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**FORTHCOMING NMR MEETINGS**

Symposium on *In Vivo* Magnetic Resonance Spectroscopy VIII, North Falmouth, Massachusetts, **March 25 - 26, 1995**; Contact: Radiology Postgraduate Education; Room C-324, University of California School of Medicine, San Francisco, CA 94143-0628; Phone: (415) 476-5731; Fax: (415) 476-9213; For registration, call (415) 476-5808.

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

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

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

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

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

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

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

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



UNIVERSITY OF CAMBRIDGE  
DEPARTMENT OF CHEMISTRY  
Lensfield Road, Cambridge CB2 1EP

Professor Bernard L Shapiro,  
Editor/Publisher TAMU NMR,  
966 Elsinor Court,  
Palo Alto,  
California 94303  
USA

11 November 94

"O Tannenbaum"

Dear Barry,

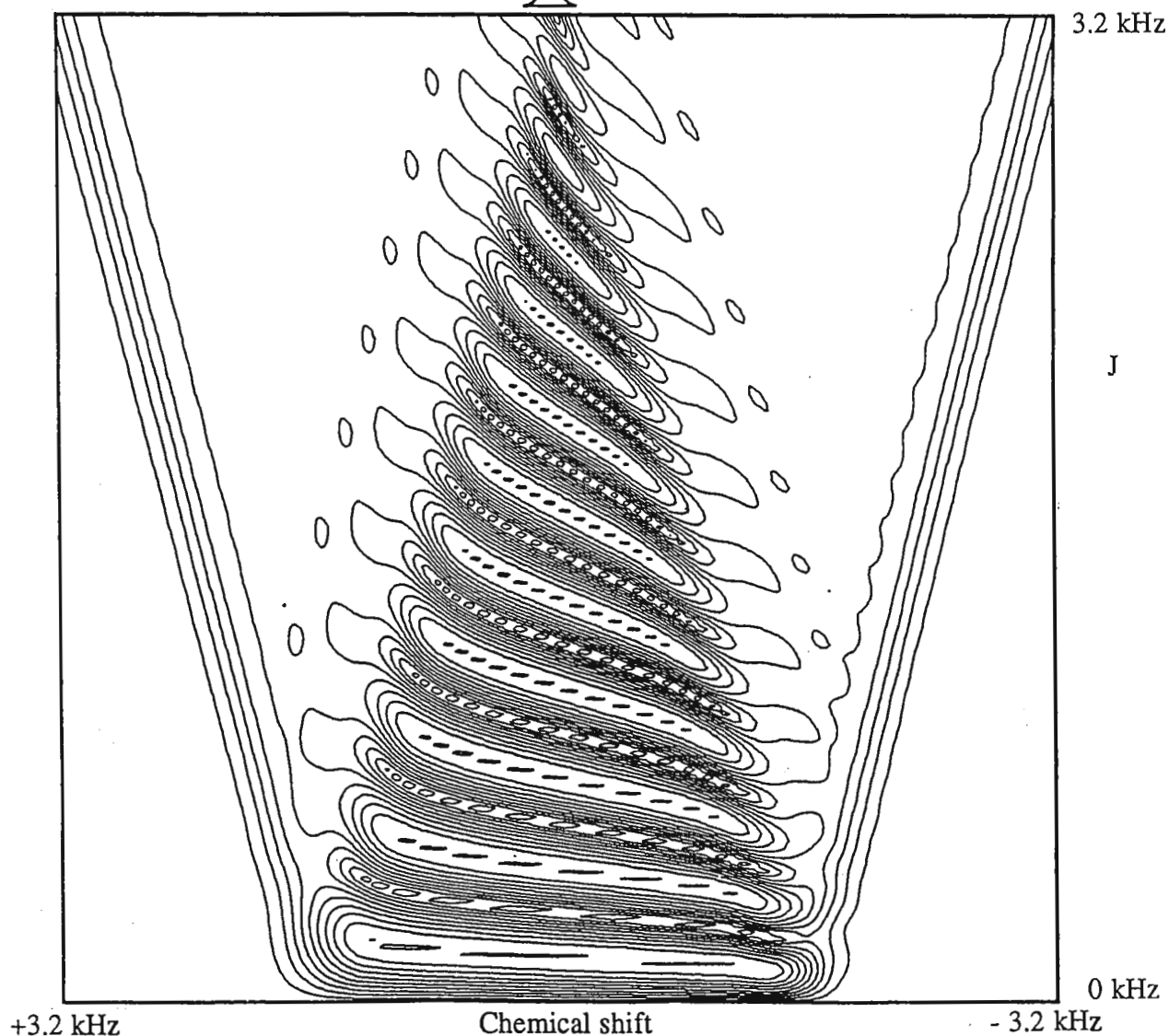
We have been investigating the utility of various soft pulses to generate antiphase magnetization ( $2I_{xy}S_z$ ) over a restricted excitation band. The application would be for band-selective long-range heteronuclear correlation experiments where we would like to measure the coupling constants. One candidate for the band-selective pulse was the hyperbolic secant {M. S. Silver, R. I. Joseph and D. I. Hoult, *J. Magn. Reson.* **59**, 347 (1984)} 6.9 ms in duration with the parameter  $\mu = 5$ , truncated at 1%. This gives a strong antiphase response for  $J = 120$  to 180 Hz and a set of responses at the odd harmonics. It seemed a suitably seasonal diagram for your December issue.

Yours sincerely,

Ray

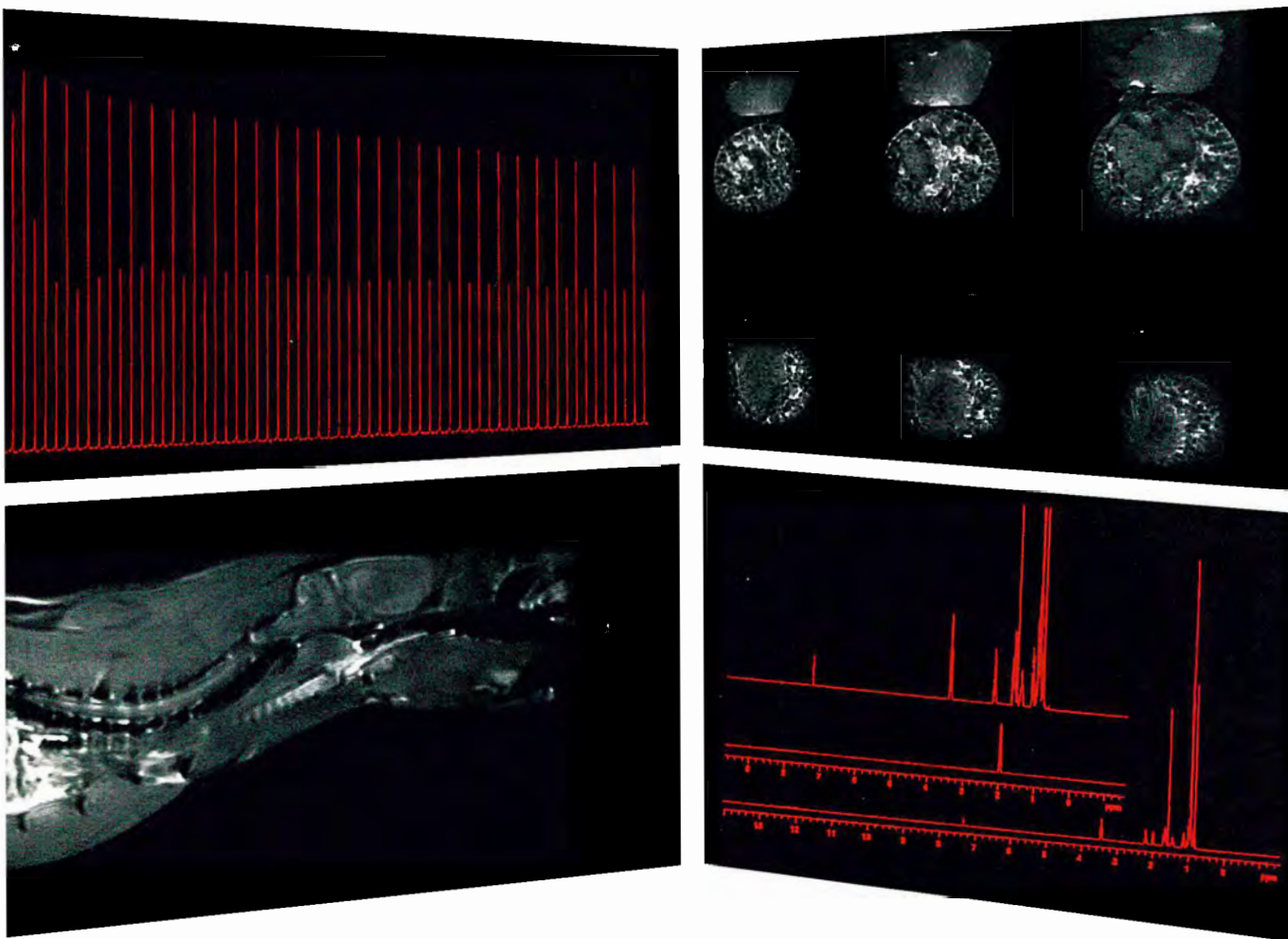


Eric





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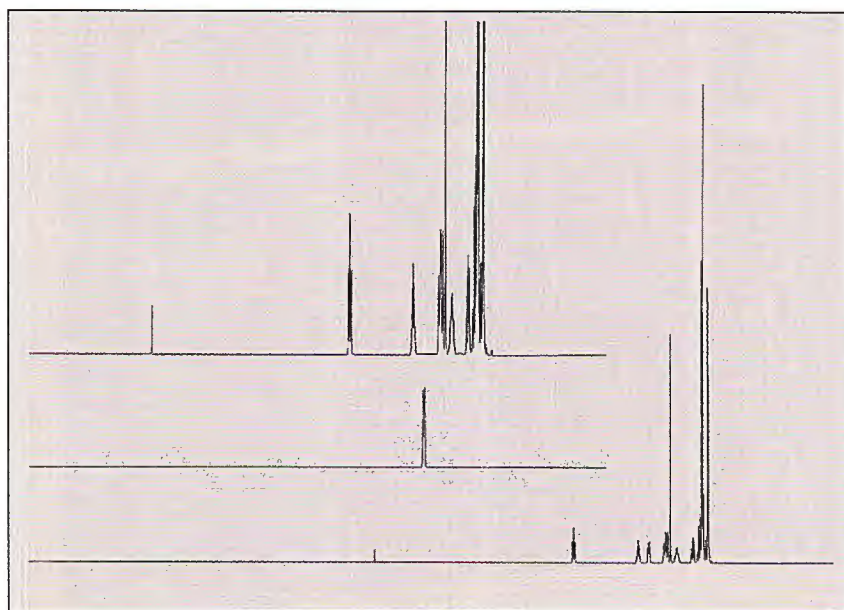
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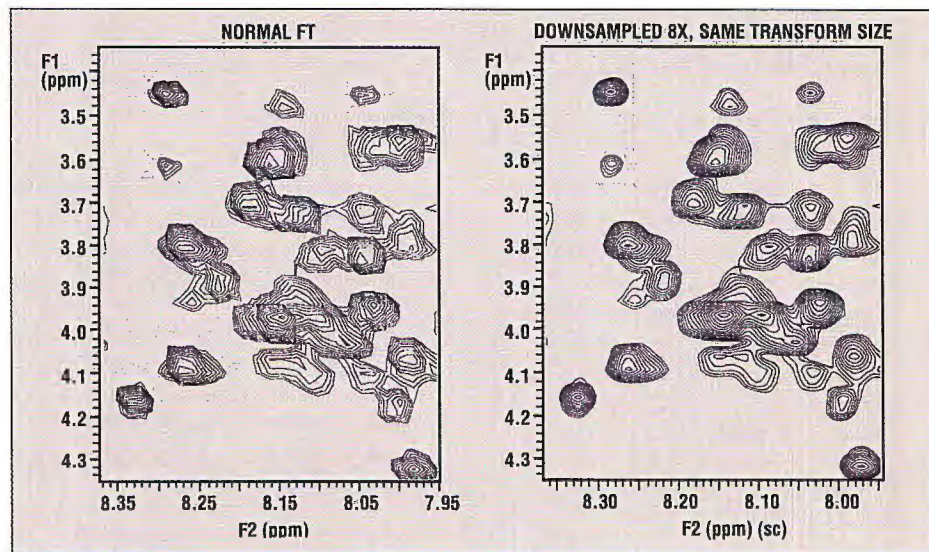
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## TAMU NMR Newsletter - Book Reviews

