

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

**No. 469**  
**October 1997**

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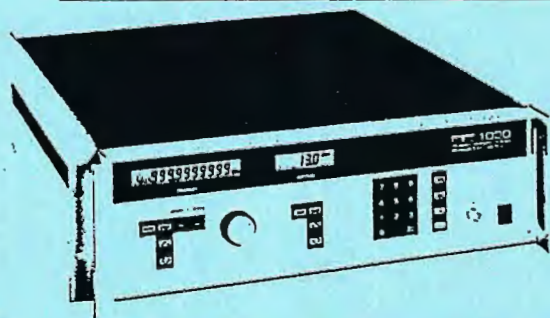




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Type 1							1 Hz resol., OCXO: \$6,425.00
PTS 310	.1-310 MHz	1 Hz	1-20 $\mu$ s	standard	3½"H×19"W	BCD (std) or GPIB (opt)	1 Hz resol., OCXO: \$5,850.00
Type 2							
PTS 500	1-500 MHz	optional .1 Hz to 100 KHz	1-20 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	\$8,720.00 (1 Hz resol., OCXO freq. std.)
PTS 620	1-620 MHz	optional .1 Hz to 100 KHz	1-20 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	\$9,625.00 (1 Hz resol., OCXO freq. std.)
PTS 1000	0.1-1000 MHz	optional .1 Hz to 100 KHz	5-10 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	\$11,830.00 (1 Hz resol., OCXO freq. std.)
PTS 3200	1-3200 MHz	1 Hz	1-20 $\mu$ s	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	\$14,850.00 (1 Hz resol., OCXO freq. std.)
PTS x10	user specified 10 MHz decade	1 Hz	1-5 $\mu$ s	standard	3½"H×19"W	BCD (std) or GPIB (opt)	\$3,000.00 (1 Hz resol., OCXO freq. std.)
PTS D310	two channels .1-310 MHz	.1 Hz	1-20 $\mu$ s	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	\$8,560.00 (1 Hz resol., OCXO freq. std.)
PTS D620	two channels 1-620 MHz	.1 Hz/.2 Hz	1-20 $\mu$ s	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	\$13,240.00 (.1 Hz/.2 Hz resol., OCXO freq. std.)



1 Switching Time is dependent on digit (decade) switched; see detailed instrument specifications.

2 For applicable digits, see detailed instrument specifications.

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### FORTHCOMING NMR MEETINGS

Missouri Magnetic Resonance Symposium (MMRS-VII), 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>. See Newsletter 467, 39.

Symposium "Magnetic Fields: Recent Advances in Diagnosis and Therapy", London, Ont., Canada, **November 14 - 16, 1997**. See Newsletter 468, 8.

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.

NATO ARW "Applications of NMR to the Study of Structure and Dynamics of Supramolecular Complexes", Sitges (Barcelona, Spain), **May 5 - 9, 1998**. Contact: Prof. M. Pons, Dept. Química Organica, Univ. de Barcelona, Mart I Franques 1, 08028 Barcelona, Spain; <http://www.ub.es/nato/nato.htm>; e-mail: miguel@guille.qo.ub.es

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.


Fifth International Conference on Heteroatom Chemistry, London, Ont., Canada, **July 5 - 10, 1998**. For details, see Newsletter 468, 40.

XIVth International Conference on Phosphorus Chemistry, Cincinnati, OH, **July 12 - 17, 1998**. For details, see Newsletter 468, 40.

Additional listings of meetings, etc., are invited.




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Worldwide

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

Wissembourg, August 20<sup>th</sup> 1997  
(received 9/2/97)

### PLAYING AROUND WITH RADIATION DAMPING AT 400 AND 800 MHz

Dear Barry,

We have recently tested the new Radiation Damping Compensation Unit (RDCU) developed in collaboration with Professor Jean-Yves Lallemand's group (Ecole Polytechnique Palaiseau) on the AVANCE 400 and 800 Mhz. It is well known that the precessing of a Magnetization of highly concentrated nuclei creates a field  $H_{RD}$  called the radiation damping field (also called FID field) as a consequence of a reaction of the detection circuit to the voltage generated in the detection coil by precessing magnetization [1]. This Field is perpendicular to the detected transverse Magnetization and is responsible of an additional "relaxation" phenomenon which is at the origin of some trouble in spectra of low concentrated biomolecules in water. In presence of natural radiation damping the water resonance flips back to it's equilibrium value much more quickly (near 150 ms and 50 ms respectively at 400 Mhz and 800 MHz in presence of natural radiation damping instead of 3.2 s if radiation damping is suppressed). This results in the shortening of the  $T_2$  value and the broadening of the water peak. The protons of a protein or a peptide which are involved in exchange and/or dipolar coupling processes with the water protons show a relaxation behaviour which is influenced by the water peak radiation damping relaxation process. This results in a 2D  $^1H$ - $^1H$  NOESY spectrum in a lowering of the intensities of the exchangeable protons peaks and of the biopolymer protons which show nOe's with the water protons. At higher fields (800 MHz) these peaks are much less intense as they should be in absence of radiation damping as is shown on figure 1. The intensities of the peaks of all this protons is restored when radiation damping is compensated.

The RDCU system is built as being possible to compensate the natural radiation damping or to enhance it. Thus the Radiation Damping Compensation Unit can operate during evolution, mixing and acquisition delays in 1D, 2D or 3D experiments. If we have a look at the behaviour of the exchangeable proton peak intensities as a function of the mixing time (figure 2) at 400 Mhz in presence of natural radiation damping, when radiation damping is compensated or enhanced, we can take advantage from this situation to record fast NOESY experiments by compensating/enhancing the natural Radiation Damping every two scans with a specific phase cycle (figure 3). Thus for small mixing times ( $\leq 50$  ms) only the protons which are involved in exchange processes with the water protons are observed.

[1] XI-AN Mao and CHAO-HUI Ye, *Concepts in Magnetic Resonance*, 1997, 173-187.

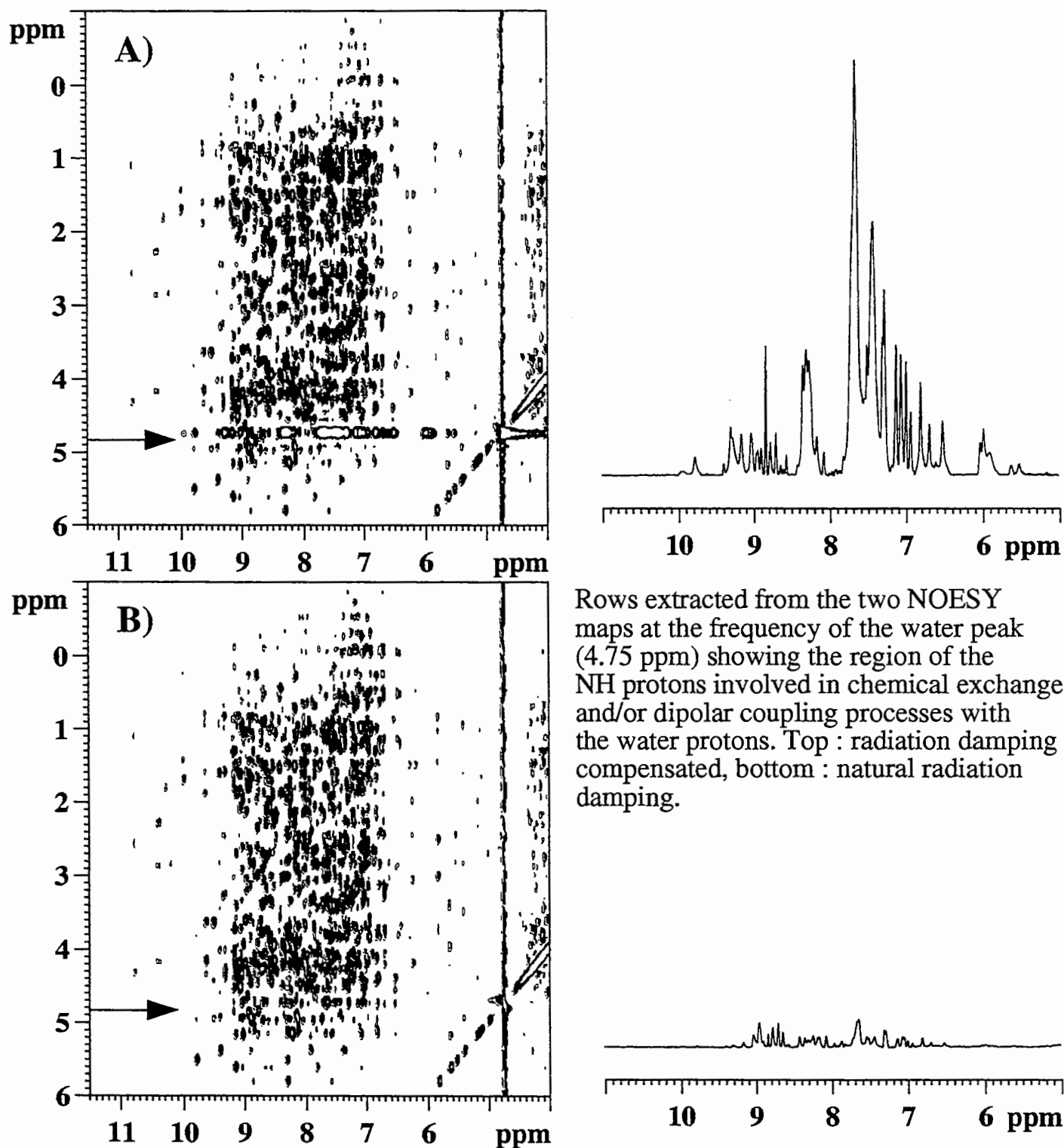


Figure 1 :  $^1\text{H}$ - $^1\text{H}$  NOESY maps recorded on the 2 mM Lysosyme sample in 90/10  $\text{H}_2\text{O}/\text{D}_2\text{O}$  (Mixing time = 150 ms) at 300 K and 800 MHz. Eight transients per 256 complex increments are taken in  $t_1$ . The water elimination is done with a WATERGATE sequence. Both maps have been processed in the same manner (F1, F2 Fourier transformation with a  $\pi/3$  shifted sinebell filter for both dimensions and no additional correction). A) The NH, CH $\alpha$  and CH $\beta$  region is shown when radiation damping is compensated during the mixing and evolution times, B) the same region when natural radiation damping occurs. Note the quasi perfect water peak elimination in both experiments.

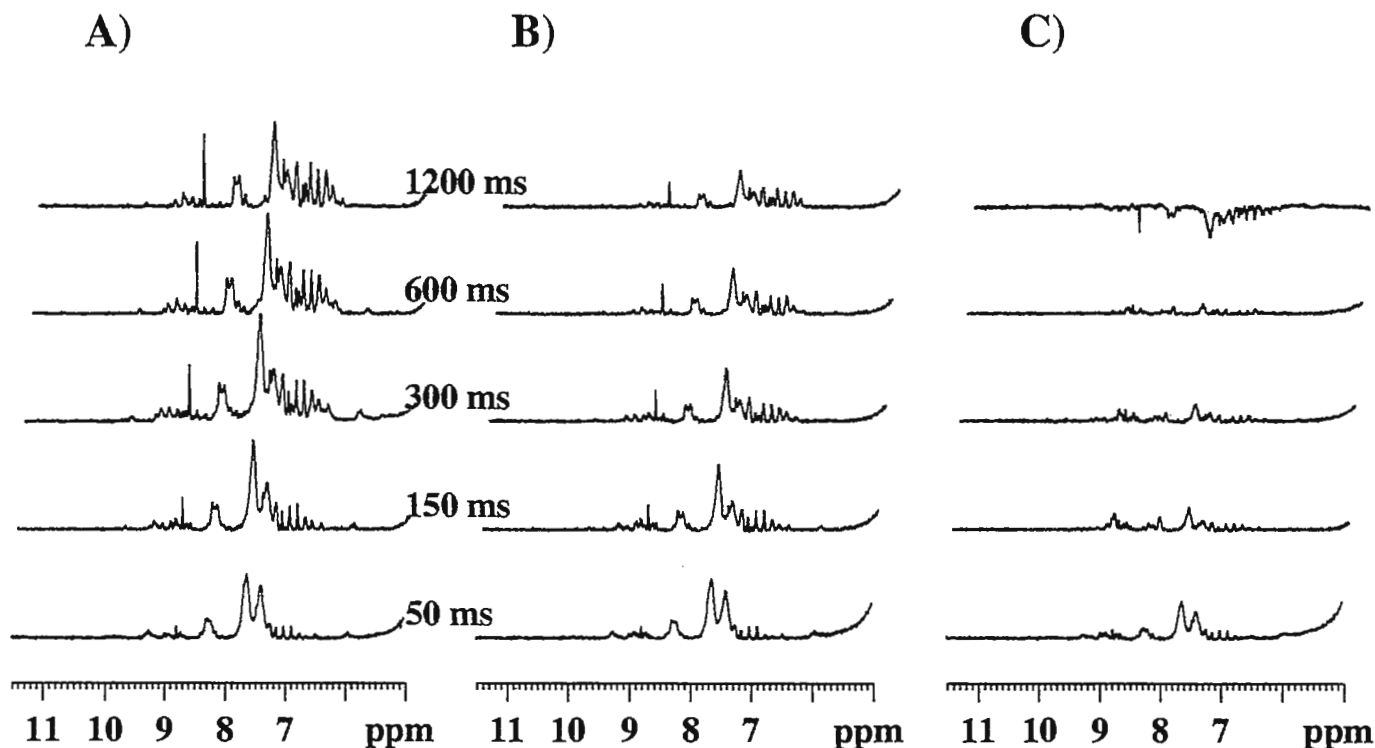


Figure 2 : Rows extracted from 2D  $^1\text{H}$ - $^1\text{H}$  NOESY maps recorded on the 2 mM Lysosyme sample in 90/10  $\text{H}_2\text{O}/\text{D}_2\text{O}$  at 300 K and 400 MHz at the frequency of the water peak (4.69 ppm). The exchangeable proton peak intensities are shown as function of the mixing time with A) the radiation damping is compensated during the evolution and mixing times, B) natural radiation damping occurs and C) the radiation damping is enhanced.

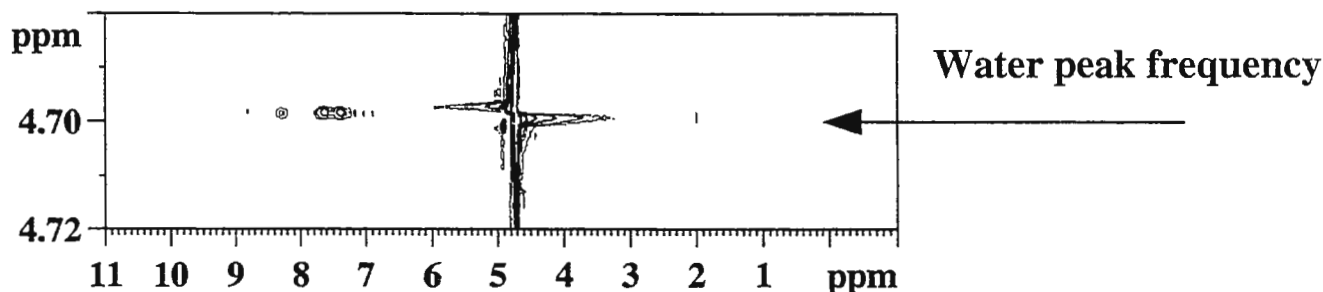


Figure 3 :  $^1\text{H}$ - $^1\text{H}$  fast NOESY map recorded on the 2 mM Lysosyme sample in 90/10  $\text{H}_2\text{O}/\text{D}_2\text{O}$  (Mixing time = 150 ms) at 300 K and 400 MHz. Full spectrum width in F2 but only 0.3 ppm in F1. Eight transients per 128 t1 increments. Radiation damping is alternatively compensated/enhanced every two scans. Only the protons involved in exchange and/or dipolar coupling processes with the water protons can be seen.

