

**THE**  
**NMR**  
**NEWSLETTER**

**No. 466**  
**July 1997**

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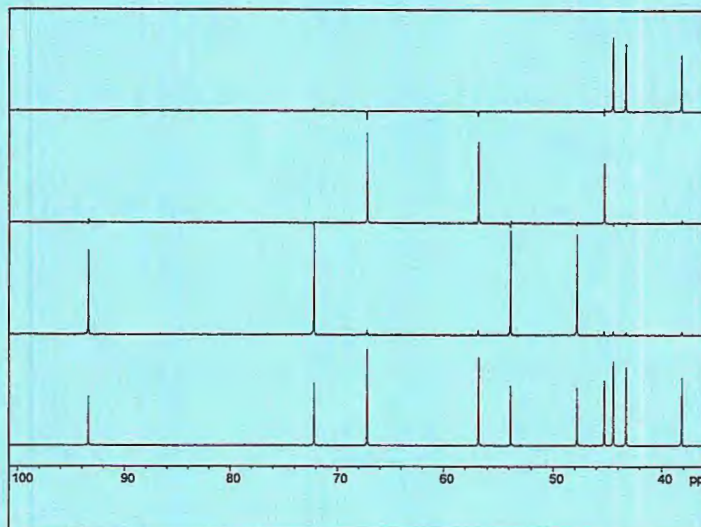
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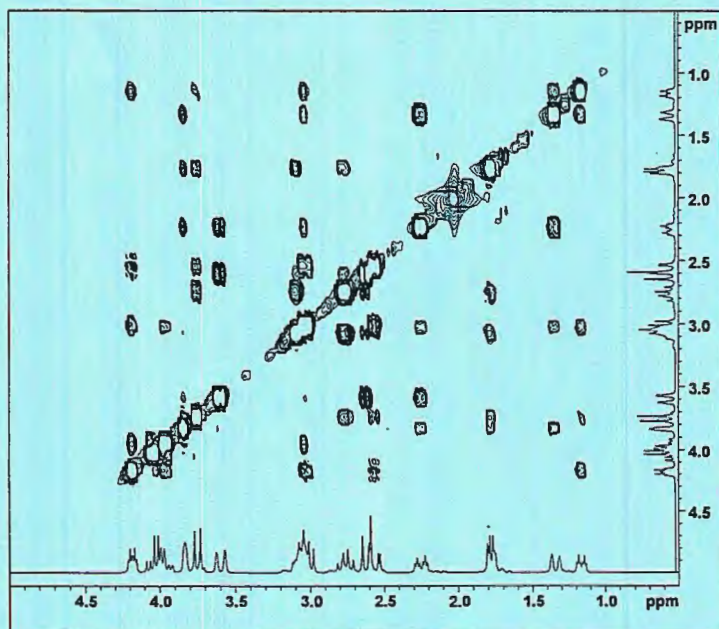
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#### FORTHCOMING NMR MEETINGS

39th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado; NMR Symposium, **August 4-7, 1997**: Contact: J. P. Yesinowski, Code 6120, Naval Research Laboratory, Washington, DC 20375-5342; 202-767-0415; fax 202-767-0594; email yesinowski@nrl.navy.mil. See Newsletter 458, 8.

8th Annual Chemmagnetics Workshop on Solid State NMR, Estes Park and Ft. Collins, CO, **August 8-9, 1997**; Contact: J. Frye, Otsuka Electronics USA, Inc., 2607 Midpoint Dr., Ft. Collins, CO 80525; (800) 468-7852; Fax. (970) 484-0487; email jimf@chemmagnetics.com; See Newsletter 465, 36.

Fourth International Meeting on Recent Advances in Magnetic Resonance Applications to Porous Media, Trondheim, Norway, **Aug. 31 - Sep. 3, 1997**; Contact: John J. Attard, SINTEF Unimed MR-Center, N-7034 Trondheim, Norway. Tel: +47 73 59 89 25; Fax: +47 73 99 77 08; Email:john.attard@unimed.sintef.no.

4th International Conference on Magnetic Resonance Microscopy "Heidelberg Conference in Albuquerque", **Sept. 21-25, 1997**: Contact: E. Fukushima, The Lovelace Institutes, 2425 Ridgcrest Drive SE, Albuquerque, NM 87108-5127; (505) 262-7155; Fax: (505) 262-7043. See Newsletter 449, 37.

Missouri Magnetic Resonance Symposium (MMRS-VIII), Tan-Tar-A Lodge, Lake of the Ozarks, Osage Beach, MO, **October 31, 1997**. Contact: Frank D. Blum, Department of Chemistry, University of Missouri-Rolla, Rolla, MO 65409-0010; 573-341-4451, fblum@umr.edu, <http://www.chem.umn.edu/midwest32.html>

39th ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, CA, **March 22 - 27, 1998**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073. See Newsletter 460, 41.

Sixth Scientific Meeting and Exhibition, International Society for Magnetic Resonance in Medicine, Sydney, Australia, **April 18 - 24, 1998**. Contact: International Society for Magnetic Resonance in Medicine, 2118 Milvia St., Suite 201, Berkeley, CA 94704; 510-841-1899.

Additional listings of meetings, etc., are invited.



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Dr. B.L. SHAPIRO  
The NMR News Letter  
966 Elsinore Court  
Palo Alto, CA 94303

5.6.1997  
(received 6/13/97)

### Beware of Manganese !

Dear Dr. Shapiro,

Recently we measured  $T_1$  and  $T_2$  relaxation times of  $^{17}\text{O}$  in a rather concentrated  $\text{Zn}(\text{ClO}_4)_2$  aqueous solution at 14.1 T of. To our big surprise,  $T_2$  was much shorter than  $T_1$  and the ratio of both varied with temperature showing a maximum at about 40° C. After some speculation about strange exchange effects, we looked for impurities in our solution. We quickly came to

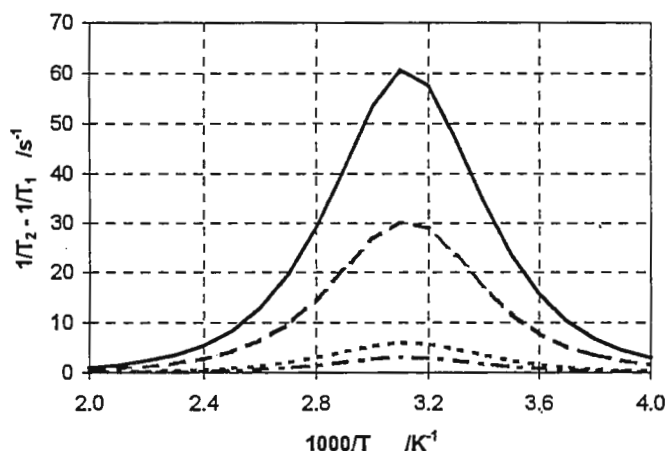


Figure 1 Excess  $1/T_2$  - relaxation due to different concentrations of  $\text{Mn}^{2+}$ :  $2 \times 10^{-5}$  m (—);  $1 \times 10^{-5}$  m; (— —);  $2 \times 10^{-6}$  m (- - -);  $1 \times 10^{-6}$  m (- · - ·);

the conclusion that the impurity could only be paramagnetic  $\text{Mn}^{2+}$ . We calculated the  $T_2$  effect of  $\text{Mn}^{2+}$  using data from experiments at 1.41 T<sup>1</sup> and got perfect agreement with  $1.1 \times 10^{-4}$  moles of  $\text{Mn}^{2+}$  in 1 mole of  $\text{Zn}^{2+}$ . The  $\text{Mn}^{2+}$  impurity was confirmed by ICP-AAS measurements. To estimate the maximum tolerable amount of  $\text{Mn}^{2+}$ , we performed a series of calculations at different temperatures (Fig. 1). The maximum excess in  $1/T_2$ -relaxation for a  $1 \times 10^{-6}$  m  $\text{Mn}^{2+}$  solution is ~3.5 % (at 50°C).

To be able to measure  $^{17}\text{O}$   $T_2$  relaxation times without contribution of  $\text{Mn}^{2+}$ , we must be sure that its content is less than  $1 \times 10^{-6}$  m. For a 1

m solution of  $\text{Zn}^{2+}$  this means that there should be less than 0.7 ppm (weight) of  $\text{Mn}^{2+}$  in the  $\text{ZnO}$  purchased. Similar considerations should be made for all  $^{17}\text{O}$  and  $^1\text{H}$  relaxation measurements on aqueous solutions of concentrated diamagnetic salts.

With best regards,

Hervé Bourgeois

Peter Caravan

Lothar Helm

Please credit this contribution to the subscription of Prof. A.E. Merbach, University of Lausanne

<sup>1</sup> Y. Ducommun, K.E. Newman, A.E. Merbach, *Inorg. Chem.* **19**, 3696 (1980).



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## School of Arts and Sciences

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Philadelphia, PA 19104-6323

May 30, 1997

(received 6/6/97)

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

Deuterium Decoupling Using the Lock Channel of a Bruker DMX Spectrometer and a Triple-Resonance Probe


Dear Barry,

The correlation time problem in NMR spectroscopy of proteins in solution presents itself most directly as broad resonance linewidths and efficient spin-diffusion. Both of these features are deleterious to multidimensional experiments and structure determination, the usual goal of the experiments, and can be unmanageable in large globular proteins and small membrane proteins in micelles in aqueous solution. After the overall correlation time of the protein is reduced as much as possible by eliminating aggregation, raising the temperature, and optimizing solution or micelle properties, isotopic labeling provides the only recourse for further improvement.

Uniform deuteration has been shown to be highly effective in combination with uniform  $^{15}\text{N}$  and  $^{15}\text{N}/^{13}\text{C}$  labeling in reducing the correlation time problem in proteins. In order to take full advantage of the effects of deuteration, it is essential to decouple  $^2\text{H} - ^{13}\text{C}$  interactions with rf irradiation at the  $^2\text{H}$  resonance frequency. We have designed and constructed a simple external circuit for a Bruker DMX spectrometer with a triple-resonance probe that enables the conventional  $^2\text{H}$  lock channel to be used for this purpose. The circuit is shown in Figure 1. The radiofrequency was generated in a PTS D620 synthesizer that was switched from the local mode when  $^2\text{H}$  decoupling irradiation was desired. After passing through the filters and the attenuator, the rf irradiation went to a high power divider and the probe. The lock signal was passed through the other port of the divider to a mixer controlled by logic from the console. The lock receiver was blanked during the  $^2\text{H}$  decoupling to avoid damage.

Figure 2 shows the pulse sequence timing diagram employed in a constant time HNCA experiment. During the  $\text{C}_\alpha$  evolution period WURST-20 modulation was applied with an effective field strength of 1 kHz. The comparison of  $\text{C}_\alpha$  free induction decays in Figure 3 demonstrates the beneficial effects of uniform  $^2\text{H}$  labeling and decoupling on a slowly reorienting protein. The sample is a 122 residue hydrophobic membrane protein in detergent micelles in solution. Correlations useful for backbone resonance assignments are available only with the use of uniform deuteration and  $^2\text{H}$  decoupling. Using only  $^{15}\text{N}$  and  $^{13}\text{C}$  labeled protein, the experiment results in essentially no useful correlations.

Sincerely,

  
M. McCoy      K. Valentine      G. Veglia      T. Le      D. Brabazon      S. Opella

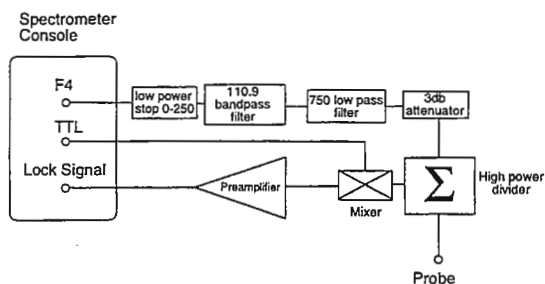


Figure 1: External circuit that enables the lock channel to be used for  $^2\text{H}$  decoupling on Bruker DMX spectrometer with a triple-resonance probe.

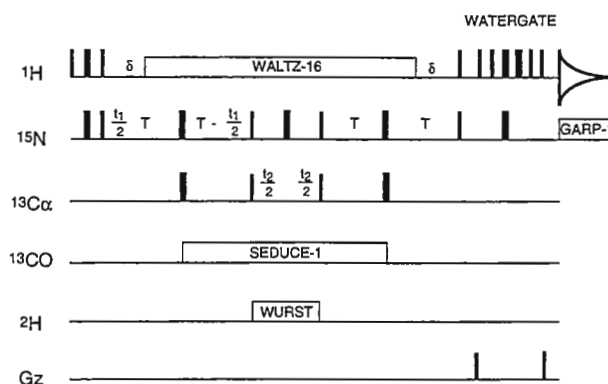


Figure 2: Timing diagram for the constant time HNCA experiment with synchronous WURST-20  $^2\text{H}$  irradiation used to decouple deuterium from carbon during the  $^{13}\text{C}_\alpha$  evolution period.

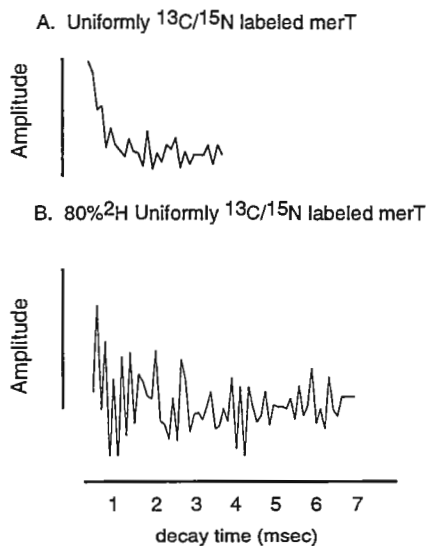


Figure 3:  $^{13}\text{C}_\alpha$  free induction decays obtained using the pulse sequence shown in Figure 2. A. Uniformly  $^{15}\text{N}/^{13}\text{C}$  labeled merT protein in SDS micelles. B. Uniformly  $^2\text{H}/^{15}\text{N}/^{13}\text{C}$  labeled merT protein in SDS micelles with deuterium decoupling. The protein has 122 residues as expressed. Approximately 80% of the hydrogen bonded to carbon was replaced by deuterium in the sample.