Book Review Editor: William B. Smith, Texas Christian University, Fort Worth, TX 76129

### "NMR and MORE" in honour of Anatole Abragam

Editors : M. Goldman and M. Porneuf

Les Editions de Physique

Avenue du Hoggar, BP 112 - F-91944 Les Ulis cedex A France ; 1994 ;

ISBN 2-86883-219-9 ; FFrs 350, 379 pages.

This book is a Festschrift in honour of Anatole Abragam, published at the occasion of his approaching 80th birthday, and companion to a Scientific Day held at Saclay, France, on June 3, 1994. It consists of articles written by old friends, young former students and middle-age disciples, all distinguished scientists.

The book begins with the Introduction of the Scientific Day by J. Friedel and an anecdotal article by M. Goldman on Anatole Abragam's "Laboratoire de Magnétisme Nucléaire" at Saclay. It ends with the closure address of the Day by N. Kurti. These articles are bilingual (French and English). The rest of the book consists of purely scientific articles in English.

A large part of the articles are devoted to Anatole Abragam's field of expertise : magnetic resonance, both nuclear and electronic. They cover many diverse modern aspects of the discipline, and among the authors are many of the most famous names of the Club, such as E.L. Hahn, B. Bleaney, C.P. Slichter, A. Pines, J. Jeener, R. Freeman, J.S. Waugh, A.G. Redfield, etc. No less brilliant personalities have contributed non-nuclear-non-electronic-non-magnetic-non-resonance articles, such as "Wet versus Dry" by P.G. de Gennes and F. Brochard, atomic laser cooling by the "Sisyphus Effect" by C. Cohen-Tannoudji,  $\mu$ SR, 2D electronic solids, one and two-electron electronics in nanocircuits, or even physical processes in mitosis, etc. Each of these articles is written in a clear and accessible way. Their interest transcends the limits of specialized domains, and they occasionally reveal unsuspected but profound analogies between completely distinct subjects. An example at hand is the "Sisyphus Effect", whose theory can be formulated in a way identical with that of magnetic relaxation in an oscillatory field, developed in the late 30's by H.G.B. Casimir and C.J. Gorter.

To sum up, this book is more than a homage to an outstanding figure in Magnetic Resonance. It is also a genuine and interesting scientific book, which has its place on every scientific book-shelf.

M. GOLDMAN

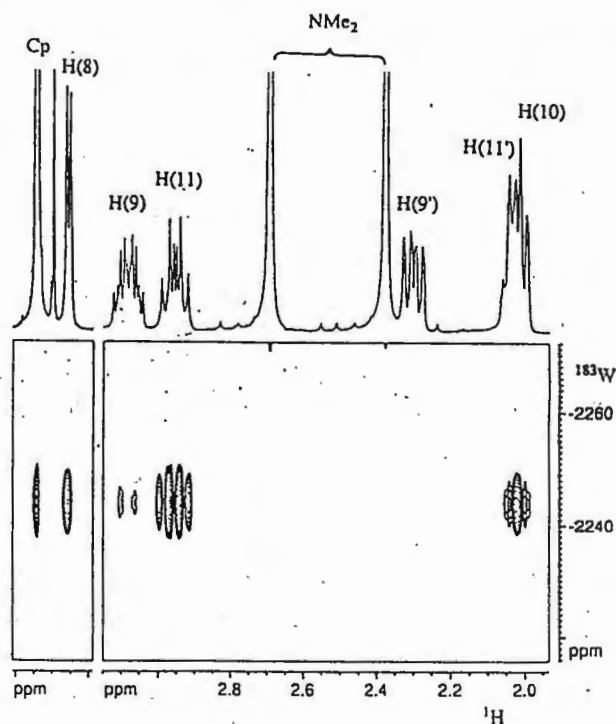
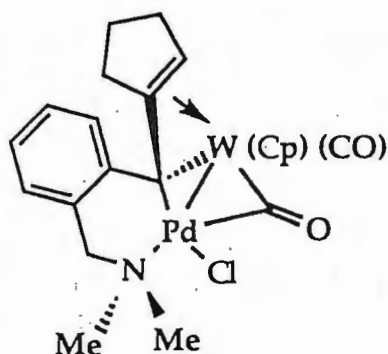
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Prof. Dr. Paul S. Pregosin  
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Prof. Barry Shapiro  
Editor/Publisher  
TAMU News Letter  
966 Elsinore Court  
Palo Alto Ca. 94303  
USA

(received 11/7/94)  
Zurich, Nov. 2, 1994

Dear Barry,

We are continuing our studies in tungsten-organometallic chemistry with the view to optimizing our  $^{183}\text{W}$  capabilities. Shown below is the  $^{183}\text{W}$  spectrum for:



It is interesting that one sees small J-values between the  $^{183}\text{W}$  and a number of spins, e.g. the Cp and cyclopentenyl protons. These coupling constants can be used to detect the metal resonance, although they are too small to be seen in the conventional 1-D spectrum.

Sincerely,

Prof. P. S. Pregosin

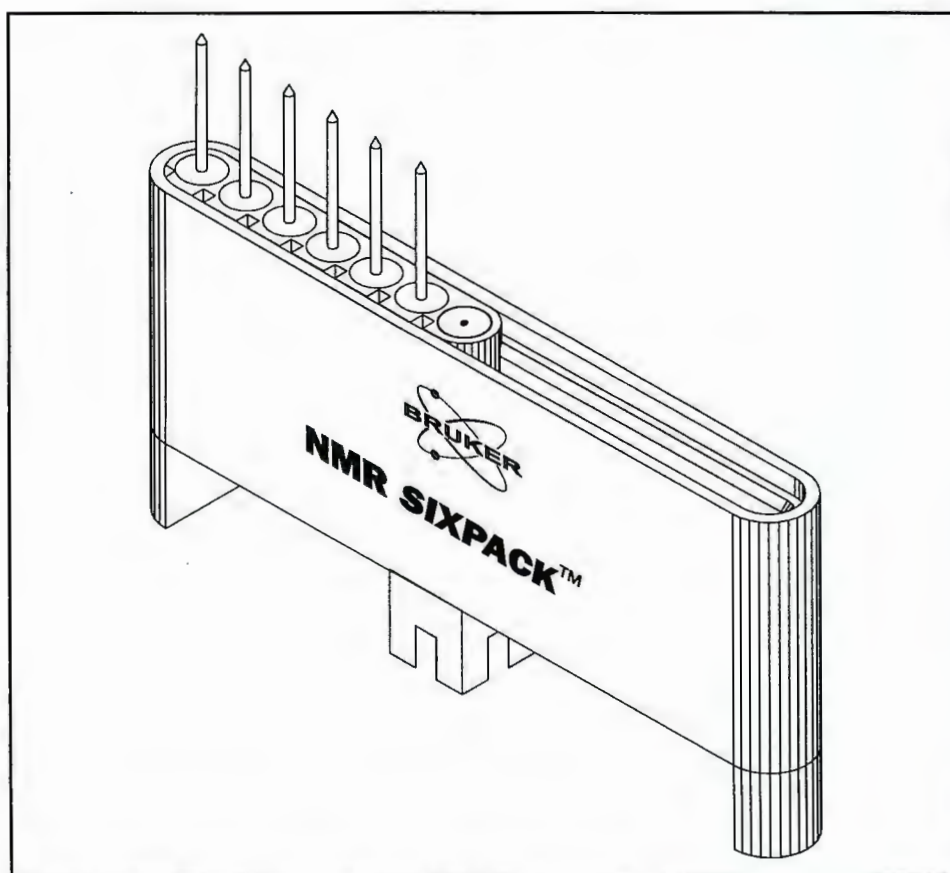


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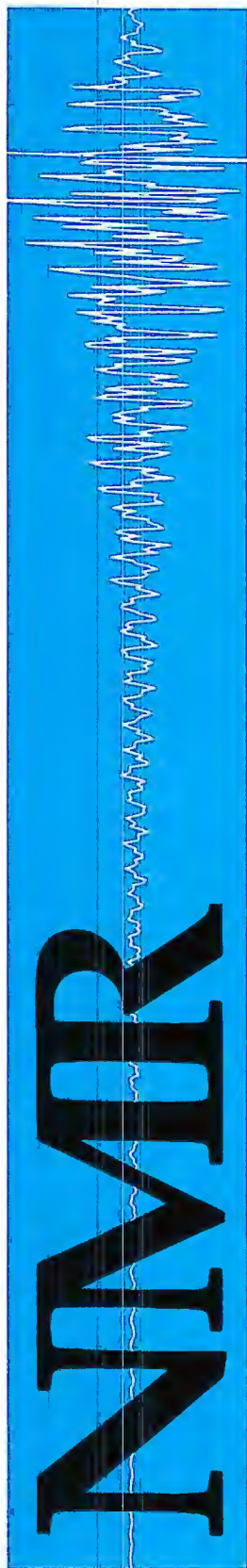
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# UNIVERSITY of PENNSYLVANIA