We are now looking for exciting new applications of our Radiation Damping Compensation Unit which should be commercially available end of september 97.

Sincerely Yours,

Dr. Philippe LUX

Dr. Christian BREVARD

Jean-Max TYBURN

# *Now what's happening to the future of solids NMR?*



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One thing hasn't changed: For more than 30 years, Bruker has remained strongly committed to solid state NMR, and today we are more committed than ever. Each generation of Bruker solids systems, from the original BKR to our latest AVANCE™ DSX, has provided superior capability, accessibility, and reliability for the fullest possible range of high power NMR experiments. Many of our unique solids features have even become classics, including pneumatic MAS sample insert/eject

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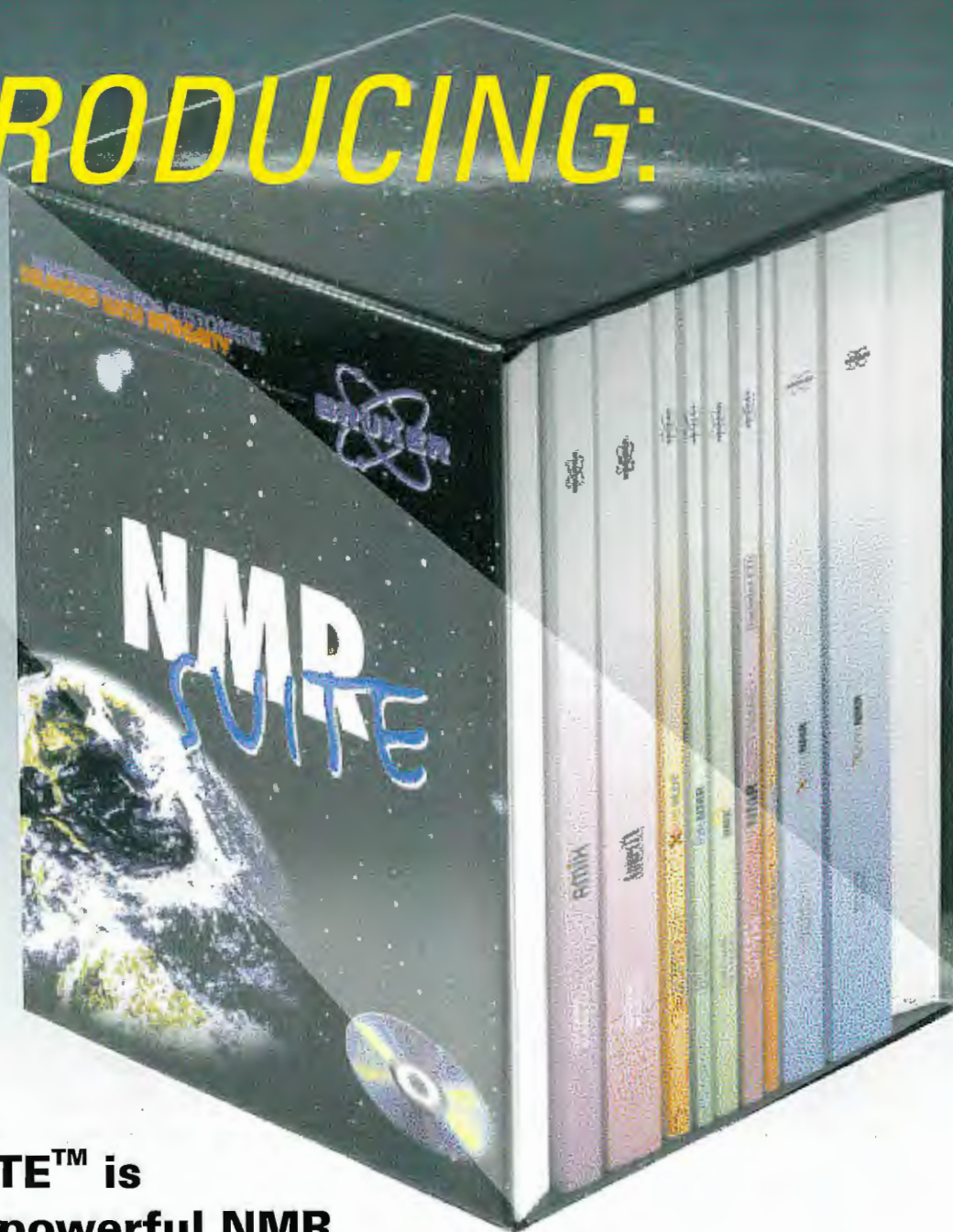


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## Boost Your Sensitivity 4X Today!

*Bruker's CryoProbe™ System Is Available Now for 400, 500, and 600MHz Spectrometers*

### BACKGROUND

Much press has been devoted in the last few years to the development of High Temperature Superconducting (HTS) materials. The advantage of using HTS materials in the construction of probe coils for NMR is a dramatic increase in the efficiency of detection. Bruker has been actively pursuing the use of HTS technology in probe coils for some time. And we're happy to report the results from our first commercial HTS CryoProbe. These probes are based on an application of HTS technology that has been recognized with a 1997 R&D 100 Award for Innovation.

### RESULTS

Spectra from Bruker's first CryoProbe show dramatic improvements in sensitivity in homonuclear experiments versus conventional probes operating at room temperature (Figure 1). The results show:

- 4X Increase in Sensitivity
- 16X Rise in Sample Throughput

If you have limited sample quantities or diminished sample solubility, these probes will be of particular interest to you as replacements for your current room temperature probe. The

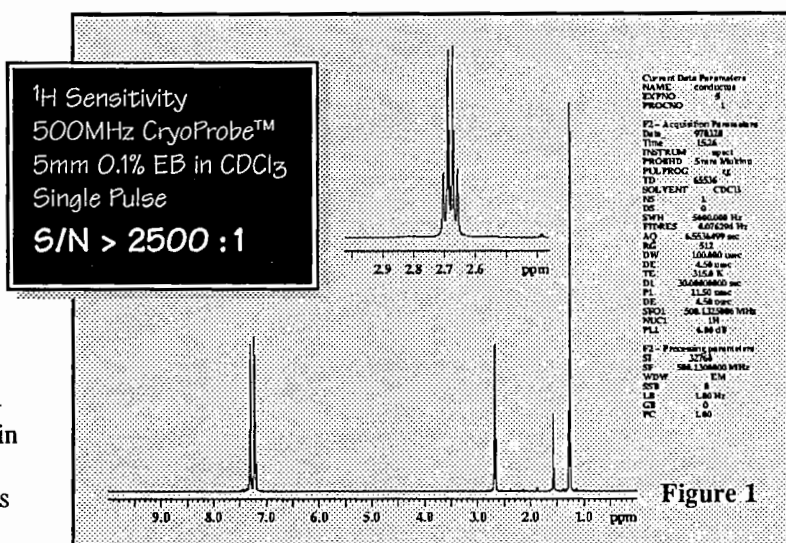


Figure 1

greatest improvements have been obtained with polymeric and biological samples dissolved in low-dielectric organic solvents, such as CDCl<sub>3</sub> or Acetone-d<sub>6</sub>.

When combined with a sample changer, a single acquisition that required a weekend can now be combined with others in unattended overnight runs (closed-cycle system, see below).

The performance characteristics of these probes is excellent. Demanding homonuclear 2-D experiments such as NOESY and TOCSY and pulse sequences requiring solvent suppression are easily accessible. Sample concentrations ≤100nmol can be examined routinely using Bruker's CryoProbe.





# ...The NMR <sup>Re</sup>volution advances

## HARDWARE

<sup>1</sup>H and <sup>19</sup>F homonuclear CryoProbes and a <sup>13</sup>C Observe / <sup>1</sup>H Decouple CryoProbe are now available for Bruker 400, 500, and 600MHz standard bore spectrometers equipped with low-noise (LNA) preamplifiers. All probes are equipped with Deuterium lock.

There are two systems for cooling the thin-film superconducting R<sub>f</sub> coils of the Bruker CryoProbes™ to the required 25K:

- Open-Cycle System - a low cost that consumes approximately 1 liter of high purity liquid Helium per hour from a dewar.

- Closed-Cycle System - a premium that provides extended periods of unattended use. The addition of a refrigeration unit that requires a small amount of lab space allows cryogen-free operation.

Cool-down times following probe installation are approximately 1 hour or less. Sample temperatures are easily maintained between 0 and 60°C using a standard VT System of your Bruker spectrometer.

## Bruker CryoProbe Performance Specifications

Probe	Sensitivity	Non-Spinning Lineshape (50, 0.55, 0.11% Peak Height)	Decoupling Power
<sup>1</sup> H - 400MHz	2000 : 1	1 / 10 / 20Hz	
<sup>1</sup> H - 500MHz	2500 : 1	1 / 10 / 20Hz	
<sup>1</sup> H - 600MHz	3000 : 1	1 / 10 / 20Hz	
<sup>19</sup> F - 400MHz	2000 : 1		
<sup>19</sup> F - 500MHz	2800 : 1		
<sup>19</sup> F - 600MHz	3300 : 1		
<sup>13</sup> C - 400MHz	600 : 1		≤25μs 90° pulse (f <sub>DEC</sub> ≥10KHz) @ 50W
<sup>13</sup> C - 500MHz	750 : 1		≤25μs 90° pulse (f <sub>DEC</sub> ≥10KHz) @ 50W
<sup>13</sup> C - 600MHz	900 : 1		≤25μs 90° pulse (f <sub>DEC</sub> ≥10KHz) @ 50W

**Bruker  
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Inc.**

Fortune Drive  
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September 24, 1997  
(received 9/25/97)

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

**Re:    *Bruker Acquires Conductus HTS Technology***

Dear Barry:

On August 5, 1997, Bruker Instruments, Inc. acquired the product rights and associated assets of the NMR spectroscopy and NMR microscopy probe business from Conductus, Inc. of Sunnyvale, CA, a leading developer and manufacturer of high-temperature superconducting (HTS) electronics. Key Conductus personnel associated with NMR probe development have joined Bruker as regular employees at Bruker's Fremont, CA facility. Conductus will continue to manufacture HTS coils for the NMR probes and the coils will be sold to Bruker on an OEM basis for incorporation into HTS probes.

Cryogenic HTS probes will open up new fields of application and new markets for NMR spectroscopy and microscopy. Bruker is committed to accelerate the commercial availability of this revolutionary technology as part of the Bruker CryoProbe™ product line, and Bruker will also continue research and development on cryogenic HTS NMR probes.

The Conductus rights acquisition compliments existing Bruker efforts in HTS technology. The result is immediate availability in the Bruker CryoProbe product line of  $^1\text{H}$  or  $^{19}\text{F}$  homonuclear 5mm probes and  $^{13}\text{C}$  observe,  $^1\text{H}$  decouple 5mm probes for 400, 500, and 600MHz spectrometers.

Regards:

Mark Chaykovsky  
Director of Marketing and Sales  
Analytical NMR

**Biophysical Faculty Position  
The University of Texas Medical Branch  
Galveston, Texas**

In continuing development of Structural Biology at the University of Texas Medical Branch, we are pleased to announce a tenure-track faculty opening at the Assistant Professor level.

***Nuclear Magnetic Resonance Spectroscopy - We seek candidates with strong backgrounds in NMR spectroscopy who make extensive use of NMR to solve important biological problems. The successful candidate is to establish an independent research program that may involve determination of macromolecular structures and/or innovative application of NMR spectroscopy in the study of biological macromolecules. The NMR facility at UTMB consists of new 400, 600 and 750 MHz Varian Spectrometers housed in a newly renovated building.***

Candidates must have outstanding potential in research with a record and stature in their field of expertise. They are expected to attract funding from national agencies and to fully participate in teaching and service with the Department of Human Biological Chemistry and Genetics. Affiliations with The Sealy Center for Structural Biology, other UTMB Centers and relevant Departments are possible.

Structural Biology currently consists of 10 biophysical faculty members in areas of NMR spectroscopy, x-ray crystallography, computational biology, and biophysical chemistry with facilities directed by Drs. David Gorenstein, Werner Braun, Robert O. Fox and James Lee, respectively. Structural Biology received financial support from UTMB and endowed support from the Sealy Center for Structural Biology. Facilities consist of a newly renovated building and additional laboratories, along with excellent computational, NMR, crystallographic, and biophysical equipment.

