient to trap the residual moisture). The major portion of the water in the air condenses in cold trap (17) rather than in the drying tubes. At the end of the low temperature experiment, the ice bucket is removed. When the ice in the trap melts, it is blown out by opening stepcock (15).

Our initial problem with this system was maintaining the 40 psi pressure without a leak. This was solved by using screw clamps on the ball joints. The major advantages of this apparatus are (1) not having to purchase nitrogen for spinning and lifting the NMR tube (2) it is a grease-free system (3) operation is continuous. There is no need to disassemble the system for removing moisture. Air flow can continue through one of the drying tubes when the other is recharged with fresh calcium chloride. We have used this apparatus at −60°C with no condensation or freezing of water inside the probe.

Sincerely,

Laurine A. LaPlanche
Associate Professor of Chemistry

Ed Hyland
Glassblower

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1. Ball joint (50/30 mm) and clamp
2. Ace thread glass connector #7 (Ace Cat. No. 5027-05); O-rings wrapped with teflon tape
3. Thermometer wrapped with teflon tape on the O-ring
4. Fischer and Porter teflon inner seal (15 mm) Cat. No. 571-158 with glass outer seal (15 mm) Cat. No. 571-190.
5. Glass drying tube (1" O.D. tubing, 16" long)
6. Glass drying tube (1" O.D. tubing, 16" long)
7. 120° 3-way teflon 4mm stopcock
8. 1" O.D. glass tubing, 14" long (or any convenient length)
9. Glass inlet tube for compressed air with hose connection
10. Glass outlet tube for dry air with hose connection
11. Glass 4" x 10" trap for warming air (if needed)
12. Heater tape
13. Drain tube consisting of 4 mm stiff teflon tubing
14. Teflon attached to 7 mm glass tube
15. 2-way teflon 2 mm stopcock
16. Insulated bucket for dry ice/acetone mixture
17. Glass 4" x 10" trap for condensing moisture
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England: BRUKER SPECTROSPIN LTD., Coventry, Tel. (02) 655200
France: SADIS BRUKER SPECTROSPIN SA, Wissembourg, Tel. (089) 94 98 77
India: BRUKER INDIA SCIENTIFIC PVT. LTD., Andheri (West), Bombay, Tel. 22 62 72 32
Italy: BRUKER SPECTROSPIN SRL, Milano, Tel. (02) 2 36 40 32
Japan: BRUKER JAPAN CO. LTD., Tatsukawa, Tel. 03-37-37-234
Netherlands: BRUKER SPECTROSPIN NV, Wormer, Tel. (75) 28 52 51
Scandinavia: BRUKER SPECTROSPIN AB, Åkersberga, Sweden, Tel. (07 64) 6 80 60
Spain: BRUKER ESPANOLA S.A., Madrid, Tel. 34-259-20-71
Switzerland: SPECTROSPIN AG, Fällanden, Tel. 1-82 59 111
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BRUKER ANALITISCHE MESSTECHNIK GMBH, Karlsruhe, Tel. 0721-5967-0
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Regional Offices in Chicago, IL, (312) 971-4300/Wilmington, DE, (302) 478-8110
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To

Professor Bernard L. Shapiro
Editor - TAMMUN NMR Newsletter
966 Elsinore Court
Palo Alto, CA - 94303

May, 1989
(received 5/6/89)

Dear Professor Shapiro:

TO SEE OR NOT TO SEE PEAKS BY CP

It is well known that the rate at which various carbons polarize, $1/T_{CH}$, in a carbon-13 CPMAS experiment can dramatically change as a function of the environment of the carbon and the nature and extent of molecular motion. Obtaining reasonably correct relative signal intensities thus requires (among other things) that $T_{CH} < \text{contact time}(CT) < T_1$. In general, contact times of 1-5 ms is usually adequate for most organic compounds.

We describe here an exceptional case where presence of $^{13}$C-$^{14}$N quadrupole interactions drastically affects cross polarization which, in turn, can selectively preclude the observation of certain $^{13}$C signals. The bottom trace in Figure 1 shows such a spectrum of benzyltriethylammonium (BTEA) cation used to pillar a synthetic clay, Laponite. The complete absence of the $\alpha$-methylene signals is in sharp contrast to the top trace which represents a spectrum of the corresponding bromide salt. Both spectra were acquired using contact times of 3 ms.

![Figure 1](image)

Figure 1  50.3 mHz $^{13}$C CPMAS spectra of BTEA-Br (top trace) and BTEA-Laponite (bottom trace).
Notwithstanding some variations in the Hartmann-Hahn match, we interpret this result as reflecting the severe deformation of bond angles about the $^{14}\text{N}$ quadrupolar center that must be necessary to accommodate the BTEA cation within the layers of the clay. The nitrogen atom in BTEA bromide, on the other hand, experiences a relatively spherical charge distribution. As the CT is progressively reduced from 3 to 1 msec, the $\alpha,\alpha'$-methylene signals begin to appear (Figure 2). In fact, the Bloch decay experiment detects signals at 60 and 51 ppm that integrate in a ratio of 1:3, as expected. We would like to thank Drs. H. Eckert and J. P. Yesinowski for the use of the former NSF NMR facility at Caltech to record the spectra.

Trust this contribution will suffice in avoiding close encounters with the dreaded ultimatum. Please credit this to the account of Dr. P. S. Iyer.

Yours Sincerely,

Mario L. Occelli
Sr. Research Associate

Pradeep S. Iyer
Sr. Research Chemist
Z•Spec Probes

Indirect Detection Probes with...

* High $^1$H and X channel efficiency
* Exceptional Lineshape and Resolution
* Full X Channel Tuning ($^{31}$P-$^{109}$Ag)
* Outstanding water suppression capability

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* Multiple Plug in Coil Sizes
* $^1\text{H}/X$ Detection or Decoupling $X(^{31}\text{P}-^{13}\text{C})$
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Z•Spec Probes are available with Nalorac Cryogenics Quest 4000 series NMR Spectrometers and as after-market Probe products for all major NMR spectrometer systems. For more information about Z•Spec Probe products contact Toby Zens, manager of Special Products Group.

NALORAC CRYOGENICS CORPORATION
837 Arnold Drive Suite 600, Martinez, CA 94553
Tel: (415) 229-3581 FAX (415) 229-1651
April 25, 1989
(received 4/29/89)

Dr. B.L. Shapiro
966 Eslinore Court
Palo Alto, CA 94303
U.S.A.

Intensity Variations in Triple Quantum Spectroscopy

Dear Barry:

After examining triple quantum spectral arrays for many commonly occurring organic radicals the following features were found to occur in every case.