School of Arts and Sciences

Department of Chemistry  
Chemistry Building  
Philadelphia, PA 19104-6323

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

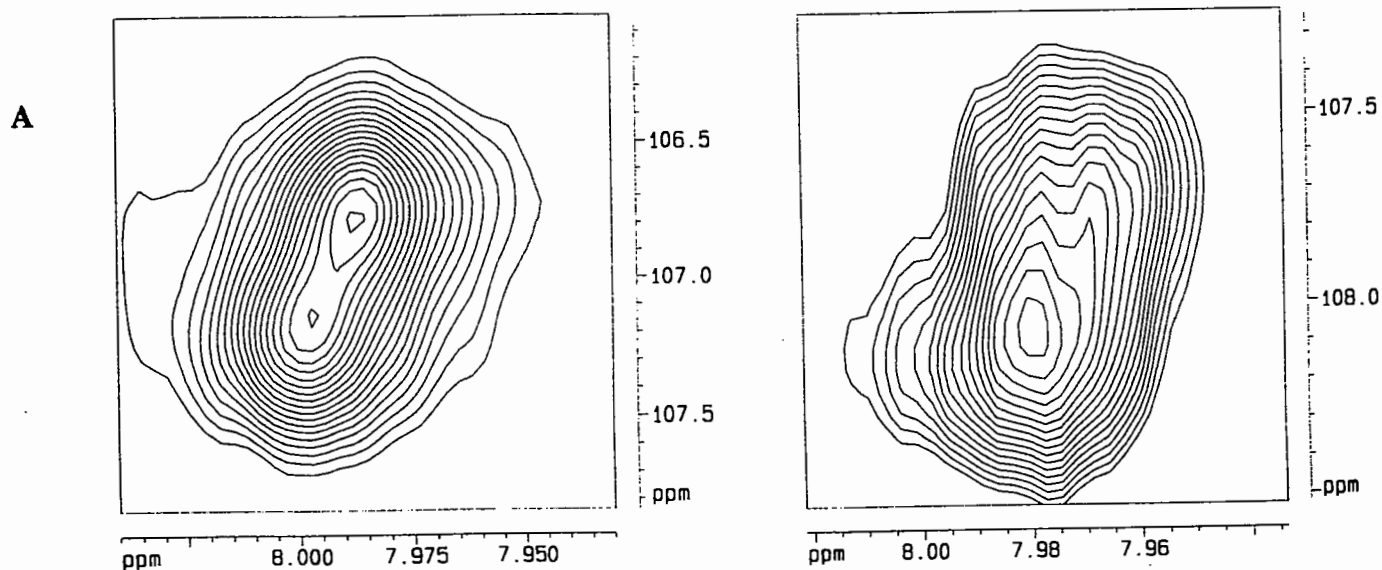
November 18, 1994  
(received 11/19/94)

Dear Barry,

## Sensitivity and Resolution of Protein Spectra at 750 MHz

Our Bruker DMX 750 spectrometer with a Magnex 750/54 magnet has been in operation since March. We have observed enhancements in resolution in studies at 750 MHz of both large proteins and small peptides in solution compared to 600 MHz. However, the increases in sensitivity in double and triple resonance 3D spectra have been even more encouraging, especially when the 3 axis gradients are employed. We present some comparisons of protein NMR spectra at 600 and 750 MHz in this letter.

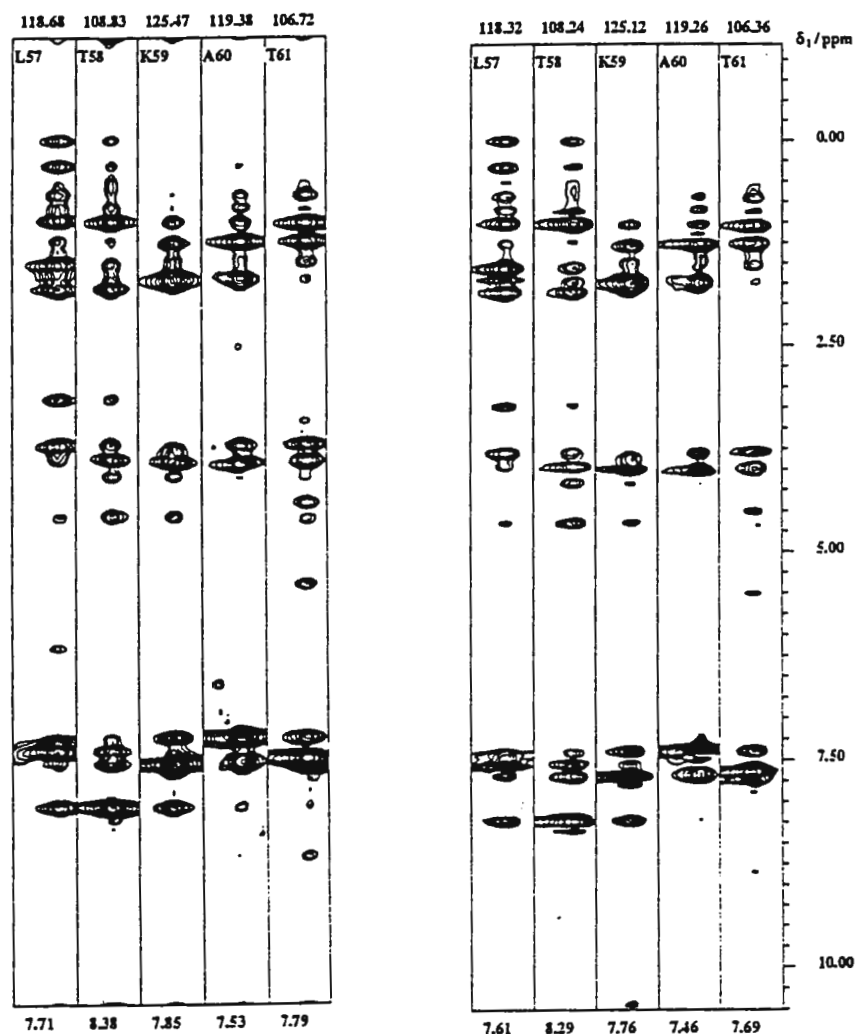
Figure A contains expansions from HMQC spectra of a uniformly  $^{15}\text{N}$  labeled 112 residue module of the  $\alpha$  subunit of spectrin which is a dimer in solution. The resonances have been assigned to Gly-75 and Gly-106. The spectrum on the left was collected at 750 MHz and that on the right at 600 MHz. All other conditions were identical. Watergate gradients were used for solvent suppression; data were collected as 8 scans in 128  $^{15}\text{N}$  increments for a spectral width of 30 PPM and with 4096 complex points in the  $^1\text{H}$  dimension of a spectral width of 12 PPM. The data are multiplied by a  $45^\circ$  shifted sine bell function and then zero-filled twice to give the final matrix size of 2K by 0.5K. The two resonances are much more clearly resolved at 750 MHz than at 600 MHz.





The spectral strips in Figure B were taken from a 3D NOESY-HMQC spectra of the uniformly  $^{15}\text{N}$  labeled reduced form of merP, a 72 residue monomeric protein. The data on the left were acquired at 750 MHz and those on the right at 600 MHz. All processing conditions were identical. The NOESY-HMQC spectra were run with Watergate gradients with 8 scans collected for each of the 1024 complex points collected in the f3 dimension at 12 PPM spectral width, 150 points collected for the  $^1\text{H}$  evolution in f1 and 64 points in  $^{15}\text{N}$  evolution at a 30 PPM spectral width. The data were processed as a 1024x256x128 matrix using an exponential multiplier of 5Hz in the f3 dimension and  $90^\circ$  shifted sine bells in both the f1 and f2 dimensions. The strips contain multiple NOE crosspeaks at 750 MHz that are missing at 600 MHz. Note in the amide portion of the spectrum the  $\text{N}_i, i+2$  crosspeak between Thr58 and Ala60 present at 750 MHz and not at 600 MHz.

B



We are encouraged by the improvement in spectral quality observed for all the proteins we have examined at 750 MHz and look forward to presenting more results the Newsletter in the future.

Sincerely,

*Kathleen G. Valentine*

Kathleen G. Valentine

*Ruth Steele*

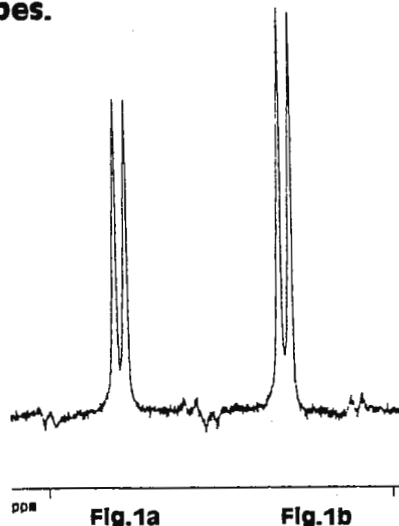
Ruth Steele

*Stanley J. Opella*

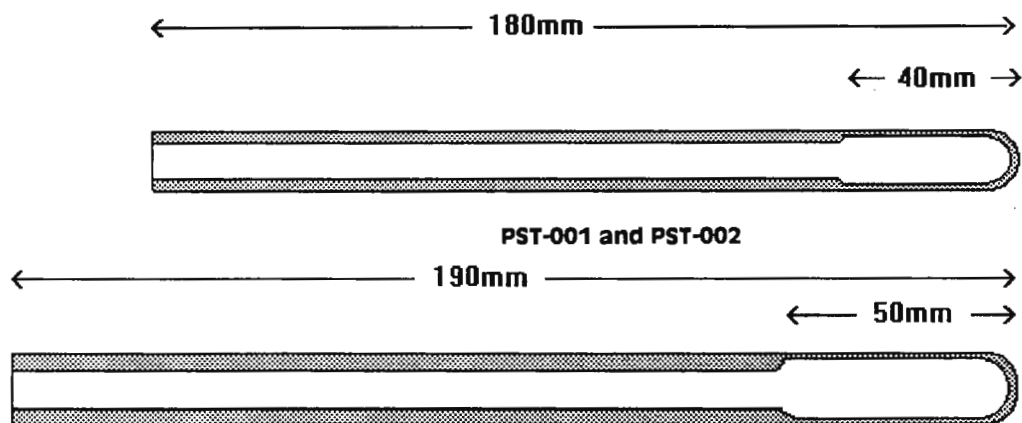
Stanley J. Opella

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The spectra of 20mm sucrose in D<sub>2</sub>O were obtained with a single scan without apodization prior to Fourier transformation on a Bruker AMX-600 spectrometer at 298 K. By using Shigemi high quality 5mm standard tube (Fig.1a) and the Shigemi highly sensitive thin wall 5mm tube (Fig.1b), the spectra confirms a sensitivity enhancement of about 10%.