Applications should include a complete curriculum vitae, a description of research interests and names and addresses of at least three references. Review of applications will begin October 1 and continue until the position is filled. Applications should be addressed to:

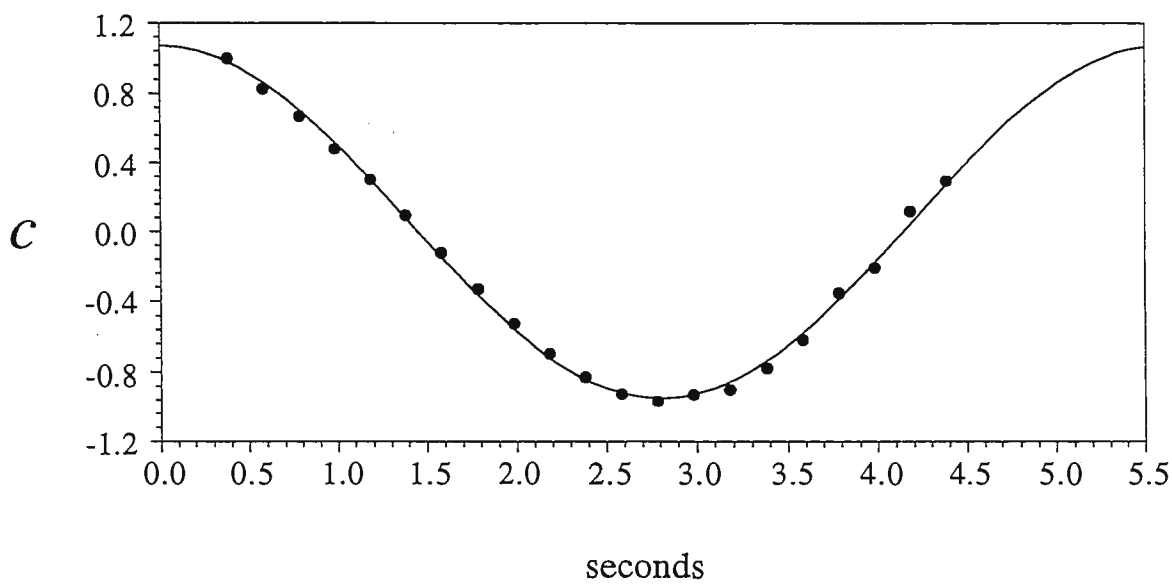
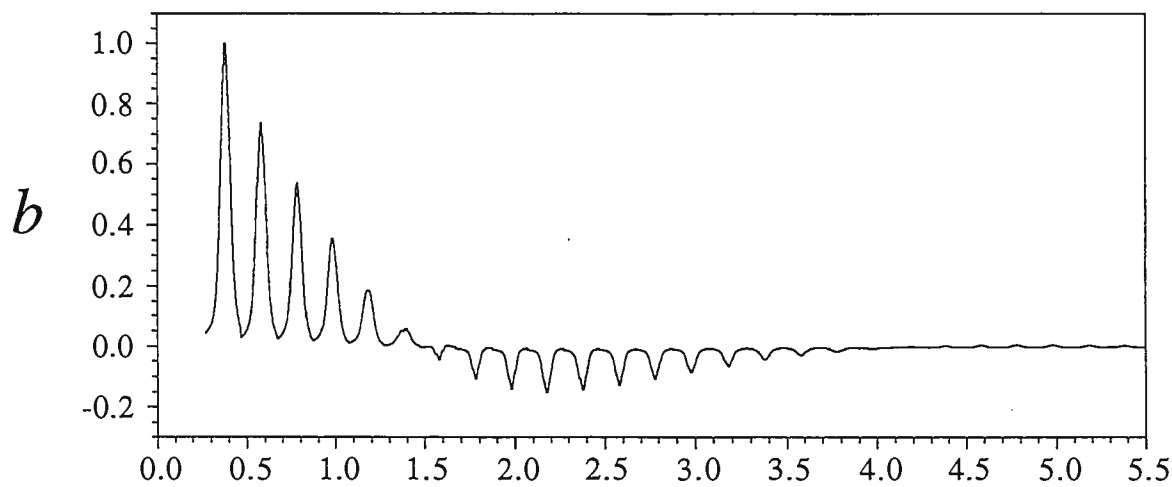
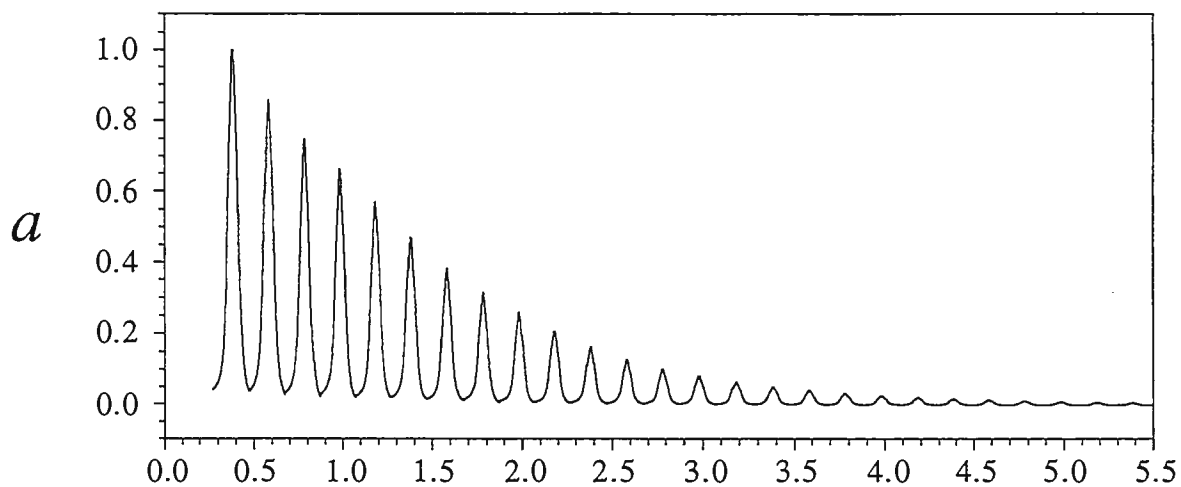
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- (1) E. L. Hahn and D. E. Maxwell, Phys. Rev. **88**, 1070 (1952).
- (2) R. Freeman and H. D. W. Hill, J. Chem. Phys. **54**, 301 (1971).
- (3) H. Geen and R. Freeman, J. Magn. Reson. **93**, 93 (1991).

## Three Little Burps (page 2)



# Technical Specifications

## 200 - 600MHz Narrow Bore NMR Magnet Systems

Oxford Instruments, NMR Instruments are the pioneers of superconducting magnet technology for NMR spectroscopy and are widely regarded by the world's 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							
Magnet	200/54		300/54		400/54	500/51		600/51
Operating Field (Tesla)	4.7		7		9.4	11.7		14
NMR Operating Frequency (MHz <sup>1</sup> H)	200		300		400	500		600
Field Stability (Hz/hour <sup>1</sup> H)	<2		<3		<8	<10		<10
5 Gauss Stray Field Contour						Standard	Shielded	Standard
								Shielded
Axial (Metres)	1.75		2.2		2.8	3.5	1.8	4.0
Radial (Metres)	1.5		1.7		2.2	2.8	1.3	3.2
								1.75
Cryostat	Standard	Compact	Standard	Compact				
Standard Cryostat Minimum Helium Refill Interval (Days)	235	80	235	80	183	150		150
Standard Cryostat Helium Refill Volume (Litres)	79	26	79	26	62	52		80
Year Hold Cryostat Option Available	✓	X	✓	X	✓	X		X
Nitrogen Refill Interval (Days)	14	14	14	14	14	17		18
Nitrogen Refill Volume (Litres)	61	32	61	32	61	84		131
Nominal Room Temperature Bore Diameter (mm)	54	54	54	54	54	51		51
Minimum Operational Ceiling Height (Metres)	2.9	2.5	2.9	2.5	2.9	3.1		3.4
System Weight (kg) Including Cryogen's	315	120	325	133	380	533		1100

## 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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France  
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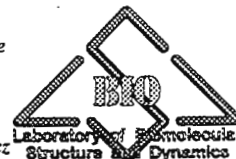
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 fax. \*\*420-5-41129506  
 e-mail: {fiala,sklenar}@chemi.muni.cz  
 (received 9/10/97)



## Automatic $^1\text{H}$ , $^{13}\text{C}$ , $^{15}\text{N}$ and $^{31}\text{P}$ referencing

With the rapidly growing database of reported  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{15}\text{N}$  chemical shifts of biomolecules in recent years, we have witnessed a renewed interest in using this type of information in the structure determination. A prerequisite of any meaningful study utilizing chemical shift is that the shifts are reported accurately and consistently. Based on careful measurements of absolute frequencies for various standards, Wishart et al. [1] proposed standard frequencies for indirect referencing of  $^{13}\text{C}$  and  $^{15}\text{N}$  chemical shifts.

To facilitate the referencing in our laboratory, we have developed a set of programs that reference proton spectra based on HDO resonance and  $^{13}\text{C}$ ,  $^{15}\text{N}$  or  $^{31}\text{P}$  in indirect dimensions of 2D or 3D spectra. All the calculations are based on data from [1] except for the  $^{31}\text{P}/^1\text{H}$  ratio, which was measured by Dr. David Live as 0.404808688 [personal communication]. The proton reference is corrected for temperature, pH and salt concentration dependence. The reference compounds for 0 ppm are DSS, DSS, liquid ammonia and TMP for  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$  and  $^{31}\text{P}$ , respectively. The reference routines exist in the versions for the current Bruker and Varian spectrometers and are available free of charge for any non-commercial use from our website [2]. Because of the different philosophy of Bruker and Varian software, as well as different history of development, the Bruker and Varian routines differ somewhat. Therefore, their operation will be described separately in the following paragraphs. For more detail information, please refer to README files on our website.

The Bruker reference package consists of four *au* programs that have to be placed in the appropriate *au/src* directory and compiled to become operational. The programs are:  
*ref* - the control module; it calls the program *wref* for proton reference and in the case of 2D or 3D spectra calls corresponding programs (*deceref\_2D*, *deceref\_3D*) to reference the indirect dimensions.  
*wref* - interactively asks the operator for the information on the pH and salinity of the sample and references proton in the direct dimension. If the spectrum is not centered on the water resonance, the program suggests the chemical shift for the water line and waits for the operator to reference it before proceeding further.

*deceref\_2D* - references  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$  or  $^{31}\text{P}$  in the indirect dimension of a 2D spectrum. The reference frequencies are derived from the reference in the directly detected dimension, which is assumed to be a proton spectrum. *deceref\_2D* can be called directly by the operator without using *ref* provided the direct dimension has already been referenced. If the nucleus in the indirect dimension is  $^1\text{H}$ , the program asks the operator whether the center of the spectrum in this dimension is the same as in the direct one. In the case of a shift, the program requests its value in Hz.

*deceref\_3D* - references  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$  or  $^{31}\text{P}$  in the indirect dimensions of a 3D spectrum. It applies the same procedure as *deceref\_2D* independently to each of the indirect dimensions.

The programs recognize and correctly reference strip Fourier transformed spectra.

The Varian package consists of five macros that have to be placed in the appropriate *maclib* directory so that the *vnmr* program can locate them. There is no "control" module and it is up to the spectrometer operator to call the macros in the appropriate order. The individual macros are:

*wref* - references proton in the direct dimension. The sample pH and salt concentration in mM have to be supplied as parameters in brackets: *wref*( pH, salt concentration ) . The macro assumes that the water resonance is at the position of the cursor. Therefore, to get a correct referencing the cursor has to be placed on the water line prior calling the macro.

*decreef* - references the nucleus ( $^{13}\text{C}$ ,  $^{15}\text{N}$  or  $^{31}\text{P}$ ) from channel 2 (i.e. decoupler 1) in the (first) indirect dimension.

*dec2ref* - references the nucleus ( $^{13}\text{C}$ ,  $^{15}\text{N}$  or  $^{31}\text{P}$ ) from channel 3 (i.e. decoupler 2) in the (first) indirect dimension.

*decreeff2* - references the nucleus ( $^{13}\text{C}$ ,  $^{15}\text{N}$  or  $^{31}\text{P}$ ) from channel 2 (i.e. decoupler 1) in the second indirect dimension.

*dec2reff2* - references the nucleus ( $^{13}\text{C}$ ,  $^{15}\text{N}$  or  $^{31}\text{P}$ ) from channel 3 (i.e. decoupler 2) in the second indirect dimension.

Again, the reference frequencies for heteronuclei are derived from the proton frequency. Therefore the direct dimension has to be referenced before calling the macros for referencing the indirect dimensions. Please note that the Varian macros cannot reference proton in the indirect dimensions. The macros check for *lsfrq1* and *lsfrq2* parameters and produce a correct reference even if the spectrum was shifted during processing.

For the referencing routines to produce correct results, it is necessary that the parameters should be set to the values the data were collected with. If you suspect an error, please check the temperature and frequencies in all channels first.

- [1] Wishart, D. S., Bigam, C. G., Yao, J., Abildgaard, F., Dyson, H. J., Oldfield, E., Markley, J. L., Sykes, B. D. *J. Biomol. NMR* (1995), **6**, 135-140.
- [2] <http://www.chemi.muni.cz/nmr/nmr.html>



Radovan Fiala



Vladimír Sklenář

### Position Available in NMR Applications and Sales

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09/26/97

**Magnetic Field Density Functional Studies of NMR Parameters**

Dear Colleagues,

(received 9/29/97)

Work is being carried out in the laboratory of Professor Alexander Pines using hyperpolarized xenon as a probe atom and SPINOEs (Spin Polarization-Induced Nuclear Overhauser Effect) to increase sensitivity in NMR experiments<sup>[1]</sup>. This has lead to an interest in developing new methods for the calculation of the chemical shifts and J couplings of xenon attached to a variety of systems especially van der Waals complexes. A few years ago, a new type of density functional theory of magnetic responses was constructed<sup>[2]</sup>, known as Magnetic Field Density Functional Theory (BDFT). It was suggested that both the calculation and interpretation of NMR parameters in terms of molecular quantities would be simpler in this new theory than in extant theories.

Recently, in collaboration with Professor Pines, an effort has been mounted to bring BDFT to fruition. In work which will shortly appear in the literature, we established a practical framework from which one can calculate chemical shifts and magnetic susceptibilities<sup>[3]</sup>. We have applied our method, with some success, to the determination of hydrogen chemical shieldings in a variety of molecules<sup>[4]</sup>. While our method is still under development, we believe it will be useful in calculations of xenon chemical shifts and J couplings. To this end we are calculating the contact J coupling between Xe and a hydrogen in a cage molecule. In applying our new method, we have deviated from the framework mentioned above used to calculate molecular chemical shieldings. The method is a Gordon-Kim type theory<sup>[5]</sup> which is designed for weakly bound systems. It has been used, with some success, in the determination of both electric and orbital magnetic responses in weakly coupled complexes<sup>[6,7]</sup>.

The inputs are the electron densities of the separated atoms, their Fermi contact interaction-induced spin densities, and a simple Thomas-Fermi-Dirac spin density functional. For the perturbation due to the Fermi contact interaction we have taken the one electron theory of Harris and Pitzer<sup>[8]</sup> and extended it to arbitrary atomic number and quantum number.

In order to check our new method we first used a simple molecular orbital estimate of the J coupling. The result, believed to be accurate to an order of magnitude, was estimated at around a few  $\mu\text{Hz}$ . We are currently running calculations using this method and soon we will be able to verify our molecular orbital estimates. If there is theoretical confirmation we shall begin estimating the J couplings in other xenon van der Waals complexes and also we shall test more sophisticated energy functionals.



Sincerely,

  
Fred Salsbury

  
Robert Harris

- [1] Y.-Q. Song, B.M. Goodson, and others, *Angew. Chem. Int. Ed. Engl.*, in press.
- [2] C. J. Grayce, and R.A. Harris, *Phys. Rev. A*, 50 (1994) 3089.
- [3] F.R. Salsbury, and R.A. Harris, *J. Chem. Phys.*, 107 (Nov 8 1997).
- [4] F.R. Salsbury, and R.A. Harris, *Chem. Phys. Lett.*, in press
- [5] R.G. Gordon, and Y. S. Kim, *J. Chem. Phys.*, 56 (1972) 3122.
- [6] R. A. Harris, D. F. Heller, and W. M. Gelbart, *J. Chem. Phys.*, 61 (1974) 3854.
- [7] C.J. Grayce, and R.A. Harris, *Mol. Phys.*, 72 (1991) 523.
- [8] R. A. Harris, and R. M. Pitzer, *Phys. Rev. A*, 38 (1988) 3104.



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Applications with curriculum vitae and the names of two references should be sent to:

Dr J. Haverkamp or Dr J.P.M. van Duijnhoven  
Unilever Research Laboratory Vlaardingen, P.O. Box 114, 3130 AC Vlaardingen, The Netherlands, Tel: +31 10 460 5150; fax: +31 10 460 5671.