(1) For a spin system $A_n B_m$, the $A$ signals will be more intense for the transitions which arise from $\delta A + 2\delta B$ or $3\delta B$ than for those arising from $2\delta A + \delta B$. The reverse is found for the $B$ signals.

(2) For $m > n$ the projection of the intensities for the $A$ transitions will be weaker than the intensities for the $B$ transitions. The greater the difference between $m$ and $n$ the greater the difference in intensities. For the isopropyl group the CH proton gives a strong signal while the $CH_3$ signals are barely visible.

The triple quantum spectral array of triethylamine is shown as an example. The strong signals from the $CH_2$ resonances are at $3\delta CH_3$ and the strong $CH_3$ signals are at $2\delta CH_2 + \delta CH_3$.

Yours truly,

S. Brownstein

SB/la
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They’re all checked by $^1$H NMR at 400 MHz and they’re not only *highly enriched*, they’re very **CLEAN** and very **DRY**.

<table>
<thead>
<tr>
<th>CAT. NO.</th>
<th>PRODUCT</th>
<th>ENRICHMENT</th>
<th>QUANTITY*</th>
<th>PRICE†</th>
</tr>
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<tbody>
<tr>
<td>82-00732</td>
<td>Acetone-d$_6$</td>
<td>99.9</td>
<td>10 x 1 g</td>
<td>14.</td>
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<td></td>
<td></td>
<td></td>
<td>10 g</td>
<td>14.</td>
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<td></td>
<td></td>
<td></td>
<td>5 x 10 g</td>
<td>69.</td>
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<td>10 x 10 g</td>
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<td>82-05012</td>
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<td></td>
<td></td>
<td></td>
<td>10 g</td>
<td>22.</td>
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<td>5 x 10 g</td>
<td>100.</td>
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<td>82-84077</td>
<td>Benzene-d$_6$</td>
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<td></td>
<td></td>
<td></td>
<td>10 g</td>
<td>18.</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>5 x 10 g</td>
<td>75.</td>
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<td></td>
<td></td>
<td></td>
<td>10 x 10 g</td>
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<tr>
<td>82-80556</td>
<td>Chloroform-d</td>
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<td>50 x 1 mL</td>
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<td></td>
<td></td>
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<td>100 g</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>5 x 100 g</td>
<td>80.</td>
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<tr>
<td></td>
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<td></td>
<td>10 x 100 g</td>
<td>142.</td>
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<td>1 kg</td>
<td>135.</td>
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<tr>
<td>CAT. NO.</td>
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<td>ENRICHMENT</td>
<td>MIN. ATOM % D</td>
<td>QUANTITY</td>
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<td>------------</td>
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<tr>
<td>82-70001</td>
<td>Deuterium Oxide</td>
<td>99.9</td>
<td>100 g</td>
<td>45.</td>
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<td></td>
<td></td>
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<td>5 x 100 g</td>
<td>215.</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>10 x 100 g</td>
<td>390.</td>
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<td></td>
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<td>1 kg</td>
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<td>82-70901</td>
<td>Deuterium Oxide</td>
<td>99.8</td>
<td>min. 10 kg</td>
<td>3500.</td>
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<td></td>
<td></td>
<td></td>
<td>25 kg</td>
<td>8500.</td>
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<td></td>
<td></td>
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<td>50 kg</td>
<td>16250.</td>
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<tr>
<td>82-70002</td>
<td>Deuterium Oxide &quot;100%&quot;</td>
<td>99.96</td>
<td>10 g</td>
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<td></td>
<td></td>
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<td>5 x 10 g</td>
<td>50.</td>
</tr>
<tr>
<td>84-70001</td>
<td>Deuterium-depleted Water</td>
<td>&lt;5 x 10^-5</td>
<td>25 g</td>
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<td></td>
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<td>4 x 25 g</td>
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<td>20 x 25 g</td>
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<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>10 g</td>
<td>12.</td>
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<tr>
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<td></td>
<td>5 x 10 g</td>
<td>56.</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>10 x 10 g</td>
<td>110.</td>
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<tr>
<td>82-00809</td>
<td>Dimethyl-d₆ Sulfoxide</td>
<td>99.9</td>
<td>10 g</td>
<td>12.</td>
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<tr>
<td></td>
<td>(multi-dose septum vials)</td>
<td></td>
<td>5 x 10 g</td>
<td>56.</td>
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<tr>
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<td></td>
<td></td>
<td>10 x 10 g</td>
<td>110.</td>
</tr>
<tr>
<td>82-00061</td>
<td>Methanol-d₄</td>
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<td></td>
<td></td>
<td></td>
<td>10 g</td>
<td>43.</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>5 x 10 g</td>
<td>171.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>10 x 10 g</td>
<td>325.</td>
</tr>
</tbody>
</table>

*PACKAGING: Organic solvents are sealed in glass ampoules to maintain the high quality of the solvents. Dimethyl-d₆ sulfoxide is also available in multi-dose septum vials. The waters are packaged in glass screw-cap bottles, except for the "100%" deuterium oxide, which is packaged in multi-dose septum vials.

†Prices are FOB Miamisburg, Ohio for delivery in North America; please request prices for delivery to the other continents. Minimum order, $50.

Please request prices for quantities larger than are listed.
Please allow us to bid on your annual NMR solvent requirements.

The other NMR solvents will soon be available; we will keep you informed. In the meantime, please ask us for any compounds in which you are interested. We have more compounds than are listed in our new Price List 5/89 and we are now in a position to carry out many custom syntheses. Please call us with your requests.
Phase Properties of the $^1$H Linear Amplifier on VXR-500S

Dear Barry:

The Varian VXR-500S spectrometer that we installed last year is equipped with linear amplifiers which allow rapid changes of the power level of the pulses. Varian's design is quite simple and consists of a computer controlled attenuator on the input to the linear amplifiers. We were interested in the phase properties of the pulses for different power levels. Figure 1 shows plots of the proton spectra of a nonspinning D$_2$O sample taken at various power levels and pulse lengths. The spectra were taken with a single scan and the spectra a-f correspond to 90° pulses of 7.0, 9.8, 23.0, 39, 88, 160 µs respectively. These pulse lengths correspond to 0, 5, 9, 14, 18 and 21 dB attenuation of the input pulse into the high power proton amplifier. Although the amplifier does not have a truly linear response, we are still able to adjust the power to the desired level. We were curious to see if the phase of the pulse changes at various power levels. Figure 1 shows that phase is constant (within the experimental precision of this measurement) over the power ranges corresponding to a 90° pulse of 10 - 90 µs. At the shorter pulse length (Figure 1a) the resonance is out of phase by 10°. However under these conditions we are running the amplifier near its maximum input and we have found poorer overall performance of the system under these conditions. Therefore we normally adjust the power level to give approximately 10µs 90° pulses. Thus the system produces pulses of constant phase for power levels corresponding to an order of magnitude change in pulse width. At lower power levels (160µs 90° pulse, Figure 1f) the resonance is again out of phase by 10°. We have not tried to determine the source of the phase problem here but are guess that it arises from Varian's use of cross diodes which prevent noise from the linear amplifier leaking into the receiver. Thus the standard configuration is only "linear" at higher power and we use the decoupling channel with a directional coupler to perform low power selective excitation experiments. Varian reportedly has developed a pin diode switch to replace the cross diode network but we have not tested this.

