The NMR Newsletter

No. 444

September 1995

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### FORTHCOMING NMR MEETINGS

- **Western Biotech Conference**, San Diego, CA, October 18 - 21, 1995; Contact: Western Biotech Conf. Registr’n., c/o Tom Lobl, Tanabe Research, 4540 Towne Centre Court, San Diego, CA 92121; Tel: (619) 622-7035; Fax: (619) 622-7089; E-mail: info@wbconf.net.
- **37th ENC (Experimental NMR Conference)**, Asilomar Conference Center, Pacific Grove, California, March 17 - 22, 1996; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.
- **NMR Symposium at the 38th Rocky Mountain Conference on Analytical Chemistry**, Denver, Colorado, July 22-25, 1996; Contact: Dr. Joel R. Garbow, Monsanto Company, 700 Chesterfield Parkway North, St. Louis, MO 63198; (314) 537-6004; Fax: (314) 537-6806; e-mail: jrgarbow@snr.monsanto.com.
- **XVIIth International Conference on Magnetic Resonance in Biological Systems**, Keystone, Colorado, August 18 - 23, 1996; 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; 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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By now, you should have received your subscription renewal invoice. If you have not, please contact us. If you have received your invoice, please initiate payment promptly. Your cooperation will be appreciated.
Catalytic enantioselective allylic alkylation using Pd-catalysts can be complicated by unwanted geometric isomerization reactions. Specifically, an isomerization from a syn/syn isomer to a syn/anti isomer can lead to reduced ee's. 2-D $^1$H-exchange NMR is a useful method for detecting these isomers and we show a section of one such spectrum below. The 1,3-diphenyl allyl cationic Pd-complex shown, contains one of our new P,S-chiral chelates based on an exo-norborneol fragment; the protons in the two major isomers (both syn/syn) represent those from the allyl CH-proton trans to the S-atom. The point to note is that, without the exchange spectrum, one would not have thought additional syn/anti isomers to be present.

Suggested Title: $^1$H 2-D Exchange in Pd-allyl Complexes.
Announcing the World’s First Customer Installation of a Persistent 800 MHz Magnet

In July 1995, Bruker successfully energized the world’s first persistent ¹H 800 MHz (18.79 Tesla) 54 mm superconducting high-resolution NMR magnet. The magnet utilizes the innovative and patented super-stabilization technology which was first pioneered by Bruker with the introduction of the world’s first 750 MHz (54 mm) magnet in 1991.

System’s Description:
The super-stabilization technology cools the superconductors to ~ 2° Kelvin permitting higher field magnet designs with less drift and larger homogeneous regions. Additional benefits include immunity to external pressure fluctuations and even safer helium fills than with conventional cryostats due to the isolation of the “cold” helium bath surrounding the coil. A detailed description of this conservatively designed, low drift, super-stable magnet can be found in the “Technical Description of Bruker 800 MHz 54 mm Cryomagnet”, version 1.0, dated August 1995.

The Bruker 800/54 magnet is part of an AVANCE 800 NMR spectrometer system installed in the laboratory of Professors Rueterjans and Griesinger. It is equipped with a BOSS™ 2 high-performance shim system, a Digital Lock, a 100 W linear ¹H amplifier, three linear 300 W X-nucleus channels, an ACUSTAR™ 3 x 10 amp ultra-stable gradient power supply, and other accessories. In addition to in-line, real-time oversampling and digital filtering, the AVANCE 800 also features the unique new DQD (Digital Quadrature Detection) technology for artifact elimination without phase cycling.

All spectra shown in this application note were measured using a “triple-triple” 5 mm TXI GRASP III probe (triple resonance with actively-shielded x, y, and z-gradients).
System's Performance:
Approximately three weeks after energization, the $^1$H magnet drift rate has fallen to about 1 Hz/hr or less. The homogeneity of the magnet is indeed excellent, and non-spinning lineshape with chloroform is better than 7/14 Hz.

Figure 1 shows 1D increments of a presat-NOESY and a WATERGATE-NOESY to illustrate the excellent lineshape and water suppression capabilities.
The sensitivity on 0.1% EB using the 5 mm GRASP III TXI probe is better than 1,200:1 (see figure 2).

Figure 2: 0.1% EB sensitivity test

S/N = 1,234:1
Doubly-labeled Ribonuclease T1 (2.0 mM in 90% H₂O/10% D₂O at temperature 308 K) was used as a test sample for ¹H/¹³C and ¹H/¹⁵N gradient-enhanced HSQC experiments. Figure 3 shows an overview spectrum of a ¹H/¹³C-HSQC experiment with two expansions shown in figures 4a and 4b. Note the extremely good resolution in the heteronuclear dimension, as well as the excellent sensitivity (both spectra were run in approximately 20 minutes with 4 scans).
Figure 4a: 2.0 mM Ribonuclease T1, $^1$H/$^1$C-HSQC

Figure 4b: 2.0 mM Ribonuclease T1, $^1$H/$^1$C-HSQC
Figure 5 shows a $^1$H/$^{15}$N gradient-enhanced HSQC experiment without $^{13}$C-decoupling for the same sample, with an expansion in figure 6.

Figure 5: 2.0 mM Ribonuclease T1, $^1$H/$^{15}$N-HSQC, without $^{13}$C decoupling.

Figure 6: 2.0 mM Ribonuclease T1, $^1$H/$^{15}$N-HSQC, without $^{13}$C decoupling.
Several homonuclear experiments were run on a 7 mM sample of flavodoxin from *Desulfovibrio vulgaris*, a protein studied in the group of Prof. Rueterjans. The 2D presat-NOESY (figure 7) and 2D WATERGATE-NOESY (figure 8) were recorded in 40 minutes each using 4 scans and 512 increments. In the WATERGATE sequence all three gradients were used for defocussing. Both NOESY spectra show the excellent resolution and sensitivity of the AVANCE 800.

Figure 7: 7 mM Flavodoxin in 90% H2O/10% D2O, NOESY
Summary:

An *AVANCE 800* has been successfully installed and the persistent 800/54 super-stabilized magnet exhibits outstanding performance. The NMR experimental results are of extremely good quality and virtually artifact free. The data benefits from the improved S/N and dispersion of the new highest-field *AVANCE* NMR spectrometer.