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

July 8, 1997  
 (received 9/4/97)

## Modification of the rotor drive ring for the Nano•nmr probe

Dear Dr. Shapiro,

The Varian Nano•nmr probes can be used to obtain high-resolution spectra on samples bound to solid-phase-synthesis resins.<sup>1-3</sup> We are using a <sup>1</sup>H Nano•nmr probe for the development of solid-phase synthesis in combinatorial chemistry. To fix it's position, the 40 µl sample tube used with the Nano•nmr probe is inserted into a rotor drive ring, which is then glued onto the tube. The idea to glue the rotor drive ring onto the tube is tedious and some users prefer not to use any glue at all. The problem now is that you need to find a drive ring that perfectly matches your sample tube so that the tube does not move from the optimal position or, in the worst case, slides out of the drive ring into the probe interior; this has happened in our experience.

To eliminate this problem we decided to try to machine a groove inside the rotor drive ring and insert an O-ring to grip the sample tube. After some experiments with the groove diameter, we have successfully modified our drive rings as follows:

We chose a standard Viton O-ring Ø 4.0 x 1.5 / FPM 80.

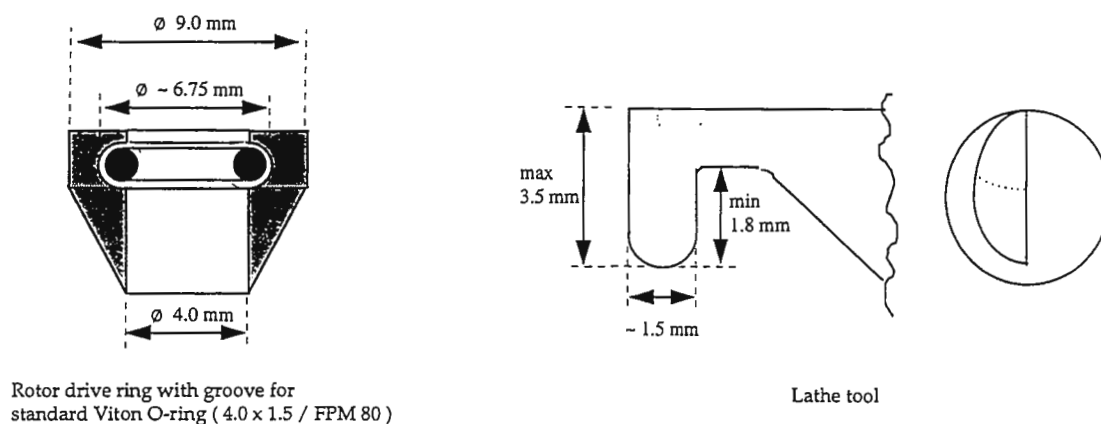
A special lathe tool with neutral cutting angle was cut from a Ø 4 mm HSS rod.

The drive ring must be held in a Ø 9.0 mm clamping sleeve - a regular lathe chuck will deform the drive ring. The drive ring diameter might shrink if excessive force is applied to the clamping sleeve.

Cutting the groove should be performed carefully; due to the small dimensions and almost no clearance it is impossible to see what is really happening. During the cutting we blew the shavings (rather like a powder) away with compressed air. The groove diameter should be 6.75 mm and the width must be 0.1-0.2 mm wider than the O-ring.

Mounting the O-ring requires some patience. It is important that the inner surface of the drive ring is not scratched and the O-ring is not twisted.

Figure 1. Rotor drive ring modification and lathe tool.



The modified drive rings are just as easy to use with the 40  $\mu$ l sample tubes as are the standard spinners for ordinary 5 mm NMR tubes. Our modification did not affect the spinning performance.

Another problem with the solid-phase resins used in combinatorial chemistry concerns the filling and emptying of sample tubes. The resin is often difficult to wash out from the Nano•nmr sample tube after analysis. To facilitate cleaning the tubes we use a cannula with a U-bend, and a syringe held over a beaker. A suitable cannula is "21G x 3 1/4 (0.80 x 80 mm)" (disposable needle) where the tip is filed flat and the edge smoothly rounded. Resins used for solid-phase chemistry have been successfully removed with this simple method.

Filling the Nano vials with dry resin can be performed easily with a thin-walled piece of tubing (i.e.  $\varnothing$  1.40 / 1.0 mm) and an internal plunger.

1. P. A. Keifer, L. Baltusis, D. M. Rice, A. A. Tymiak, J. N. Schoolery, *J. Magn. Res. A*, **119**, 65-75, (1996).
2. P.A. Keifer, *J. Org. Chem.*, **61**, 1558-1559 (1996).
3. W. L. Fitch, G. Detre, C. P. Holmes, J. N. Schoolery, P. A. Keifer, *J. Org. Chem.*, **59**, 7955-7956 (1994).

Sincerely yours,

*Torbjörn Halvarsson*  
Torbjörn Halvarsson

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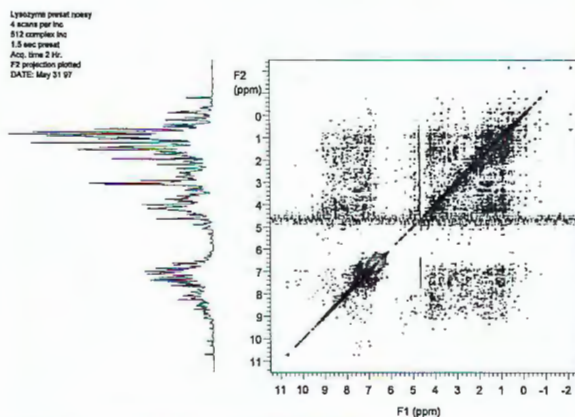
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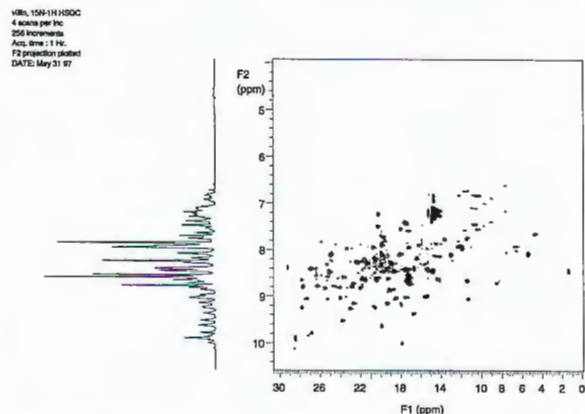
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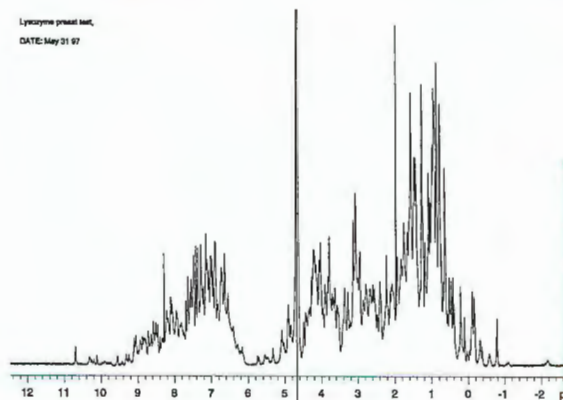
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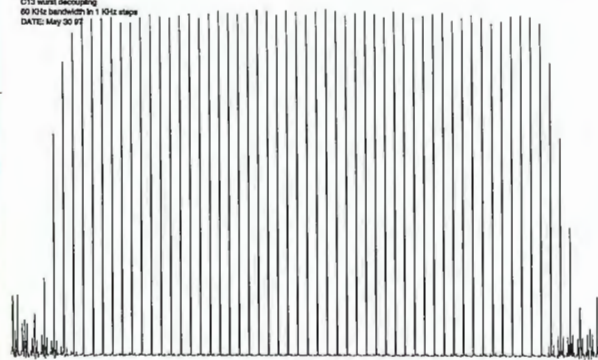


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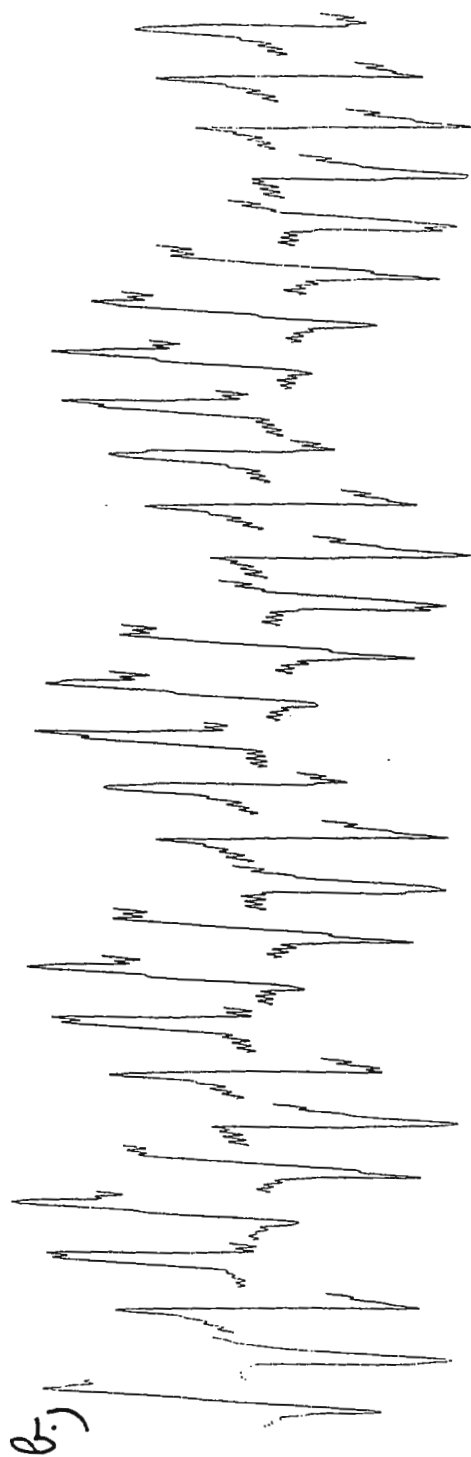
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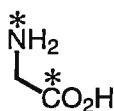
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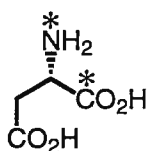






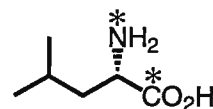
**Glycine-1-<sup>13</sup>C, <sup>15</sup>N**

>99 atom % <sup>13</sup>C  
>99 atom % <sup>15</sup>N



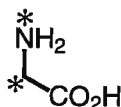
**L-Aspartic Acid-1-<sup>13</sup>C, <sup>15</sup>N**

>99 atom % <sup>13</sup>C  
>99 atom % <sup>15</sup>N



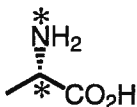
**L-Leucine-1-<sup>13</sup>C, <sup>15</sup>N**

>99 atom % <sup>13</sup>C  
>99 atom % <sup>15</sup>N



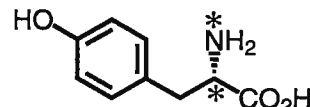
**Glycine-2-<sup>13</sup>C, <sup>15</sup>N**

>99 atom % <sup>13</sup>C  
>99 atom % <sup>15</sup>N



**L-Alanine-2-<sup>13</sup>C, <sup>15</sup>N**

>99 atom % <sup>13</sup>C  
>99 atom % <sup>15</sup>N



**L-4-Hydroxyphenylalanine-2-<sup>13</sup>C, <sup>15</sup>N**

>99 atom % <sup>13</sup>C  
>99 atom % <sup>15</sup>N

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Dr. B. L. Shapiro  
NMR Newsletter  
996 Elsinore Court  
Parlo Alto, CA 94303

Phone: +886-2-3668287  
E-mail: nmra@po.iam.s.sinica.edu.tw

September 8, 1997  
(received 9/13/97)

Dear Barry,

**Characterization of the Benzene Molecule Adsorbed in Faujasite Zeolite Using  
Double Quantum Filtered NMR Spectral Analysis<sup>†</sup>**

We have been interested in using  $^2\text{D}$  double quantum filtered NMR (DQF) spectroscopy<sup>1</sup> to characterize the benzene adsorption in zeolite. The pulse program is shown in Fig. 1. In a previous letter, we reported the  $\text{C}_6\text{D}_6/\text{USY}$  system and currently we are examining the other faujasite system. The DQF spectra of  $\text{C}_6\text{D}_6/\text{NaY}$  and  $\text{C}_6\text{D}_6/\text{USY}$  systems are shown in Fig. 2.

In accordance with simulation, the fine structure of the wiggling peaks shown in the DQF spectrum of  $\text{C}_6\text{D}_6/\text{USY}$  system indicates the orderly directed residual quadrupolar interaction in  $\text{C}_6\text{D}_6/\text{USY}$  zeolite due to the adsorption between the benzene ring and aluminum hydroxyl group in USY zeolite. In  $\text{C}_6\text{D}_6/\text{NaY}$  the simple pattern appeared in DQF spectra is attributed to a much less orderly adsorption interactions for benzene adsorbed among tetrahedral arranged  $\text{S}_{\text{II}}$  and 12-ring window sites. The conclusion is consistent with the observation from IR studies<sup>2</sup>.

We believe this DQF NMR spectroscopy analysis can be applied to the detection of the site distribution, adsorption strength and adsorbate dynamics. DQF spectral analysis along with the usual relaxation studies may be a better combination of NMR method to characterize the zeolite systems.

Sincerely Yours,

Mei-Po Suen

Yu-Huei Chen

Lian-Pin Hwang  
Professor of Chemistry

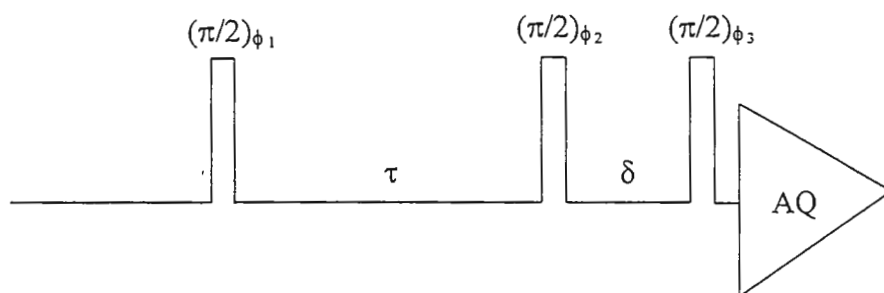


Fig. 1 The pulse program for  $^2\text{D}$  DQF NMR spectroscopy.  $\tau$  is creation time for generating DQ and  $\delta=10\text{ }\mu\text{s}$  is used in the experiments..