Since most commercial instruments use nonlinear Class C amplifiers for producing high power proton pulses we have not compared these results with other systems but would be curious to see how other linear systems perform on such tests.

Sincerely,

Arthur Pardi
Dear Barry,

NIC/GE and Bruker Data Processing with PCNMR+

Last year we acquired the University of Wisconsin PCNMR+ program for processing NMR data on a PC/AT clone. We needed to use PCNMR+ for data acquired on our 3 spectrometers built around Nicolet 1100 and 1200 computers as well as Bruker NMR's. There are two problems with NMC or GENT files: Transfers to a PC and getting PCNMR+ to deal with non-Bruker data. The first problem can be solved by using Nicolet's version of KERMIT. This works but requires the NMR program be exited to start KERMIT. We prefer a simpler way. In the past I added a Nicolet Filtran compatible background transfer routine to NMC and GENT to send data to a 1200 Data Station. Unlike the GE version it can be invoked on the instrument and can send 2D data. We started writing a Filtran compatible program for the PC running at 38.4 Kbaud. A 16k file transfers in about 20 seconds to a 10 MHz AT clone.

The second problem requires more work. Parameters need to be translated to the Lycrics format and the 20 bit data need to be converted to 32 bit words interleaved as real and imaginary points. Not all parameters are used, we translate the important ones such as SF, SW, pulses and delays. The lack of spectrum reverse in PCNMR+ is overcome by looking at the SRFLAG in the parameter sector. If needed we invert the imaginary part of the FID.

Since our Bruker spectrometers are equipped with ethernet, we started writing a program to convert files transferred to a VAX with BRUKNET and retrieved on a PC via FTP. The same program also converts files transferred via the Bruker Kermit.

I worked on the PCNMR+ menus to include some undocumented features. Base line correction (BC) and Magnitude calculation (MC) are added to the PROC menu. Other parts of the MENU220.TXT file can be edited. The two numbers mean 12 lines, 13 characters wide. The number of lines must be correctly set or the PC locks up! The first column is the screen dispaly, the second is the command to be executed and the third is the corresponding help file (if it doesn't exist you can make your own). It is possible to include commands with parameters, e.g. "PJ Proton.250".

Sincerely,

Rudi Nunlist

9 15

( == FILE ==) (TITLE) (file.hlp)
( Read File) (MOUSEGET)(mouseget.hlp)
( Write File) (WR) (wr.hlp)
( Floppy Disk) (FLOPPY) (floppy.hlp)
( Hard Disk C:) (HDISK) (hdisk.hlp)
( Hard Disk D:) (DU Disk) ()
( DU Disk Unit) (DU) (du.hlp)
( ChangeDir) (MOUSEDIR)(mouseedir.hlp)
( Network Disk) (NETWORK)(netwrk.hlp)
( Exec DOS Cmd) (DOS) (dos.hlp)
( ) ()()
( EXIT PCNMR!) (MD) (mo.hlp)
FMR and Hare Research are developing and marketing an alternative for off-line NMR data processing using IBM-compatible PCs. These turnkey data stations come configured ready to process NMR data utilizing Hare Research’s Felix/PC(tm) software. This software is supplemented with a mouse-oriented, menu-based, user interface and expanded plotting capabilities supplied by FMR. Menu software greatly increases the user friendliness of the processing software for both new and occasional users. A command line interface remains in place at all times for the experienced user.

Existing PCs, printers and plotters can be used with the software modules provided for processing and plotting NMR data for as little as $1000.00. Alternatively, FMR can provide a complete turnkey PC system starting at $5000.00.

### 1DFT Processing Software

### Multi-D Processing Software

### Data transfer translation programs

### Printers (dot matrix, Postscript or PCL lasers)

### HPGL Compatible Plotters

### Ethernet communications systems

### Software Support

- Expanded user manuals.
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- Organized Bug report collection and feedback.
- New feature request incorporation process.

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- 1.2 MB Floppy
- 40 MB Hard Disk
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- 5 second 8k point CFFT
- 20 MHz 80386/80387
- 1.2 MB Floppy
- 1 MB Main Memory
- 80 MB Hard Disk
- VGA Graphics & Mouse
- DOS 3.3 & 1D NMR Software

#### PCDS-3

- 0.25 second 8k point CFFT
- 25 MHz 80386/80387
- 1.2 MB Floppy
- 2 MB Main Memory
- 8 MFLOP Floating Point Array Processor
- 130 MB ESDI Hard Disk
- VGA Graphics & Mouse
- DOS 3.3 & 1D NMR Software
Shimming can be very difficult! This statement is a given. However, the user can make it easier by making sure the instrument is properly configured. Thousands of hours of time have been wasted and many grey hairs have been gained struggling with shimming. In the previous note, the influence of the sample on the shimming process was discussed. Another area which influences the shimming process is the receiver coil.

Instrument manufacturers have always pushed for the ultimate sensitivity. The tendency to focus on this one instrumental performance parameter over others has led to a "war" of sensitivity numbers. This focus on sensitivity is being challenged by today's more sophisticated users, who judge instruments on sensitivity and other equally important performance parameters. The war has led to probes with longer receiver coils.

For a given sample tube diameter, the longer the receiver coil the more sample can be observed and the higher the potential sensitivity. Today's 5 mm ¹H probes range from 12 mm to 24 mm in length. However, a longer receiver coil generates at least two shimming problems: one from the region over which the room temperature shims can generate the proper field correction and another from the probe material's magnetic susceptibility.

Shim Correction Distance - All shim sets have a useful region of correction outside of which the supplied field correction no longer has the designed field characteristics. As the sample approaches this area, the field correction over the sample length also deviates and the shimming process becomes more difficult.

Probe Material Magnetic Susceptibility - Even when the probe is made of magnetic susceptibility corrected materials, the probe materials influence the field homogeneity in the sample area because of residual susceptibility. All materials in the probe have some susceptibility. As strange as it may seem, the glass insert has a much higher magnetic susceptibility than the copper coil. However, since the insert has cylindrical symmetry, its geometry cancels its magnetic susceptibility over the sample region to the extent that the insert is uniform. Anyone who has tried to shim a previously good probe which has developed a cracked insert knows that even when mechanical integrity of the probe is maintained, the crack in the glass makes the probe very difficult to shim. This is also why holes in the insert in the region of the sample for coil leads or light access are not practical. In many of today's probes, the materials are corrected for magnetic susceptibility and/or arranged to maintain cylindrical symmetry to minimize the influence on field homogeneity around the receiver coil.