*Bruker* acknowledges Prof. Rueterfants and Prof. Griesinger for using their NMR facilities and for providing the research samples.

[http://www.bruker.com](http://www.bruker.com)
DIRECT VISUALISATION OF FLIP ANGLE DEPENDENT B_1 INHOMOGENEITY

Dear Dr. Shapiro,

It is commonly assumed that B_1 inhomogeneity is one of the primary causes of artefacts in NMR imaging experiments, and in some instances, loss of signal to noise ratio. We have implemented a pulse sequence which allows us to observe directly the inhomogeneity in B_1 as a function of applied flip angle. The pulse sequence is a standard 2DFR Spin Echo protocol in which the selective 90 deg. shaped pulse is replaced by a non selective hard pulse whose duration is varied such that its resulting flip angle becomes:

\[ \frac{\pi}{2} + n(2\pi) \]

where n is an integer. Care must be taken in the timing of the whole sequence when comparing the images generated with this method, with those obtained by the standard 2DFR Spin Echo for a particular echo time. Additionally, RF power levels should be monitored and the field should be shimmed such that \( \chi B > \chi B_0 \). The sequence has allowed us to test the quality of our birdcage resonators, by filling the entire sample volume with a tightly fitting balloon filled with silicone oil. Figure 1 shows an image obtained in the manner described above in 64 mm diameter birdcage coil when \( n=1 \). The \( \pi/2 \) hard pulse had a duration of 50 µs and the Larmor frequency was 190 MHz. The image appears to be homogeneous and artefact free except for a narrow region close to the rungs of the resonator (slice thickness = 2 mm). Figure 2 shows an image at the same slice location when \( n=3 \) (total length of the RF pulse = 650 µs). The alternating bright and dark rings around the elements of the resonator demonstrate the loss of B_1 homogeneity at this particular flip angle. When the balloon was filled with water the electric permeability of the medium (\( \epsilon_{\text{water}} = 81 \), \( \epsilon_{\text{oil}} = 2.2 - 2.8 \)) reduced the RF wavelength at this frequency to the order of the coil diameter, generating a RF standing wave. With this method it is possible to observe such effect, as can be seen in Fig. 3 by a dark horseshoe shaped feature along the direction of the linear polarised field of the coil. We are grateful to Dr. Dieter Gross of BRUKER ANALITIK, Germany, for bringing this method to our attention.

Sincerely,

Wolfgang Roffmann
Fernando Zelaya
Stuart Crozier
Kurt Luscher
David Doddrell

Dr. Bernard Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303
USA

July 24, 1995. (received 7/26/95)
Figure 1.

Figure 2

Figure 3
Dear Dr. Shapiro,

Over the past year we have been working closely with Varian NMR Instruments Inc. toward the implementation of LC-NMR as a recognized analytical methodology within Eastman Kodak Company. One success story we would like to share has been the evaluation of a commercially available nonionic surfactant, Tergitol 15-5-5 whose generic structure is shown below:

\[
\begin{align*}
CH_3(CH_2)_o(CH_2CH_2O)_mH \\
\text{O} \\
CH_3(CH_2)_oCH(CH_2)_yCH_3
\end{align*}
\]

Tergitol 15-5-5 is comprised of a mixture of ethylene glycol units (varying length) capped at one end by a C12, a C13 or a C14 alkyl chain. In addition to the varying lengths of the alkyl chains (C12 - C14), the carbon site of attachment for the ethylene glycol segment varies along the length of the alkyl chain as well.

Shown in Figure 1 is a contour representation of the LC-NMR data set with the five LC peaks identified. On the left vertical axis is the reconstructed LC chromatogram generated from the NMR data, while positioned along the right axis are selected individual NMR spectra denoting the associated spectral changes as one moves from the “front to back edges” of the C12, C13, and C14 LC peaks and the “center cut” from the low molecular weight analog. From Figure 1 we conclude the carbon site of attachment (ether linkage) migrates from the middle of the alkyl chain, “front edge of the LC peak”, to one side of the alkyl chain (C-2) at the “back edge of the LC peak” for each of the three alkyl analogs:

- \( CH_3(CH_2)_oCH(O-EG)(CH_2)_yCH_3 \) “front edge of the LC peak”
- \( CH_3CH_2CH(O-EG)(CH_2)_yCH_3 \) “middle of the LC peak”
- \( CH_3CH(O-EG)(CH_2)_yCH_3 \) “back edge of the LC peak”

Shown in Figure 2 is a stacked plot representation of the resolution enhanced NMR spectra comprising the C13 alkyl chain region which supports our above mentioned conclusions.

While this is just one example for the applicability of LC-NMR and the collaborative interactions between Varian NMR Instruments Inc. and Kodak, many others do exist and we anticipate reporting on these in the future.

Sincerely,

Eastman Kodak Company:  Bill Lenhart  Tim Schunk
Varian NMR Instruments:  Dave Duff  Paul Keifer  Steve Patt

Eastman Kodak Company · 1669 LAKE AVE · ROCHESTER, NEW YORK 14650 · 716 458-1000
LC conditions: Zorbax ODS 25cm X 4.6mm id, 6 micron particle size
15:45 DiO:Methanol
Isocratic
Flow rate: 1ml per minute
Concentration: 50 mgs/ml 100ul injection

Figure 1

Figure 2
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Others

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Dear Barry:

A few months ago, we reported on the design and construction of a fairly simple device to permit the monitoring of actual pulse outputs and timings of current multi-channel NMR spectrometers (JMR 112A, 250-254 (1995)). The motivation behind this device was, partly, a result of our new involvement with Varian spectrometers with a C-like pulse programming language and my long standing interest in knowing whether the instruments were really doing what we ask them to do!