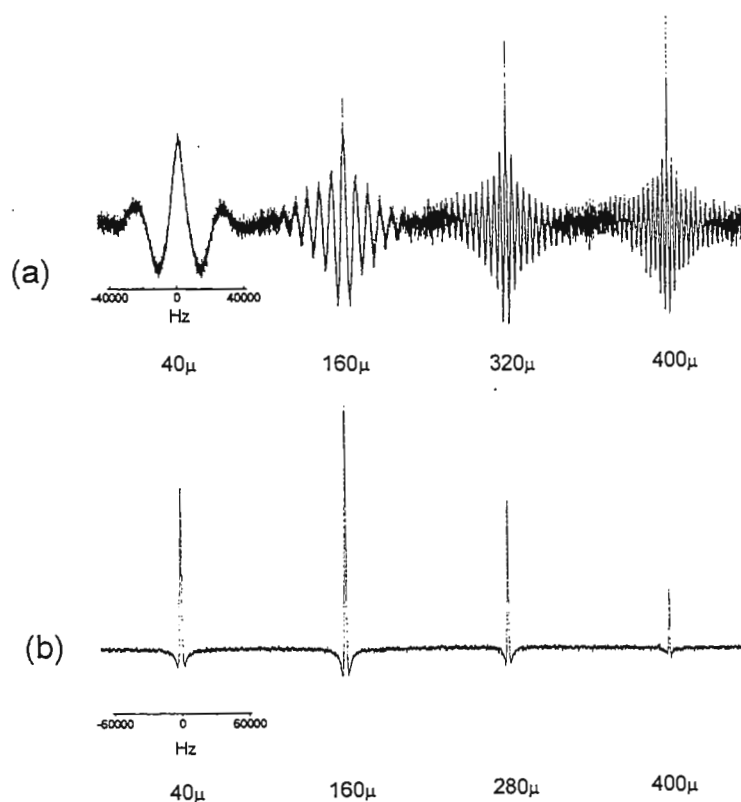


Fig. 2 DQF spectra of  $\text{C}_6\text{D}_6/\text{USY}$ (a) and  $\text{C}_6\text{D}_6/\text{NaY}$  (b). Various creation time  $\tau$  are cited below the spectrum.

#### Reference

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2. B.-L. Su, *J. Chem. Soc. Faraday Trans.*, 1997, **93**, 1449-1457.

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

September 25, 1997

Dear Barry:

CSA in proteins

We all know how exquisitely sensitive chemical shifts are to local conformation. When looking at a protein, for example, even a very conservative mutation will cause minute changes in its structure which are readily apparent from changes in chemical shift. Unfortunately, a thorough quantitative understanding of all factors influencing chemical shifts is still lacking. For proteins, an empirical correlation between the  $^{13}\text{C}^\alpha$  chemical shift and the polypeptide backbone torsion angles  $\phi$  and  $\psi$  accounts for most of the variation.<sup>1</sup> However, additional factors contribute an rms value of 1 ppm too. Oldfield and co-workers showed that *ab initio*  $^{13}\text{C}^\alpha$  shift calculations (using conformational information from a high resolution crystal structure) also agree to within about 1 ppm rms with experimental data.<sup>2</sup> Clearly, it would be desirable to get a better grip on what this residual rms deviation of 1 ppm is caused by. One approach to obtain more insight in the basis of the correlation between structure and chemical shift is to monitor the chemical shift anisotropy. In liquids this is easily done by measuring relaxation interference between the CSA tensor and a large one-bond dipolar interaction. We previously used this approach to get information on the  $^{15}\text{N}$  CSA tensor in peptide bonds,<sup>3</sup> whereas Batta et al. studied the effect for the  $^{13}\text{C}$  resonances in carbohydrates.<sup>4</sup>

Relaxation interference between the CSA tensor and the dipolar coupling tensor causes the two  $^{13}\text{C}\{-^1\text{H}\}$  or  $^{15}\text{N}\{-^1\text{H}\}$  doublet components to relax at different rates. The effect has long been known and is the subject of many dozens of papers. A particularly lucid description has been given by Goldman.<sup>5</sup> The difference in relaxation rate for the two doublet components is proportional to the size of the dipolar interaction, and to the difference in shielding ( $\sigma_{\text{par}} - \sigma_{\text{perp}}$ ) parallel ( $\sigma_{\text{zz}}$ ) and orthogonal ( $(\sigma_{\text{xx}} + \sigma_{\text{yy}})/2$ ) to the bond vector, which differs by a factor 3/2 from  $\sigma_{\text{par}} - \sigma_{\text{isotropic}}$ .

Figure 1 shows the cross-correlation effect on the  $^{15}\text{N}$  doublet components of several of the backbone amide nitrogens in the protein ubiquitin. The spectrum was recorded with a constant-time  $^{15}\text{N}$  evolution period of 100 ms, and if the relaxation rates of the two doublet components are given by  $\lambda \pm \eta$ , the ratio of the doublet component intensities equals  $\exp(2\eta/100 \text{ ms})$ . If the  $^{15}\text{N}$  CSA tensor is assumed to be axially symmetric, with its symmetry axis making an angle  $\theta$  with the N-H bond, the relaxation interference effect is proportional to  $S^2(3\cos^2\theta - 1)/2$ , where  $S^2$  is the generalized order parameter, accounting for fast internal motions. Values for  $\text{CSA}_{\text{red}} = (\sigma_{\parallel} - \sigma_{\perp}) S^2(3\cos^2\theta - 1)/2$  obtained in this manner are in quite good agreement with those reported earlier<sup>3</sup> on the basis of a different relaxation interference experiment (Figure 2). The correlation between the  $\text{CSA}_{\text{red}}$  values and the isotropic  $^{15}\text{N}$  shifts indicates that, on average, an upfield change in  $\sigma_{\parallel}$

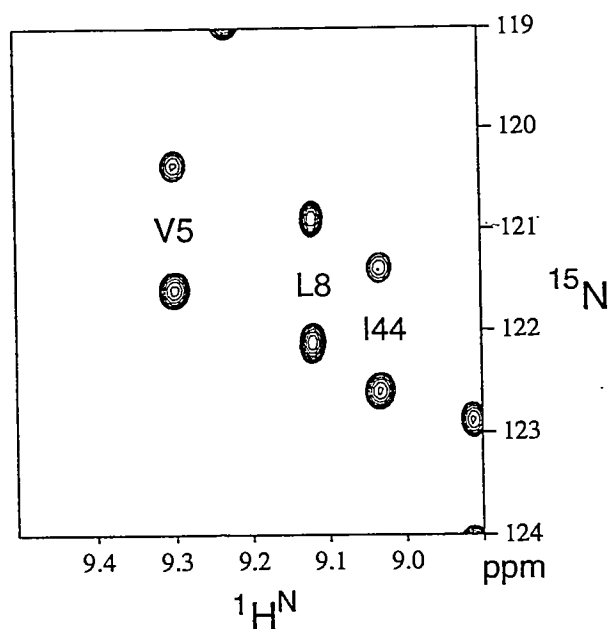


Figure 1. Small region of the constant-time  $^1\text{H}$ - $^{15}\text{N}$  correlation spectrum of ubiquitin,  $^1\text{H}$ -coupled in the  $F_1$  dimension. The spectrum was recorded at 750 MHz  $^1\text{H}$  frequency, with a  $^{15}\text{N}$  constant time duration of 100 ms. The ratio of the doublet component intensities is due to relaxation interference between the CSA and dipolar mechanisms and, as the dipolar contribution is known, provides information on the  $^{15}\text{N}$  CSA

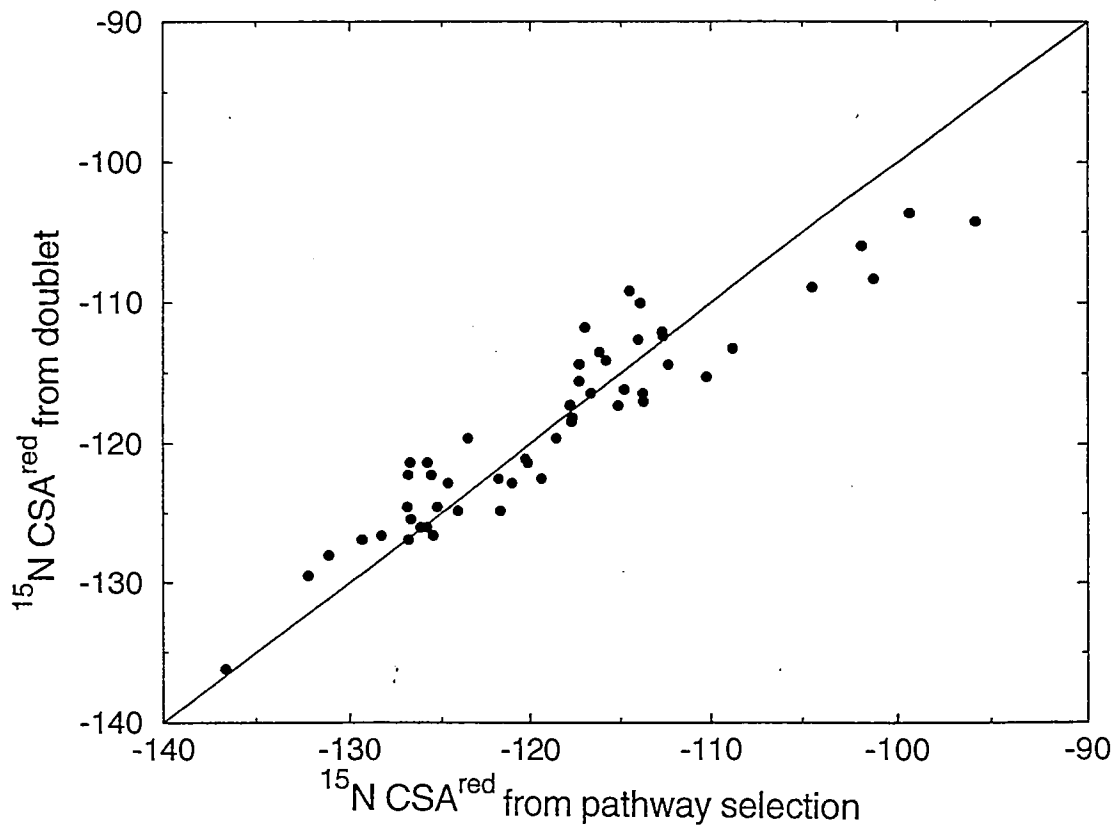


Figure 2. Agreement between  $^{15}\text{N}$  CSA<sup>red</sup> values measured from the relative intensities of the doublet components, and from the experiment described in reference 3.

correlates with a downfield change in  $\sigma_{\perp}$ ; i.e., variations in the  $^{15}\text{N}$  shift tensor tend to be more pronounced than variations in the isotropic  $^{15}\text{N}$  shift. However, we were unable to establish a simple relation between either the  $^{15}\text{N}$  isotropic shift or its CSA and any single structural parameter.

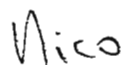
In contrast, both the amide proton CSA showed a clear correlation with hydrogen bonding (as does the isotropic shift). Again, a decrease in  $\sigma_{\parallel}$  appears to correlate with an increase in  $\sigma_{\perp}$ , and values measured for the backbone amides in ubiquitin range from nearly zero for non-hydrogen bonded amides to almost 15 ppm for strongly hydrogen bonded ones, albeit with considerable scatter.<sup>6</sup>

The most unambiguous correlation between polypeptide structure and CSA is observed for  $^{13}\text{C}^{\alpha}$  nuclei, however. Although the  $^{13}\text{C}^{\alpha}$  CSA is relatively small, its effect on relaxation interference is magnified by the large one-bond  $^{13}\text{C}^{\alpha}\text{-}^1\text{H}^{\alpha}$  dipolar coupling, making it straightforward to measure. Typical values for  $\text{CSA}_{\text{red}} = S^2(\sigma_{\text{par}} - \sigma_{\text{perp}})$  range from  $-6 \pm 5$  ppm in  $\alpha$ -helices to  $-27 \pm 4$  ppm in  $\beta$ -sheet for all residues except Gly (not measured), Ser, and Thr. Thr and Ser were found to change sign for  $\text{CSA}_{\text{red}}$ , with values of  $\sim +10$  (Thr) and  $+25$  (Ser) in  $\alpha$ -helices and values comparable to the other residues in  $\beta$ -sheet. Note that these values are only indirectly related to the magnitude of the  $^{13}\text{C}^{\alpha}$  CSA as  $\text{CSA}_{\text{red}}$  is sensitive to both the magnitude and orientation of the CSA tensor. Preliminary *ab initio* calculations by Oh-Young Kwon (NHLBI) show much better correlation ( $R^2 = 0.87$ ) between experimental and calculated values for  $\text{CSA}_{\text{red}}$  than for the difference between the isotropic  $^{13}\text{C}^{\alpha}$  shifts and their random coil values ( $R^2 = 0.72$ ).

It is clear that detailed and very useful information on the CSA tensor can be obtained from relaxation interference experiments in solution. It is relatively straightforward to measure such effects in a quantitative manner, not just for  $^{15}\text{N}$  and  $^{13}\text{C}^{\alpha}$ , but for all protons and  $^{13}\text{C}$  nuclei, including backbone carbonyl and sidechain methyl groups. This opens exciting new opportunities to study the exquisitely sensitive relation between chemical shift and protein structure, and, ultimately, to increase the accuracy of protein structures determined by NMR.



Ad Bax



Nico Tjandra

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- (2) de Dios, A. C.; Pearson, J. G.; Oldfield, E. *Science* **1993**, *260*, 1491-1496.
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- (4) Batta, G.; Gervay, J. *J. Am. Chem. Soc.* **1996**, *117*, 368-374.
- (5) Goldman, M. *J. Magn. Reson.* **1984**, *60*, 437-452.
- (6) Tjandra, N.; Bax, A. *J. Am. Chem. Soc.* **1997**, *119*, 8076-8082..



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(received 9/23/97)

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

RE: Unknown Plant Extract

Dear Barry,

Recently we were given a small sample of a plant extract to analyze, the  $^1\text{H}$  spectrum of which is shown in Fig. 1a. It was immediately obvious that the sample contained at least two sugars, possibly more, the anomeric protons of which come at 5.05ppm (unit A) and 5.31ppm (unit B).

The coupling constant of both anomeric protons is about 3.3 hz, consistent with the  $\alpha$  configuration at C-1. The stereochemistry at the other carbons was determined using a simple 1d tocsy experiment with Z filter<sup>1</sup>. Selective inversion of the anomeric protons at 5.05 ppm and 5.31 ppm using an IBURP-2 pulse gave the spectra shown in Figs. 1b and 1c respectively. The chemical shifts and approximate coupling constants are collected in Table I. The large coupling constants for the protons at C-2, C-3 and C-4 show that these sugars are  $\alpha$ - glucose units.

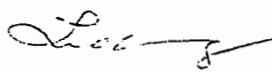
The lack of a  $\beta$  anomer would suggest that two glucose units are linked. The nature of the linkage is still unknown.

Table I

unit	$\nu_1$	$\nu_2$	$\nu_3$	$\nu_4$	$\nu_5$	$\nu_6$	$\nu_6'$	$J_{12}$	$J_{23}$	$J_{34}$	$J_{45}$	$J_{56}$	$J_{56'}$
A	5.05	3.44	3.61	3.31	3.78	3.64	3.76	3.3	9.9	9.4	9.4	4.9	a
B	5.31	3.45	3.55	3.33	3.81	3.68	3.74	3.3	9.9	9.4	9.6	4.8	a

a) Not resolved

Further work is in progress.