Department of Physics

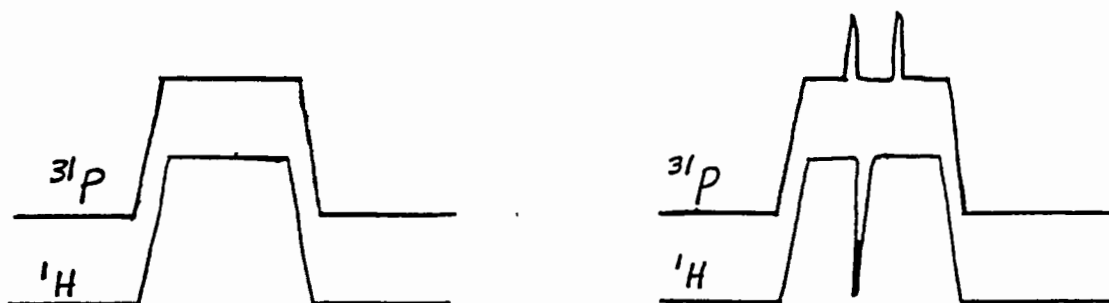
28 May 1997  
(received 6/2/97)

High Resolution NMR in Crummy Fields

Dear Dr. Shapiro:

We have used the intermolecular NOE between a solute and the solvent to yield high-resolution, traditional-appearing spectra of the solute, even in inhomogeneous fields.

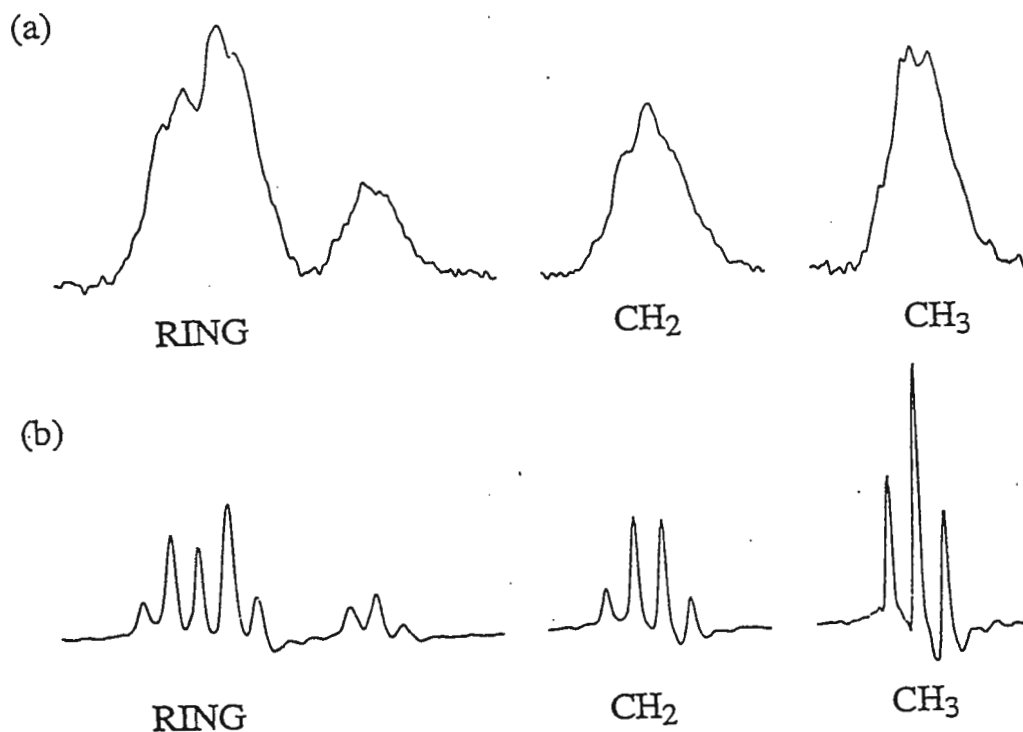
The one-dimensional version of this experiment is easiest to understand. Consider a solute with two distinct  $^{31}\text{P}$  resonances with  $\text{H}_2\text{O}$  as the solvent. We assume (though it is not necessary) that the broadening due to field inhomogeneity greatly exceeds the relative shift of the two  $^{31}\text{P}$  lines. Thus, the widths and shapes of the proton and  $^{31}\text{P}$  resonances are nearly the same (in gauss or ppm units); see left-hand sketch.



A narrow hole is now burned into the proton resonance, as in the right-hand sketch above, saturating the spins only in those regions with a specific value of field  $H$  (*i.e.*, on resonance with narrow-band irradiation). The solute-solvent NOE then causes 2 anti-holes to appear in the  $^{31}\text{P}$  spectrum, because the NOE-enhanced magnetization occurs only in those spatial regions where the solvent protons are saturated. The desired spectrum is the difference of the two  $^{31}\text{P}$  spectra—just the anti-holes. Thus one performs a difference experiment, with and without the proton hole-burning irradiation.

The experiment just described has worked with solutions of  $\text{H}_3\text{PO}_4$  plus ATP in water at 2.0 Tesla. Line-narrowing of a factor of 20 has been obtained. Because one selects (by hole-burning) a small fraction of the available spins, the  $S/N$  is poor.

A 2-D version of the experiment is hetero-NOESY. We used  $C_6F_6$  solvent with  $^{19}F$  in the first (incremented) dimension and ethylbenzene protons in the second (real-time) dimension. Inhomogeneous broadening makes the cross-peaks appear as long streaks along the diagonal, as has long been known. By projecting the 2-D spectrum parallel to this diagonal, one obtains a traditional-appearing spectrum of the solvent without inhomogeneous broadening. The spectrum of Figure a shows the normal spectrum (with intentionally poor magnet shimming) while Figure b is the result of the 2-D hetero-NOESY with diagonal projection, with the same inhomogeneous field. Clearly the resolution is much better. The  $S/N$  of the 2-D experiment depends mainly on the size of the solvent-solute NOE, which may be quite small. Also, all lines in the solute spectra may not be represented equally, if the NOE magnitudes are unequal.



Both experiments 1-D and 2-D, use the single solvent resonance line as a local gaussmeter, to reference the local solute spectrum to the local solvent resonance frequency.

We believe this experiment may have applications in *in vivo* NMR, where the sample/patient distorts the otherwise uniform field.

Sincerely,

Mark S. Conradi  
David P. Cistola

John J. Balbach  
Changguo Tang

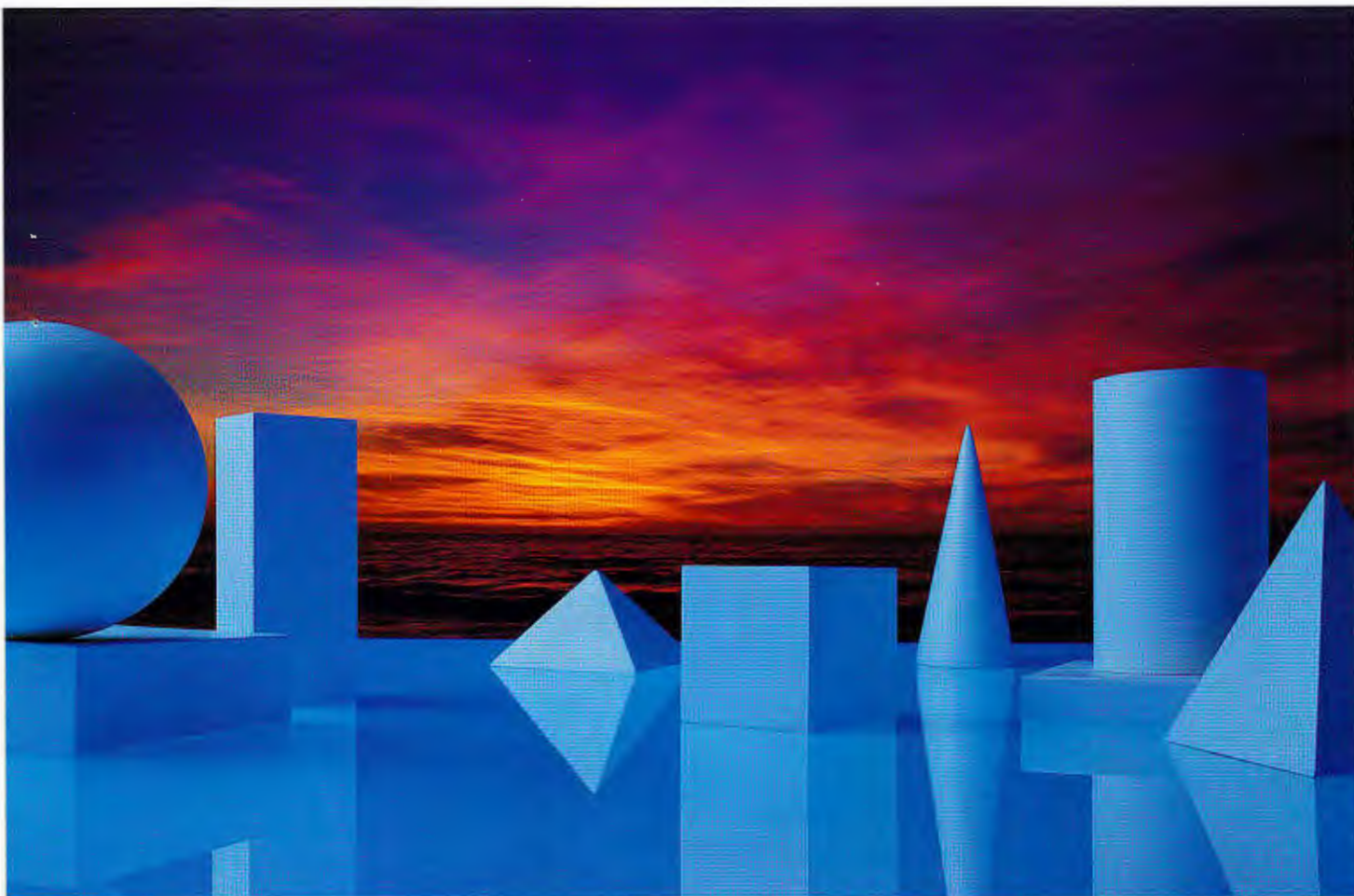
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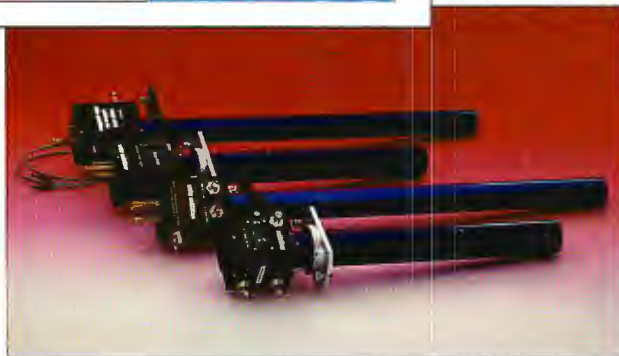
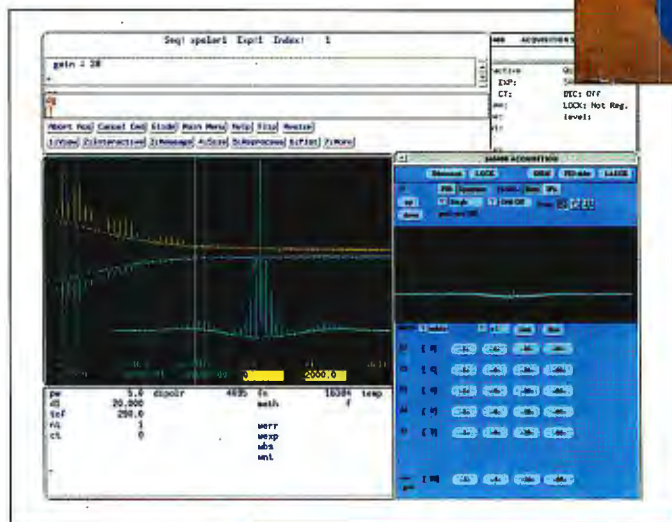
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Dr Barry Shapiro  
The NMR Newsletter  
966 Elsinore Court  
Palo Alto, CA 94303

June 24, 1997  
(received 6/26/97)

### Recent Results from Varian INOVA 800

Dear Barry,

We thought that your readers would be interested in some recent results obtained on the INOVA 800. The mid-bore (63mm) has really given our probe engineers some space in which to work, permitting the use of higher voltage capacitors necessary to produce the stronger RF fields necessary. We are pleased to note that this has resulted in  $^{13}\text{C}$  90 degree pulses times of 10.5 usec using a  $1\text{H}\{-^{13}\text{C},^{15}\text{N}\}$  triple-resonance pulsed-field gradient probe (5mm). The proton 90 degree pulse width measured 4.8 usec (at 3 dB down from maximum power).

Magnet performance is also excellent, in particular non-spin lineshape (6.0 and 8.1 Hz at 0.55% and 0.11%). The proton sensitivity on 0.1% ethylbenzene in a normal wall NMR tube is 1525:1, showing the expected increase over comparable technology probes at 600 and 750 Mhz.