We reported several examples of the utility of the monitor to visualize high-frequency (500 and 600 MHz) pulses with modest electronic equipment, the timing of elements within a sequence, and the old trick of A.G. Redfield’s to monitor the rotating frame magnetization. Perhaps a more direct example of the utility of the device to develop and/or de-bug a complicated pulse sequence element is shown in this letter. Specifically, there are several pulse sequences which require a change in ordering of two or more pulses as an evolution time is incremented through the course of the experiment. For example, the work of Palmer, et al. (JBNMR 2, 103 (1992)) and Vuister and Bax (JACS 115, 7772 (1993)) present pulse sequences where the sequential order of two pulses must be changed during the incrementation of an evolution time. Since the incremented time is an evolution time, it is critical that the timing be exact from one ordering to the other, otherwise significant frequency artifacts can be introduced into the resulting spectrum. Current pulse programming languages purport to make this a simple task involving a logical test. Some care is required to insure that pulses do not overlap, or to deal with such a case. Despite the approach, it is essential to know that what is requested is executed.

The figure shows an example of the HNHA sequence of Vuister & Bax, actually implemented with the flip-back and Jump-Return technology of Kuboniwa, et al. (JBNMR 4, 871 (1994)). The top panel shows the entire sequence, including the $^1$H, $^{15}$N, and z-axis shaped PFG pulses. The reduction generates some distortion in the figure and the JR sequence appears to be a single pulse. The lower four panels illustrate the monitoring of the exact timing of the $^{15}$N 180° pulse relative to the
\[1^H\] 180° pulse for four increments (dwell = 138.8 usec) in the latter part of evolution of nitrogen magnetization where the cross-over occurs. The pulse sequence code which handles this crossover is shown to the right of the figure.

It is clear that the ability to monitor the actual output is a real time-saver in developing such sequences!

```c
/* now have to handle the two cases of order for the 1H and 15N 180° */
if ((bigT + tau2 + pw >0.5e-6 < (zeta - pw))
  delay(bigT - gt3 - tau2 - pwx);
/* 2nd part of N15 evolution */
  dec2rgpulse(2*pwx,zero,0.2e-6,0.2e-6);
  delay(zeta - bigT - tau2 - pwx - pw);
  rgpulse(2*pw,zero,0.2e-6,0.2e-6);
  delay(zeta - gt2 - pw - 200.0e-6);
  if (dps_flag)
    zgradpulse(gzvl2,gt2);
  else
    shapegrad(gzvl2,gt2);
  delay(200.0e-6);
}
else
  delay(zeta - gt3 - pw);
  dec2rgpulse(2*pwx,zero,0.2e-6,0.2e-6);
  delay(200.0e-6);
  dec2rgpulse(2*pw,zero,0.2e-6,0.2e-6);
  delay(200.0e-6);
  if (dps_flag)
    zgradpulse(gzvl2,gt2);
  else
    shapegrad(gzvl2,gt2);
  delay(200.0e-6);
}
/* now convert HMQC into anti-phase Hn magnetization */
```

Best regards,

Andy

R. Andrew Byrd
Structural analysis of carboxymethyl inulin by $^{13}$C and $^1$H NMR spectroscopy.

Dear Dr. Shapiro,

Within the framework of our research to convert inulin (a $\beta$(2➔1)-linked fructan with one glucose unit at the reducing end) into useful materials with industrial applications, we have been studying its carboxymethylation in aqueous medium. An important issue in the analysis of carboxymethyl inulin (Figure 1) is the distribution of substituents in the fructose units, i.e. the relative reactivity of the three available hydroxyl groups in the internal fructose units (C-3, C-4 and C-6).

In order to study the distribution of substituents, carboxymethyl inulin with a low degree of substitution ($ds = 0.70$) was prepared. The product was hydrolyzed into monosaccharides. As established with HPLC analysis, the mixture consisted of fructose, monosubstituted fructose (3-, 4- and 6-) and a smaller amount of disubstituted fructose (3,4-, 4,6- and 3,6-). Each monosaccharide consisted of an anomeric mixture in aqueous solution (C-pyranose, C-furanose and $\alpha$-furanose). Trisubstituted fructose was not present at this low degree of substitution. The glucose units were not further taken into account as their amount is less than 5%. Fructose was removed from the mixture by incubation with *Saccharomyces cerevisiae*. A fermentation product, glycerol, was formed.

Although the resulting mixture seems very complicated, the $^{13}$C NMR spectrum revealed one predominant monosubstituted product (> 50%), showing that one position of the fructose ring is much more reactive than the others during carboxymethylation.

The structure of this product was elucidated and the $^{13}$C and $^1$H NMR chemical shifts were completely assigned. The chemical shift of the C-2 anomeric carbon of this product (δ 99.4 ppm) established the C-pyranose form, which implies that C-6 is not substituted as this carbon is part of the pyranose ring. The $^{13}$C NMR chemical shift of C-1, C-6 and C-2 could simply be assigned by comparison with the unsubstituted $\beta$-D-fructopyranose (Table I). From a HETCOR experiment (Fig. 3), we were able to assign the protons on C-1 and C-6. The chemical shifts of the C-6 protons were used to assign the other protons of the pyranose ring using a COSY spectrum. The HETCOR spectrum revealed then the chemical shifts of the pyranose ring carbons.

The large upfield shift of H-4 and downfield shift of C-4, and the smaller opposite shifts of the neighbouring atoms (H-3, H-5, C-3, C-5) demonstrate that the substituent is on the C-4 position (Table I). Similar substituent effects were observed in O-carboxymethyl derivatives of glucose. As shown by the HETCOR spectrum, the $^1$H chemical shifts of the substituent (CH$_2$COONa) coincide at 4.05 ppm. Additional evidence for this assignment was obtained by an H-coupled $^{13}$C spectrum, which shows a triplet for the carbon at 69.8 ppm. This triplet collapses into a singlet on selective irradiation of the $^1$H resonance at 4.05 ppm. Conclusively, the main product formed by hydrolysis of carboxymethyl inulin is 4-O-carboxymethyl $\beta$-D-fructopyranose (Fig. 2), showing that the most reactive position during carboxymethylation is C-4. This high selectivity can be explained by the relatively higher acidity of secondary alcohols compared with primary alcohols and by steric effects occurring in the inulin chain.

When responding please quote our reference.
Figure 3.