The longer the receiver coil, the more difficult the shimming process. The first empirical observation of a long coil is a tendency to have a split field (a doubling of the peaks). This makes the Z₁ and Z₂ shim settings very sensitive, thereby putting high demands on instrumental and environmental stability.

One parameter the user should know is the length of his receiver coil. This can be determined without opening the probe as follows:

1. For a ¹H probe, put the smallest drop of water in the bottom of a 5 mm NMR tube. For probes of other nuclei, use the smallest possible sample with a strong signal on that nucleus.
2. Place this tube in a working spectrometer such that the drop of water is in the center of the receiver coil.
3. Take and process a 16K Word +/-10,000 Hz sweep width spectrum using magnitude calculation.
4. Adjust the ends of the processed spectrum to remove all DC and tilt.
5. Take an integral of the entire sweep width and set it to a value of 100.
6. Set up a MACRO or LINK to repeat this process with the same processing and integration normalization constants.
7. Record the sample position and its integral value.
8. Move the sample up 1 mm and repeat the acquisition, processing and integration. Record sample position and integral value.
9. Repeat this process until the integral value goes to zero and back up again as the water drop is moved away from the coil center.
10. Repeat the process, lower the sample in 1 mm steps.
11. Plot the integral value versus the sample position. The plot shows:
   a. The length of the receiver coil. This value can be a surprise. For proper shimming the normal sample length needs to be at least twice the length of, and centered about, the receiver coil.
   b. The position of the receiver coil relative to the bottom of the spinner. The receiver coil center should be at the center of the RT shims (to be discussed later) and in the center of the magnet's most homogeneous region.
   c. Any receiver coil pickup in a region outside of the main coil (above or below). This can be seen by the integral going to zero and back up again as the water drop is moved away from the coil center. These remote pickup areas are often at a different field value and can lead to low level lineshape distortions and poor water saturation.

![Signal Integral Versus Position Below Spinner](image-url)
Dear Professor Shapiro:

Mass NMR in Agricultural Research

Plant breeding and genetic engineering of vegetable seeds are presently being used in the development of specialty vegetable oils. These oils have derived the name specialty because they contain special or enhanced properties as a result of altering the oil composition or more specifically the carboxylic acid profile. Agricultural researchers involved in this type of work are interested in reliable non-invasive methods which can help define the acyl distribution of triacylglycerols within whole seeds. This information can then be used to screen seeds so that time and money are not wasted on the breeding of nonviable seeds, i.e. seeds which do not contain the desired acyl distribution.

Mass NMR has been shown to be a method which can nondestructively define the oil composition in single seeds (1-3). Mass NMR reduces inhomogeneous line broadening due to differences in magnetic susceptibility (4). These differences in magnetic susceptibility arise from voids or solid masses surrounded by discrete pools of oil. The heterogeneous nature of seeds is evident from an NMR u-image (0.5 mm thick horizontal slice) of a sunflower seed (Figure 1). The light shades define the geometric location of the oil and the dark shades the voids or solid masses. This particular sunflower seed was soaked in water for 12 hours. The water, which was absorbed exclusively into the shell, shows a chemical shift image 3 ppm down field from the oil. The irregular voids or solid masses shown in this image have a different bulk magnetic susceptibility (BMS) compared to the oil BMS. The BMS variation results in induced dipolar fields analogous to glass beads dispersed in liquid as previously described by Garroway (5). Under Mass conditions the dipolar interactions reduce to zero since the oil is in a liquid state and these induced dipolar fields have a geometric relation of \(3 \cos^2 \theta - 1\). Consequently, narrower line widths are observed which ultimately increase both sensitivity and resolution over non-Mass experiments.

We have used both \(^1\text{H}\) (Figure 2a-c) and \(^{13}\text{C}\) (Figure 3a-d) Mass NMR to examine a variety of our high oleic sunflower seeds developed by Agrigenetics. The unsaturated distribution data given in Table 1 represents the oleic:linoleic range that we observed from randomly selected seeds. This set of sunflower seeds was produced from a breeding program designed to yield an oil which is at least 80% oleic. This can be compared to unaltered or natural sunflower seeds which are approximately 70% linoleic and 15% oleic. The natural sunflower seeds are used to produce more typical sunflower oil, e.g. Wesson® vegetable oil. To verify the quantitative NMR results, the oil from four sunflower seeds previously examined by Mass NMR was extracted and transesterifed to the corresponding methyl ester. The methyl carboxylate distribution calculated by gas chromatography is in excellent agreement with the Mass \(^1\text{H}\) and/or \(^{13}\text{C}\) NMR results given in Table 1.

The \(^{13}\text{C}\) olefinic distribution was obtained from the integration of the oleic C-9/10 and linoleic C-13/9 peaks relative to the fully resolved linoleic C-10/12 peaks. The molar unsaturate to saturate content can also be obtained from the integral difference between the carbonyl and the olefinic regions in the Mass \(^{13}\text{C}\) NMR spectrum. This requires a 3 fold increase in acquisition time since the carbonyl carbon \(T_1\)'s (1,3 = 3.0 sec.; 2 = 2.6 sec) are approximately three times the oleic C-9/10 olefinic carbons.

The \(^1\text{H}\) olefinic distribution was obtained from the integration of the linoleic H-11 CH\(_2\) protons -CH=CH-CH\(_2\)-CH=CH- at 2.79 ppm and the oleic H-9/10, linoleic H-9,10,12,13 olefinic protons. In general the \(^1\text{H}\) NMR data is more accurate since the S/N is considerably better, especially when quantifying low levels of...
an the spectral window using a spinning rate of 2-3 KHz at our observe frequency of 400 MHz. These spinning speeds are not always trivial since the seeds do not have a symmetrical weight distribution. Furthermore, the $^1$H narrow chemical shift range is not suitable for the characterization of seeds which have a more complex oil composition. One such seed is the rape seed variety which naturally contains an unsaturated acyl distribution of oleic (18:189), linoleic (18:269,12), linolenic (18:369,12,15), eicosenoic (20:1611), and erucic (22:1613).