RF homogeneity is also excellent, showing 87% and 67% for 450/90 and 810/90 for protons. Carbon gives 87% and 76% for 360/0 and 720/0. The good non-spin lineshape permits high quality presaturation performance, as noted in the 1mM phenylalanine and lysozyme results (see figures). Magnet stability is essential and is measured by the quality of the presat NOESY spectrum of a 1mM lysozyme sample in 90%  $\text{H}_2\text{O}$ . Indirect detection performance is demonstrated by a high-resolution N15 HSQC of labelled villin in 90%  $\text{H}_2\text{O}$ . Details are given in the figure captions.

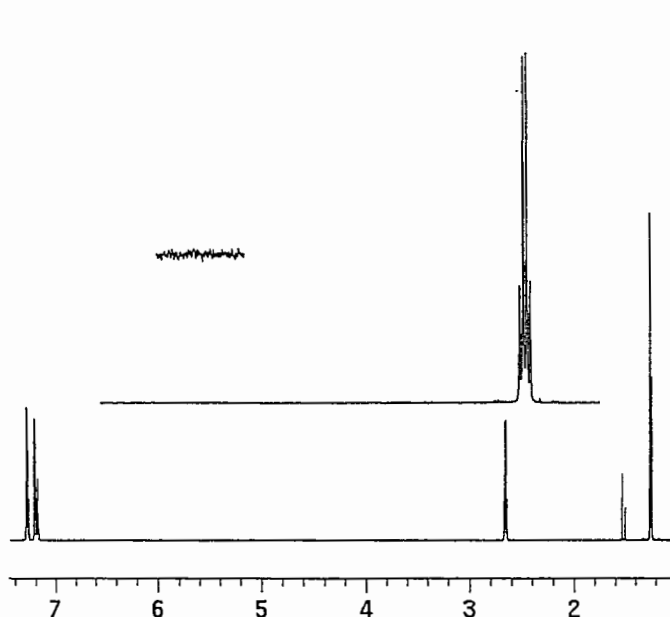


Figure 1.  $1\text{H}$  s/n=1525 for 0.1% ethylbenzene using standard wall sample. Inset noise x40.

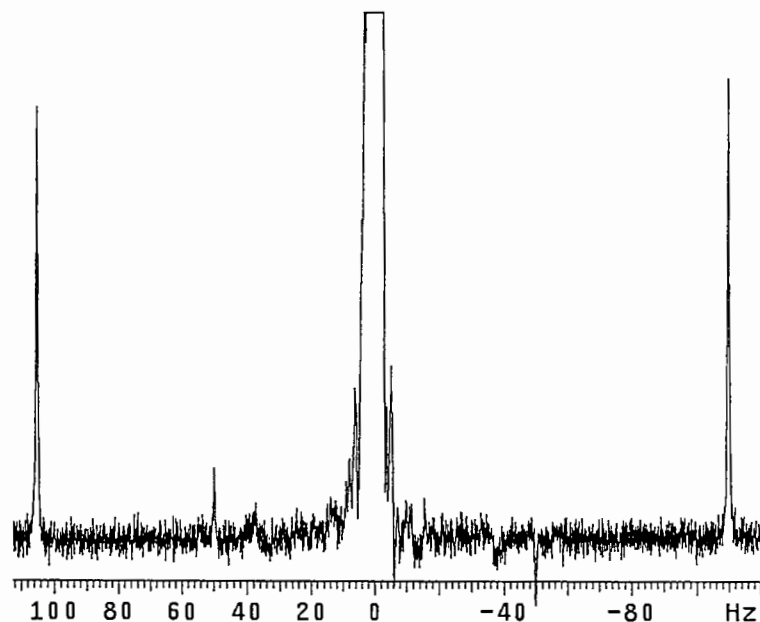


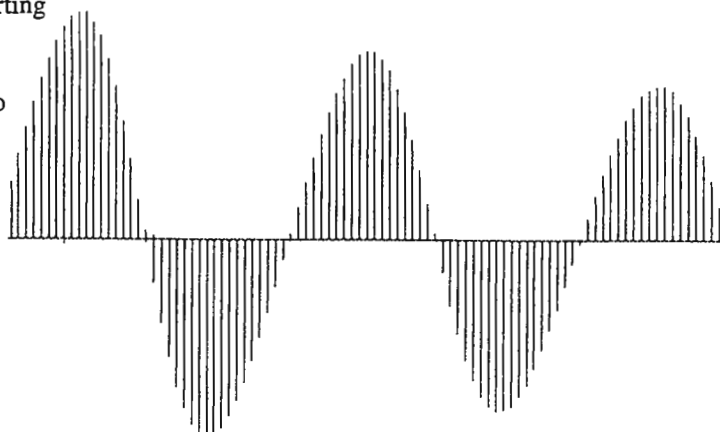
Figure 2.  $1\text{H}$  lineshape (nonspin) at 800 MHz. 6.0 Hz at 0.55%, 8.1 Hz at 0.11% (chloroform)

**Figure 3.**  $^1\text{H}$  RF Homogeneity on doped  $\text{H}_2\text{O}$ .

Pulse width array in 0.5 usec increments, starting

at 1 usec. Measured  $450/90 = 0.87$  $810/90 = 0.68$ 

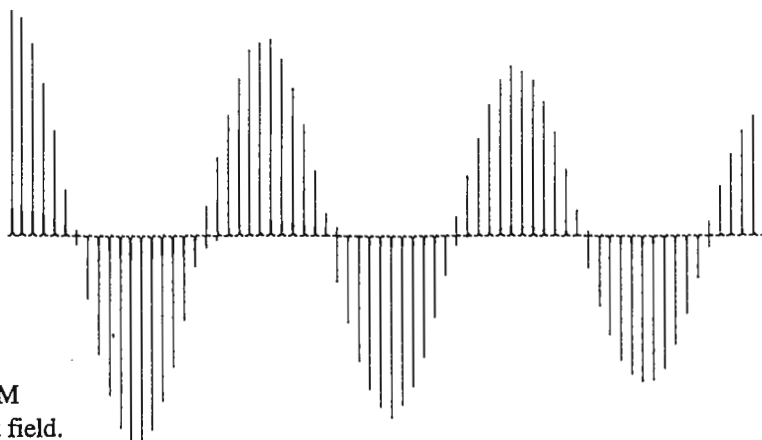
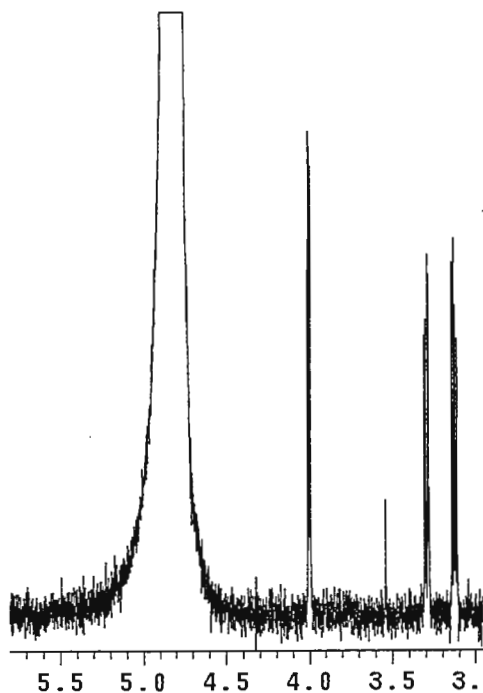
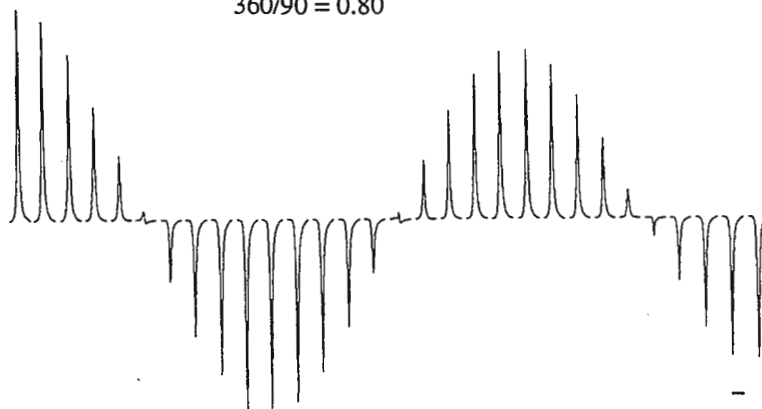
(sample slightly radiation-damped, leading to asymmetry of lobes)

**Figure 4.**  $^{13}\text{C}$  RF Homogeneity on  $^{13}\text{CH}_3\text{I}$ .

Pulse width array in 2 usec increments, starting

at 0 usec. Measured  $360/0 = 0.87$  $720/0 = 0.76$ 

(400 watt amplifier)

**Figure 5.** Presaturation lineshape test. 1 mM phenylalanine in 90%  $\text{H}_2\text{O}$ . 65 Hz presat field. non-spin.**Figure 6.**  $^{15}\text{N}$  RF Homogeneity on  $^{15}\text{N}$  [15N]-benzamide in  $\text{dms}\text{-d}_6$ . Measured  $360/90 = 0.80$ 

- continued



# Technical Specifications

## 200 - 600MHz Wide Bore NMR Magnet Systems

Oxford Instruments, NMR Instruments are the pioneers of superconducting magnet technology for NMR spectroscopy and are widely regarded by the worlds research community as the foremost manufacturer and supplier. A worldwide base of over 4500 successfully installed magnet systems serves as testament to an innovative magnet design approach and quality manufacturing processes. As an integral part of a high resolution NMR spectrometer, Oxford Instruments magnets are recognized the world over for their superior performance in chemical, pharmaceutical, biological, and materials research applications.

### Advanced Design and Manufacture

State of the art superconducting magnet and cryostat designs provide superior NMR performance, as demonstrated in lineshape, resolution, magnetic field stability, and system siting:

- Advanced electromechanical modelling and system design tools optimise magnetic field homogeneity (to at least eighth order, on-axis) while at the same time minimising residual transverse gradients
- Designed for optimal system siting, minimising ceiling height, weight, service access, stray field and system footprints
- High efficiency room temperature shims provide optimal field homogeneity with low heat dissipation
- Rigorous magnet system testing in Oxford Instruments' extensive cryogenic and NMR test facilities ensure that optimal magnet performance is achieved on site
- All systems supplied with a wide range of safety features, providing worry-free operation at all field strengths
- All manufacturing processes certified to the internationally recognised ISO 9001 quality standards



### Performance Excellence

Oxford Instruments magnet and room temperature shim systems provide outstanding performance in a complete range of NMR applications. Sample sizes from 37  $\mu$ l magic-angle-spinning (MAS), 10 mm high-resolution biomolecular to 60 mm microimaging, as well as all other commonly used NMR sample types, are supported:

- Exceptionally low magnetic-field drift rates provide superior stability
- Full range of room temperature shim options for all magnet systems deliver unparalleled lineshape and resolution for both large and small samples
- Minimal cryogen usage provides the lowest possible cost of operation with a range of standard or year hold helium cryostat options available
- All Oxford Instruments magnets feature a virtually unlimited lifetime (with proper maintenance and cryogen service)

See reverse for full technical specifications

# Specifications

Specification	System Type					
	200/89	300/89	300/150	400/89	500/89	600/89
<b>Magnet</b>						
Operating Field (Tesla)	4.7	7	7	9.4	11.7	14
NMR Operating Frequency (MHz <sup>1</sup> H)	200	300	300	400	500	600
Field Stability (Hz/hour <sup>1</sup> H)	<2	<3	<15	<10	<12	<12
Axial 5 Gauss Stray Field Contour (Metres)	2.6	2.7	4.2	3.3	4.5	5.0
Radial 5 Gauss Stray Field Contour (Metres)	2.0	2.2	3.3	2.6	3.5	3.9
<b>Cryostat</b>						
Standard Cryostat Minimum Helium Refill Interval (Days)	203	203	120	180	150	90
Standard Cryostat Helium Refill Volume (Litres)	68	68	101	60	80	135
Year Hold Cryostat Option Available	✓	✓	X	X	X	X
Nitrogen Refill Interval (Days)	14	14	22	14	18	18
Nitrogen Refill Volume (Litres)	61	61	135	67	131	121
Nominal Room Temperature Bore Diameter (mm)	89	89	150	89	89	89
Minimum Operational Ceiling Height (Metres)	2.9	2.9	4.1	2.9	3.4	3.4
System Weight (kg) Including Cryogen's	391	399	1050	410	1075	1200

## Customer Support

Oxford NMR Instruments provides a worldwide network of subsidiary companies and representatives available to provide technical siting, service and sales support.