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	PST-002	0.21	40/15	4.96 + 0.00 - 0.01	4.54 ± 0.01	\$13.00	\$12.00
8	ST8-001	0.25	40/ 8	8.00 + 0.00 - 0.01	7.52 ± 0.01	\$31.00	\$28.00
	ST8-002	0.25	50/15	8.00 + 0.00 - 0.01	7.52 ± 0.01	\$27.00	\$25.00
10	ST10-001	0.25	40/ 8	9.98 + 0.00 - 0.01	9.52 ± 0.01	\$36.00	\$32.00
	ST10-002	0.25	50/15	9.98 + 0.00 - 0.01	9.52 ± 0.01	\$32.00	\$28.00

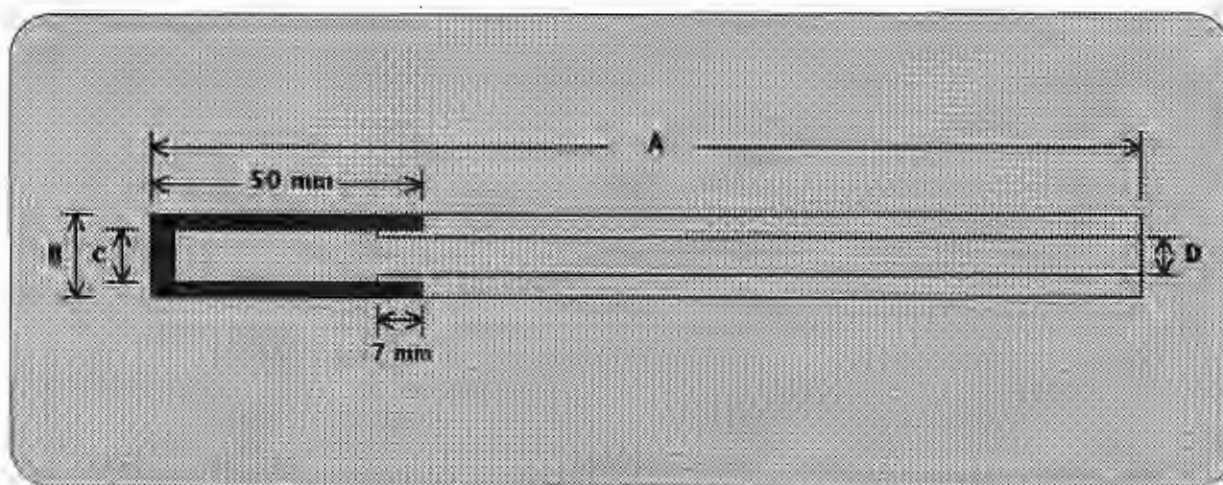
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National Institutes of Health  
 Building 5, Room 112  
 9000 Rockville Pike  
 Bethesda, MD 20892  
 October 28, 1994  
 (received 11/3/94)

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

### Asymmetric Spectra in Optically-Pumped NMR

Dear Barry:

I am currently setting up a new laboratory in the Laboratory of Chemical Physics of the National Institute of Diabetes and Digestive and Kidney Diseases (NIDDK) at the NIH. My new phone number and e-mail address are given below. Visitors are always welcome.

For the past two years, in my old lab at AT&T Bell Laboratories, Sean Barrett (now Assistant Professor of Physics at Yale University), Gary Dabbagh, and I were exploring the use of optical pumping as a means of enhancing NMR signals in directly-detected NMR experiments on semiconductor thin films and heterostructures. We carried out measurements on GaAs samples and on GaAs/AlGaAs quantum wells, and found that NMR signals could be enhanced by roughly two orders of magnitude (or more, if you include reductions in the effective  $T_1$ ) by irradiation of interband transitions with circularly polarized infrared light at low temperatures. Some of our initial results on the quantum wells were published in Phys. Rev. Lett. **72**, 1368 (1994). More recent results, in which we use NMR spectra and relaxation measurements to probe the properties of two-dimensionally confined electrons in the quantum wells at low temperatures and in high magnetic fields, are now being written up.

There is one feature of these experiments that has not been and probably will not be included in our papers, and that I would like to point out here. Early on, in optical pumping experiments on variously doped GaAs films grown on GaAs substrates, we found that the optically-pumped  $^{69}\text{Ga}$ ,  $^{71}\text{Ga}$ , and  $^{75}\text{As}$  spectra obtained at low temperatures ( $< 10\text{ K}$ ) showed three resolved lines, with splittings on the order of 10 kHz. Although all three nuclei are spin-3/2, there should be no quadrupole splitting of the NMR lines in ideal bulk GaAs, due to the cubic symmetry of the crystal lattice. At low temperatures, however, strains can develop in the sample that are sufficient to produce the observed splittings, unless care is taken to mount the sample in a strain-free manner. This effect has been seen before, and was not a big surprise to us (although the uniformity of the strain-induced splittings across the sample's surface still puzzles me). What was surprising was the fact that the spectra were strikingly asymmetric. The two quadrupole satellites, corresponding to the  $m=-3/2$  to  $m=-1/2$  and the  $m=1/2$  to  $m=3/2$  transitions, had very different intensities at times, sometimes differing by a factor of two or more.

It took me a while to realize what was going on. I think the explanation is that the nuclear spins were being cooled by optical pumping to spin temperatures  $T_s$  where the usual high temperature limit (defined by  $kT_s \gg h\nu_0$ , where  $\nu_0$  is the NMR frequency) did not apply. In addition, the pulses we were using to excite the NMR FID signals were not  $90^\circ$  pulses. Under these conditions, asymmetric spectra are expected.

If one knows the pulse flip angle and measures the ratio of the areas of the two satellite lines, one can determine  $T_s$ . This is sometimes useful, particularly for estimating the absolute number of nuclear spins that are contributing to the optically-pumped NMR signals. I've calculated the relative signal amplitudes for the three transitions of a spin-3/2 nucleus for arbitrary flip angle  $\theta$  and for arbitrary initial populations  $p_m$  of

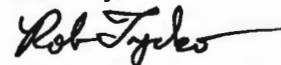
the four spin sublevels. The initial populations follow a Boltzmann distribution if there is a well-defined spin temperature (i.e.,  $p_m \propto \exp(-mh\nu_0 / kT_S)$ ). The results are as follows:

$$\begin{aligned} A_{-3/2,-1/2} = & \frac{3}{16} p_{-3/2} [\sin(3\theta/2) + \sin(\theta/2)] [\cos(3\theta/2) + 3\cos(\theta/2)] \\ & - \frac{3}{16} p_{-1/2} [3\cos(3\theta/2) + \cos(\theta/2)] [\sin(3\theta/2) + \sin(\theta/2)] \\ & + \frac{3}{16} p_{1/2} [-3\sin(3\theta/2) + \sin(\theta/2)] [-\cos(3\theta/2) + \cos(\theta/2)] \\ & + \frac{3}{16} p_{3/2} [-\cos(3\theta/2) + \cos(\theta/2)] [\sin(3\theta/2) - 3\sin(\theta/2)] \end{aligned}$$

$$\begin{aligned} A_{-1/2,1/2} = & \frac{3}{8} p_{-3/2} [\sin(3\theta/2) + \sin(\theta/2)] [-\cos(3\theta/2) + \cos(\theta/2)] \\ & + \frac{1}{8} p_{-1/2} [3\cos(3\theta/2) + \cos(\theta/2)] [3\sin(3\theta/2) - \sin(\theta/2)] \\ & + \frac{1}{8} p_{1/2} [-3\sin(3\theta/2) + \sin(\theta/2)] [3\cos(3\theta/2) + \cos(\theta/2)] \\ & - \frac{3}{8} p_{3/2} [-\cos(3\theta/2) + \cos(\theta/2)] [\sin(3\theta/2) + \sin(\theta/2)] \end{aligned}$$

$$\begin{aligned} A_{1/2,3/2} = & \frac{3}{16} p_{-3/2} [-\sin(3\theta/2) + 3\sin(\theta/2)] [-\cos(3\theta/2) + \cos(\theta/2)] \\ & + \frac{3}{16} p_{-1/2} [-\cos(3\theta/2) + \cos(\theta/2)] [3\sin(3\theta/2) - \sin(\theta/2)] \\ & + \frac{3}{16} p_{1/2} [\sin(3\theta/2) + \sin(\theta/2)] [3\cos(3\theta/2) + \cos(\theta/2)] \\ & - \frac{3}{16} p_{3/2} [\cos(3\theta/2) + 3\cos(\theta/2)] [\sin(3\theta/2) + \sin(\theta/2)] \end{aligned}$$

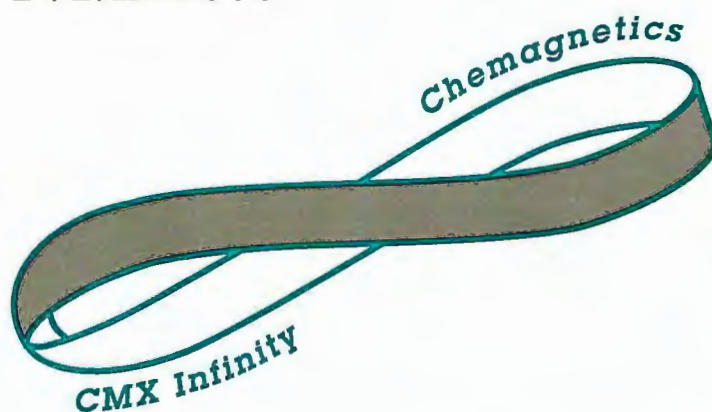
Sincerely,



Dr. Robert Tycko  
phone: 301-402-8272  
fax: 301-496-0825  
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# ARGONNE NATIONAL LABORATORY

9700 SOUTH CASS AVENUE, ARGONNE, ILLINOIS 60439

October 15, 1994  
(received 10/22/94)

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

Re: **Chemical-Shift Selective Imaging of Binary Systems**

Dear Barry,

We have devised a simple pulse sequence to selectively and independently image two different sample regions within a binary system employing differential chemical shifts. The transmitter is centered between the two resonances of a binary system. For purposes of illustration we constructed a phantom consisting of a 5-mm NMR tube placed inside of a 10-mm NMR tube. The inner tube contained acetone (2.04 ppm) and the annular region between the two concentric tubes contained chloroform (7.24 ppm). A small amount of relaxation agent was added to each solvent to reduce the spin-lattice relaxation time,  $T_1$ , by approximately an order of magnitude.

The pulse sequence employs a chemical-shift filter consisting of two  $90^\circ$  pulses prior to the imaging experiment. An initial  $90^\circ_x$  pulse tips the proton magnetization for both resonances into the xy plane. A dephasing time equal to  $1/4$  of the precessional period in the rotating frame of each resonance is allowed to elapse so that the magnetizations from both components (acetone and chloroform) are allowed to be mutually out of phase by  $180^\circ$ . A second  $90^\circ_y$  pulse (or  $90^\circ_{-y}$ ) stores the magnetization of one of the components along the  $+z$  axis while inverting the other spin system. A delay equal to  $1/eT_1$  for the inverted spins is allowed to evolve after which the only net magnetization present is that of the other component stored along the  $+z$  axis. At this point we begin a standard 2D spin-echo imaging pulse sequence and thereby obtain a selective image of only one component of the binary system.

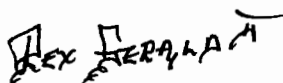
It is noted that this method is suitable for a two component system with different chemical shifts, is independent of the spin-lattice relaxation rates for the two components, and does not require equal spin concentrations.