The olefinic carbon region of two different rape seed varieties is shown in Figure 4a,b. The top spectrum (4a) is of a high oleic rape seed variety and the bottom is of a high erucic (4b) rape seed variety. The most obvious difference between the two spectra is the oleic C-10/9 peaks in the top spectrum and the unresolved erucic C-13/14 and eicosenoic C-11/12 peak in the bottom spectrum. Each acyl group which contributes a peak to an olefinic region is defined in the bottom spectrum. The additional unsaturated acyl groups in rape oil produce a more complex olefinic spectrum when compared to sunflower seeds which have an unsaturated acyl composition of only oleic and linoleic. The small size of the rape seed as well as the complex acyl composition exacerbates the already unfavorable $^{13}$C sensitivity. In order to acquire a suitable signal intensity within an acceptable experimental time period, a sensitivity enhancement of 1 Hz line-broadening was applied to both spectra. The applied sensitivity enhancement, however, resulted in an unacceptable loss of resolution. For this reason we are presently examining polarization transfer techniques, e.g. DEPT, as a practical method to increase sensitivity thus eliminating the need for a mathematical sensitivity enhancement.

Please credit this contribution to Horton Dunn's account.

Sincerely,

Kurt F. Wollenberg

References:
2) Rutar, V.; Bugar, M.; Bline, R. J. Magn. Reson. 27,91 (1977)

Figure 1: NMR u-image of a 0.5mm thick horizontal cross section of a sunflower seed at 400MHz. The light shade is the oil, the dark shade is the voids or solid masses. The outer ring is the shell which was soaked in water and shows a chemical shift image 3 ppm down field from the oil.
Table 1: Normalized oleic (18:1) to linoleic (18:2) and saturate to unsaturate molar composition for five different sunflower seeds. The values were derived from MASS $^1$H and $^{13}$C NMR spectra of the seeds and GC analysis of the extracted oil from the corresponding seed which the MASS data was derived.

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<th>18:1 / 18:2</th>
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<td>95 : 5</td>
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<td></td>
<td>80 : 20</td>
<td>91 : 9</td>
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<td>7 : 93</td>
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<td>sunflbl</td>
<td>22 : 78</td>
<td>84 : 16</td>
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<td></td>
<td>17 : 83</td>
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<td></td>
<td>96 : 4</td>
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<td></td>
<td>97 : 3</td>
<td>92 : 8</td>
<td>GC</td>
</tr>
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</table>

Figure 2: 400.1 MHz MASS $^1$H NMR spectra of three different sunflower seeds which correspond to a) sunflbl b) sunflwh c) sunflbl in Table 1. The normalized molar oleic to linoleic concentration is given to the left of each spectrum.

Figure 3: 100.3 MHz MASS $^{13}$C NMR unsaturated spectra of four different sunflower seeds which correspond to a) sunflwh b) sunflbl c) sunflwh d) sunflbl in Table 1.

Figure 4: 100.3 MHz MASS $^{13}$C NMR unsaturated spectra of a) a high oleic rape seed and b) a high erucic rape seed. The bottom spectrum defines the generalized regions which the different acyl groups contribute olefinic peaks.
Exchange rate of proline cis/trans isomerism in calbindin D9k

Dear Dr. Shapiro:

We have recently acquired a GE Omega 500 spectrometer, the first such spectrometer installed in Europe. One of the first experiments we successfully have performed on the new spectrometer is the determination of the rate of interconversion between the two conformations we have found for our pet protein calbindin (75 amino acid residues). This conformational heterogeneity is caused by cis/trans isomerism at Pro43 (1) and forthcoming publications will present complete sequential resonance assignments for both forms.

1D spectra were recorded using a special preparation of calbindin in which all amino acids but serine and valine are deuterated (2). This sample allows the observation of resolved cis and trans signals even in one-dimensional experiments and in figure 1A the signals from one cis- as well as the corresponding trans-Val61 methylgroup are shown at different temperatures. The signals start to coalesce above 70 °C and from lineshape analysis, figure 1B, the exchange rates (trans → cis) can be determined to 1 s⁻¹ (70 °C), 3.3 s⁻¹ (80 °C), and 6.7 s⁻¹ (90 °C) respectively. Extrapolating this to room temperature we arrive at a value of 0.005 s⁻¹ which agrees with rates observed for proline isomerism in peptides (3) indicating that the loop in which Pro43 resides must be flexible.

Sincerely Yours,

Johan Kördel
Torbjörn Drakenberg
Sture Forsén
HOHHA of Strychnine on an Omega 600

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Figure 1. Determination of the Pro43 cis-trans exchange rate in calbindin D9k. Depicted at different temperatures in A) are the 500 MHz 1D signals of the Val61 C'YH3 groups from the cis-Pro43 (c-V61) and trans-Pro43 (t-V61) isoforms of 1H-Ser,Val-2H-calbindin (2). In B) lineshapes fitted to the experimental data are shown. These lineshapes were calculated using a simple model of two exchanging AX spin systems including J-coupling and the fitted trans-form residence times are given.

Subject: An Inexpensive, Stable VT Controller.

Greetings, Barry,

For 2D-FT-NMR, for precise relaxation time measurements, and for very high resolution NMR experiments it has become increasingly important to maintain very stable sample temperatures and to realize very low thermal gradients across the sample volume. Using a low cost (less than $500.00) PC-XT type computer, an inexpensive and easily available Intersil 14 bit ADC chip, a very stable constant current source and a small (about 3mm by 1 cm) platinum resistor, we have built a temperature controller which operates in the range of -160°C to +120°C with a temperature stability of better than 35 millidegrees for periods of up to about 36 hours. The temperature is measured 10 times in three seconds and an average is taken. That temperature and the two previously measured temperatures are then used to calculate the increment or decrement needed for the heater in the gas-flow heating system. The heater current is adjusted every three seconds; heater increments or decrements can be made in steps as small as of one part in 2^16. The +/- 35 mdeg temperature stability is maintained even when the room temperature is changing by +/- five degrees. The thermal gradients over the sample are small enough to obtain carbon-13 resolution of 30 to 40 mHz (this of course depends upon the temperature and the relaxation time). The total cost of the system is about $700.00. Anyone interested in the plans for this system can obtain them by writing to me at the above address.

Happy* *golfing!

Best regards,

Thomas C. Farrar
Professor of Chemistry

*Tom - Not being a golfer yourself, you might not know that 'happy' is not a useful parameter. One golfs for therapy, to avoid more intellectually supportable but less esthetic activities, or for a host of other reasons, including merely succumbing to the addiction. Sort of like mountain climbing, sailing, etc. In fact, there is only one thing less likely to make one happy than golfing, and that is - you guessed it - not golfing. Thank you, however, for your good wishes and the most rare opportunity to contribute to your education.
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<th>Typical 12 Peak Curve Fit (minutes)(^3)</th>
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</table>

\(^1\) With ca. 300 MB disk, cartridge tape, color graphics system, printer, plotter, accessories, software.
\(^2\) Starting from 256 x 2k dataset.
\(^3\) Estimated for several systems, where the software porting is underway.
\(^4\) Anticipated.