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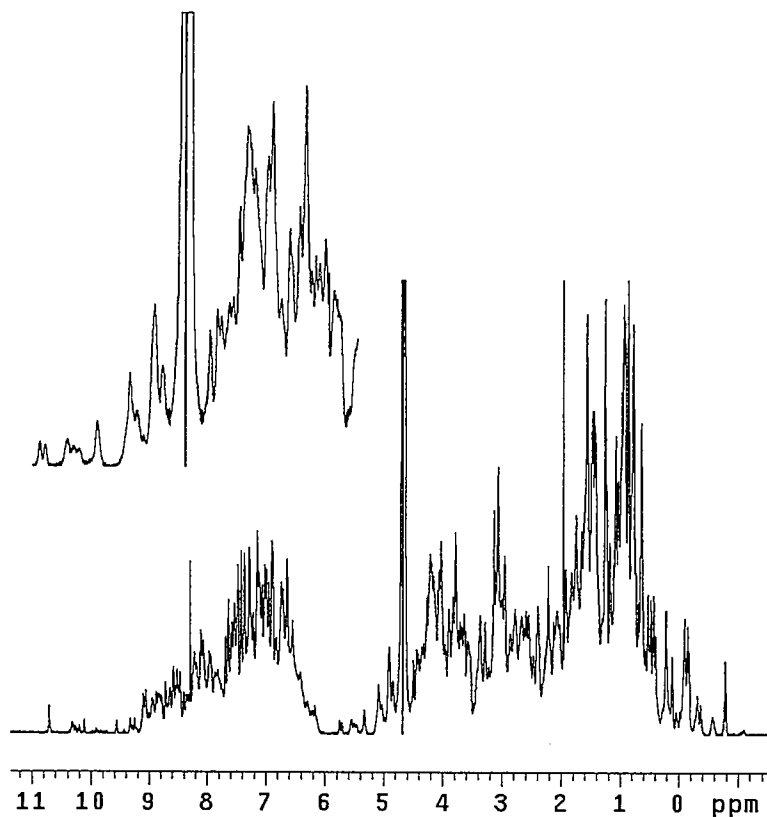
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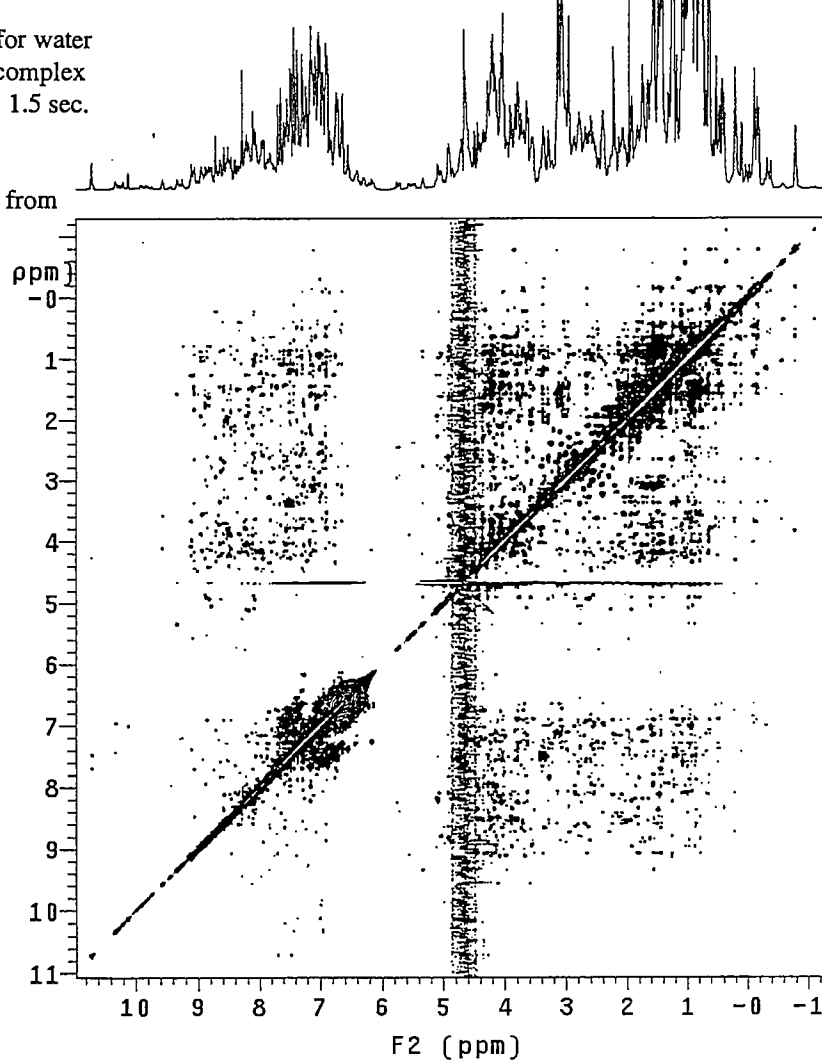




**Figure 7.** Presaturation spectrum of 1mM lysozyme in 90% H<sub>2</sub>O. (No solvent filter applied).

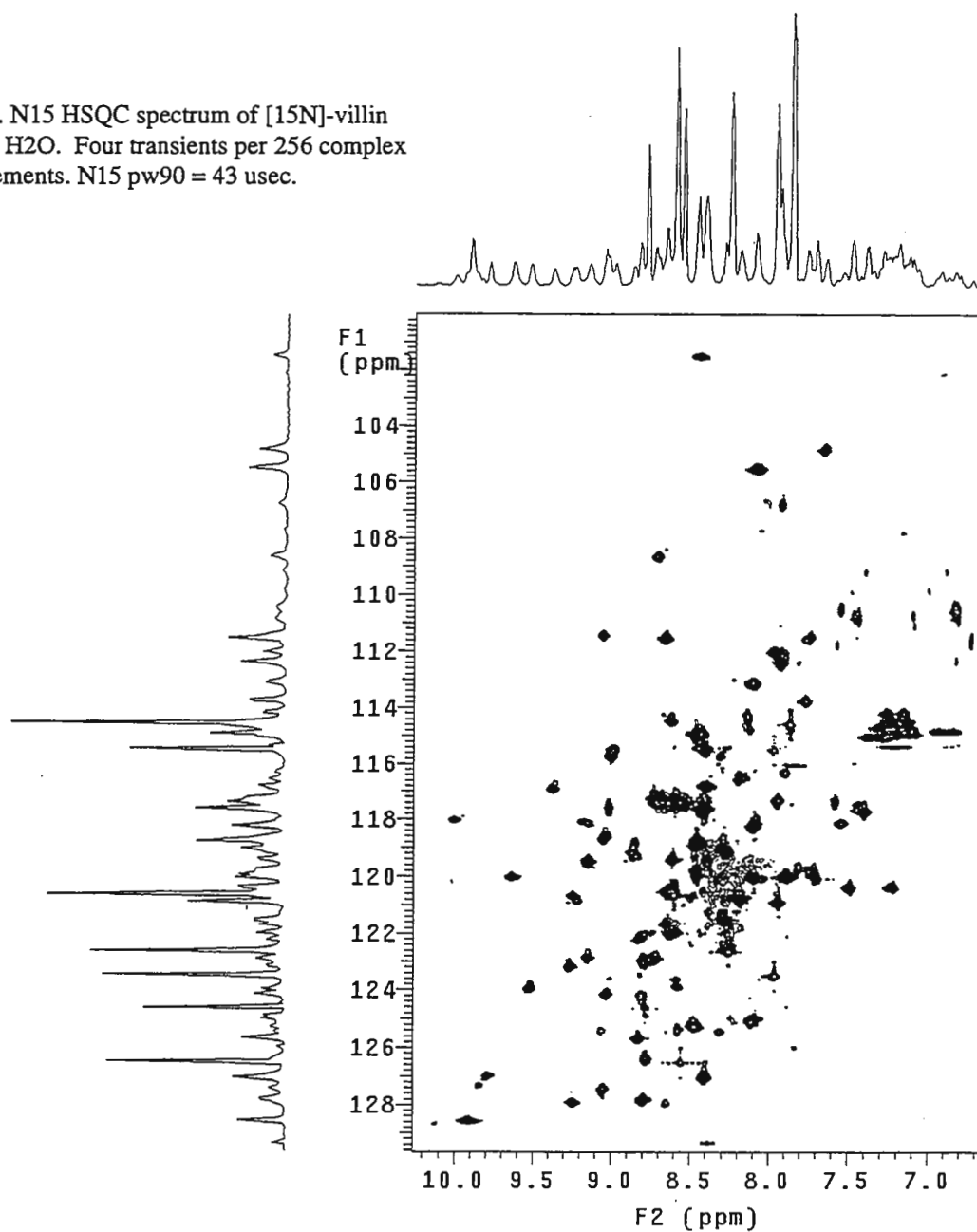
**Figure 8.** NOESY using presaturation for water suppression. Four transients per 512 complex increments in t<sub>1</sub> Presaturation period, 1.5 sec. (10 Hz solvent filter applied)

The 1D spectrum is the F2 projection from the 2D data.





**Figure 9.** N15 HSQC spectrum of [15N]-villin in 90% H<sub>2</sub>O. Four transients per 256 complex t1 increments. N15 pw90 = 43 usec.



We are pleased with the overall performance of the 800. System deliveries will begin this summer.


Sincerely Yours,

*George A. Gray*

George A. Gray  
NMR Applications Lab

*Ali Behbin*

Ali Behbin, Manager      Mark Van Crielinge      Eriks Kupce  
R&D Probe Engineering    R&D System Engineering    Varian, UK



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

July 1, 1997

### **Varian Acquires the Chemagnetics' NMR Business**

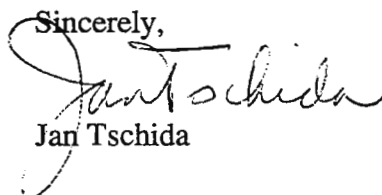
Dear Barry,

As you have heard, on May 30, Varian completed the acquisition of the assets of the Chemagnetics NMR business in Fort Collins, Colorado. Operationally, Varian will remain in the Fort Collins site and in fact have signed a five year lease on the facility. The Fort Collins group will continue to focus on solid state NMR technology and we will maintain development, manufacturing and applications activities there. Many of the key technical people have joined the Varian Team and they are already working with their counterparts in Palo Alto. We will be working hard to support the many loyal Chemagnetics users worldwide and we welcome them to the Varian family at large. All service and applications support for Chemagnetics products will continue to be handled out of Ft. Collins. In Europe, the existing Chemagnetics team has merged with the Varian organization. We plan to train several of the Varian service personnel on the Chemagnetics products in order to expand our support in Europe. This training is scheduled in Ft. Collins in July and will include US service as well.

Chemagnetics' excellent reputation for providing products and support to the solid state NMR community, together with Varian's stronger presence in the liquids and imaging areas, provides an ideal basis for a truly well-rounded family of products. With the traditional boundaries between liquids, solids and imaging becoming far less clear, Varian recognizes that the need for such a balanced approach is more important than ever.

Clearly, the coming months are going to be very busy for many of us here at Varian as we work towards integrating the Chemagnetics' activities into the Varian environment. Already, we have begun a comprehensive review of all of the technologies that are now available to us and in that regard, our longer term goal is very clear; to ultimately offer a range of products that offer outstanding performance in all of the liquids, solids and imaging applications areas.

Sincerely,



Jan Tschida



**Carleton**  
UNIVERSITY

**Department of Chemistry**

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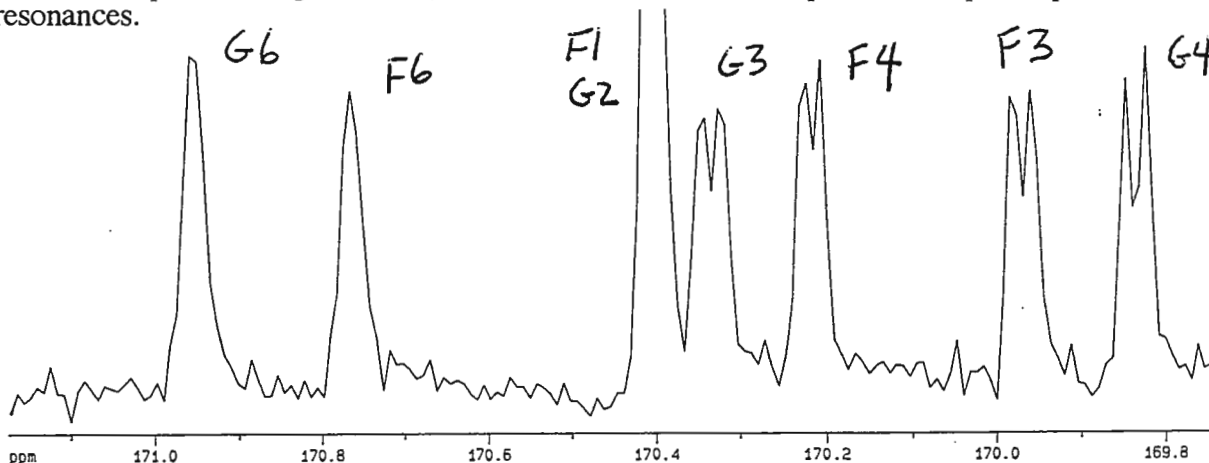
Dr. B.L. Shapiro  
The NMR Newsletter  
966 Elsinore Court  
Palo Alto California, 94303 USA

May 27, 1997  
(received 6/17/97)

**Title: Vicinal  $^1\text{H}$ - $^{13}\text{C}$  Couplings in Sucrose Octaesters: Probes for Vitamin Interaction**

Sucrose octaesters (palmitates, oleates, linolenates etc.) are the major components of the non-caloric fat substitute marketed as "Olestra". There has been some controversy regarding the inhibition by Olestra of absorption of fat soluble vitamins A and E. We are looking at specific Olestra components via NMR methods and the use of the vicinal H-C-O-C coupling to the carbonyl carbons struck us as a possible probe for monitoring conformational changes due to interactions with the vitamins.

Several years ago<sup>1</sup>, the specific assignments of the 8 carbonyl resonances of sucrose octaacetate were published as an illustration of the power of the HMBC experiment. We have used this material as a model compound for the determination of the  $^3J$  values. The experiment was carried out with selective  $^1\text{H}$  decoupling of the methyl protons of the ester functions. As the spectrum shown below illustrates, there is well resolved coupling for four of the ester carbonyl carbons, with the maximal coupling being 2.36 Hz for the high field carbonyl attached to the C4 site of the glucose moiety. Similar resolution (although a different sequence of resonances) was found for sucrose octapalmitate again with selective  $^1\text{H}$  irradiation of the proximal aliphatic proton resonances.



Preliminary experiments with the introduction of Vitamin A and E indicate that there are substantial conformational alterations in the sucrose octaesters as monitored by this method.