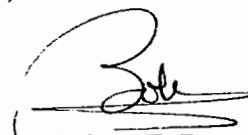
We illustrate chemical shift selective imaging in Figure 1. The top image is the result of a normal 2D imaging experiment. The bright central region reflects the larger magnetization for the acetone protons,

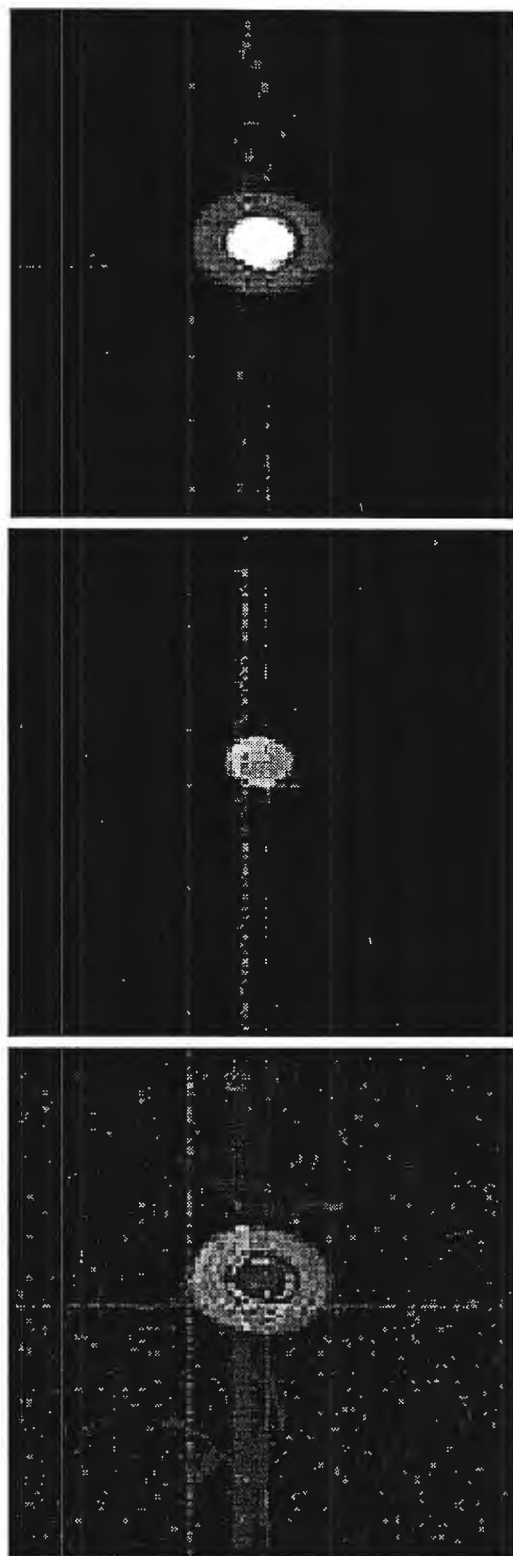
$$\frac{\bar{M}_{\text{Acetone}}}{\bar{M}_{\text{Chloroform}}} \cong 15.$$

The annular region is the chloroform. The middle image is a selective image of the acetone wherein the chloroform is completely obscured. The bottom image is the chloroform signal in the annular region with the acetone nearly completely attenuated in the center.

Sincerely,

  
Rex E. Gerald II  
Chemistry Division

  
Robert E. Botto  
Chemistry Division



**Figure 1.** Proton MRI images of acetone-chloroform phantom: (top) normal 2D spin-echo image; (middle) chemical-shift selective image of 5-mm NMR tube containing acetone; (bottom) chemical-shift selective image of annular region containing chloroform.



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Pulse width	10 ms	20 ms
Duty cycle	Up to 10%	Up to 10%
Amplitude droop	5% to 10 ms typ.	5% to 20 ms typ.
Harmonics	Second: -25 dBc max. Third: -12 dBc max. to 30 MHz -25 dBc max. above 30 MHz	
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	100% max.	
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Phone# 298-47-7474 (Project Office)

-8909 (Design Group)

-0607 (Engineering Group)

-9017 (Characterization Group)

F a x# 298-47-3599

November 7, 1994

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

## Quasi Spectral Density Function Analysis for N-15 nuclei in Proteins

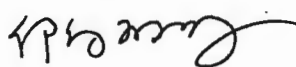
Dear Dr. Shapiro:

Mapping of spectral densities of NH motions in proteins has been experimentally given by Drs. Peng and Wagner(1,2). It set us free from the model free approach (3,4). But, this mapping has been suffered from the overestimation of antiphase relaxation rates caused by exchange processes of the proton spin, spin diffusion, and chemical exchange. As an alternative to avoid the problem, we propose quasi spectral density function (QSDF) analysis, where we take the advantage to use nitrogen nuclei by putting the spectral densities of  $J(\omega_H - \omega_N)$ ,  $J(\omega_H)$ , and  $J(\omega_H + \omega_N)$  equal. Under this assumption, three relaxation times of  $T_1$ ,  $T_2$ , and cross relaxation suffice to derive three spectral densities of  $J(0)$ ,  $J(\omega_N)$  and  $J(\omega_H + \omega_N)$ . The assumption seems to beautifully hold for the N-15 which has ten times lower and opposite sign of gyromagnetic ratio to that of proton. The data in the figure correspond to  $J(0)$ ,  $J(\omega_N)$ , and  $J(\omega_H + \omega_N)$  of the main chain amide N-15 of calmodulin, of which original relaxation times were taken from the paper by Barbato *et al* (5). As a convenient tool for analysis, we define new indices  $J(0; \omega_N)$  and  $J(\omega_N; \omega_H + \omega_N)$  as a difference of two related spectral densities in order to evaluate motions slower than  $\omega_N^{-1}$  and  $(\omega_H + \omega_N)^{-1}$ , respectively. These indices are introduced to express spectral density functions, which reflect many internal motions inseparably appearing and include those slower than overall tumbling motion of the protein molecule. Application of the QSDF to interpret relaxation data of various proteins shows interesting aspects of the internal protein dynamics (6,7).

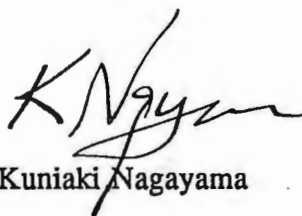


1. Peng, J. W., and Wagner, G. (1992) *J. Magn. Reson.*, **98**, 308-332
2. Peng, J. W., and Wagner, G. (1992) *Biochemistry*, **31**, 8571-8586
3. Lipari, G., and Szabo, A., (1982) *J. Am. Chem. Soc.*, **104**, 4546-4559
4. Lipari, G., and Szabo, A., (1982) *J. Am. Chem. Soc.*, **104**, 4559-4570
5. G. B. Barbato, *et al.*, *Biochemistry* **31**, 5269 (1992)
6. Ishima, R. and Nagayama, K., (1994) abstract in the 33th NMR symposium in Japan.
7. Ishima, R. and Nagayama, K., (1994) *Biochemistry*, submitted.

Sincerely



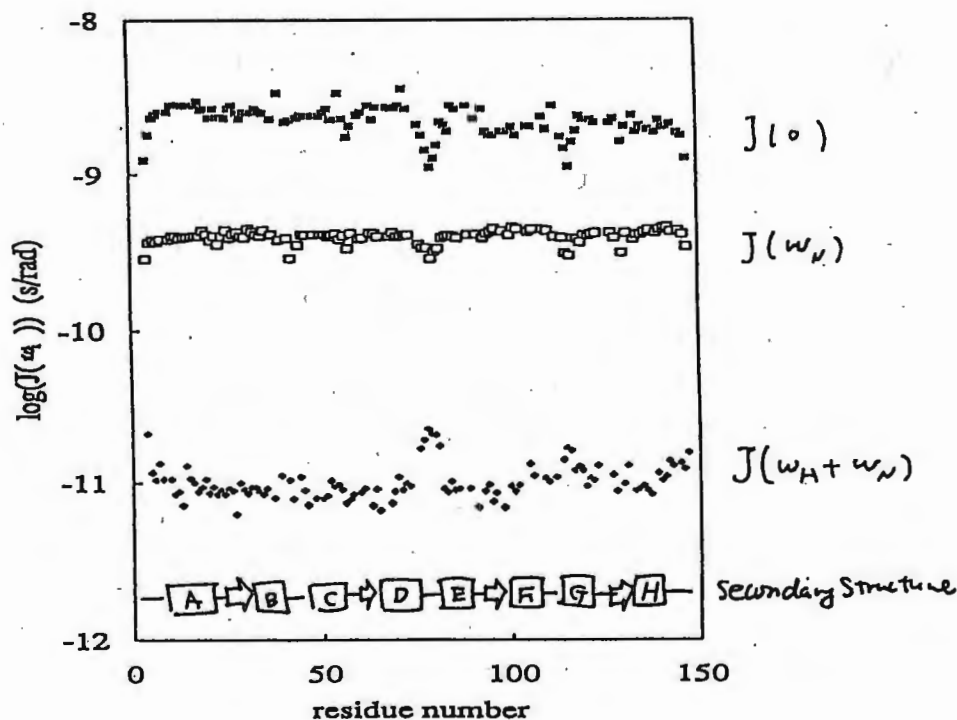
Rieko Ishima



Kuniaki Nagayama

ERATO Nagayama Protein Array Project,  
Tsukuba Research Consortium, Pilot lab.,  
5-9-1 Tokodai, Tsukuba 300-26 Japan

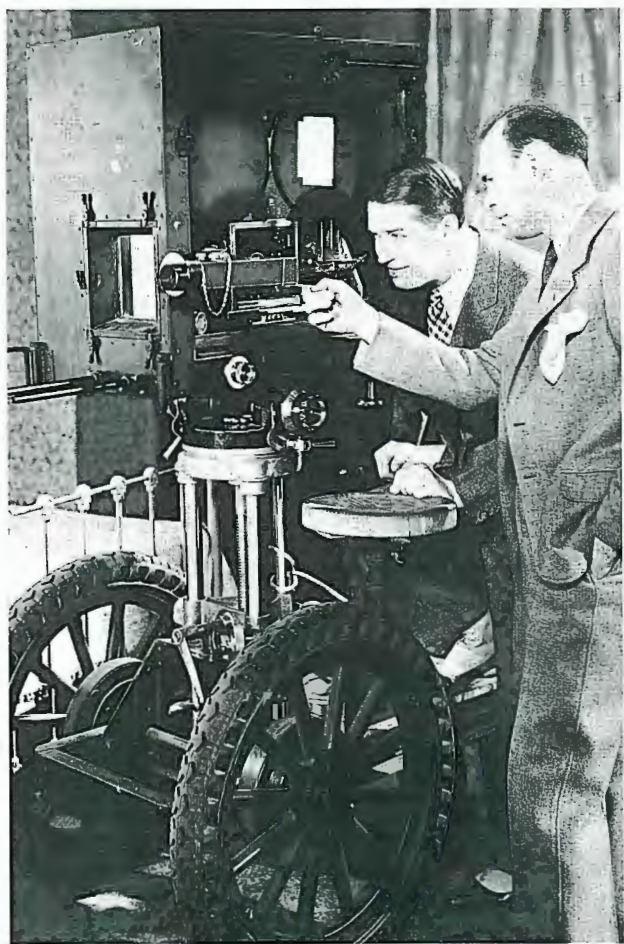
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200	54	2	365	2.8
100	54	1	365	2.8
500	89	15	120	3.4
400	89	10	180	2.8
360	89	10	365	2.8
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# OXFORD

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*Henry Ford Hospital*



Department of Neurology

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Professor B.L. Shapiro,  
TAMU NMR Newsletter,  
966 Elsinore Court,  
Palo Alto, CA 94303

November 3, 1994  
(received 11/7/94)

#### FIELD MAPPING/CHANGE OF ADDRESS

Dear Professor Shapiro,

Please note my recent change of address. Proton MR spectroscopic imaging (SI) is gaining increasing popularity amongst the research community as a means of evaluating cerebral metabolism in numerous different pathological conditions. The NIH group have recently developed a multi-slice SI sequence which allows most of the human brain to be scanned in a single, 30 minute data acquisition (1). A pre-requisite for high resolution proton spectroscopy of large volumes is successful adjustment of magnetic field homogeneity; this may be done automatically on the GE Signa system (2).