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"Two-Dimensional NMR Methods for Establishing Molecular Connectivity, A Chemists Guide to Experiment Selection, Performance, and Interpretation."

by
Gary E. Martin and Andrew S. Zektzer

This past semester I taught a course in NMR for our graduate students. While we spent a lot of time in the assigned textbook, I found as the semester progressed that I was drawing increasingly on material covered in this book for my lectures. It is not an introductory text, but it picks up where Sanders and Hunter and/or the text by Derome leave off. It is not so detailed to bring you the ability to program your own pulse sequences, but it will allow you to choose among the large number of available sequences to solve the problem at hand. The literature is covered right up to the publication date.

After the obligatory introductory chapter, the book takes up successively the subjects of establishing proton-proton connectivities, heteronuclear chemical shift correlations, relayed coherence transfer and related experiments and the 2-D INADEQUATE experiment. The final two chapters are a set of application problems and the solutions thereto.

As with all such complex subjects, there are places where the individual reader wants more information or clarification. While pulse duration is discussed, on page 42 the term flip angle is suddenly tossed in without definition. Similar lapses in terminology occur elsewhere. Thus, one suddenly is awash in coherence and coherence transfer which are concepts not defined anywhere that I can find. I worked the first application problem (6.1) and found that the assignments for H2 and H3 needed to be reversed and that the chemical shift for H10 looked more like 7.54 ppm than 7.51.

Having said that, I find this to be my single most used book on 2-D NMR. For those who have started to produce 2-D spectra and want to go further into the subject, it offers an inexpensive entre' into the recent literature with many good examples.

W.B.S.
Bernard L. Shapiro, Ph.D.
966 Elsinore Court
Palo Alto, CA 94303

Dear Dr. Shapiro:

In conjunction with our studies on the factors which control electron transfer in cytochromes, we have been interested in the motions of various forms of horse heart cytochrome c. We have recently found that the alkaline form of this protein (that appearing with an apparent pK, of about 9.5) is not one, but two new forms of the protein ("NMR Study of the Alkaline Isomerization of Ferricytochrome c", X. Hong and D.W. Dixon, FEBS Lett., 1989, 246, 146-148). This is true in both phosphate and borate buffer.

Our very recent work in this area at 400 MHz (Varian VXR-400, Georgia State University) and at 600 MHz (General Electric Omega 600, Emory University) has allowed us to establish more connectivities. Two additional spin sets, each with four resonances, can be seen in the COSY spectrum (45 °C; spin set 1: 14.8, 9.4, 10.6 and 3.58 ppm; spin set 2: 14.3, 9.0, 10.6 and 3.61 ppm).

Of the peaks in these spin sets, the two at 14.3 and 14.8 ppm are most clearly seen. The chemical shifts indicate that protons responsible for these resonances are near the ferric moiety. These peaks integrate to two protons each (with respect to the heme methyl resonances of the alkaline forms of the protein, which are found in approximately equal amounts at 45 °C).

The alkaline form of cytochrome c is thought to have a lysine as the axial ligand and there is evidence that more than one lysine can assume this role. The chemical shifts and integrations of the spin sets reported herein are consistent with assignment of these as due to two different axial lysine moieties. However, alternative interpretations (i.e. that these are due to other spin sets near the heme such as the heme propionates) are not yet eliminated.

Sincerely yours,

Dabney White Dixon

Xiaole Hong

'H NMR of cytochrome c at pH 9.25 (45 °C; \( \mu = 0.1 \) M phosphate in D_2O, 600 MHz). Resonances due to native form of cytochrome c: A and G, heme 7\( \alpha \) propionate; D, His-18 \( \beta \). Resonances due to alkaline forms: E and F: ring 3-Me of two forms of protein; B and H: spin set 1; C and H: spin set 2.
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<td>HOUSTON 8” single pen digital/analog plotter - LIKE NEW!</td>
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Policies and Practical Considerations
(Revised February 1989)

1. Policy:

The TAMU NMR Newsletter is a means for the rapid exchange of information among active workers in the field of NMR spectroscopy, as defined broadly, including imaging. As such, the Newsletter will serve its purpose best if the participants impart whatever they feel will be of interest to their colleagues, and inquire about whatever matters concern them.

Since the subscriber/participant clearly is the best judge of what he or she considers interesting, our first statement of policy is "We print anything." (This usually is followed by the mental reservation, "that won't land us in jail.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. I trust that the reasons for this policy are obvious.

The TAMU NMR Newsletter is not, and will not become, a journal. We merely reproduce and disseminate exactly what is sent in. Foreign participants should not feel obliged to render their contributions in English.

2. Public Quotation and Referencing:

Public quotation of Newsletter contents in print or in a formal talk at a meeting, etc., is expressly forbidden (except as follows), and reference to the TAMU NMR Newsletter by name in the scientific literature is never permissible. In order to quote results or use material from the Newsletter, it is necessary, in each individual case, to obtain the prior permission of the author in question and then to refer to the material quoted as a "Private Communication". If your copy of the Newsletter is shared with other readers, it is your obligation as the actual recipient of the Newsletter to see that these other readers of your copy are acquainted with, and abide by, these statements of policy.

3. Participation is prime requisite for receiving the TAMU NMR Newsletter:

In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.

We feel that we have to be quite rigorous in this regard, and the following schedule is in effect: Eight months after your last technical contribution you will receive a "Reminder" notice. If no technical contribution is then forthcoming, ten months after your previous contribution you will receive an "Ultimatum" notice, and then the next issue will be your last, absent a technical contribution. Subscription fees are not refunded in such cases. If you are dropped from the mailing list, you can be reinstated by submitting a contribution, and you will receive back issues (as available) and forthcoming issues at the rate of nine per contribution.

Frequent contributions are encouraged, but no "advance credit" can be obtained for them. In cases of joint authorship, either contributor, but not both, may be credited. Please indicate to whose account credit should be given. Please note that meeting announcements, as well as "Position Available," "Equipment Wanted" (or "For Sale"), etc., notices are very welcome, but only on a not-for-credit basis, i.e., such items do not substitute for a bona fide technical contribution. Similar considerations must occasionally be applied to a few (quasi-)technical items.

4. Finances:

The Newsletter is wholly self-supporting, and depends for its funds on advertising, donations, and individual subscriptions.

The Subscription fee is currently US$120.00 per year, with a 50% academic or personal discount. Subscriptions are available only for the twelve monthly issues which begin with the October issue and run through that of the following September. However, a subscription can be initiated at any time, and the issues back to the previous October will be provided as long as copies remain available.