Table I. $^{13}$C and $^1$H NMR chemical shifts of $\beta$-D-fructopyranose and 4-O-carboxymethyl $\beta$-D-fructopyranose in D$_2$O.

<table>
<thead>
<tr>
<th></th>
<th>C-1</th>
<th>C-2</th>
<th>C-3</th>
<th>C-4</th>
<th>C-5</th>
<th>C-6</th>
<th>CH$_2$COONa</th>
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<tbody>
<tr>
<td>$\beta$-D-fructopyranose$^4$</td>
<td>65.4</td>
<td>99.6</td>
<td>69.1</td>
<td>71.2</td>
<td>70.7</td>
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<tr>
<td>4-O-carboxymethyl-$\beta$-D-fructopyranose</td>
<td>65.4</td>
<td>99.4</td>
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<td>$\beta$-D-fructopyranose$^5$</td>
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<td>0</td>
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</table>

Yours Sincerely,

Dorine L. Verraest

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

Specially designed
Thin Wall NMR Sample Tube

Shigemi's high precision thin wall NMR sample tube has a unique construction. The wall thickness of this particular tube is reduced only around the position of the detection coil. The result of this new invention allows an increase in the sample volume and higher sensitivity without sacrificing its mechanical strength. Therefore, there is no need for special handling during routine usage of our Shigemi NMR tubes.

The spectra of 20mm sucrose in D₂O 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%.

---

### Price Each

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<td>20/8</td>
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SHIGEMII, INC.
Suite 21, 4790 Route 8 • Allison Park, PA 15101 • USA
Tel:(412)444-3011 • Fax:(412)444-3020
Shigemi has recently developed a unique alumina tube for $^{29}$Si and $^{11}$B NMR. The tube consists of a standard glass NMR tube connected to a highly densified alumina bottom which holds your sample. By using our alumina tube, the $^{29}$Si spectrum is free from a broad $^{29}$Si signal, and the spinning sidebands are suppressed to a minimum because of the tube's precision and quality. As of now, only Shigemi can offer you this very specialized and high quality tube for a reasonable price.

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<td>Si-010</td>
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Re: Methylene Induced Line Broadening in Solid-State NMR

Dear Barry:

A protocol for solid-state spectral editing of $^{13}$C CP/MAS NMR spectra has recently been introduced by Wu et al. (1) and is currently being utilized in our laboratory to investigate the structure of fossil resins. In particular, we are interested in the chemical transformations that are important in the maturation of polylabdanoid resinites.

While testing the editing procedure on a model labdanoid, i.e. sclareol (shown below), we observed severe line broadening in the aliphatic region of the CP/MAS spectrum, a phenomenon common to NMR spectroscopy of many non-crystalline organic solids. Upon completion of the spectral editing, it was evident to us that the spectral broadening was due largely to sclareol's methylene carbons.

Figure 1 contains the four "edited" spectra for our model labdanoid and the standard CP/MAS spectrum (bottom). Methyl- and quaternary-only sub-spectra (top and penultimate spectra, respectively) are obtained as a single spectrum by depolarization of CH$_2$ and CH spins. The "edited" sub-spectra are then achieved by subtraction. In the middle of the Figure is the CH-only sub-spectrum given by subtracting CH$_2$ and residual CH$_3$ and quaternary resonances from a short (40 µs) CP experiment. Second spectrum from the top is the methylene only sub-spectrum, which is obtained by inverting methylene polarization while nulling non-methylene resonances.

In contrast to the relatively sharp peaks of the methyl-, methine- and quaternary-only sub-spectra, the methylene resonances are extremely broad and unresolved. Having isolated and examined the spectrum given by each type of carbon, it became apparent to us that the overall broadening of the spectrum could be attributed exclusively to methylenes, and these are perhaps masking signals derived from non-methylene carbons. Broadening due to low frequency molecular motions is well documented in the literature (2) and may, in fact be a predominant source of broadening in many polymeric non-crystalline materials.


Sincerely,

David J. Clifford  
Ken B. Anderson  
Robert E. Botto

Chemistry Division  
Chemistry Division  
Chemistry Division

Operated by The University of Chicago for The United States Department of Energy
Figure 1. Four "edited" spectra corresponding to sclareol's methyl, methylene, methine and quaternary carbons. The standard CP/MAS spectrum is shown at the bottom.
Oxford Instruments’ commitment to quality ~ look for the signs...

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Donney Mead
Oxford OX2 0DX, England
Telephone +44 (0) 1865 269500 Fax +44 (0) 1865 269501
August 16, 1995

Dr. B. L. Shapiro
The NMR Newsletter
966 Bsinore Court
Palo Alto, CA 94303

Dear Barry:

Using $^{13}$C NMR line shape analysis, we have measured rates of rotation about the ring-$N^+$ bonds in A and B and around the ring-$C$(t-butyl) bond of C. At 200 K,

\[ \text{A. } Y^- = \text{I}^- \quad \text{B. } Y^- = \text{CF}_3\text{SO}_3^- \quad \text{C} \]

the $N^+$-methyls, as well as t-butyl methyls give rise to three unequally spaced lines, 1:1:1, which signal average at higher temperatures. In addition, the dimethylamino's give rise to 1:1 doublets and they signal average with increasing temperature. That is the result of inversion at nitrogen together with rotation around the ring-$N$(dimethyl) bond. We previously reported some pretty peculiar results. Now having repeated the work, the activation parameters are strange but in different ways, see Table. That the inversion/rotation $\Delta H^\ddagger$'s are similar might have been expected, even in different solvents. Further, sterically, t-butyl is like $N^+(\text{CH}_3)_3$ except for the charge. One can conclude that electrostatic effects don't influence $\Delta H^\ddagger$. It's the entropies that bother me, especially for C, which is neutral. Unimolecular processes in neutral molecules come with neutral (almost zero) $\Delta S^\ddagger$ values. The line shape analysis was done pretty carefully. The $^{13}$C lines are widely spaced, 550 Hz. The intrinsic shifts and line widths do not appear to vary with temperature. The data are nicely reproducible. Was gibt hier? Plus twenty eu is too much. I wouldn't expect inversion/rotation in A to have a $\Delta S^\ddagger$ of +17 eu, but A, at least is a salt. I can only imagine that the transition state for inversion/rotation in C is highly disordered, i.e. the energy surface looks like a flat plateau. We need advice.
The other rotation data looks OK.