1. T. Nishida, C.R. Enzell and G.A. Morris. *Magn. Res. Chem.* **24**, 179 (1986).

*G.W. Buchanan*  
G.W. Buchanan  
Professor of Chemistry



# Model 3445/3446 Amplifiers from AMT

**10-130 MHz  
Bandwidth**

**1000 and 2000  
watt Models  
available**



## For High Performance NMR/NMRI Applications

Your NMR/NMRI requirements are pushing the leading edge of science and you need AMT RF power technology! The 3446 and 3445 operate from 10-130 MHz and are rated at 1000 watts for low field NMR and up to 2000 watts for NMRI applications up to 3 Tesla. AMT has brought together the highest possible RF performance at a most cost effective price. Nobody builds a better NMR/NMRI amplifier than AMT...

### Additional Features Include:

- 10-130 MHz bandwidth for use in systems up to 3T
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- CW power capability for decoupling
- Blanking delay time >1  $\mu$ s for multi-pulse



## Models 3445/3446

10-130 MHz, pulsed, solid-state,  
RF power amplifier systems

### Key Specifications:

Models:	3445	3446
Frequency range	10-130 MHz	10-130 MHz
Pulse power (min.) into 50 ohms	2000 W	1000 W
CW power (max.) into 50 ohms	200 W	100 W
Linearity ( $\pm 1$ dB to 30 dB down from rated power)	1500 W	800 W
Pulse width	20 ms	20 ms
Duty cycle	Up to 10%	Up to 10%
Amplitude droop	5% to 20 ms typ.	5% to 20 ms typ.
Harmonics	Second: -25 dBc max. Third: -24 dBc max.	
Phase change/output power	10° to rated power, typ.	
Phase error overpulse	4° to 20 ms duration, typ.	
Output noise (blanked)	< 10 dB over thermal	
Blanking delay	< 1 $\mu$ s on/off, TTL signal	
Blanking duty cycle	Up to 100%	
Protection	1. Infinite VSWR at rated power 2. Input overdrive 3. Over duty cycle/pulse width 4. Over temperature	

### Supplemental Characteristics:

Indicators, front panel	1. AC power on 2. CW mode	4. Overdrive 5. Over pulse width	6. Over duty cycle 7. LCD peak power meter
System monitors	1. Forward/Reflected RF power 2. Over pulse width/duty cycle	3. DC power supply fault	4. Thermal fault
Front panel controls	1. AC power	2. Forward/Reflected power	
AC line voltage	208/230 VAC, 10%, 1Ø, 47-63 Hz		
AC power requirements	3445 1400 VA	3446 700 VA	
Size (HWL, inches)	8.75 x 19 x 24	8.75 x 19 x 24	
Net weight	110 lbs.	75 lbs.	



### Other members of AMT's NMR/NMRI Family:

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6-220 MHz, 300/1000 W

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30-310 MHz, 400/700 W

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Stockholm, 30 May 1997

(received 6/7/97)



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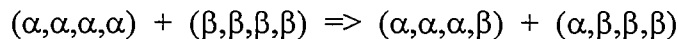
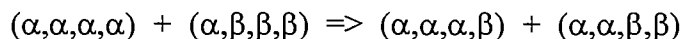
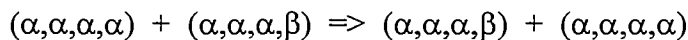
## A Spy Nucleus: For Your Eyes Only

Dear Prof. Shapiro,

A  $^{205}\text{Tl}$  NMR spectrum of a solution containing only the tetrahedral complex  $\text{Ti}(\text{CN})_4^-$  (100%  $^{13}\text{C}$ -enriched cyanide) consists of a 1:4:6:4:1 quintet.<sup>1,2</sup> We have recorded a  $^{205}\text{Tl}$  TPPI EXSY of this solution and the result is shown in Figure 1. The well-defined cross-peak pattern raises questions: what is the origin of the cross-peaks? What kind of chemical exchange can take place in a solution which contains only one species?

The  $^{13}\text{C}$  longitudinal relaxation can be excluded experimentally because the relaxation rate is about  $0.2\text{ s}^{-1}$ , that is too slow to result in cross peaks at mixing time  $\tau = 0.020\text{ s}$ . Therefore, we had to search for another process in which the spin state of the carbons can change or, with other words, magnetization is carried from one multiplet site to another.

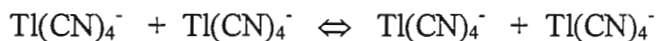
"If thou search thou will find...". Let us imagine that two  $\text{Ti}(\text{CN})_4^-$  species labeled with different carbon spins meet in a solvent cage and exchange one (or more?) cyanides. This meeting can give different result depending on the spin labels ( $\alpha$  or  $\beta$ ) of the four carbons, for example:



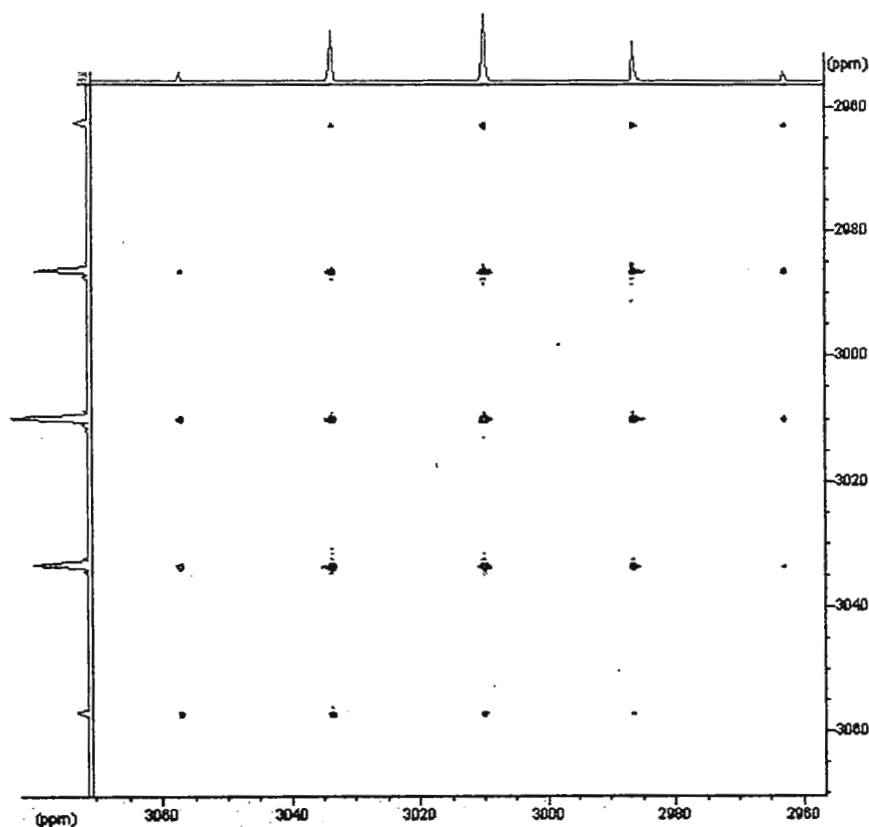
This is exactly what the 2D EXSY shows, *i.e.* from the species labeled  $\alpha\alpha\alpha\alpha$  the magnetization can travel, by means of chemical exchange, to every site except  $\beta\beta\beta\beta$ . However, a closer look at this system shows that only the cross peak between  $(\alpha, \alpha, \alpha, \alpha)$  and  $(\alpha, \alpha, \alpha, \beta)$  corresponds to a primary

exchange process for the species ( $\alpha, \alpha, \alpha, \alpha$ ). All other cross peaks are due to secondary and higher order processes. This can be inferred from the build-up curves for the mixing time range 5 - 60 ms.

In general terms, this is a chemical exchange reaction in which no macroscopic change really occurs:



According to our knowledge, it is the first example where scalar coupling allows for the study of exchange paths which would be hidden for any other experimental method (?).



**Figure 1.** A  $^{205}\text{Ti}$  NMR EXSY spectrum of an aqueous solution containing only the complex  $\text{Ti}(\text{CN})_4^-$  (100%  $^{13}\text{C}$ -enriched). Mixing time = 20 ms.

Istvan Banyai

Mateus W. da Silva

Julius Glaser

## References

1. Blixt, J., Glaser, J., Mink, J., Persson, I., Persson, P., Sandström, M. *J. Am. Chem. Soc.* **117** (1995) 5089-5104.
2. Blixt, J., Györi, B., Glaser, J. *J. Am. Chem. Soc.* **111** (1989) 7784-7791.



# UltraShield™ Magnets Win the Space War

## *Introducing the BRUKER SPECTROSPIN*

### *600 MHz/52 mm UltraShield™ High Resolution NMR Magnet*

Siting is now much easier than ever before because the space required for NMR systems has just become considerably smaller. The magnetic stray field has been significantly reduced by redesigning the coil of a standard magnet and adding a superconducting active shield. The volume enclosed by the 5 Gauss surface for an UltraShield™ magnet is ten times smaller than for a comparable standard magnet, without sacrificing any specifications.

#### **BRUKER SPECTROSPIN**

SPECTROSPIN is a member of the BRUKER family of companies and is located near Zurich, Switzerland. SPECTROSPIN is the world's largest manufacturer of superconducting NMR magnets. Our more than 30 years of experience in development and production of superconducting magnets enables us to deliver NMR magnets with exceptional performance and reliability. Many SPECTROSPIN superconducting magnets built in the late 70's and early 80's are still on field, providing quality data and dependable service.



## **Main Features**

- Active shielding technology strongly reduces stray fields and decreases the volume enclosed by the 5 Gauss surface by a factor of ten.
- Advanced magnet design and a new  $z^3$  cryoshim provide outstanding field homogeneity with excellent resolution and non-spinning lineshape.
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- Optimized cryostat design provides an extremely low helium evaporation rate.
- Lowest drift rates.
- Special sensors connected to the Automatic Cooling Device (ACD) prevent stresses during magnet cooling.
- Advanced vibration isolation system integrated in the cryostat stand provides optimal dampening of ground vibrations.
- Electronic atmospheric pressure device stabilizes the field drift and helium boil-off when changes in atmospheric pressure occur (optional).



*Innovation for customers  
delivered with Integrity*

# SPECIFICATIONS

## MAGNET

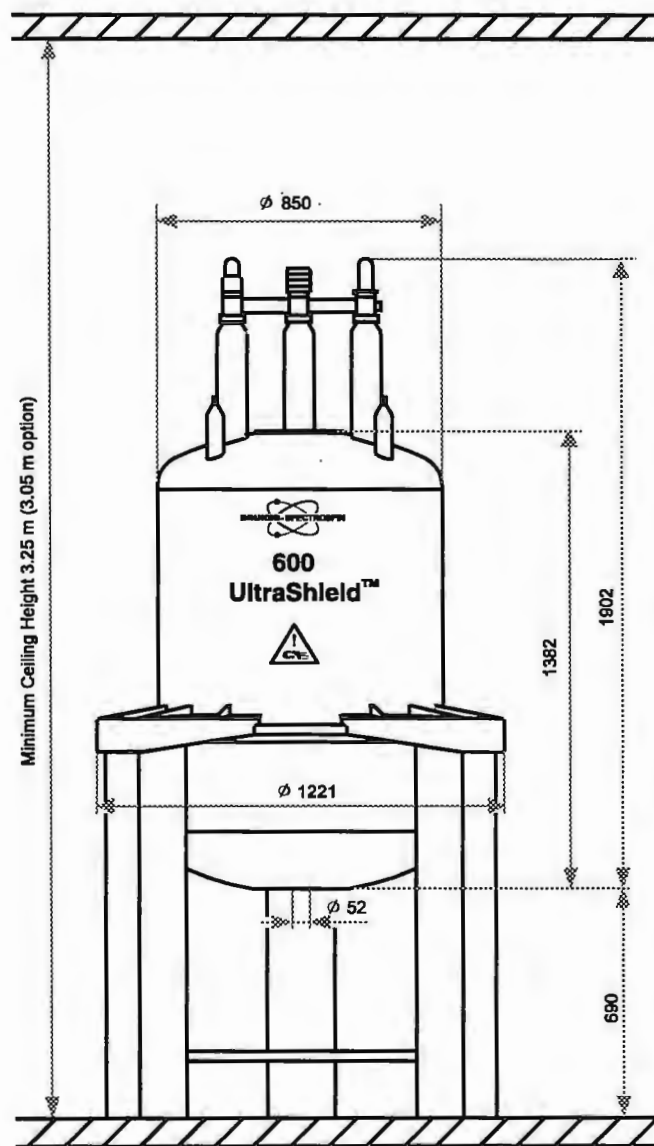
Central Field	14.1 Tesla
NMR Frequency	600 MHz
Field Drift	< 9 Hz/hr
Superconducting Shims	$Z, Z^2, Z^3, X, Y, XZ, YZ, XY, X^2-Y^2$
Axial Range with Field Homogeneity better than -10 ppm (w/o RT Shimming)	~ 60 mm
5 G Line from the Magnetic Center	
-radially	< 1.8 m
-axially	< 2.5 m
Resolution at 50% 1% CHCl <sub>3</sub> 5 mm spinning	< 0.45 Hz
Lineshape 1% CHCl <sub>3</sub> 5 mm non-spinning	
at 0.55%	< 6 Hz *
at 0.11%	< 12 Hz *
Spinning Sidebands	< 1%

\* Typical values obtained with the BOSS II™ shim system.