Companies and other organizations are also invited to consider joining the list of Sponsors of the Newsletter. Sponsors' names appear in each month's Newsletter, and copies of the Newsletter are provided to all Sponsors. The continuation of
this non-commercial Newsletter depends significantly on the interest and generosity of our Sponsors, most of whom have been loyal supporters of this publication for many years. We will be happy to provide further details to anyone interested.

Another major, indeed most essential, source of funds for the Newsletter is Advertising. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their companies. Our rates are very modest - please inquire for details.

5. **Practical Considerations:**

a) All technical contributions to the TAMU NMR Newsletter will always be included in the next issue if received before the published deadline dates.

b) Please provide **short titles** of all topics of your contributions, so as to ensure accuracy in the table of contents.

c) Contributions should be on the **minimum** (NOTE!!) number of 8.5 x 11" (21 x 27.5 cm) pages, printed on one side only. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5 - 0.75" (1.3 - 2.0 cm) on all sides. Please observe these limits. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted in place on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

Foreign subscribers are reminded that regardless of the standard paper length you use, all material - letterhead, text, figures, addresses printed at the page bottom, everything must not exceed 10" (ca. 25.3 cm) from top to bottom.

Significant savings of Newsletter pages and total space can be made by exercising close control over the formatting and type sizes of the contributions. Please consider the following:

i) For those with computers, try using a smaller type font. The body of this page is printed in 10 point type, which I believe is adequate for most purposes. Even 12 point is acceptable, I suppose. Those who are computerized can also employ non-integral spacing of lines so that sub- and superscripts don't collide with lines below and above.

ii) **PLEASE avoid excessive margins.** Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or standard! This page has margins on both sides of 0.6" (ca. 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc.

Also, please avoid large amounts of unused space at the top of letters. Give thought to the sizes of figures, drawings, etc., and please mount these so as to use the minimum space on the page.

iii) 'Position Available', 'Equipment Wanted', and Similar Notices. These are always welcome, without charge, but not for subscription credit, of course. Such notices will appear, however, only if received with these necessarily rigid constraints: a) **Single spaced**; b) both side margins 0.6 - 0.7" (1.5 - 1.7 cm).- NOT WIDER; c) the minimum total height, please, but definitely no more than 4.5" (11.5 cm.) This will let me place such notices wherever a bit of space occurs.

iv) **AVOID DOUBLE SPACING LIKE THE BLACK PLAGUE ! ! !** This is extremely wasteful of space. Even sans computer, small type and 1.5-line (if needed) spacing can be had with a little effort.

6. **Suggestions:** They are always welcome.

Address for all correspondence:

Dr. Bernard L. Shapiro  
Editor/Publisher  
TAMU NMR Newsletter  
966 Elsinore Court  
Palo Alto, California 94303  
U.S.A.  

Telephone: (415) 493-5971.

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Please contact:

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<table>
<thead>
<tr>
<th>Solvent Code</th>
<th>Solvent Name</th>
<th>Unit Size</th>
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<td>$35</td>
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A NMR Spectroscopist in the NMR Facility of the University of Missouri is immediately available. The candidate should preferably have a Ph. D. degree or comparable experience. Expertise in modern 2D and high-field NMR techniques is essential, knowledge of software and RF electronics highly desirable. The spectroscopist will have opportunity to conduct independent and/or collaborative research. Salary is commensurate with qualification. Interested applicants should send their applications and arrange for at least 2 letters of reference to be sent to

Professor T. C. Wong  
Department of Chemistry  
University of Missouri  
Columbia, Missouri 65211

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966 Elsinore Court
Palo Alto, CA 94303

Dear Dr Shapiro,

NMR-Tube Reactions

Although NMR users with a biological bent often follow metabolites etc. in crude media in an NMR tube, organic chemists are more apt to only run spectra of purified compounds. It may be that I had unusually good access to NMR while in New Zealand, but I like to do a substantial number of my exploratory reactions right in an NMR tube. After all, you know straight away if the reaction has gone, and if you have the product you expect. It can avert an unnecessary work-up.

NMR-tube reactions have an added benefit. Because NMR gives detailed and often unequivocal structural information, it can prove your claim in a controversy. The example here involves quinone methides, e.g. 1 of lignin (model) compounds, e.g. 2. These quinone methides have developed an aura of mysticism. They are reported to be extremely reactive with anything slightly nucleophilic, and extremely unstable. Some years ago we surprised some researchers by obtaining very nice NMR spectra of a range of these, and by determining their conformations in solution by NOE experiments. But the misconceptions remain. One in particular is the commonly held belief that HO· will add nucleophilically to a quinone methide (in base), and there are plenty of reports in the literature attesting to this. [The reason that these reports are incorrect will not be dealt with here, but simply involves the unexpected stability of the quinone methide under the reaction conditions, and the work-up procedure used]. I tried for many weeks to add HO· to these quinone methides and simply could not get it to happen. I am not that lousy a chemist and the explanation is simple — the quinone methides really don't have any interest in adding HO·. But how do you convince researchers of that when they are under the misapprehension that quinone methides will react with anything. How do you demonstrate unequivocally that the quinone methide is stable under these conditions? Well, by NMR of course!

The figure illustrates the preparation of a precursor bromide and then the quinone methide (over NaOH) right in a NMR tube. The reaction was just done in CDCl₃ (CD₂Cl₂ would have been preferable as it doesn't react with base) and the NaOH was simply added in H₂O as shown. The quinone methide is cleanly formed (see spectra). After 1 hour (the reaction time people usually use) the spectrum is identical - the quinone methide is still intact. After 24 hours in fact, there is little degradation. Rather than reacting rapidly with base as was traditionally expected, it seems that storing these quinone methides over base is a good way to prolong their lives!

Sincerely,

John Ralph

(spectra on next page)
Spectra from the NMR-tube reactions described in text. 

a) erythro-2b starting material. b) after adding TMSBr, the bromide 4b is formed, initially as the threo isomer. Isomerism over 30 minutes gives ca. 50:50 threo:erythro. c) Adding NaOH in H₂O (correct - no deuterated materials) generates the quinone methide 1b very cleanly. Note that the increase in intensity in the CHCl₃ peak is from reaction with the undeuterated base. This QM solution is stable for about a day under NaOH!
Position Available: NMR Spectroscopist

The University of Delaware Department of Chemistry and Biochemistry seeks a person with Ph.D. or equivalent experience for the position of NMR Spectroscopist. Duties include maintenance of NMR and ESR instruments, obtaining spectra, supervision and training of users, and aiding users with the planning and interpretation of NMR experiments. This is not a faculty position, but the spectroscopist is permitted to do independent and/or collaborative research and may apply for research support. Equipment includes: Bruker WM250, Bruker AM250, Bruker MSL-300, Chemagnetics m100S, Varian E-9 ESR, and two older NMR spectrometers, slated for replacement. Position is available July 15 of this year. Applicants should write to:

Joseph H. Noggle
Department of Chemistry and Biochemistry
University of Delaware
Newark DE 19716

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Schering-Plough Research has available a postdoctoral position which offers an outstanding scientist the opportunity to develop NMR techniques to probe the structure of recombinant lymphokine proteins generated by the Biotechnology group. The successful candidate will have a background in two dimensional NMR of biomolecules and in offline data processing and structure generation techniques. Additional experience in protein handling techniques and/or familiarity with a graphics workstation environment would be an advantage. State of the art NMR instrumentation (GN-500, XL-400, XL-300, XL-200) and computational facilities (SUN SPARC, SUN 3/60, CONVEX C-210, SILICON GRAPHICS IRIS) with appropriate software (FTNMR, dSPACE, SYBYL, AMBER) are available.