Best wishes to you and the Newsletter.

Yours sincerely,

Gideon Fraenkel  
Professor of Chemistry  

Sharon Boyd  
Graduate Associate  

Albert S. Chow  
Research Associate

<table>
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<th>Compound</th>
<th>Solvent</th>
<th>anion</th>
<th>Inv/rot</th>
<th>(\Delta H^\ddagger) kcal/mole</th>
<th>(\Delta S^\ddagger) eu</th>
<th>Rot. ring-XMe3</th>
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<td>17</td>
<td>10.2</td>
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<tr>
<td>B</td>
<td>ac</td>
<td>I(^-)</td>
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<td>8.3</td>
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<td>CF(_3)SO(_3^-)</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
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</tr>
</tbody>
</table>

nm = CD\(_3\)NO\(_3\)  
ac = acetone-d\(_6\)

---

**Postdoctoral Position at Argonne**

I will have an opening for a NMR postdoctoral scientist in my laboratory in the Chemistry Division on October 1, 1995. The position will involve research in developing new solid-state magnetic resonance microscopy and spin-diffusion methods to probe domain structures within heterogeneous polymeric materials.

The successful candidate should have a recent Ph.D. (within three years) in Chemistry, or closely related field, with some experience in hardware and software development, and with experience in solid-state NMR and its application to structure elucidation.

The Chemistry Division has state-of-the-art NMR facilities, including a new fully equipped Bruker DMX 500/200 system with a full complement of solids, liquids and imaging capabilities, a home-built Tecmag 400 imaging/spectroscopy system, GE Omega 300, Bruker AM 300, and Bruker CXP 100 spectrometers, and several IBM R-6000 and Silicon Graphics workstations for advanced data processing, calculation and data refinement.

For further information contact:

Robert E. Botto  
Chemistry Division  
Argonne National Laboratory  
9700 S. Cass Ave  
Argonne, IL  60439  
Ph: (708)252-3524  FAX: (708)252-9288  
e-mail: robert_botto@qmgate.anl.gov
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- Obtain minimum frequency separation between the X and Y channels when needed.

13C/15N REDOR with 1H decoupling, obtained on [2-13C,15N]-glycine.

1H decoupling field, stable RF and stable spinning speed are all critical for REDOR experiments.

Control experiments (1st, 3rd, etc. peaks) demonstrate high decoupling powers. Significant signal remains after 64 rotor periods, as seen in the next to last peak.

27Al/15N TRAPDOR with 1H decoupling, of monomethyl amine (MMA) on a zeolite surface, obtained at -140°C to freeze amine motion on the zeolite surface.

Stability in probe tuning and spinning speed must be maintained at -140°C in order to obtain TRAPDOR data.

The TRAPDOR technique is similar to REDOR in that distance information is obtained through dipolar couplings.

data courtesy of C. Grey, SUNY, Stony Brook.
Chemagnetics Triple Resonance Technology

$^{15}$N Double-Cross Spectra of [2-$^{13}$C,$^{15}$N]-$^{1}$H decoupling. Cross polarization is performed in the direction $^{1}$H $\rightarrow ^{13}$C $\rightarrow ^{15}$N.

As the $^{13}$C decoupling field increases from left to right, the noise level remains the same. Signal-to-noise is best with a sufficient level of $^{13}$C decoupling.

Minima in peak intensities correspond to $^{13}$C decoupling fields equal to and at twice the spinning frequency.

$^{27}$Al Observation with X/H decoupling, of AlPO$_4$-H$_2$.

Decoupling of the $^{31}$P and $^{1}$H nuclei provides enhanced resolution in the $^{27}$Al spectrum.

The signal-to-noise does not degrade with addition of $^{1}$H and $^{31}$P decoupling. This is the result of good X/Y channel isolation in the probe and filtering between the probe and receiver.

$^{13}$C CP and Double-Cross Spectra of d$_8$-PS/PMMA copolymer.

$^{1}$H $\rightarrow ^{13}$C CP shows peaks from both PS and PMMA components of the copolymer, indicating intimate mixing of the two materials. The $^{2}$H $\rightarrow ^{1}$H $\rightarrow ^{13}$C double-cross spectrum demonstrates $^{2}$H polarization transfer to $^{13}$C via $^{1}$H's. The Control Experiment, with $^{2}$H CP power off, shows that all double-cross signal originated from $^{2}$H.

*spinning sidebands. Sample/idea courtesy of N. Zumbulyadis, Eastman Kodak.

2607 Midpoint Drive · Fort Collins, CO 80525 · (970) 484-0428 · FAX (970) 484-0487 · 1-800-4-OTSUKA
gs-SELTRIP

Dear Barry,

Instead of a 3D $^1$H,$^13$C,$^{31}$P correlation [1] one can solve the assignment problem of $^{31}$P nuclei with a selective 2D experiment, which we dubbed earlier SELTRIP [2]. This "burned out" all the $^1$H,$^13$C correlation signals stemming from the selected phosphorus atom. However, we have now developed an improved sequence, gs-SELTRIP, which gives a positive answer: The spectra show all $^1$H,$^13$C-correlation signals from carbons correlated to the selected $^{31}$P-nucleus. The pulse sequence (Fig. 1) contains a selective 180° pulse, a DQ-$^{31}$P,$^{13}$C-filter and uses the echo/anti-echo method in the gradient version to get phase sensitive spectra. The experiment also works without pulsed field gradients. In that case it is necessary to cycle the first 90° $^{13}$C pulse in the TPPI manner.