## CRYOSTAT

Helium Evaporation Rate	~ 40 ml/hr
Helium Refill Volume	~ 125 liters
Helium Hold Time	> 130 days
Nitrogen Evaporation Rate	~ 400 ml/hr
Nitrogen Refill Volume	~ 163 l
Nitrogen Hold Time	> 17 days
Magnet Stand	included
Anti-Vibration Columns	included
Weight Without Cryogenics	1150 kg
Weight Including Cryogenics	1300 kg
Minimum Ceiling Height	3.25 m
Reduced Minimum Ceiling Height	3.05 m

## 600 MHz / 52 mm UltraShield™ Magnet



Dimensions in millimeters unless stated otherwise

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## Lilly Research Laboratories

A Division of Eli Lilly and Company

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Indianapolis, Indiana 46285  
(317) 276-2000

June 17, 1997  
(received 6/21/97)

### Control of Spinning Sidebands in High Resolution NMR Spectroscopy

Dear Dr. Shapiro,

We have a strong interest in being able to detect low molarity analytes in the presence of other analytes whose concentrations may be as much as 10,000 times greater. In order to do this routinely one needs to minimize or eliminate the presence of any resonances which do not arise directly from the constituents of the sample. The primary culprit for most NMR spectroscopists is spinning sidebands. Spinning sidebands have traditionally been minimized by iterative adjustment of the radial shims yielding residual sidebands of about 0.1%. Once the spinning sidebands have been minimized to these levels methods to further reduce the amplitude of the sidebands have been described which vary the spinning speed in a linear<sup>1</sup> or non-linear<sup>2</sup> fashion during data acquisition. The effect of these techniques is to spread the spinning sideband intensity over a range of frequencies so that the final result is a spinning sideband whose shape reflects the distribution of spinning speeds. For these methods to be effective the spinning sideband intensity should respond in a predictable way to variations in spinning sidebands. The theory of spinning sidebands as originally developed by Williams and Gutowsky<sup>3</sup> assumed, quite correctly at the time, that spinning sidebands were due primarily to samples spinning in the presence of linear  $H_0$  gradients perpendicular to the axis of rotation. The theory of Williams and Gutowsky also predicts the behavior of spinning sidebands as the spinning speed increases. The ratio of integrated intensities for two values of the modulation index ( $k = \gamma H_1^{(0)} / 2\pi\nu_s$ ),  $k$  and  $k'$ , is given by  $A_{\pm n}(k)/A_{\pm n}(k') = (k/k') = (n'/n_s)$ . Consider the implications to the intensity and appearance of spinning sidebands. For a linear field gradient of 4 Hz and a spinning speed of 20 Hz,  $A_{\pm 1}$  and  $A_{\pm 2}$  should be 0.5% and 0.0008 %, respectively, clearly indicating that only first-order spinning sidebands should be observed. This also predicts that  $A_{\pm 1}$  should decrease from 0.5% ( $k=0.2$ ) to 0.33% as  $n_s$  increases from 20 Hz to 30 Hz. From the standpoint of detection of low molarity analytes the spinning sideband peak height,  $P_n$ , is more important than the integrated intensity,  $A_n$ . Under a linear variation of  $n_s$  between a lower limit of  $n_s^L$  and upper limit  $n_s^U$ , each spinning sideband will undergo a broadening of  $|n|(n_s^U - n_s^L)$ , and a resulting decrease in  $P_n$ . The ratio of  $P_n$  at a fixed  $n_s$  and in the presence of spinner speed variation between  $n_s^L$  and  $n_s^U$  is approximately  $P_n^F/P_n^V = W_{ex}^{-1} [W_{ex} + |n|(n_s^U - n_s^L)]$  where  $P_n^F$  and  $P_n^V$  are the values of  $P_n$  at a fixed  $n_s$  and in the presence of spinner speed variation, respectively, and  $W_{ex}$  is the linewidth at half-height for fixed  $n_s$ . The practical limit on values of  $(n_s^U - n_s^L)$  which maintain high resolution and stability is about 10 Hz. Therefore, the upper limit to the reduction of  $P_n^F$  by spinner speed variation for  $W_{ex} = 1.0$  Hz, a typical value for proton linewidths, is about a factor of  $10|n|$ . Given this constraint it is important to optimize the manner in which the spinner speed variation is implemented.

The previous theoretical discussion on the formation of spinning sidebands focused on samples spinning through linear  $H_0$  gradients perpendicular to the axis of rotation. In the absence of  $H_0$  gradients, or when  $H_0$  gradients are minimized, other mechanisms exist for the formation of spinning sidebands.<sup>2,4</sup> Dadok has described in some detail these mechanisms which involve  $H_1$  inhomogeneity and variable coupling of the precessing magnetization to the receiver coil.<sup>4</sup> On our spectrometer, the overwhelming mechanism for spinning sidebands are  $H_0$  gradients perpendicular to the axis of rotation. This conclusion is supported by several observations. Only first-order spinning sidebands are visible in spectra acquired with optimal  $H_0$  homogeneity ( $P_{\pm 1} \leq 0.1\%$ ). A substantial degradation of  $H_0$  homogeneity is required before second-order sidebands become visible, but higher order sidebands have not been observed.

A key factor discovered during initial experiments was that the spinner in this instrument, and undoubtedly in spinner assemblies for other commercial instruments, does not respond quickly to changes in spinner air flow. The response is especially sluggish when the spinner has to change from decelerating to accelerating. Presumably, this is because there is very little friction between the gas flow and the spinner, and the spinner has a correspondingly high inertia. For the same reason, the rate of deceleration with a step decrease in flow rate was found to be greater than the rate of acceleration with a step increase in flow rate. These factors make it difficult to program a planned change in spinner speed with time. Specifically, a linear variation of  $\nu_s$  would not result in an equal amount of time at each spinner speed. Rather, the sample would spend more time spinning at the slower

spinner speeds leading to more intensity in the spinning sideband as  $\nu_s$  approaches  $\nu_s^L$ . The quickest change in gas flow rate would have to be much slower than the slowest response time of the spinner so that the spinner speed always matches the flow. This turns out to be a very low frequency ( $\ll 0.001$  Hz) resulting in a very long acquisition time to complete one period of spinner speed variation; therefore, to completely distribute  $P_n$  it becomes advantageous to collect data over more than one period of  $\nu_s$  variation.

Another consideration that must be taken into account when choosing a spinner speed profile is the behavior of  $P_n$  as a function of spinner speed. The intensity of a given spinning sideband increases as  $\nu_s$  becomes smaller. In order to uniformly spread out a given spinning sideband, it is necessary to design a spinner speed profile where more time is spent spinning at higher  $\nu_s$  and less time is spent spinning at lower  $\nu_s$ . Unfortunately, the inertia of the spinner causes a long lag at low  $\nu_s$  as the gas flow rate changes from decreasing to increasing. To effectively resolve this dilemma, and compensate both for spinner inertia and the behavior of spinning sidebands with respect to  $\nu_s$ , we designed a custom spinner speed profile whose overall shape is an exponential increase that is intended to counterbalance the change in spinning sideband intensity with  $\nu_s$ . We have added a pulse following the drop in flow at the beginning of the exponential increase. This pulse compensates for the lag at low  $\nu_s$  caused by the spinner's inertia and actually causes the spinner speed to temporarily increase again. The temporal position, length of time, and intensity of the pulse have all been empirically optimized to counterbalance the lag. If the rate of change becomes too fast, the spinning speed profile cannot change fast enough to correlate to the changing air flow. For example, a period of two minutes with the same air flow profile results in a distorted  $\nu_s$  profile that does not have the desired effect on the sideband shape. It was also observed that as the period is increased, the characteristics of the pulse in air flow have to be modified to maintain the same effect on the sideband shape.

A manuscript describing this work in greater detail will be submitted to the Journal of Magnetic Resonance, Series B. Please credit this contribution to Doug Dorman's account.

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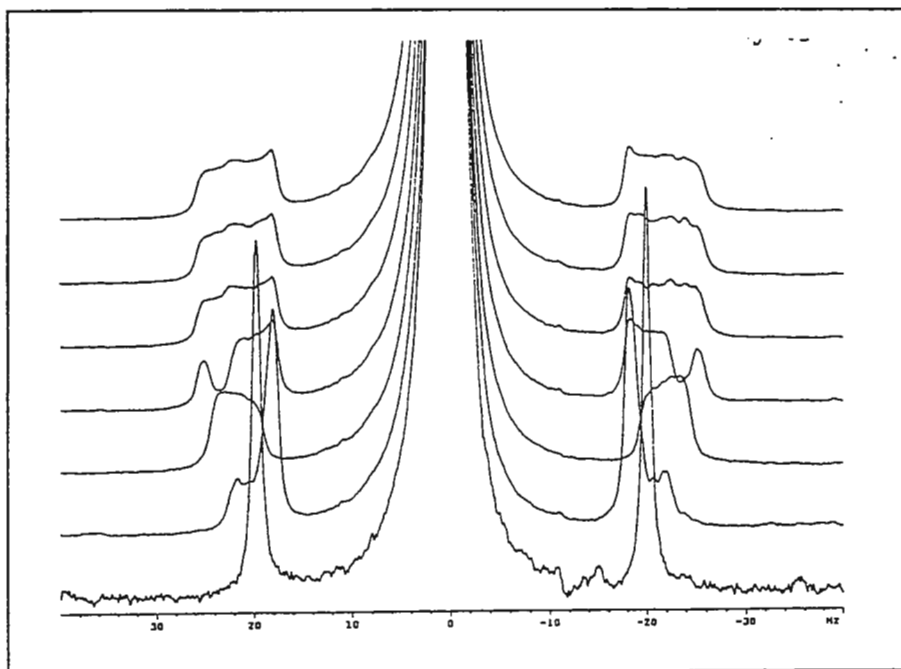
*Steven R Maple*

Steven R. Maple, Ph.D.

*Matthew W Borer*

Matthew W. Borer, Ph.D.

The figure shows a proton resonance and the first order spinning sidebands with increasing data collection time. The bottom trace corresponds to a single scan of about 5.8 sec. The next spectrum is the average of 26 scans corresponding to 25% of the ten minute period of spinning speed variation. Subsequent spectra are 52, 78, 104, 208, and 416 scans corresponding to 50%, 75%, 100%, 200%, and 400% of the variation period.



<sup>1</sup>B. Bammel, R. E. Avilia, *Anal. Chem.* 52, 1999 (1980).

<sup>2</sup>S. R. Maple doctoral dissertation, Indiana University, 1989.

<sup>3</sup>G. A. Williams and H. S. Gutowsky, *Phys. Rev.* 104, 278 (1956).

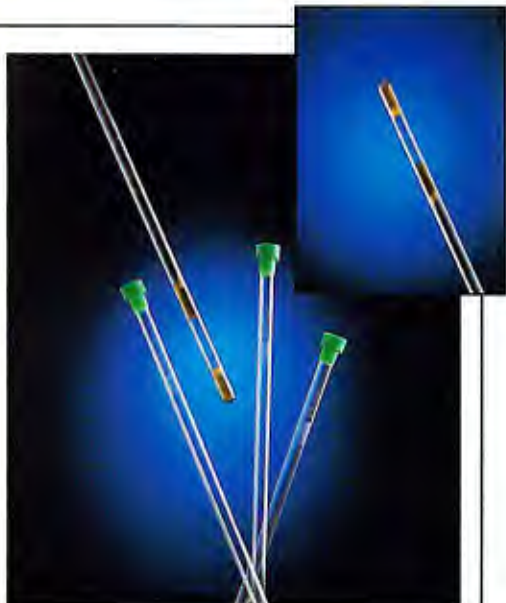
<sup>4</sup>J. Dadok Presented at the Twelfth Experimental NMR Conference, Gainesville, FL, February, 1971.



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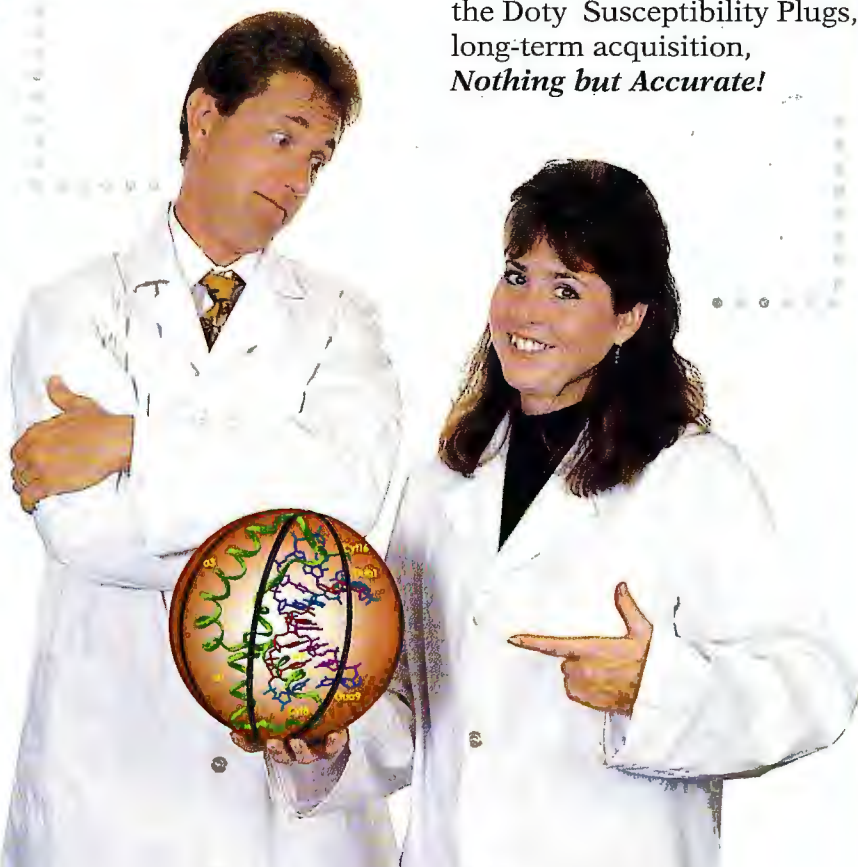
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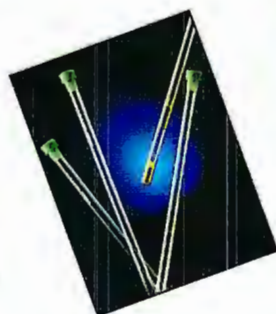
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
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Dr. B. L. Shapiro;

Multiple Quantum MAS Correlation Experiments and  
t<sub>1</sub>-Proportional Phase Shifts in NUTS<sup>†</sup>