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Subscription Renewals for the October 1989 - September 1990 Newsletter Year - Again.

Subscription renewal invoices for the October 1989 - September 1990 Newsletter year will be mailed out on or about June 30, 1989. If you should receive an invoice and do not get one by, say, July 15, please contact me without delay. Your kind prompt attention to these invoices will be greatly appreciated. In order to keep costs and my work load down to a tolerable level, please initiate payment without delay, for receipt by September 5, 1989.

Kindly note that (i) prepayment is required, (ii) all payments must be in US dollars, net of all bank or other charges, (iii) checks must be drawn on a U.S. bank or a U.S. branch of a foreign bank, (iv) checks must be made out to 'Texas A&M University', but mailed to B. L. Shapiro, TAMU NMR Newsletter, 966 Elsinore Court, Palo Alto, CA 94303, U.S.A., (v) the use of commercial subscription agencies to make payment is not acceptable.

In anticipation of revenues from Advertising and Sponsorships continuing to develop, I have decided to keep both subscription and advertising rates at their current levels for the 1989-90 year. I hope that my optimism is not unwarranted. Let me once again draw your attention to the vital role which our Advertisers and Sponsors play in maintaining the fiscal viability of the Newsletter. It would be useful for our Subscribers to make their appreciation known to the Advertisers and Sponsors for this service to the NMR public. With subscription fees as the only source of funds, the Newsletter would cease to exist in short order. Additional Advertising and Sponsorships are needed to keep up with the natural increases in the costs involved in publishing the Newsletter - can your organization help??

Newsletter Publication Schedule.

I will be away from my office for the period June 20 through July 16, 1989. My incoming mail and telephone answering machine will be monitored during this period, however, so keep those technical contributions, advertising insertion orders and payments coming, please. The actual Newsletter publication schedule will remain essentially unchanged, at least to first-order. My plans for the above period include attendance at the July 10-14 Royal Society meeting at the University of Warwick, where I hope to see many old (perhaps longstanding would be more felicitous) friends and mayhap some new ones. These Royal Society meetings are always worthwhile and most pleasant, and attending these well-organized gatherings is highly recommended for both participants and ‘accompanying persons’.

B. L. S.
1 May, 19 May 1989.

Incredibly Important Notice - Again.

When a Newsletter subscriber/participant lets his (rarely hers) subscription lapse by ignoring the pink ‘Reminder’ and ‘Ultimatum’ notices, the name is removed from the mailing list according to our time-honored recipe (q.v.). More often than not, this is followed by remorse and a feeling of deprivation by the erstwhile communicant. This in turn usually results in a late technical contribution being sent in, with the request for reinstatement on the roles of the righteous. While I am willing to accommodate the tardy (with whom I readily identify), doing so makes a non-trivial amount of extra work. Extra work makes me grumpy, for it cuts into the time for other more valued activities. Thus deprived I tend to be uncharitable.

Therefore, to aid in keeping subscriptions in good standing, the following practice is now in effect: For the issue of the month after an ‘Ultimatum’ notice is sent out, the mailing label will be adorned with a large red dot or circle. This decoration means that no further issues will be sent until a technical contribution has been received. Please respond without delay to the receipt of a red dotted issue and save me the trouble of removing and re-adding your name from the mailing list. Better, of course, is to respond to the ‘Reminder’ or ‘Ultimatum’ notice. Thanks for your cooperation.

B. L. S.
CSI 2T Applications

Shielded Gradients and Localized Spectroscopy

Eddy current effects are the leading cause of errors and lack of consistent results in gradient localization methods. It is not surprising, then, that actively shielded gradients, which have dramatically reduced eddy currents, represent a significant technology advance for all forms of B_0 gradient volume localization and spectroscopic imaging methods. The fast rise time and high gradient strength characteristics of the coil used in these experiments are also important. Even without pre-emphasis, shielded gradients recover fast enough to obtain spectroscopic information at 1 msec or less after a strong gradient has been turned off (Fig. 1).

As an example, a 4DFT spectroscopic imaging technique can resolve the four frequency domains that are associated with an NMR signal from an object: x-, y-, z-spatial coordinates and chemical shift δ. The above technique can be a practical alternative to single volume localized spectroscopy. This method allows phosphorous spectra to be obtained from well-defined regions as demonstrated in the following experiment, which was carried out on a GE CSI 2T system using high-strength, shielded gradient coils (Fig. 2). The phase-encode time is kept short (on the order of the dwell-time) to minimize phase-errors in the final spectra, as well as to avoid loss of signal due to T2 decay, which is significantly short in biological phosphates.

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Fig. 1—Using an oil/water phantom, a 10 G/cm gradient will create a water frequency profile extending from 156 KHz to 280 KHz away from normal water resonance. Residual gradient effects of less than 0.01% (50 Hz at 10 G/cm) are observed in a spectrum acquired beginning 1 msec after a 20 msec gradient pulse.

Fig. 2—Stacked plot showing 512 phosphorous spectra from 60 mm cubed region of a live rat. Each trace corresponds to 7.5 mm cubed region (voxel) from within the region of interest. The offset traces clearly show the achievable spectra and spatial resolution of the technique, as well as demonstrating localization of the liver phosphorous metabolites from that of overlying skeletal muscle. Total acquisition and processing time was two hours.
Subject: Rf STABILITY

All of the automation, elegant experiments, and high speed computer processing will do nothing for an NMR experiment if the spectrometer is not stable. The Rf section of the spectrometer must be reproducible and clean of spurious signals over periods of days for some experiments.

One of the most demanding experiments for spectrometer stability is the reverse detection (H [Cl]) experiment without C decoupling. Between the relatively sharp lines and the low magnitude of the satellites, this experiment graphically demonstrates the stability of the spectrometer. As the data below shows, a standard JEOL GSX Spectrometer has the Rf stability to do these experiments.

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