It is also useful to have a field map, after shimming has been completed, which may be used to correct residual frequency shifts in the SI data sets ("susceptibility correction"). This information may be used to improve the quality of metabolic images which are reconstructed from the SI data. We have been using a simple double gradient echo sequence for this purpose. The data acquisition parameters are as follows: 1 NEX, TR 250 msec, TE = 10 and 20 msec, 45° flip angle, 256x128 matrix size, four 15 mm slices (prescribed at the same locations as the four slices of the SI data set), giving a 33 sec data acquisition time. Images of the frequency of the water resonance are calculated from  $\Delta f = \arg(E_1/E_2)/\Delta TE$ . The figure shows a field map from one of the four axial-oblique slices, and an NAA SI image reconstructed with and without susceptibility correction:

Note the relative degradation of field homogeneity in the most anterior and posterior brain regions (linear shim corrections only). The worst region occurs in the anterior mid-brain section of the lowest slice, resulting from the air-tissue interfaces of the petrous air spaces. The NAA image created without susceptibility correction shows artifactual hyper- and hypo-intensities resulting from field inhomogeneities; the corrected image shows an essentially uniform NAA distribution at this level of the brain.

Sincerely Yours,

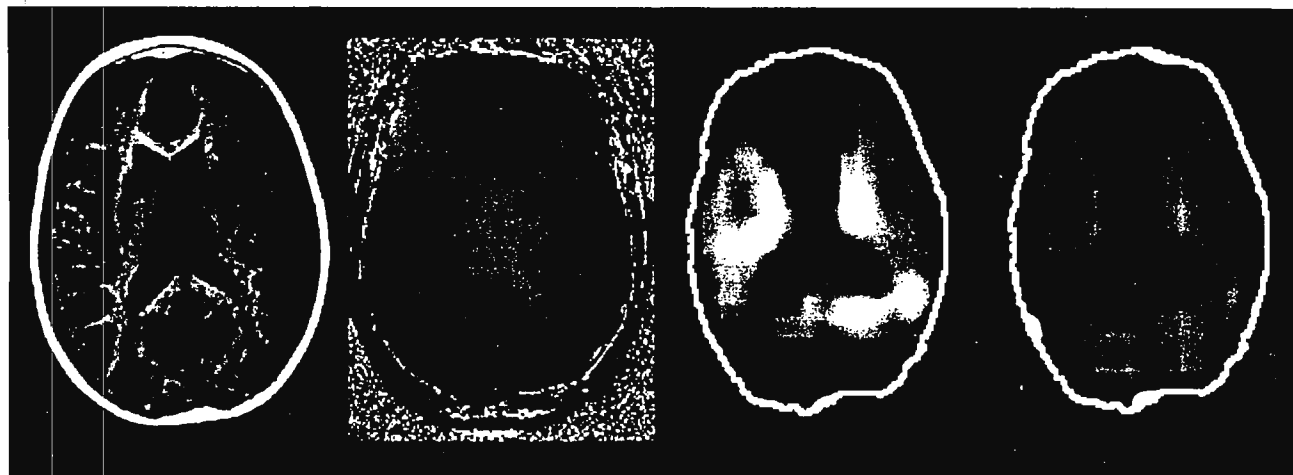
*Peter Barker*

Peter B. Barker, D. Phil.,

(1) Duyn, JH, Gillen J, Sobering G, et al.. Radiology 188, 277-282, (1993).

(2) Glover, GH, Schneider, E, Magn. Reson. Med., 18, 371-383, (1991), Webb, P, Macovski, A, Magn. Reson. Med., 20, 113-120, (1991).



**T<sub>1</sub> MRI****Field Map****No Correction****Corrected**

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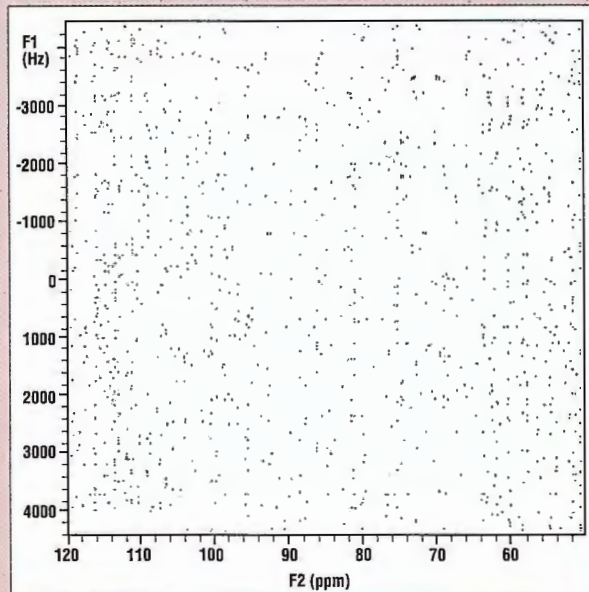
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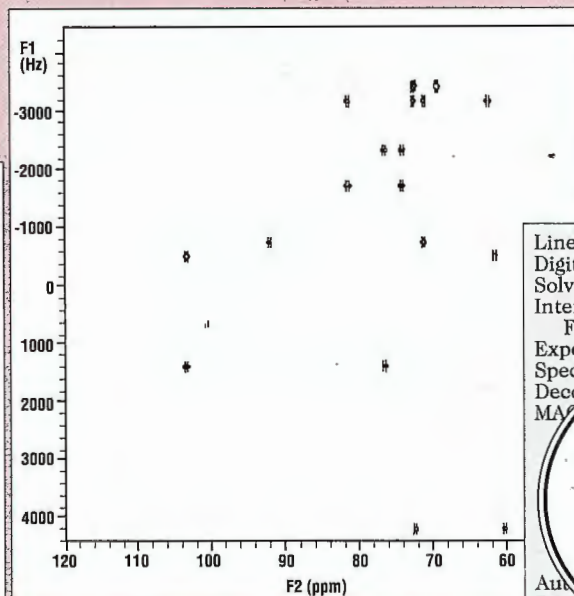
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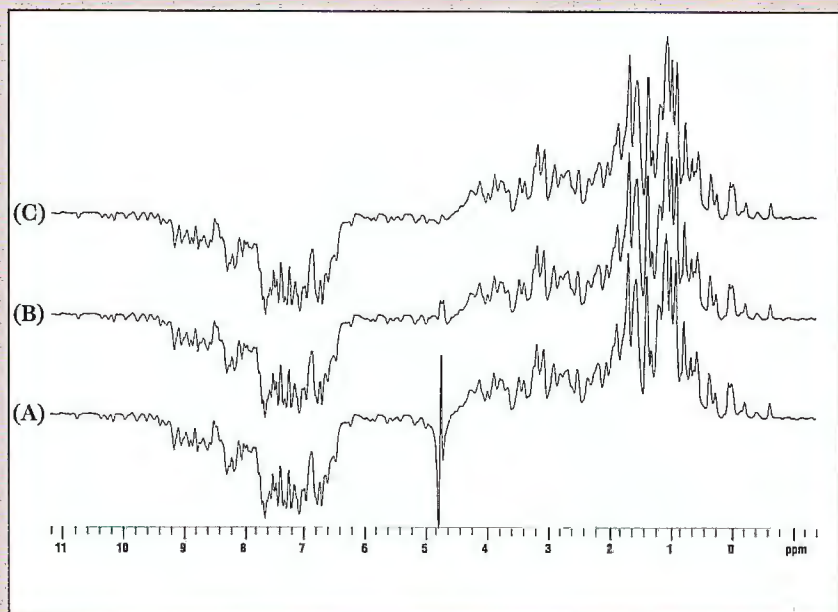


# Featured at This Year's ICMRBS\*

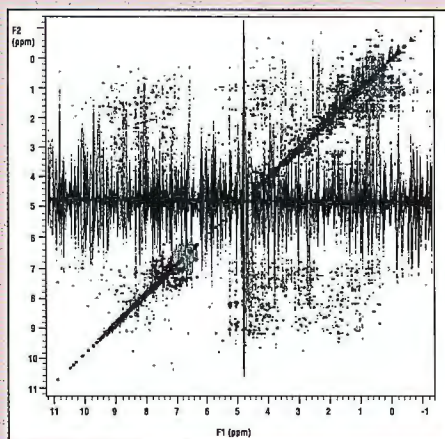
\*International Conference on Magnetic Resonance in Biological Systems

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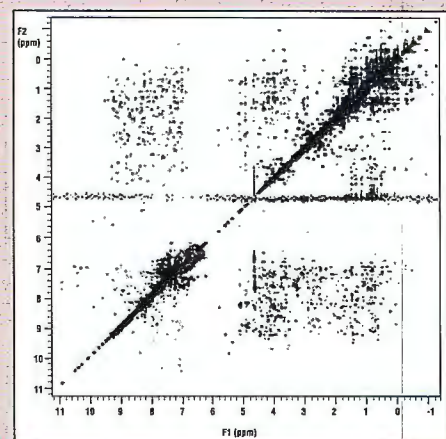
*The residual water signal in a first increment SSNOESY experiment, shown after (A) Simple FT (B) Low-frequency subtraction (LFS) and (C) Zero-frequency subtraction (ZFS).*



*A non-excitation 2D sequence (SSNOESY) on 1 mM lysozyme, dramatically improved in (B) by Zero-frequency subtraction processing in  $t_2$ . Note the peaks extremely close to the water in F2.*



(A)



(B)

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Department of Chemistry

Professor Gideon Fraenkel

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(received 11/14/94)

November 7 1994

Missing Link

Benzyllithium:

 $^1J(^{13}\text{C}, ^6\text{Li})$ 

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

Dear Barry:

Interpretation of coupling constants has always been a theoretical muddle, in part due to their small size. Coupling between  $^{13}\text{C}$  and directly bonded  $^6\text{Li}$  in many organolithium compounds  $\text{R}_n\text{Li}$  falls into a remarkably simple pattern.  $^1J(^{13}\text{C}, ^6\text{Li})$  is ca.  $17/n, \text{Hz}$ ,  $n$  being the state of aggregation and appears to be largely independent of the nature of  $\text{R}$ : Allylic lithium compounds, believed to be ion-pairs, do not exhibit carbon-lithium coupling. The absence of observable coupling may be due to fast lithium relaxation (not likely for  $^6\text{Li}$ ) or fast intermolecular  $\text{C}_\omega\text{Li}$  bond exchange. One might expect that between the "common pattern"  $\text{R}_n\text{Li}$  species and the ion-pairs there should be a continuum of decreasing covalent character to the  $\text{C}_\omega\text{Li}$  bonds with accompanying decreasing values of  $^1J(^{13}\text{C}, ^6\text{Li})$ . A set of benzylic lithium compounds we have studied lately, provide precisely such a missing link, exhibiting the expected multiplicity of the benzylic  $^{13}\text{C}$  resonance or just a broad line which narrows on irradiation at the  $^6\text{Li}$  frequency. All these compounds have similar ring  $^{13}\text{C}$  shifts indicating, qualitatively, similar electronic structures and significant negative charge delocalized into the aromatic rings. In the case of benzyllithium- $^6\text{Li}$  itself, use of dilute (0.001 M)  $^{13}\text{C}_\alpha$  enriched, complexed to  $\text{N,N,N,N}$ -tetramethylethylenediamine and low temperature, 180 K, provides the same  $^{13}\text{C}$  signal/noise ratio as a  $^{13}\text{C}$  natural abundance sample, 0.1 M, and reduces the rate of intermolecular  $\text{C}_\omega\text{Li}$  exchange, compared to the latter sample, by a factor of 100. The faster exchange process in the 0.1 M natural abundance sample would be due to bimolecular exchange of lithiums between  $^{13}\text{C}_\alpha$  and  $^{12}\text{C}_\alpha$ . In fact, the dilute enriched sample shows a clean 1:1:1  $^{13}\text{C}_\alpha$  multiplet at 180 K due to  $^{13}\text{C}_\alpha\text{Li}$  coupling of 3 Hz whereas in the case of the normal ( $^{13}\text{C}$  in natural abundance) 0.1M sample the  $^{13}\text{C}_\alpha$  resonance is just a slightly broadened single line, due to fast  $\text{C}_\omega\text{Li}$  exchange.

What this means in the cosmic scheme is that in most secondary benzylic lithiums and benzyllithium itself the  $\text{C}_\alpha\text{Li}$  bond has a detectable degree of covalence with some associated "s" character and so the arrangement about  $\text{C}_\alpha$  deviates from planar  $\text{sp}^2$ . This is the missing link alluded to above.

The trick has been to use dilute  $^{13}\text{C}_\alpha$  enriched samples at low temperature to minimize the intermolecular  $\text{C}_\omega\text{Li}$  exchange rate. This is accomplished by internal solvation of lithium in **1**, the first time we saw  $^{13}\text{C}_\omega\text{Li}$  coupling in a benzylic lithium compound, 2.8 Hz.

With best wishes,

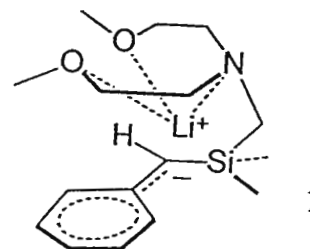
Yours sincerely,

Gideon

Gideon Fraenkel  
 Professor of Chemistry

K. M

Kevin Martin  
 Research Associate



GF:klk.



National Institutes of Health  
 Building 5, Room 112  
 9000 Rockville Pike  
 Bethesda, MD 20892  
 November 8, 1994

(received 11/14/94)

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

### A New Book on NMR and Molecular Dynamics

Dear Barry:

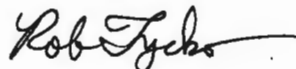
I would like to call your attention to a book that I have edited, entitled "Nuclear Magnetic Resonance Probes of Molecular Dynamics". The book deals with applications of NMR to studies of molecular dynamics in a very wide variety of chemical and biochemical systems and with recent developments in techniques and theory that make these applications possible. The chapter titles and authors are as follows:

- I. How Does Nuclear Magnetic Resonance Probe Molecular Dynamics? (R. Tycko)
- II. Deuterium NMR Studies of Dynamics in Solids and Liquid Crystals (R.R. Vold)
- III. Molecular Dynamics in Polymers Studied by Multidimensional Solid State NMR (B.F. Chmelka, K. Schmidt-Rohr, and H.W. Spiess)
- IV. Dynamic Magic Angle Spinning NMR Spectroscopy (S. Vega)
- V. Relaxation-Induced Transfer of Nuclear Spin Polarization as a Probe of Molecular Structure and Dynamics in Mobile Phases (L.G. Werbelow)
- VI. Pressure as an Experimental Variable in NMR Studies of Molecular Dynamics (J. Jonas)
- VII. Connections between NMR Measurements and Theoretical Models of Structural Dynamics of Biopolymers in Solution (R. Brüschweiler)
- VIII. Investigating Furanose Ring Dynamics in Oligonucleotides with Solid State  $^2\text{H}$  NMR (D.L. Mattiello and G.P. Drobny)
- IX. Protein Mobility from Multiple  $^{15}\text{N}$  Relaxation Parameters (J. Peng and G. Wagner)
- X. Transport Ordered 2D NMR Spectroscopy (C.S. Johnson, Jr.)
- XI. PGSE NMR and Molecular Translational Motion in Porous Media (P.T. Callaghan and A. Coy)
- XII. NMR Spectroscopy and Dynamics at Catalytic Surfaces (T.M. Duncan)

The book is due to be published on November 24 by Kluwer Academic Publishers. The list price is \$252, but the publisher has agreed to sell the book for \$69 in quantities of six or more. Discounts for personal copies may also be obtained by contacting Dr. David Larner, Science and Technology Division, Kluwer Academic Publishers, P.O. Box 17, 3300 AA Dordrecht, The Netherlands (e-mail: wil.bruins@wkap.nl).

The authors have all done an outstanding job of presenting their material. The book should serve as an excellent introduction to the field for novices and as a valuable reference for experts.

Sincerely,



Dr. Robert Tycko  
 301-402-8272  
 tycko@helix.nih.gov

# Automatic NMR Sample Preparation

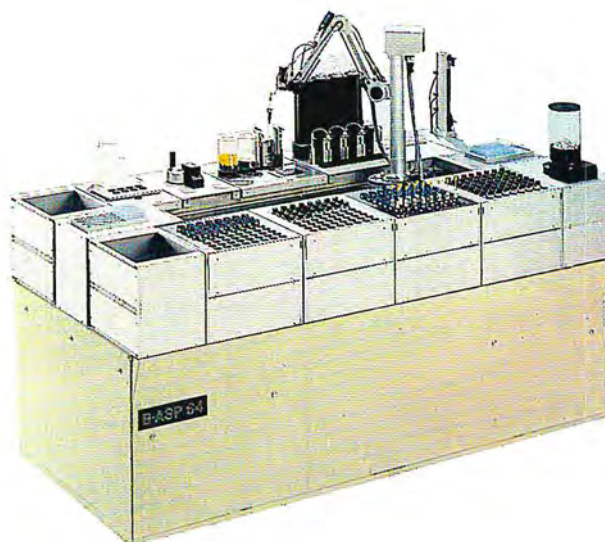


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Output trays:	holding 64 sample tubes each
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NMR sample tube dispenser and assembly station:	available for 5 mm tubes
Sample filtration and dosage station:	ca. 128 filters, 128 caps
Barcode collar dispenser:	ca. 150 collars
Dimensions:	with stations 1800 x 970 mm
Height closed:	170 mm
Height open:	210 mm
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# ROSKILDE UNIVERSITY

Associate professor Poul Erik Hansen, Department of Life Sciences and Chemistry

e-mail: Poulerik@mmf.ruc.dk

P.O.Box 260, DK-4000 Roskilde, Denmark



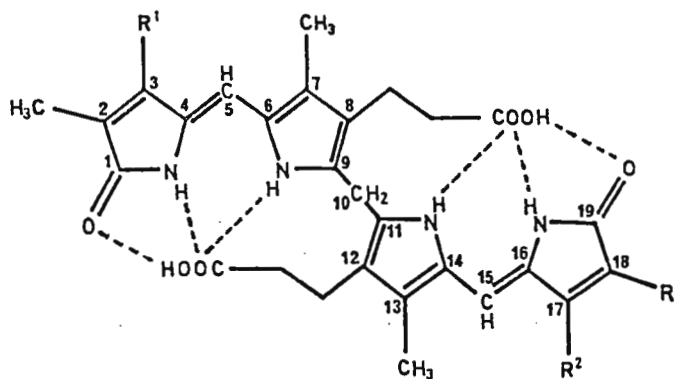
Prof. B.L.Shapiro  
966 Elsinore Court,  
Palo Alto, CA 94303  
USA

November 2 1994  
(received 11/7/94)

Mile high  $pK_a$  values for the carboxylic acid groups of bilirubin. Help!

Dear Prof. Shapiro

Somebody said long ago "I thought bilirubin had been figured out by now". But no, especially the determination of the  $pK_a$  values continues to cause debate.



Bilirubin is very insoluble, so fifteen years ago we determined the  $pK_a$  values by co-titration in DMSO using  $^{13}C$  NMR. The result was quite normal  $pK_a$  values around 4.4 for both carboxylic acid groups after correction for solvent effects.<sup>1</sup> More recently, Ostrow *et al.* have found, that the  $pK_a$  values are 8.1 and 8.4.<sup>2</sup> Recently, the values have been estimated by micellar electrokinetically capillary chromatography.<sup>3</sup> The values were between 6.2 and 6.5, but claimed not to be very accurate. In other words, quite a spread has been established. We are presently attempting to determine the values in cholate micelles by NMR.

Hydrogen-bonding of the OH groups was claimed by Ostrow to be the reason for the unusually high  $pK_a$  values.

I would be glad to know, if anybody has found unusually high  $pK_a$  values for carboxylic acids in e.g. proteins or other biomolecules.

Yours sincerely

*Poul Erik Hansen*  
Poul Erik Hansen

<sup>1</sup> P.E.Hansen, H.Thiessen, R.Brodersen, *Acta Chem.Scand.* B33, 281 (1979)

<sup>2</sup> J.-S.Hahm, J.D.Ostrow, P.Mukerjee and L.Celic, *J.Lipid Res.* 33, 1123 (1992)

<sup>3</sup> A.D.Harman, R.G.Kibey, M.S.Sablik, Y.Fintschenko, W.E.Kurtin and M.M.Bushey, *J.Chromatograph A*, 552, 525 (1993).