To demonstrate the efficiency of gs-SELTRIP we choose a sample of 80 mg NAD+ (Fig. 2) in D$_2$O. In the $^{31}$P-NMR spectrum of NAD+ the phosphorus signals are separated by only 40 Hz. Nevertheless the gs-SELTRIP spectra (Fig. 4 and 5) show only the filtered correlation signals.

\[ y \quad 1/(2J_{ep}) \quad DQF \quad 1/(2J_{ep}) \quad 180 \quad y \quad \Phi_3 \]

\[ \Phi_1 : X, -X \]
\[ \Phi_2 : y, y, -y, -y \]
\[ \Phi_3 : X, -X, -X, X \]

Fig. 2  NAD$^+$

Fig. 3  $^{31}$P NMR of NAD$^+$

Fig. 4  gs-SELTREP of NAD$^+$, sel. pulse on A

Fig. 5  gs-SELTREP of NAD$^+$, sel. pulse on N

With best regards,

Ronald Wagner

Stefan Berger
July 25, 1995 (received 8/7/95)

Dr. Barry Shapiro
NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

Dear Dr. Shapiro

Vibration Problems: Go to the Source
Improving Baselines in Presaturation Solvent Suppressed Spectra

For several years our VXR 4000 had been suffering from a vibration problem. There were 3% sidebands at 22.7 Hz, which did not have a constant phase. They tended to average away after multiple transients. We could even see ripples in a beaker of mercury placed under the magnet. Estimated costs for an anti-vibration platform ran from $14,000 to $24,000, plus the downtime of the instrument. So rather than molest the magnet, we decided to locate the vibration source. Our building engineer switched-off various machines, while I watched the sidebands on an HDO signal. Sure enough, we found that an air conditioner three floors above, on the other side of the building was the problem. The solution was to install a set of springs under the compressor. Needless to say, the cost of this was far less than $14,000.

Presaturating the water signal with the decoupler before acquiring $^1$H spectra is an easy and effective way to reduce its intensity and increase the signal to noise ratios of the peaks of interest. However, if the signals of interest are of very low intensity, in our case dissolved starch, the residual water signal presents a slight problem. Residual $xy$ coherence at the water frequency in phase with the rf read pulse will result in a residual signal randomly out of phase with the rest of the spectrum. This can greatly distort the baseline in the region of the water signal and can make integration more difficult. This will also detrimentally affect 2D spectra. This problem can be greatly alleviated and the appearance of the $^1$H spectrum can be greatly improved. Waiting about 16 msec after the decoupler is gated off before applying the rf pulse allows time for the $xy$ coherence to decay. This does not reduce the suppression if the T-1 of the water is long enough and results in a much flatter baseline. The residual water signal can be suppressed even more by applying an 8 msec homospoil pulse at the beginning of the 16 msec delay. However, if the spectrum contains sharp signals, eddy currents from the homospoil may result in distorted peaks. To see the results, notice the water signal and baseline of Figure 1, in which the rf pulse was applied immediately after presaturation. Then compare this spectrum to Figure 2 where the 16 msec delay and homospoil were applied.

Sincerely

[Signature]

Gary Juneau
Figure 1  Standard Presaturation

Figure 2  Presaturation with 16 msec Delay and Homospoil
AMT’s NEW Solid State RF Power Amplifiers for High-Field NMRI imaging systems

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ELECTRICAL/MECHANICAL SPECIFICATIONS AT 25°C:

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<td>11 dB typ., with 0 dB attenuation at the RF input</td>
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SUPPLEMENTAL CHARACTERISTICS:

- Connectors, rear panel
  - RF input: BNC (F)
  - RF output: 7/16 EIA (F)
  - Noise blanking: BNC (F)
  - Interface: 25 pin DIF, EMI filtered
- Water: Brass Swagelok™ fitting P/N B-810-6
  Requires mating Swagelok/adapter P/N B-PB8-TA8 for a #8 hose (500°, 12 mm OD)

Indicators, front panel
- System "ON"
- Overdrive
- Peak power meter
- CW mode
- Over pulse width
- Over duty cycle
- Over temperature

System monitors
- Forward/Reflected RF power
- Over pulse width/duty cycle
- DC power supply undervoltage
- Over thermal

Front panel controls
- AC power
- Pulse width
- Duty cycle
- Forward/Reflected power

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  - 3495/3446
  - 10-130 MHz, 2000/1000 W

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3080 Enterprise Street • Brea, California 92621 • Phone: (714) 993-0802 • Fax (714) 993-1619
Solid-State NMR Pulse Sequence That Scales-Up Chemical Shift Frequencies

Dear Barry,

It is a tantalizing to contemplate the prospect of increasing (or scaling up) the frequency separation between chemically shifted resonances by applying a pulse-sequence rather than employing a higher magnetic field. The chemical shift concertina was originally invented to scale down chemical shifts in solid-state NMR (1), and a number of variations of scale-down pulse sequences have been described since then. Scale-up has been demonstrated in multi-dimensional solution NMR experiments (2, 3). In this letter we demonstrate a pulse sequence that scales-up chemical shift frequencies in solid-state NMR spectra.

A two-dimensional pulse sequence that correlates scaled-up and unscaled chemical shift resonance frequencies is diagrammed in Figure 1. This pulse sequence is equally applicable to $^{13}$C, $^{15}$N, or other dilute spin nuclei. After cross-polarization from the abundant $^1$H spins, the $^{15}$N magnetization evolves according to its chemical shift during $t_1$. This is followed by a constant time period, $\Delta$, during which a $\pi$ pulse is used to control the evolution of the $^{15}$N magnetization under the chemical shift and the factor $\alpha$ determines the second incremental time for each $t_1$ experiment. As a result, the $^{15}$N magnetization evolves for a time $(1 + \alpha)t_1$ with a frequency $(1 + \alpha)\omega_N$ which leads to the $^{15}$N chemical shift frequency scaled by a factor of $(1 + \alpha)$. The factor $\alpha$ has to be carefully selected to accommodate the scaled chemical shift interaction in the spectral width set by the $t_1$ increment while avoiding loss of magnetization due to $T_2$ relaxation. The phase of the $\pi/2$ pulse preceding data acquisition is selected for quadrature detection in the $\omega_1$ dimension.