(received 6/17/97)

Many of the new multi-dimensional solid state NMR experiments are based on the refocusing of anisotropic interactions and require a "shearing" of the resulting 2D spectra in order to obtain an isotropic projection in one of the dimensions. A good example is the multiple-quantum (MQ) MAS correlation experiments presented by Frydman and Harwood<sup>1</sup> for the investigation of quadrupolar nuclei. Here at Sandia National Laboratories we have begun using these MQMAS correlation experiments to investigate a variety of materials. In Fig. 1A is the 2D <sup>23</sup>Na triple-single quantum (3Q-1Q) MAS correlation experiment for our MQMAS setup sample Na<sub>2</sub>SO<sub>4</sub>. The quadrupolar interaction refocuses during acquisition period t<sub>2</sub> along the t<sub>2</sub> = k\*t<sub>1</sub> line, with k = 7/9 for <sup>23</sup>Na (I = 3/2). Because the echo forms at different times during t<sub>2</sub>, a t<sub>1</sub>-proportional phase shift is produced during a simple 2D FT. As an example, the contour plot Fig. 1B shows the results for Na<sub>2</sub>SO<sub>4</sub>, in which the quadrupolar anisotropic interaction in the 3Q dimension (vertical) is correlated with the 1Q dimension, but retains some anisotropic component. This anisotropic component can be removed by a "shearing" transformation, easily obtained by a t<sub>1</sub>-proportional first-order phase correction prior to the final Fourier transform in t<sub>1</sub>.

$$S(t_1', \omega_2) = e^{i\phi(t_1, \omega_2)} S(t_1, \omega_2)$$

where the phase angle in degrees is given by

$$\phi(t_1, \omega_2) = k \times (dw_1 / dw_2) \times 360^\circ$$

Fig. 1C demonstrates this process, with the final correlation contour plot in Fig. 1D giving an *isotropic* projection in the 3Q dimension. Unfortunately, this type of incremental phase shift is not available in older versions of vendor software, making it more difficult to obtain a sheared transform. While this problem will be remedied in our lab by a console upgrade this summer, we still wanted to pursue these types of MQMAS experiments. Woody Conover (ACORN NMR) was gracious enough to program a new incremental phase shift into the NUTS processing software, allowing off-line analysis of our MQMAS experiments. The macro for the implementation of the t<sub>1</sub>-proportional first order phase correction is included below. This incremental phase shift should be beneficial for other types of solid state multi-dimensional experiments requiring a shearing transformation.

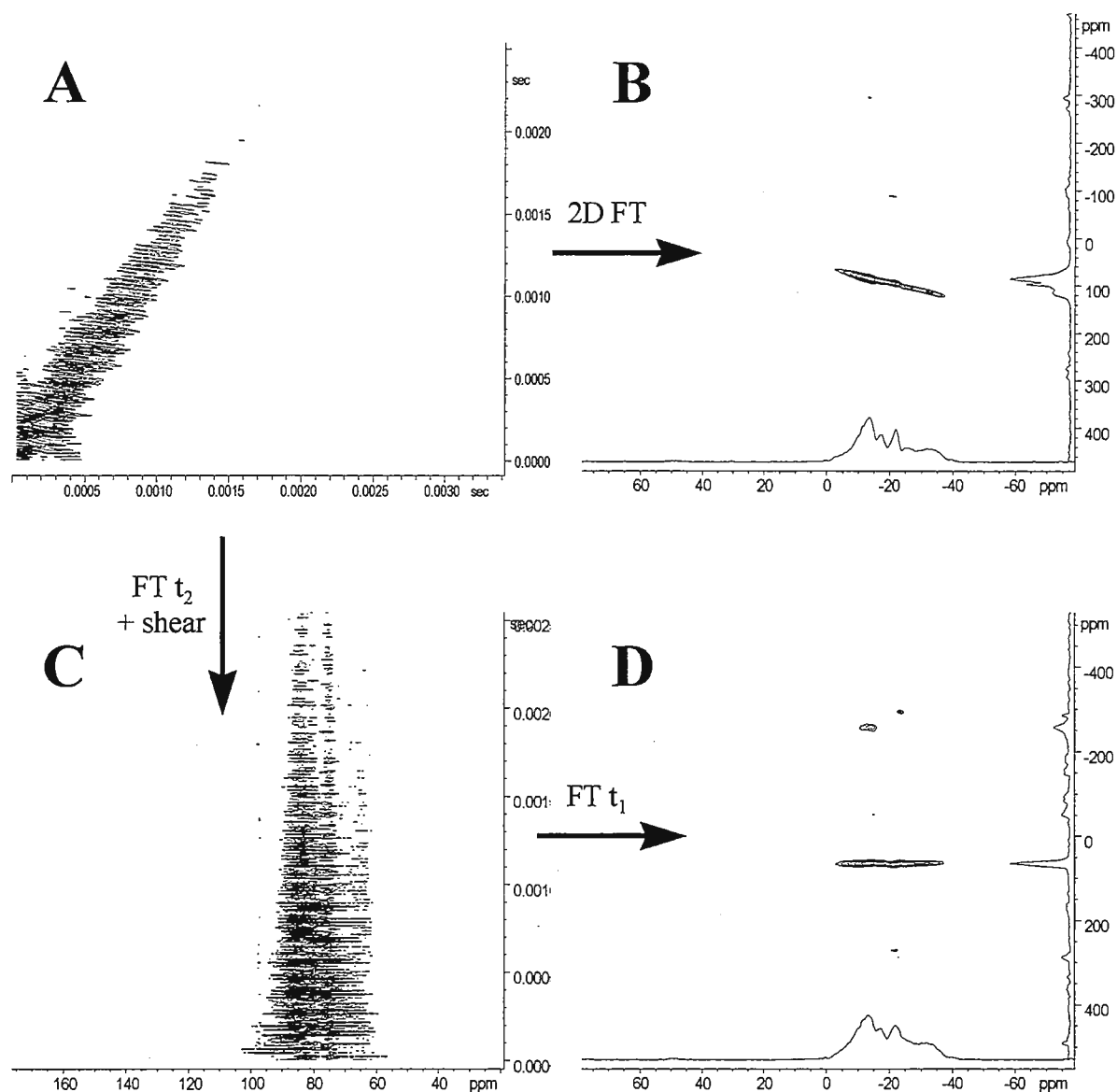
```
NUTSMacro  MQ_shear
; Transform for shearing during 2D transform
; Transform in F2 already completed
; set working directory and file - ft1.2d
ask filea
ga
; set output file
set filec shear.2d
; perform shear, phase increment in degrees set in PB and = k*dw1/dw2*360
set sl 1
ga pi sc in
; transpose data prior to final FT
set filea skew.2d
set filec td.2d
set sl 1
ga td
; data is now ready for last transform
end
```

  
Todd M. Alam

<sup>†</sup> Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

<sup>1</sup> L. Frydman, J. Harwood, *J. Am. Chem. Soc.* **1995**, *117*, 5367.





**Figure 1:** Two-dimensional  $^{23}\text{Na}$  triple quantum (3Q) MAS NMR of  $\text{Na}_2\text{SO}_4$ . A) 3Q-1Q multiple quantum (MQ) correlation experiment using a simple 2 pulse experiment.<sup>1</sup> B) 2D contour spectra of MQ correlation following Fourier transform in both dimensions *without* shearing. Note the lack of an isotropic projection in the F1 dimension (vertical). C) Data following Fourier transform in  $t_2$ , plus a  $t_1$ -incremented first-order phase correction (shear). D) Final 2D correlation experiment following a simple Fourier transform of the sheared data set shown in C. Note that the F1 projection now contains the narrow isotropic resonance for 3Q-1Q correlation.



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May 28, 1997

Barry Shapiro  
The NMR Newsletter  
966 Elsinore Court  
Palo Alto, CA 94303

Re: 4.7 T Magnet

Dear Barry:

I have a wide bore 4.7 T Oxford magnet vintage 1985 that we have used for years on our solids instrument which we have just decommissioned.

Although this is a convenient field for a number of experiments and the large bore is very handy, we must make room for new hardware and are interested selling this magnet.

Anyone who is interested may contact me directly.

Sincerely,



Robert G. Bryant



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**Richard A. Komoroski, Ph.D.**  
 Professor

May 30, 1997  
 (received 6/5/97)

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



Title:  $^{31}\text{P}$  NMR of Phospholipids in Human Brain

Dear Barry:

Phospholipids (PLs) are important components of biological membranes that vary substantially in their molecular structure. Disease processes can be expected to affect phospholipid composition and membrane function.  $^{31}\text{P}$  NMR has been used to characterize PL mixtures by class or headgroup by dissolving in aqueous detergents or a special solvent system to yield narrow  $^{31}\text{P}$  resonances.<sup>1</sup> High resolution  $^{31}\text{P}$  NMR has the advantages of simultaneous detection and quantitation of numerous PLs, detection of unsuspected and unidentified PLs, and its nondestructive nature. We used this method to characterize PLs in human amniotic fluid as a measure of fetal lung maturity.<sup>2</sup> We recently resolved the  $^{31}\text{P}$  NMR signals of phosphatidylcholine (PC) molecular species (different acyl chains) of differing unsaturation or chain length<sup>3</sup> by solubilization in an aqueous solution of bile salt (BS) under very specific conditions, and applied this technique to amniotic fluid.<sup>4</sup> Here we report application of the sodium-cholate-solubilization technique to the observation of different PL species in extracts of human brain.

The Figure shows the  $^{31}\text{P}$  spectra obtained at 121.65 MHz on a GE GN-300WB FTNMR spectrometer of PL profiles of human frontal gray matter using the special organic solvent system (top) and sodium cholate solubilization (bottom). PL classes, including PC, sphingomyelin (SM), phosphatidylserine (PS), phosphatidylinositol (PI), phosphatidylethanolamine (PE), PE plasmalogen, and diphosphatidylglycerol (DPG, or cardiolipin), are resolved in the top spectrum.<sup>1-3</sup>

The bottom spectrum was optimized for resolution of PC species and PL classes. Dipalmitoylphosphatidylglycerol (DPPG) was added as an internal quantitation standard. Good resolution was obtained for PCs with various combinations of saturated and unsaturated acyl side chains. Resolved resonances were seen for PC molecular species with 0 (dsPC), 1 (suPC), and 2 (duPC) unsaturated acyl side chains,<sup>3</sup> with additional potential for peak resolution at 121 MHz suggested by observation of an unidentified PC species ( $\text{PC}_u$ ). Preliminary work indicates that molecular species of other common PLs such as PE and PG can be resolved. Unidentified PE resonances are observed, as well as lyso-PE (LPE) and lyso-PC (LPC). Specific assignments of PE and PE plas are not made in the bottom spectrum because of possible overlap of individual molecular species of the two PLs.

The Table gives the relative amounts of the various PL classes and PC species for human brain. There is approximate agreement with previous results for PL classes. The relative amounts of the three major PC species types are consistent with the distribution reported for rat brain PC.

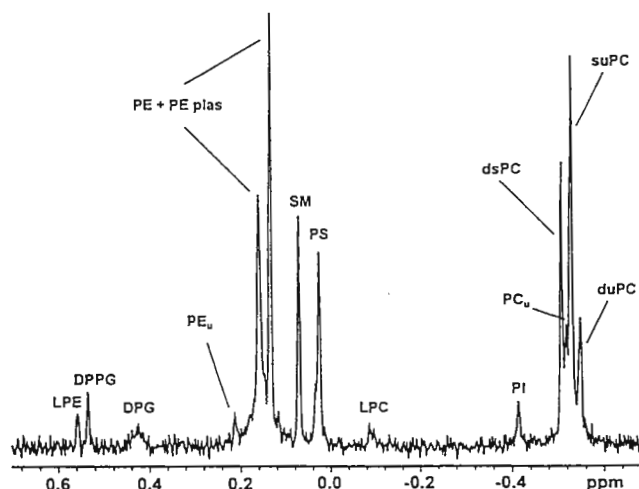
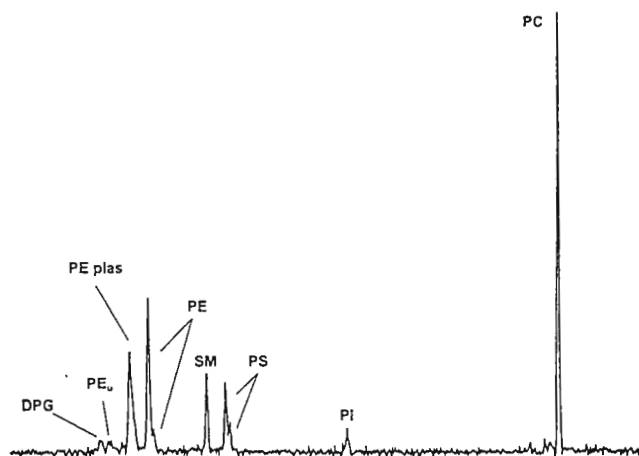
At least for saturated acyl chains in PC, shorter chain length moves the  $^{31}\text{P}$  resonance upfield.<sup>3</sup> Thus there is potential to resolve PL mixtures with side chains of different lengths.

The chemical shift of  $PC_u$  is consistent with dimyristoyl PC (C-14),<sup>3</sup> but this species has not been reported as a significant component of brain PC in other studies. We are pursuing resolution and identification of additional PL species and application of both methods to PLs in postmortem brain samples of schizophrenic patients.

1. Glonek, T., in *Phosphorus-31 NMR Spectral Properties in Compound Characterization and Structural Analysis*, Quin, L.D., Verkade, J.G., Eds., Ch. 22, pp. 283-294, VCH, New York, 1994.
2. Pearce, J.M., Shifman, M.A., Pappas, A.A., Komoroski, R.A., *Magn. Reson. Med.* **21**, 107-116(1991).
3. Pearce, J.M., Komoroski, R.A., *Magn. Reson. Med.* **29**, 724-731(1993).
4. Pearce, J.M., Krone, J.T., Pappas, A.A., Komoroski, R.A., *Magn. Reson. Med.* **30**, 476-484(1993).