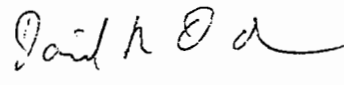
Lydia Chang



Don Bowler



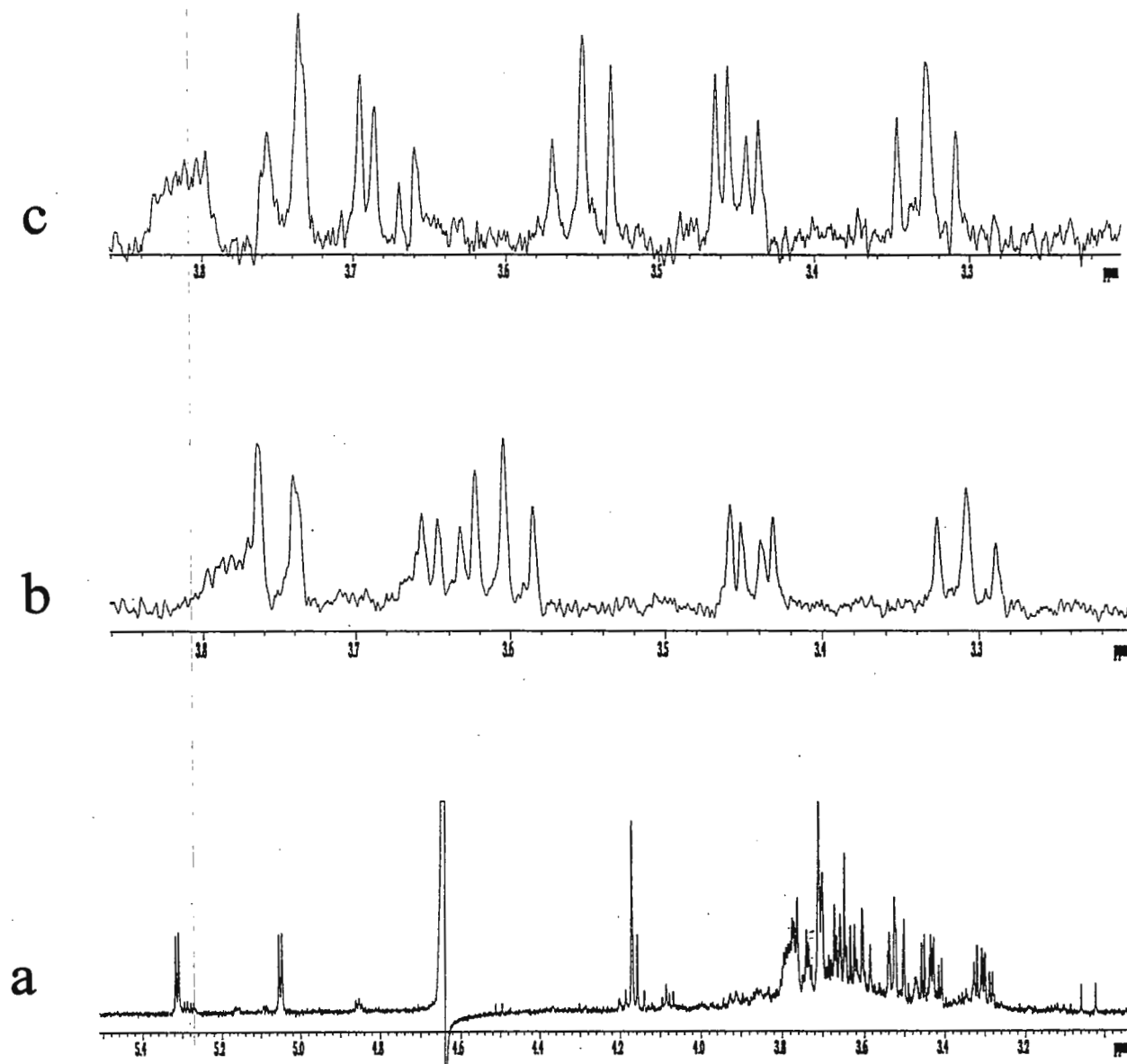
Joe Snyder



Dave Dohn

1. S. Subramanian and A. Bax, *J. Magn. Reson.* 71, 325 (1987)

Fig. 1



NMR spectrum of unknown plant extract. a)  $^1\text{H}$  NMR of extract at 500 MHz in  $\text{D}_2\text{O}$ . b) Tocsy spectrum obtained by selective inversion of the anomeric proton at 5.05 ppm. c) Tocsy spectrum obtained by selective inversion of the anomeric proton at 5.31 ppm. All tocsy data was acquired with z filter using z delays of 3, 5, 7, 9 and 11.1 msec, mix time of 250 msec, , and 128 scans. An IBURP-2 pulse was used to selectively invert the anomeric proton.





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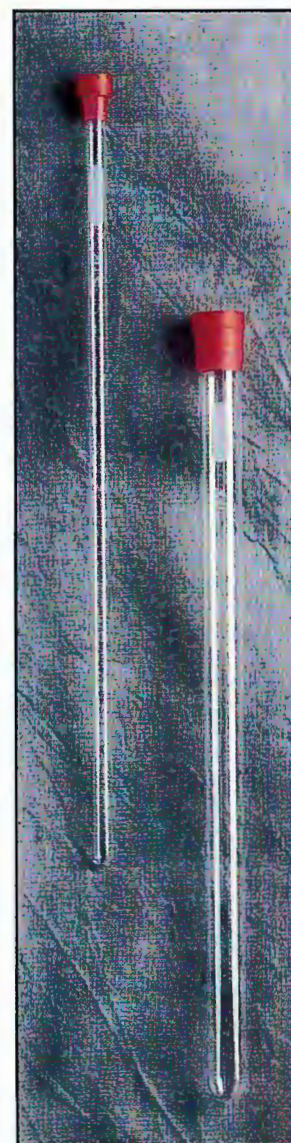


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† T.I.R. = TOTAL INDICATOR READING IN INCHES.





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Article Number	Approx. MHz	Length, Inches	Wall Thickness	Wall Variation T.I.R.†	Camber	1997 Price Pkg./5
897840-0000	>400	7	0.01118	0.0005	0.00025	53.75
897840-0008		8	0.01118	0.0005	0.00035	60.00
897840-0009		9	0.01118	0.0005	0.00045	66.25
897835-0000	360	7	0.01118	0.0010	0.0005	46.60
897835-0008		8	0.01118	0.0010	0.0006	51.75
897835-0009		9	0.01118	0.0010	0.0007	58.00
897830-0000	270	7	0.01118	0.0015	0.0010	41.90
897830-0008		8	0.01118	0.0015	0.0011	46.60
897830-0009		9	0.01118	0.0015	0.0012	52.25
897825-0000	200	7	0.01118	0.0020	0.0005	41.90
897825-0008		8	0.01118	0.0020	0.0006	46.60
897825-0009		9	0.01118	0.0020	0.0007	52.25
897820-0000	150	7	0.01118	0.0020	0.0010	37.70
897820-0008		8	0.01118	0.0020	0.0011	41.90
897820-0009		9	0.01118	0.0020	0.0012	47.00
897805-0000	100	7	0.01118	0.0025	0.0020	35.70
897805-0008		8	0.01118	0.0025	0.0020	39.30
897805-0009		9	0.01118	0.0025	0.0020	44.50
897800-0000	80	7	0.01118	0.0030	0.002	34.90
897800-0008		8	0.01118	0.0030	0.002	38.80
897800-0009		9	0.01118	0.0030	0.002	43.50

O.D. INCHES - 0.1186" (+0.0000/-0.0005)

I.D. INCHES - 0.0945" (+0.0005/-0.0000)

## NMR Tubes, 5 mm

Article Number	Approx. MHz	Length, Inches	Wall Thickness	Wall Variation T.I.R.†	Camber	1997 Price Pkg./5
897240-0000	>400	7	0.01475	0.0005	0.00025	66.75
897240-0008		8	0.01475	0.0005	0.00035	73.75
897240-0009		9	0.01475	0.0005	0.00045	80.25
897235-0000	360	7	0.01475	0.0010	0.0005	49.40
897235-0008		8	0.01475	0.0010	0.0006	54.25
897235-0009		9	0.01475	0.0010	0.0007	60.00
897230-0000	270	7	0.01475	0.0015	0.0010	42.10
897230-0008		8	0.01475	0.0015	0.0011	46.70
897230-0009		9	0.01475	0.0015	0.0012	53.00
897225-0000	200	7	0.01475	0.0020	0.0005	42.10
897225-0008		8	0.01475	0.0020	0.0006	46.70
897225-0009		9	0.01475	0.0020	0.0007	53.00
897220-0000	150	7	0.01475	0.0020	0.0010	31.90
897220-0008		8	0.01475	0.0020	0.0011	35.20
897220-0009		9	0.01475	0.0020	0.0012	39.20
897205-0000	100	7	0.01475	0.0025	0.0020	25.70
897205-0008		8	0.01475	0.0025	0.0020	29.40
897205-0009		9	0.01475	0.0025	0.0020	33.10
897200-0000	80	7	0.01475	0.0030	0.002	22.30
897200-0008		8	0.01475	0.0030	0.002	23.40
897200-0009		9	0.01475	0.0030	0.002	24.90

O.D. INCHES - 0.1955 (+0.0000/-0.0005)

I.D. INCHES - 0.1655 (+0.0005/-0.0000)

† T.I.R. = TOTAL INDICATOR READING IN INCHES.

Pkg./25

897193-0000	60	7	0.015			45.10
897194-0000	60	7	0.002			45.10

O.D. INCHES: 0.190-0.197

I.D. INCHES: 0.161-0.169

† T.I.R. = TOTAL INDICATOR READING IN INCHES.

## NMR Tubes, 10 mm

Article Number	Approx. MHz	Length, Inches	Wall Thickness	Wall Variation T.I.R.†	Camber	1997 Price Pkg./5
897335-0000	>360	7	0.0182	0.0015	0.0005	21.10
897330-0000	150	7	0.0182	0.0020	0.0010	17.90
897325-0000	80	7	0.0182	0.0030	0.0015	15.50
897320-0000	60	7	0.0182	0.0090	0.0020	10.70

O.D. INCHES - 0.3937 (+0.0000/-0.0005)

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# ARGONNE NATIONAL LABORATORY

9700 SOUTH CASS AVENUE, ARGONNE, ILLINOIS 60439

September 3, 1997

(received 9/11/97)

Bernard L. Shapiro  
The NMR Newsletter  
966 Elsinore Court  
Palo Alto, CA  
U.S.A. 94303

Re: **Xenon Gas Imaging of Mesoporous Aerogels**

Dear Barry,

Aerogels represent a new class of open-pore materials with pore dimensions in the nanometer range, typically between 2 and 50 nm, and are thus classified as mesoporous materials. Silica aerogels exhibit many unusual properties, including extremely low densities ( $0.003\text{--}0.35\text{ g/cm}^3$ ), high thermal resistance, low refractive index and sound velocity, and high surface area. These unique properties allow for their use in many new applications, such as insulated windows for solar applications, catalysts, gas separation media and Cherenkov counters.

In this communication, we report the first direct evidence linking xenon chemical shifts to xenon occluded within a microporous system. We show that the combination of NMR spectroscopy and chemical-shift selective magnetic resonance imaging (MRI) can be used to resolve some of the important aspects regarding the structure of silica aerogels.

Ampules for NMR investigations were prepared in the following manner. Three different specimens of silica aerogel (nominal dimensions: 3 mm x 3 mm x 3 mm) were placed in a heavy-wall borosilicate glass sample tube. The sample tube was evacuated and then the bottom of the tube was immersed in liquid nitrogen. An amount of xenon gas was allowed to enter the tube to create a pressure of 30 atmospheres at room temperature, a small amount of relaxation agent ( $\text{O}_2$  gas) was added to enhance the spin-lattice relaxation rate of xenon nuclei, and the tube was then flame sealed.

Chemical-shift selective imaging was accomplished using the SECSI method of Gerald and Botto (see *The NMR Newsletter*, 1994, 435, 17). The selection sequence is  $90^\circ_X - \tau_{CH} - 90^\circ_X - \tau_{null} - 90^\circ - \text{Image}$ . The resonance(s) of the spin(s) to be imaged is placed at (or near) resonance. Because  $T_1$  relaxation times of the three xenon resonances in the sample were similar, it was possible to suppress two resonances simultaneously; this was accomplished by implementing the SECSI sequence in the appropriate fashion, as described below. The  $\tau_{null}$  times used in the experiments ranged from 0.7-0.9 seconds, and the recycle delay time used was 5 s. Images of either occluded or free xenon gas were obtained by setting the value of  $\tau_{CH} = 150\text{ }\mu\text{s}$  in the experiment. 64 phase-encoding steps; each step required 200-600 transients in order to obtain an adequate S/N ratio. The time to echo  $\tau_E = 3.8\text{ ms}$ . The in-plane pixel resolution was  $420\text{ }\mu\text{m} \times 420\text{ }\mu\text{m}$ . Slice selection was accomplished by substituting a three-lobe, sinc- $90^\circ$  pulse in place of the third hard- $90^\circ$  pulse in the SECSI sequence. Slices were taken perpendicular to the main field axis of the superconducting magnet.

The  $^{129}\text{Xe}$  NMR spectrum of the high pressure ampule containing xenon, where the chemical shift reference of  $\delta = 0\text{ ppm}$  corresponds to the shift of pure xenon gas extrapolated to zero concentration, exhibits a resonance at 17 for free (or bulk-phase) xenon gas. Two smaller resonances at 46 and 52 ppm are observed for xenon occluded within the aerogel specimens.

$^{129}\text{Xe}$  chemical-shift imaging provides the first conclusive evidence of the direct correlation between  $\delta_{\text{Xe}}$  and the location of xenon within aerogel samples. Figure 1 shows  $^{129}\text{Xe}$  imaging results for the high-pressure xenon/aerogel sample. Slices of SECSI images of occluded and bulk-phase xenon gas are shown in Figures 1A and 1B, respectively. Chemical-shift selected spectra are shown directly below each of their respective images. A photograph of the three aerogel specimens inside the high-pressure ampule is shown in Figure 1C for comparison.

The ability to suppress two xenon resonances simultaneously in the imaging experiment makes it possible to image either of the two occluded xenon resonance lines independently. The  $^{129}\text{Xe}$  chemical-shift selective images utilizing this protocol are depicted in Figure 2. Figure 2A clearly demonstrates that xenon having a chemical shift of  $\delta = 52$  ppm resides within the middle specimen exclusively. On the other hand, Figure 2B shows that xenon at  $\delta = 47$  ppm is present in the other two aerogel specimens. This spectroscopic information has important implications about the pore structure of aerogel networks, and clearly illustrates that the two xenon chemical shifts are associated with entirely different pore structures.

Figure 2D represents the composite image from both experiments, in which Figures 2A and 2B have been added together. The sum of the two images allows a direct comparison of signal intensity of both bulk-phase and occluded xenon. The most striking feature seen in the composite image is that entire regions devoid of xenon signal can be identified; in particular, regions are evident at the edges of images at the left and middle of the figure. Moreover, the total concentration of xenon in the specimen at the right is significantly greater than the concentration of xenon in the surrounding free gas; the concentration of occluded xenon is found to be nearly twice the concentration of xenon in the bulk phase.