Conventional and scaled-up two-dimensional correlation spectra are shown in Figures 2A and B, respectively. These spectra result from 64 $t_1$ experiments with 8 acquisitions, a recycle time of 3.5 sec and a cross-polarization mix time of 1 msec. The spectrum in Figure 2A was obtained with a dwell time of 25 µsec while that in Figure 2B had a dwell time of 12.5 µsec. For spectrum B, $\alpha$ was set to a value of 1.0 to achieve a scaling factor of two in the first dimension. Therefore, the scale along the $\omega_1$ dimension is twice that along the $\omega_2$ dimension in Figure 2B. Significantly there is a net decrease in linewidth after taking scaling into account with this procedure because of the constant time interval in the pulse sequence. Therefore, there is a real increase in resolution. The experiments were performed on a home-built spectrometer with $^1$H and $^{15}$N resonance frequencies of 550.09 MHz and 55.74 MHz, respectively. The sample was a single crystal of N-acetyl-L-$^{15}$N-Valine-L-$^{15}$N-Leucine placed at an arbitrary orientation with respect to the external magnetic field in a solenoidal coil double tuned to the $^1$H and $^{15}$N resonance frequencies.
Figure 1. Two-dimensional solid-state NMR pulse sequence that correlates the scaled and unscaled chemical shifts.

Figure 2. A. Two-dimensional correlation spectrum of $^{15}$N chemical shifts without scaling. B. Two-dimensional correlation spectrum of $^{15}$N chemical shifts with scaling in $\omega_1$ using the pulse sequence in Figure 1.


Sincerely,

A. Ramamoorthy

M. McCoy

S. J. Opella
PGSE-NMR has developed into a very powerful method for studying the dynamics of complex multi-component systems. Perhaps the most significant advantages the NMR technique has over other commonly used techniques, such as viscosity or neutron scattering, are that no special sample preparation is required and that the dynamics of each individual component can be simultaneously measured. This is particularly important in fields such as polymer-surfactant interactions. However, the biggest limiting factor in the applicability of this NMR technique has been the necessity for non-overlapping spectral lines. Hence, the systems most studied have been simple molecules with few lines in the spectra. C.S. Johnson, Jr. and coworkers have suggested the so-called DOSY-method, which is a way to achieve a separation of diffusion components of a FT-PGSE dataset.

We recently have developed a quite different mode of analysis of PGSE-NMR data (CORE-NMR, Component-Resolved NMR) which effectively not only increases the range of systems to which FT-PGSE can be applied but also the accuracy of the technique itself. We illustrate our point with an testing example - the determination of the diffusion coefficients of a solution containing a protein - gelatin - and a non-ionic surfactant based on a sugar. The NMR spectra of these two components show considerable overlap.

The Experiment is shown in Figure 1. The surfactant lines are much sharper than the protein lines. As may be clearly seen, the main signal around 1.5ppm decays rapidly at first and subsequently adopts a much slower decay. A typical analysis of this system would be to fit the data contained in this peak to a double exponential and thus, extract two diffusion coefficients. It should be remembered that all peaks pertaining to the protein should attenuate at the same rate within their own noise (P.Stilbs, Anal.Chem. 53 (1981) 2135). Similarly, for the surfactant. The question is thus, "can one use all this spectral information?" The answer is "yes". The key to this analysis is as mentioned above - all the resonances pertaining to one component decay with the same constant. Further details of the computational procedures are described in a manuscript that was...
submitted to Journal of Magnetic Resonance in late June. Also, instead of using only one data channel but using all the channels above a threshold, the data quality may be significantly improved simply by the fact that many more decays must give the same result, so there is a very significant gain in effective Signal/Noise - like a factor of 10, using the same dataset!

Having obtained these more accurate diffusion coefficients, the analysis program then goes through a routine which determines how much signal is present in each decay given those diffusion coefficients. In other words, the analysis separates the data not only into components based on their diffusion coefficient but also returns the fitted spin-echo spectra for comparison with the real thing. This analysis has been tested on a number of systems ranging from proteins and block copolymers to simple alcohols and the improvement in accuracy is very noticeable. More importantly, the analysis has "paved the way" for the study of a number of complex-component systems.

Yours Sincerely

Peter Stilbs  Kim Paulsen  Pete Griffiths

Figure 1.
Now Offers Spinner Turbines!

Starting in March, 1995, Wilmad can provide the most common 5mm and 10mm Spinner Turbines for Varian and Bruker NMR Spectrometers. Manufactured by RotoTec in Germany, these Spinner Turbines are prepared to precision standards that exceed those provided by the Spectrometer maker. Save time by ordering your Spinner Turbines along with the most valued NMR Sample Tubes in the world... From Wilmad!

Varian Spinner Turbines.....Made from high density GFK (fiberglass), these Turbines incorporate a single O-Ring imbedded at the top of the turbine that holds the NMR sample tube firmly.

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Bruker Spinner Turbine......Equivalent to Bruker's 'Selected' quality of NMR Spinner Turbine for 500 and 600MHz NMR Spectrometers, you'll find the price is attractive for even 200-400MHz spectrometers. Prepared from high quality ABS (polyamide), the design incorporates two O-Rings for ease-of-use and snug fit on the NMR tube.

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ASK FOR CUSTOMER SERVICE
The $^{13}$C Shielding Tensor of $^{13}$CO Bonded to Haemoglobin

Dear Barry,

As part of our ongoing studies on the local geometry of the Fe-C-O unit in heme proteins and model compounds$^{1,2}$ we have recently measured$^{3}$ the $^{13}$C shielding tensor of rabbit Hb$^{13}$CO (molecular weight ca. 64,500). Such a measurement poses a significant challenge to the solid-state NMR spectroscopist because in the normal $^{13}$C CP/MAS NMR spectrum there is considerable natural abundance $^{13}$C signal intensity from the protein which masks spinning side-bands from the $^{13}$CO resonance - see Fig. 1(a). The protein signal is reduced to some extent by application of the non-quaternary suppression technique (NQS) - Fig. 1(b). In Fig. 1(c) we show the $^{13}$C CP/MAS spectrum of Hb$^{13}$CO acquired with a combination of NQS and the SELDOM (selectivity by destruction of magnetisation) sequence$^{4}$ (Fig. 2) which works by storing the selected magnetisation along the $z$-axis and allowing unwanted magnetisation to decay due to short $T_2$ values normally found in solids. Excellent selectivity (although at a cost in sensitivity) was achieved. The spinning side-bands from the $^{13}$CO site can readily be identified and there is little interference from the rest of the protein. These results show great promise for the measurement of $^{13}$C shielding tensors in other complex heme biological systems.