TABLE  
Mole % of Various PL Classes  
and PC Species

PI	2.1
PS	10.4
SM	7.4
PE	18.6
PE plas	19.9
$PE_u$	2.9
DPG	2.5
PC	36.2
dsPC	24.7
$PC_u$	8.7
suPC	46.8
duPC	19.9



*Rich*

Richard A. Komoroski

*John M. Pearce*

John M. Pearce

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Date: May 28, 1997  
(received 6/7/97)

*Long-time VT-experiments at low temperatures*

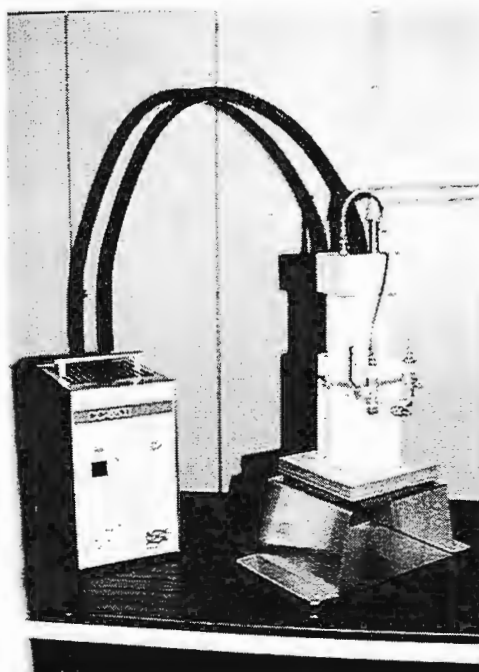
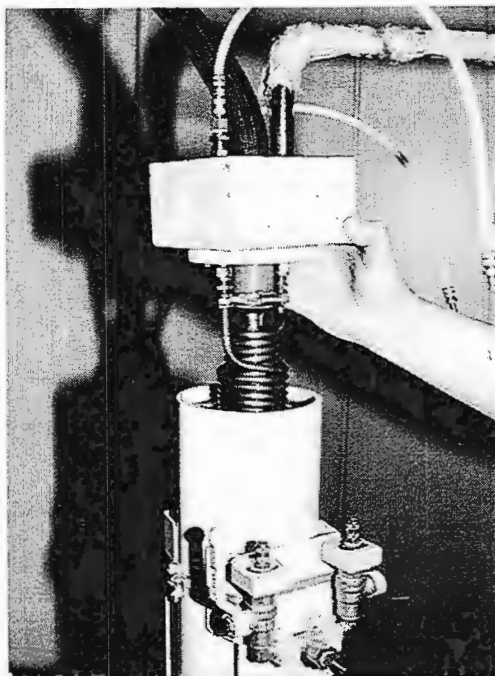
Dear Barry,

if one needs to do low-temperature nmr-experiments, one usually uses a heat exchanger filled with either liquid nitrogen or a cooling mixture (acetone/dry ice) or one evaporates liquid nitrogen and directs the cool gas into the nmr-probe. All this methods require a periodical refill which might disturb an long-time experiment. For example, we performed 2D-MAS experiments at moderate low temperatures (around -20°C) and found it very inconvenient to stop the experiment, refill liquid nitrogen, stabilize the spinning speed and continue the experiment.

Now, we added to our VT-setup a device that in principle allows unlimited experimental time without any stop due to refill of the cooling liquid. It comprizes of an Immersion Heat Exchanger (nicknamed 'Cold Finger'), a heat exchanger for the cooling gas and a transfer dewar. In the heat exchanger, we wound our copper air tube twice around the 'Finger' and immersed it into a dewar (volume about 2 liters) filled with a 1:1 mixture of a non-freezing liquid and water. For the non-freezing liquid, we took the one which is used in the cooler of a car. This design enables a good thermal contact between the 'Cold Finger' and the copper tube. We additionally put a bypass into the air line (the valves seen in the photographs) to allow adjustments at ambient temperatures and control of the cooling process. Mixing the non-freezing liquid with water reduces the viscosity of the mixture and results in a better transfer of the heat between air and 'Cold Finger'. Since the latter contains heavy iron parts, we placed it about 2m away form our 400WB-magnet. The transfer dewar is about 1,5m long and connects the heat exchanger with the inlet of the bearing air of our CPMAS-probe.

We used for these experiments a 7mm CPMAS-probe with typically 4kHz spinning speed and a flow of 17 lpm bearing air. 15 min after switching on the power of the 'Cold Finger', we reached -15°C at the position of the sample, -22°C after 30 min and -25°C after 1 hour. The temperature of the liquid in the heat exchange was around -32°C. We used pressurized air (dried by an adsorption dryer) rather than the expensive nitrogen gas. Besides the financial gain, it avoids the necessity to change the N<sub>2</sub>-gas bottles or lN<sub>2</sub>-evaporator, too.

We performed CPMAS-experiments as long as four days and we did never faced any problems so far. The accessible temperature is rather limited, however, the temperature region between room temperature and  $-25^{\circ}\text{C}$  is of potential interest for many dynamical investigation of organic solids and polymers.



Yours sincerely

Detlef Reichert

A handwritten signature in cursive script that reads "Detlef Reichert".

Horst Schneider

A handwritten signature in cursive script that reads "Horst Schneider".





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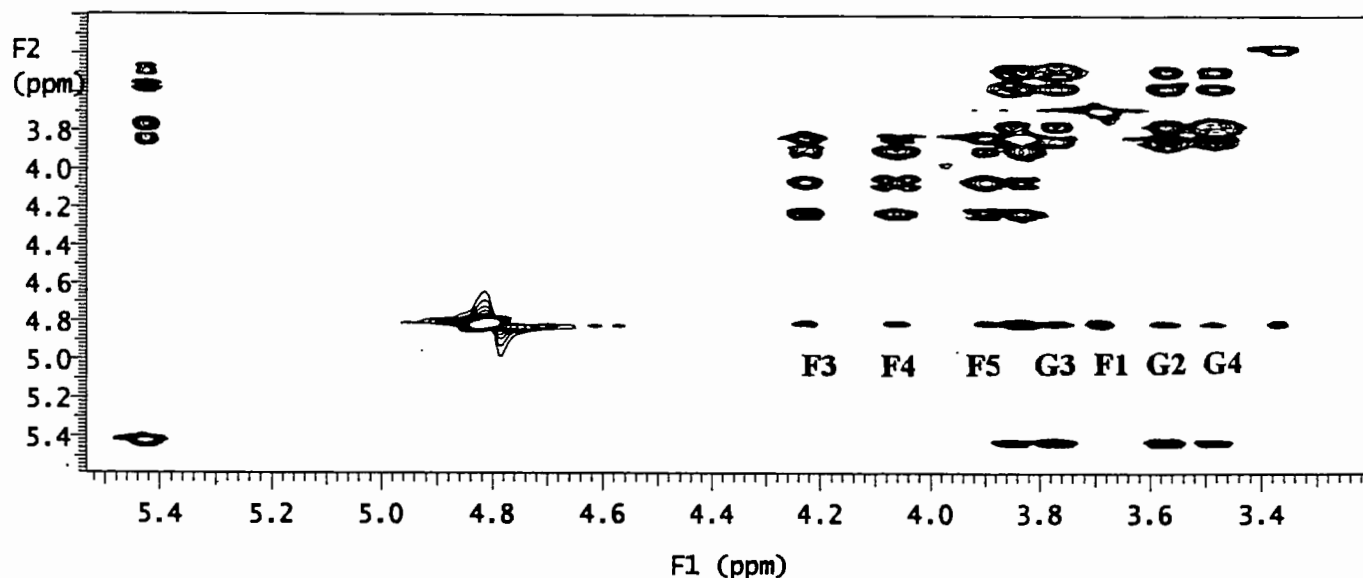
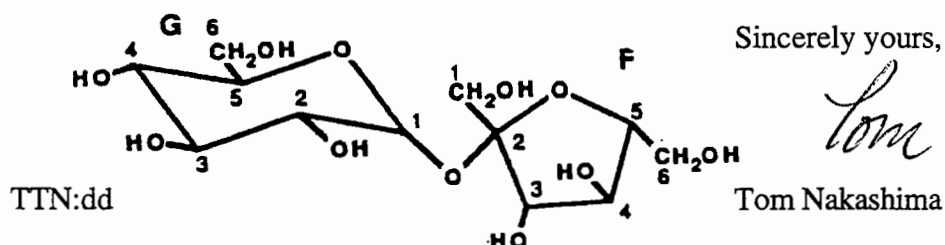
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(received 6/30/97)

Dear Barry :

**RE: INFORMATION FROM THE WATER STRIPE  
IN TOCSY EXPERIMENTS ON SYSTEMS WITH EXCHANGEABLE PROTONS**

When exchange between water and -XH protons, is fast, the resonances of the -XH and water protons are coalesced into a single line. Under these conditions it is impossible to trace J connectivities to the exchanging protons. However, if the water magnetization is saturated completely before the start of a TOCSY experiment, then any magnetization which appears at the water proton frequency in f2 can arise in one of two ways — recovery of the water magnetization or proton magnetization resulting from exchange with the -XH protons. The latter carries with it chemical shift information from the coupling partners of the -XH protons. Thus we are able to use the water resonance as a reporter to obtain chemical shift information about protons coupled to the exchangeable -XH protons. This method should prove to be a useful assignment technique.

As an example, the results of a TOCSY experiment obtained for sucrose (structure shown; 16 mg in ~80% H<sub>2</sub>O/20% D<sub>2</sub>O with some methanol) with suppression of water with the WET sequence are shown in the Figure. The water trace in the contour map clearly shows correlations to all protons  $\alpha$  to -OH groups. Full details are reported elsewhere.



NMR Spectroscopist

Texas A&M University Department of Chemistry has an opening for an NMR spectroscopist to work in the departmental NMR facility. This position is responsible for the operation, maintenance, instruction, and service spectroscopy on an MSL-300 solid state spectrometer, user instruction and assistance on a variety of solution state NMR spectrometers (8 systems ranging from 200 to 500 MHz), LHe servicing on all the cryomagnets in the facility, and assisting users in the planning and interpretation of NMR experiments. The successful candidate will be a highly motivated individual with an advanced degree in chemistry, excellent communications skills, training in solid state NMR spectroscopy, and at least three years experience running a solid state NMR spectrometer. The starting salary is \$32,000 per year, commensurate with experience, and includes an excellent benefits package. This position is available immediately, and will remain open until filled. Applicants should send a resume and three references to Ms. Judy Ludwig, Department of Chemistry, Texas A&M University, College Station, TX 77843-3255. Refer to vacancy number 971200.

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## Postdoctoral Position Available

We have funding for a postdoctoral research assistant to work on a project in high-resolution liquid-phase NMR to study methods for simplifying proton spectra. A Ph.D. in chemistry or physics is required, together with proven experience in magnetic resonance methodology and computer programming. Please send a *curriculum vitae* and arrange to have two letters of recommendation sent to Professor Ray Freeman, Department of Chemistry, Lensfield Road, Cambridge CB2 1EW, United Kingdom. Cambridge is an Equal Opportunity Employer.

**THE**  
***NMR***  
**NEWSLETTER**

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 415-493-5971\* - Please call  
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**Deadline Dates**


No. 467 (Aug.) 25 July 1997

No. 468 (Sept.) 22 Aug. 1997

No. 469 (Oct.) 26 Sept. 1997


No. 470 (Nov.) 24 Oct. 1997

No. 471 (Dec.) 21 Nov. 1997

\* 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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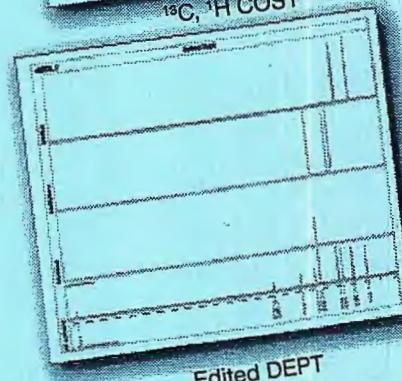
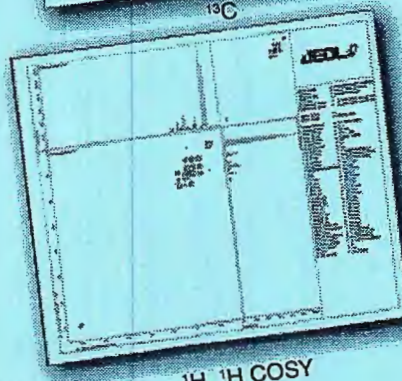
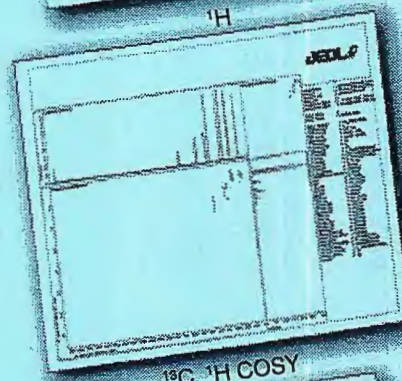
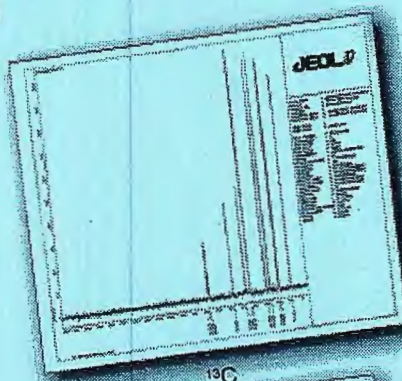
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