The images presented in Figures 1 and 2 illustrate the ability of the xenon MRI method to study heterogeneity associated with the pore structure of aerogels. The most exciting aspect of this work is the power of xenon chemical-shift imaging for elucidating differences in the physical properties of aerogels. In particular, we have been able to correlate MRI results with optical properties of the specimens. Three distinct regions in the specimens can be clearly distinguished on the basis of differences in the xenon NMR parameters, and imaging facilitates their visualization. Recall that the middle specimen appears translucent, while the other two specimens are largely transparent, and that the specimen on the left has translucent regions at two of its outer edges (see Figure 1C). Adsorbed xenon in the middle, uniformly translucent specimen is found to resonate at a higher frequency, and exhibits a broader resonance than xenon adsorbed in the other two specimens, and thus is readily distinguished in the image depicted in Figure 2A. The images highlighted in Figure 2B for the outer two specimens constitute a second type of pore structure associated with optical transparency. A third pore regime can be ascribed to regions that are devoid of xenon signal, which occur at the edges of the center and left images, see Figure 1D. These regions may have resulted from physical damage to the fragile aerogel specimens. They apparently are regions inaccessible to xenon and are consistent with a collapsed pore network.

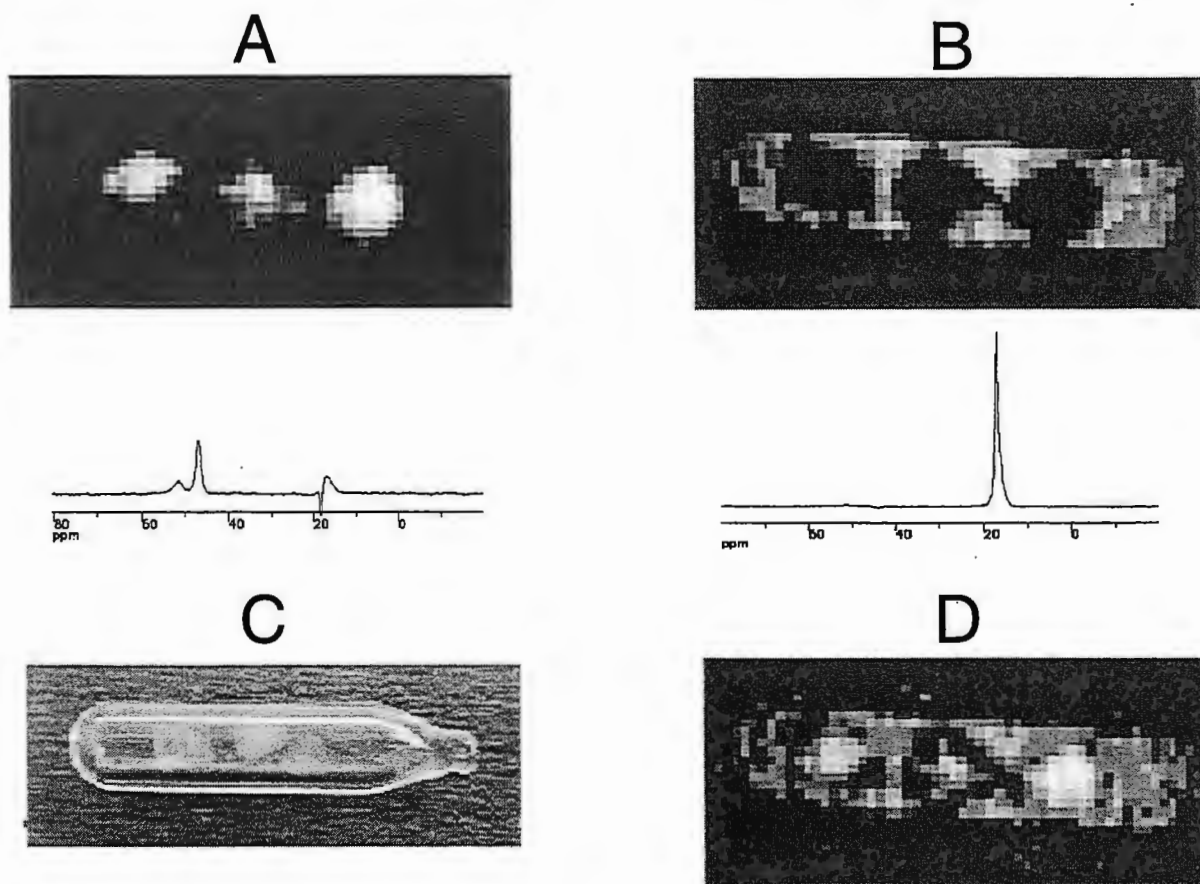
Sincerely,

*David M. Gregory*

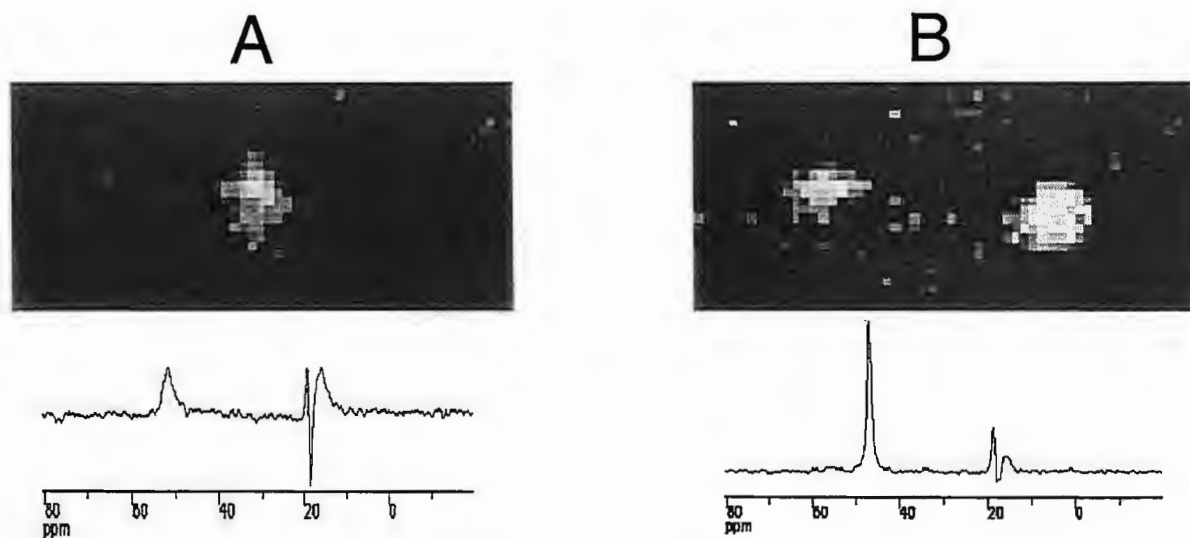
David M. Gregory  
Chemistry Division

*Robert E. Botto*

Robert E. Botto  
Chemistry Division



**Figure 1.** Slices (thickness of 1 mm) of chemical-shift selective images; in-plane resolution is 420  $\mu\text{m}$ . Lighter pixels indicate more signal intensity: (A) Image of the xenon adsorbed inside the aerogel samples; (B) Image of the xenon gas outside the aerogel samples; (C) Photograph of the high pressure sample showing the three aerogel samples. Opaque and translucent regions appear brighter in the photograph; (D) Image showing the direct addition of the data shown in (A) and (B).



**Figure 2.** Slices (thickness of 1 mm) of chemical-shift selective images: (A) Signal from the resonance at 52 ppm has been selected while the other resonances have been suppressed; (B) Signal from the resonance at 46 ppm has been selected.



University of Illinois  
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School of Chemical Sciences  
142B RAL, Box 34-1  
600 S. Mathews Avenue  
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Telephone: (217) 244-0564  
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mainzv@aries.scs.uiuc.edu

September 23, 1997 (received 9/24/97)

TITLE: 750 Installation, Baseline Noise and Sidebands

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

Dear Barry:

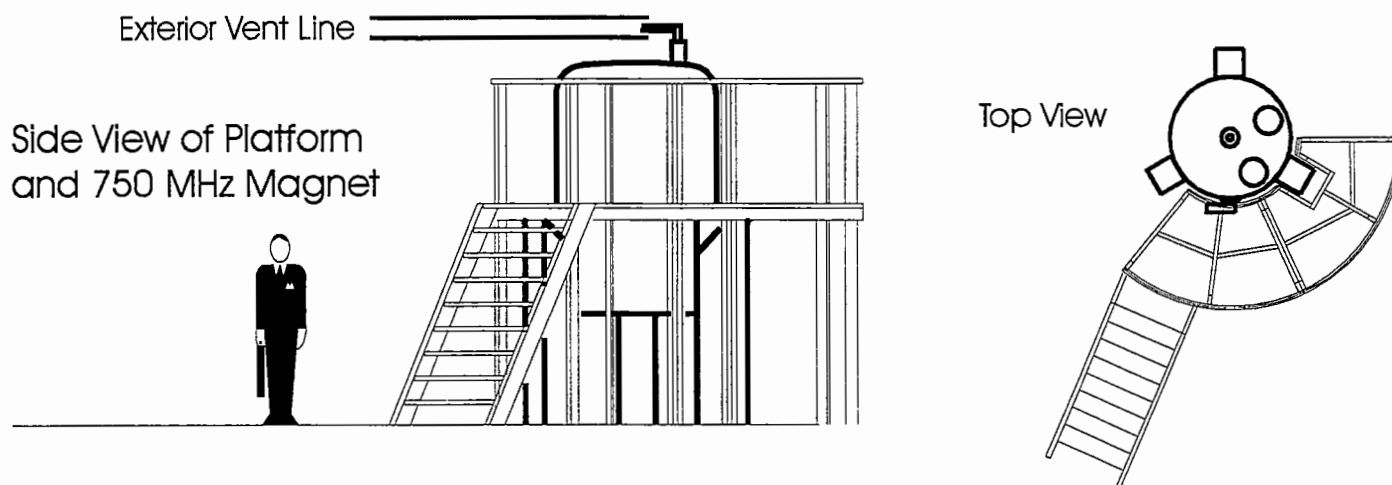
I thought you might be interested in what's been happening in the NMR Lab in the School of Chemical Sciences at the Univ. of Illinois at Urbana-Champaign over the past year. Last year we installed three new systems (Varian Unity Inova 750, Unity Inova 600, and Unity Inova 500/89) replaced the consoles on two 500 magnets (now a Unity Inova 500 and Unity 500), upgraded our Unity 400 with gradients, de-commissioned four systems (XL200, QE300, GN300NB, and NT360), and moved into two additional labs (one newly renovated and one part of the new Chemical and Life Sciences Laboratory). All of this actually went fairly smoothly, considering that we moved three systems to new locations, added anti-vibration legs or tables to three magnets, as well as the hassle of getting new spectrometers up and running.

For our 750, we had a wooden platform built (with brass screws!) to make access easier and safer. Speaking as one who has perched at the top of a 10 foot stepladder, balancing on one foot while putting a sample in the magnet a good four feet away, it is not a comfortable feeling. A schematic view from the top and side of the platform is shown below. This platform was built by carpenters from the UIUC Mill Shop for about \$4,000. I am sure our Mill Shop would be happy to build one for someone outside of the Univ. of Illinois, however, the shipping costs might be prohibitive. If anyone is interested in building one of their own, I can send you a full set of plans either as hardcopy, an AutoCad file, or both.

One of the shake-down experiences we have had with our 750 might also be useful. We had one probe installed with our Inova 750, a Varian 5mm HCN Z probe (0.1% Ethylbenzene S/N ~ 1300:1), and from the beginning observed while not spinning what we described as  $\pm 40$  Hz noise in the baseline around all peaks, with sidebands at  $\pm 20$  and 40 Hz. (See figure 1, below. NOTE: All spectra are taken non-spinning.) As time went on we installed a manostat on the magnet, but the problem did not improve. I must now digress.

When our 750 was installed, Oxford insisted upon a vent line from the magnet quench vent to the outside of the building. This was installed as an aluminum duct, hanging from the ceiling supports, and encompassing but not touching the quench vent on the magnet. At the time, the vent went straight outside, with no barrier. As a result, we had wind coming down the vent into the room. We placed foam packing material between the magnet quench vent and the exterior vent line to cut down the wind. When the foam was removed, the  $\pm 40$  Hz noise disappeared, but ~20% sidebands at  $\pm 1.1$  Hz appeared due to the wind tunnel effects. We eventually concluded that our 40 Hz noise problem was due to vibrations transmitted to the magnet from the exterior helium vent line, and contributed to by the wind. Our next move was to place a barrier in the exterior vent line where it met the outside world. Our current choice of material is aluminum foil, but we are searching for a 5 psi carbon disk to replace the aluminum. With the foil in place, we no longer had a wind tunnel effect, so we removed the foam packing material, and lo and behold, the  $\pm 40$  Hz noise went away (see figure 2 below). Success at last, we thought!

Lurking around the bend was another problem. About two weeks later, we noticed that the sensitivity of an HMQC experiment seemed to have been seriously compromised. Investigating further revealed 10-15% sidebands at the  $\pm 1.5$  Hz position, which were constantly changing phase and intensity, and were similar to those observed when the exterior vent line was open to the outside (see figure 4). At first we thought it was an unstable supply of



compressed air for the anti-vibration legs. However, the problem persisted when we switched to compressed air cylinders. We did find the problem disappeared when the magnet was taken off of the anti-vibration legs. We then noticed that when standing on the platform by the magnet, you could feel cold air blowing, but the source was impossible to localize. This led us to investigate stray air currents in the room.

Another digression - the room is a 40 ft. x 40 ft. with 18 ft. ceiling. The magnet is centered in the room. The A/C is ducted, with no ducts above or within 8 ft. of the magnet, and the returns are 10-15 ft. from the magnet. We can turn off the A/C but it is difficult to turn off the exhausts.

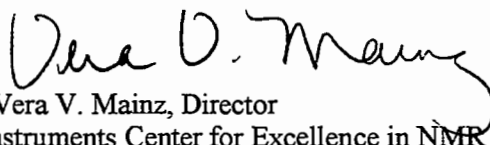
With the help of our A/C people, we turned off the room A/C, and anxiously watched our data. No change! I told the A/C people to take a break, and then one of my spectroscopists (Feng Lin) and I started discussing the problem again. Since we never saw this problem when we were observing the  $\pm 40$  Hz noise, it occurred to me that maybe the packing material between the magnet quench vent and the exterior vent line was enough to damp the very small vibrations we were now observing. We tested this hypothesis by replacing the foam packing material, and lo and behold, the sidebands disappeared (see figure 3). They reappeared momentarily when the A/C people opened the door into the room and caused a very large (since we now had no A/C but full exhaust) pressure differential, but as soon as the door closed, the sidebands again disappeared. So, we are currently trying to better balance the air flows in the room, but it is interesting to me that a several ton magnet, granted, floating on air, can be so easily affected by very small air currents.

How you control the temperature in a space meant for magnets, especially large magnets, has always been with us. My particular problems seem to involve unbalanced air flow in a room. The imbalance could be related to the fact that the A/C is vented (a plenum ceiling (forced A/C into a pressurized ceiling) would give a lot more flexibility) and that the exhaust air is part of the Building exhaust, and therefore out of my control. We may still have to build an airlock to control the pressure changes when people enter the room. It has also been suggested that we could hang a plastic curtain (i.e., a very large shower curtain) around the magnet to resolve the problem - a practical albeit ugly alternative.