Yours sincerely,

G.E. Hawkes

I.P. Gerothanassis

QMW is a college of the federal University of London, incorporated by Royal Charter, engaged in undergraduate and postgraduate teaching and research in Arts, Medicine, Engineering, Informatics and Mathematical Sciences, Laws, Natural Sciences and Social Sciences. Patron: Her Majesty The Queen.
Figure 1. $^{13}$C CP/MAS NMR spectra of solid Hb$^{13}$CO recorded at 75.5 MHz on a Bruker MSL-300 spectrometer (1.5 ms contact time, 0.4 s recycle delay, 3240 Hz spinning speed, processed with 100 Hz line-broadening):
(a) normal CP/MAS spectrum, 10,000 scans;
(b) CP/MAS with NQS, 40 µs interrupted decoupling, 10,000 scans;
(c) CP/MAS with NQS and SELDOM, 12 SELDOM loops with 250 µs dephasing time, 40 µs interrupted decoupling, 126,000 scans.

Figure 2. Pulse sequence for the combined NQS and SELDOM methods.
Fri, Jul 28, 1995
(received 8/8/95)

Dr. B.L. Shapiro,
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA
94303

Dear Dr. Shapiro,

New Paradigm for NMR Demonstrations

We have recently been through the exercise of evaluating a Bruker DPX 300 vs a Varian Inova 300 for use with both solutions and solids. As part of the evaluation we had their service managers in with their schematics to evaluate the circuitry with our electronics people and we had both companies demonstrate their software on campus so that we could get feedback from faculty who would not be going to factory demos and more importantly from students who would use the systems "hands on". The vendors chose to do this by bringing down a workstation with the software preloaded. However data acquisition and the instrument interface could not be demonstrated since the software was aware that no console was attached. The effort in writing a "virtual console" for such demonstrations, as several mass spectrometry vendors have done, could better be spent on implementing new features of use to existing and potential spectrometer owners/purchasers.

However, the software was accessible internally at both companies from other workstations or Macs or PCs running X-server software. Such access was part of our own requirements for access from all parts of the campus, office, lab and classroom. We were typically being shown at a second workstation the working up of T1 data etc while the machine console computer was being used to acquire more data or demo something else to another one of the three of us who were present at the demo site. Full instrument control was available from these remote workstations. We quickly realized that there is no reason not to have done this for the demo at home, especially given our reasonably fast internet connection.

Since, among other things, to increase instrument throughput, we were almost certainly going to buy a small sample changer for solutions (6 samples for Bruker's, sequential and 9 for Varian, random access), we realized as we tested the changers their further potential for remote demonstrations. On the day of our "at home" software demo, the sample changers (presumably the vendor's larger random access unit attached to a demo machine) could have been preloaded with tuning standards as well as with the samples previously sent down to the demo lab from four or five people who would not be attending the Boston or Palo Alto demos, although known to be major users of the new system. Thus some of the samples that we brought down with us could have been demonstrated at home to the individuals concerned - data acquisition, processing plotting etc, done from any Unix box, or X-server equipped Mac or PC here at Brock via the internet. The individual whose sample is being run gets the chance for instant feedback to the vendor's application chemist who is running the demo from the customer's site rather than back at the actual instrument, and he/she knows immediately if the system being evaluated can do what he/she expected.
Given time zone differences such use could significantly increase the throughput on demo lab instruments and be used in demos for countries around the world, where there may be no nearby demo site. Thus many more people can see much of the capabilities of the instrument that is being proposed. Thus the lucky (unlucky if they make the wrong choice) person(s) who get the trip to the demo site would also get more direct feedback from the users.

Junior faculty can often find themselves in the position as "expert" in a particular form of spectroscopy or instrumentation and therefore have to make a choice. If they make a poor choice, often because they choose what they used as a graduate student or as a postdoc (which may be obsolete now), or may have been a poor choice on the part of their original supervisor, and they thus risk their future tenure. It is for this reason that one of us (JMM) teaches an advanced instrumentation course facetiously subtitled "A Course in Applied Sales Resistance".

Since the vendors often hook in via the net to a customer's lab for diagnosis, why not carry this one stage further and use the same type of hookup for demonstrations. Then those of us present at the demo in the factory don't have to second guess what our colleagues want. In a situation where many have to be served, that is a consideration in the choice of system. Since those who are present at the actual demo site are often computer and hardware experts, they can spend more time inside the system hardware and software rather than just rushing samples through the instrument as spectators making periodic comments or suggestions.

For the local customer's site demo, once the three to six hour period of access to the spectrometer and sample changer back at the demo site is over, the customer could be given remote log-in privileges at another workstation at the vendor's site. The experimental data would have been transferred to this machine, and the potential customers could then allowed to play with their raw data for a fixed period of time, e.g. a week. That would give them a chance to get a feel for the variations in data workup and plotting available, and a real feel for the power and user friendliness or unfriendliness of the software. There would be no requirement that the customer had the same brand of Unix workstation that was needed to operate the spectrometer since any X-server would work for X-compliant software.

This proposed model would certainly work at most university sites, and at many industries since they are also getting fast internet connections for their Web servers. In the absence of a hard internet connection locally, a very fast modem might well be adequate. This would have to be determined by the vendors in experimentation on different sites around the world.

Yours sincerely,

Jack M. Miller
Prof. of Chemistry

J. Stephen Hartman
Prof. of Chemistry

Tim Jones
Coordinator of Analytical Services
Address all Newsletter correspondence to:

Dr. B. L. Shapiro
The NMR Newsletter
956 Elsinore Court
Palo Alto, CA 94303.

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

Deadline Dates

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Fax: (415) 493-1348, at any hour. Do not use fax for technical contributions to the Newsletter, for the received fax quality is very inadequate.

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Mailing Label Adornment: Is Your Dot Red?

If the mailing label on your envelope is adorned with a large red dot: this decoration means that you will not be mailed any more issues until a technical contribution has been received.
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.