To summarize, we currently have an aluminum foil barrier between the outside world and the 750 room and inside the room foam is between the exterior vent line and the magnet quench vent.

If anyone else has had problems of this nature, I would appreciate hearing about them.

Sincerely,

  
Vera V. Mainz, Director

Varian Oxford Instruments Center for Excellence in NMR

Figure 1.  
VENT OPEN, FOAM IN PLACE

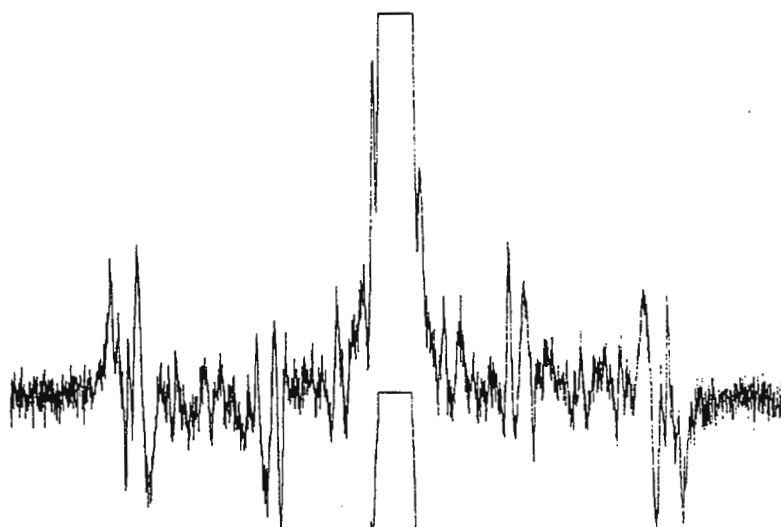


Figure 2.  
VENT CLOSED, FOAM OFF

Lasted for about two weeks.

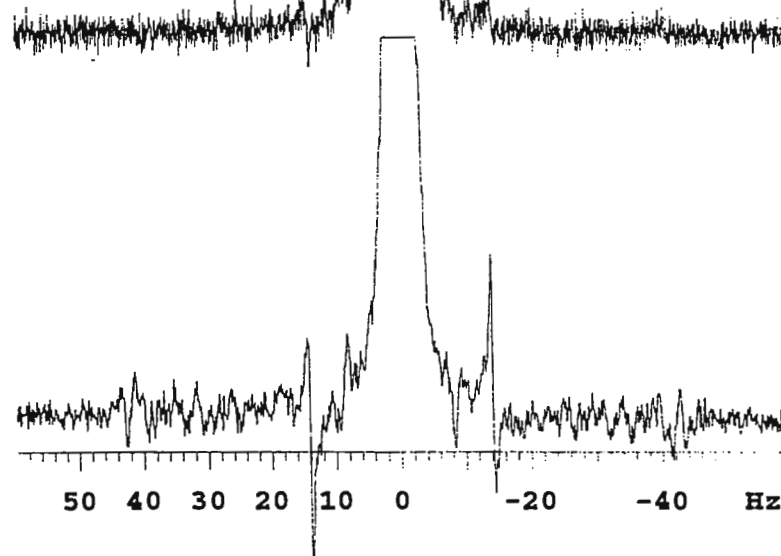


Figure 3.  
VENT CLOSED, FOAM BACK  
IN PLACE

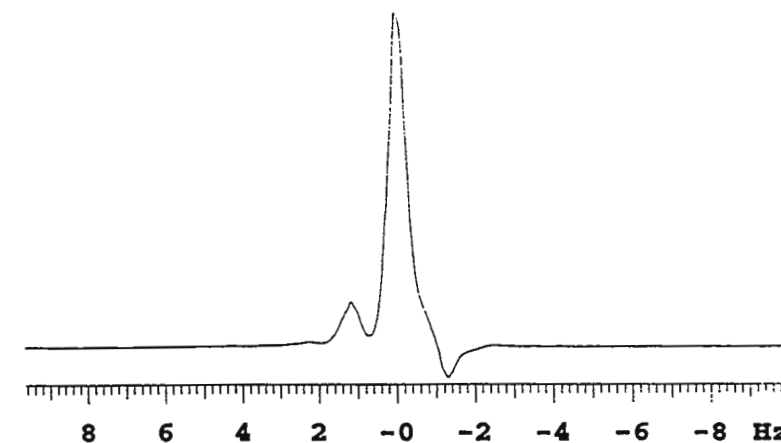


Figure 4.  
VENT CLOSED, FOAM OFF

Similar in appearance to when  
the vent was open and the foam  
was off.

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NMR S 97



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IN REPLY REFER TO:

(received 9/25/97)  
24 September 1997

## Zero-Field $^{14}\text{N}$ NMR Relaxation and Molecular Motions in RDX

Dear Barry:

We are happy to report that our new Bruker DMX-500 with a self-shielded wide-bore Magnex magnet is currently being installed, and our MSL-300 has been recently upgraded to a DSX-300. Meanwhile, it might be of interest to report some  $^{14}\text{N}$  NMR results obtained at the other extreme of magnetic field, zero field.

Zero-field  $^{14}\text{N}$  NMR (a.k.a. "pure NQR") relaxation studies can be used to study slow molecular motions in solids, although relatively few examples have been reported. It may seem surprising at first that measurements of the  $^{14}\text{N}$  spin-lattice relaxation time  $T_1$  in zero-field could provide information about slow molecular motions, since the  $^{14}\text{N}$  transitions often occur around 2-5 MHz or so. However, as first discussed by Alexander and Tzalmona (*Phys. Rev.*, **138**, A845 [1965]), the zero field situation differs in a fundamental way from standard BPP relaxation in high field. This is because a slow molecular motion (such as a jump, which can be viewed as occurring instantaneously) can cause the *main Hamiltonian* to suddenly change, due to the change in direction of the electric field gradient (efg). In BPP, it is a *minor perturbing Hamiltonian* which changes. Since the Hamiltonian in zero-field can change non-adiabatically, the nuclear spin wave-functions remain the same, while the Hamiltonian is changed due to the new efg direction. The result is a calculable transition probability to a new eigenstate leading to a  $T_1$  value that is comparable to the motional correlation time  $\tau_c$ . Alexander and Tzalmona measured  $\tau_c$  values as long as 1 s for the tetrahedral jump process in hexamethylenetetramine (HMT) from the zero-field  $^{14}\text{N}$   $T_1$ .

The symmetrical molecule hexahydro-1,3,5-trinitro-s-triazine, also known as RDX (see Figure) is one of the most common "energetic materials" and has been extensively studied experimentally and theoretically. Nonetheless, surprisingly little is known about possible molecular motions taking place in the solid state. The zero-field  $^{14}\text{N}$  NMR measurements of both  $T_1$  and  $T_2$  show a large decrease (from seconds to milliseconds) going from about  $-30^\circ\text{C}$  to  $+60^\circ\text{C}$ , temperatures well below the melting point of  $200^\circ\text{C}$ . The graph below shows an Arrhenius plot of the  $T_1$  data for one of the 3 crystallographically-inequivalent ring nitrogen peaks at 3.41 MHz (the so-called  $\nu_-$  line), and the linear fit over this relatively narrow temperature range suggests the presence of a thermally activated process with an activation barrier of ca. 80 kJ/mole (19 kcal/mole). A similar slope is obtained for the corresponding  $\nu_+$  peak at 5.2 MHz. The  $(1/T_1)$  intercept of approximately  $10^{16}\text{ s}^{-1}$  cannot directly give the pre-exponential factor for the motion, which requires knowing the correlation time, but the latter must be  $\geq 10^{16}\text{ s}^{-1}$ . This value is much higher than that expected from lattice librational motion, and can be taken to indicate that either concerted motions or lattice expansion effects upon the activation barrier are present.

The nature of the thermally-activated motions is still unknown. A chair-chair interconversion does not seem possible because the intermolecular interactions in the solid state would result in an

anisotropic potential. Conformational isomerization by ring inversion has recently been identified in solid cyclohexane (McGrath & Weiss, *Langmuir*, 13, 4474 [1997]), but the process is slow ( $51 \text{ s}^{-1}$  at 222 K), and occurs only in the plastic phase. No such phase exists for RDX. Different conformations due to ring puckering are a possibility, but no evidence of additional peaks from "frozen" conformations in the zero-field  $^{14}\text{N}$  NMR have ever been reported, even though data has been obtained down to 77 K (Karpowicz & Brill, *J. Phys. Chem.*, 87, 2109 [1983]). Additional measurements at lower temperatures and on other transitions (for both ring and nitro group nitrogens) may provide some further clues to the nature of the motion. As always, any suggestions are welcome.

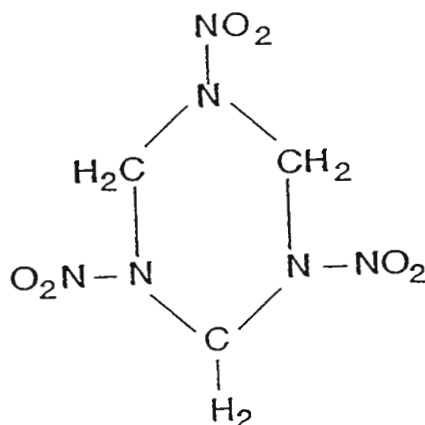
Sincerely yours,



James P. Yesinowski  
yesinowski@nrl.navy.mil



Kenneth J. McGrath  
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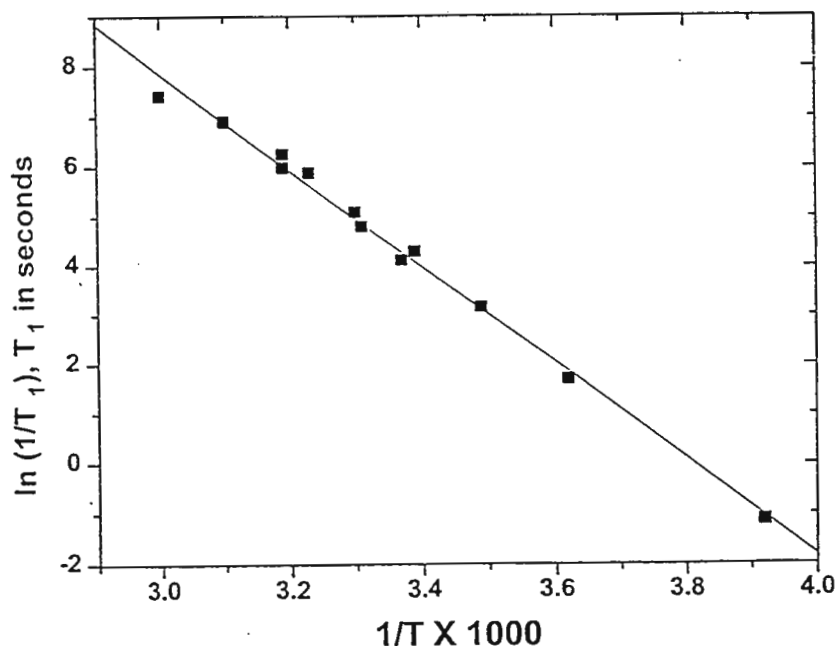


**RDX**

Ring (Amine) Nitrogen Transitions:

- $3 \nu_+$  (~5.2 MHz)
- $3 \nu_-$  (~3.4 MHz)
- $3 \nu_0$  (~1.8 MHz)

$^{14}\text{N}$   $T_1$  Data for 3.41 MHz RDX Transition





# THE NMR NEWSLETTER

B. L. Shapiro, Publisher  
The NMR Newsletter  
966 Elsinore Court  
Palo Alto, CA 94303-3410

650-493-5971  
650-493-1348 fax  
shapiro@nmrnewsletter.com  
<http://www.nmrnewsletter.com>

## Policies and Practical Considerations

(Slightly revised October 1997)

*The NMR Newsletter* (formerly the TAMU NMR Newsletter, the IIT NMR Newsletter, and originally, the Mellon Institute NMR Newsletter), now in its fortieth year of consecutive monthly publication, continues under the same general policies as in the past.

### 1. Policy:

*The NMR Newsletter* is a means for the rapid exchange of information among active workers in the field of NMR spectroscopy, as defined broadly, including imaging. As such, the Newsletter serves its purpose best if the participants impart whatever they feel will interest their colleagues, and inquire about whatever matters concern them. Technical contributions should always contain a significant amount of information that has not already been published or that will appear in the formal literature within a few weeks of the appearance in the Newsletter.

Since the subscriber/participant clearly is the best judge of what he or she considers interesting, our first statement of policy is "We print anything." (This is followed by the reservation, "that won't land us in jail or bankruptcy court.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. The Newsletter is not, and will not become, a journal. We merely reproduce and disseminate exactly what is submitted.

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*continued*



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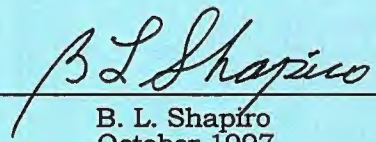
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6. **Suggestions:** They are always welcome.

  
B. L. Shapiro  
October 1997

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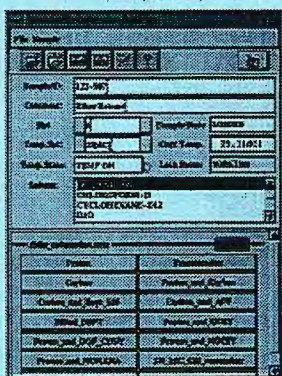
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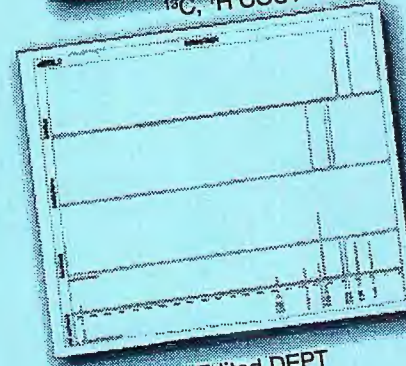
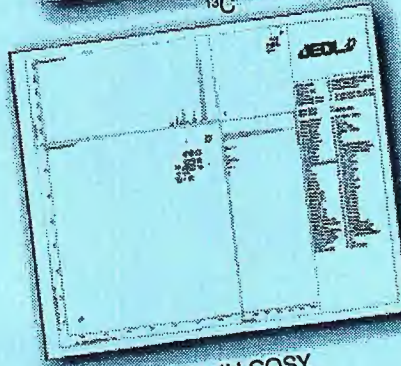
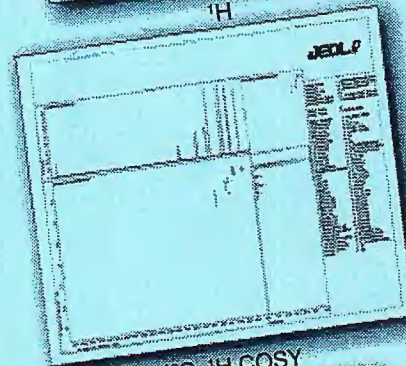
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