## **MAGNETIC RESONANCE AND THE ENVIRONMENT**

### **CONFERENCE/WORKSHOP**

**Richland, Washington**  
**April 19 & 20, 1995**

A new scientific collaborative user facility, the Environmental Molecular Sciences Laboratory (EMSL), is under construction at Pacific Northwest Laboratory (PNL). Full operation is scheduled to begin in late 1997. The primary mission of this DOE-sponsored laboratory is to develop, refine, and use state-of-the-art research methods for investigating the molecular processes that control complex environmental processes. It is expected that the research at EMSL will contribute significantly to a safe, effective, and efficient cleanup of hazardous waste sites.

#### **Magnetic Resonance Facility**

Part of the EMSL will be a major magnetic resonance facility, which together with a mass spectroscopy facility constitutes the EMSL Macromolecular Structure and Dynamics (MS&D) group. The research in this group will focus on the following topics:

- the effects of exposure to toxic chemicals and/or ionizing radiation on the molecular, cellular, and tissue level
- catalytic destruction and conversion of chemical and radioactive wastes
- characterization of the processes that control the biological degradation of contaminants
- in situ and ex situ characterization of highly toxic stored waste and contaminated groundwater.

The MS&D group will be equipped with a large variety of liquid-state and solid-state NMR spectrometers, operating in different fields, including a 1-GHz, high-resolution NMR spectrometer and state-of-the-art EPR spectrometers.

#### **Conference/Workshop**

On April 19-20, 1995, a conference/workshop entitled "Magnetic Resonance and the Environment" will be held at PNL. The purpose of this event is twofold:

- In the scientific part of the meeting, lectures will be held on the MS&D research program and invited speakers will give seminars on related topics.
- In the workshop part of the meeting, the possibilities of the NMR facility as a user's facility will be discussed. Up to 50% of the operational time of EMSL facilities and equipment will be made available to external scientists, and in the workshop the realization of collaborative work and the establishment of a user's review committee will be discussed.

#### **Preliminary program:**

##### **Wednesday, April 19:**

Morning: Introductory and keynote lectures  
Afternoon: Tour of the NMR facility and the Hanford site  
Evening: Informal dinner

##### **Thursday, April 20:**

Morning/Afternoon: Specialized lectures  
Evening: User's Session/Discussion

#### **Registration**

Details about the program and the costs will be given in the second announcement. If you are interested in attending the conference/workshop, please contact Ginny Woodcock or Robert Wind no later than January 30, 1995.

---

#### **Registration information:**

Ginny Woodcock  
Pacific Northwest Laboratory  
P.O. Box 999, MSIN P7-55  
Richland, WA 99352

Telephone: (509) 372-3878  
Facsimile: (509) 376-2303  
E-mail: vd\_woodcock@pnl.gov

#### **Technical information:**

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0.2 mg/ml 0.1% 1%	Gadolinium Chloride DSS (Sodium 2,2-dimethyl-2-silapentane-5-sulphonate) Water in Deuterium Oxide	<sup>1</sup> H	Homogeneity
1%	Chloroform in Acetone-d <sub>6</sub> (min. 99.9%D)	<sup>1</sup> H	Line Shape
5%	Chloroform in Acetone-d <sub>6</sub> (min. 99.9%D)	<sup>1</sup> H	Line Shape
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Professor B.L. Shapiro  
TAMU Newsletter  
966 Elsinore Ct  
Palo Alto, CA 94303

November 9, 1994  
(received 11/16/94)

**Optimized shims in 20 minutes, ..... starting from cryoshims**

Dear Barry,

It was a typical Californian afternoon in our laboratory. Mark was making socially-acceptable coffee, Peter was trying to remove the key chain from his new Macintosh power book, Ralph was running up and down the stairs trying to time his appearance in agreement with the usual sounds of coffee that is ready to be poured and Tim was smoking outside.

I had nothing to do for about 20 minutes and it seemed like a good idea to zero all the room temperature shims and reshim our 500 MHz from cryoshim. The spectrum with just cryoshim (Fig. 1) looked promisingly disgusting for this ultimate test of our new 3D image-based field mapping approach (J. Magn. Reson. A 111, 203-207 (1994), December issue). In this approach we map the inhomogeneous field in a 3D image and, using previously-established field maps of our 28 shims, optimize the shims in a computerfit. This procedure is initiated and completed with one single 6-letter command. For the sucrose lineshape sample (2 mM sucrose in 90/10 H<sub>2</sub>O/D<sub>2</sub>O) this gradient shimming took 20 min for five iterations (five field maps). The resulting presaturation spectrum (Fig. 2) showed a very symmetric solvent lineshape with a bottom linewidth of 30 Hz at half the height of DSS, much better than any of the usual values (about 60 Hz) after one to two days of intense and stressful shimming. The splitting in the anomeric sucrose (5.4 ppm) was down to 85% of the top, also much better than any previous hand shimming. While the coffee was being poured, we decided that it would be cute to shim a BPTI sample from cryoshim also (Fig. 3, 4 iterations = 16 min). For researchers that are worried about the fact that 4 iterations are needed, we are happy to report that when the experiment is started at standard magnet shims, only one iteration is necessary.

As you see, shimming will no longer be a time-consuming problem and savings in man-hours (and thus billions of \$\$\$\$\$\$\$\$) for companies and universities will be astronomical. However, all we are worrying about is what to do during the next coffee preparation. Mark is considering to buy a new ultrafast coffee machine. Together with a warning mechanism in the other building, this approach may improve Ralph's timing for the final pouring.

**Shimming, it isn't magic, it's ..... imaging**

Subramanian Sukumar

Mark O'Neil Johnson

Ralph E. Hurd  
GEMS

Peter C.M. van Zijl  
JHU

Tim Kelly



FIGURE 1

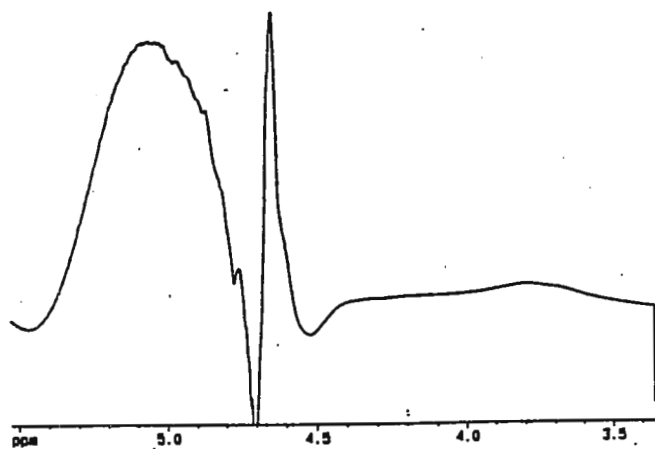
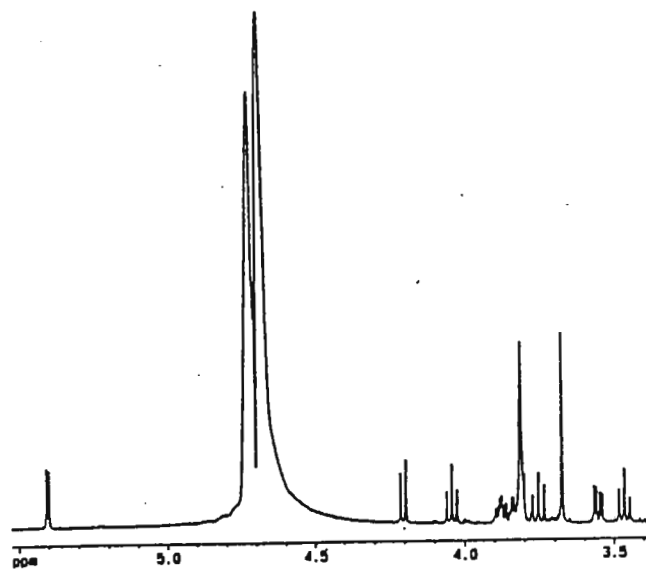


FIGURE 2



Presaturation spectra for 2mM Sucrose in 90/10 H<sub>2</sub>O/d<sub>2</sub>O with cryoshims only (Figure 1) and after five iterations of gradient shimming (Figure 2)

FIGURE 3

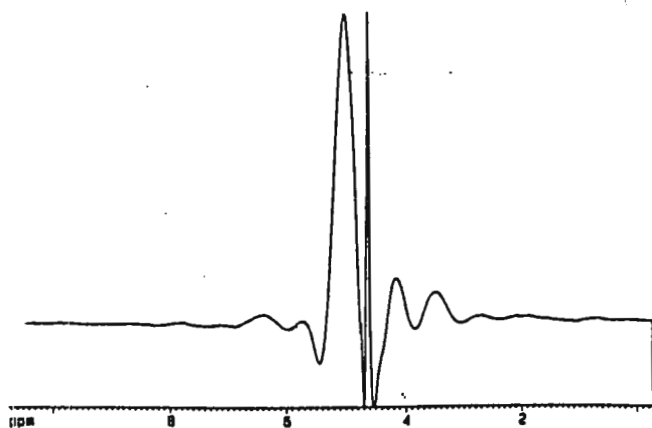
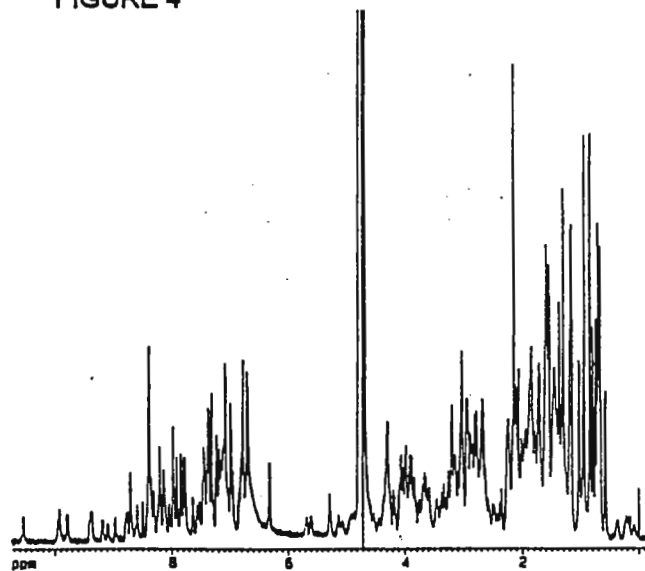


FIGURE 4



A presaturation spectrum for 1.5 mM BPTI in 90/10 H<sub>2</sub>O/D<sub>2</sub>O with cryoshims only (Figure 3) and after four iterations of gradient shimming (Figure 4)

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

Dear Barry,

## Positions Available

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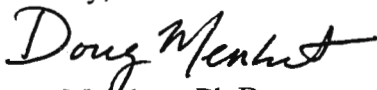
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Sincerely,



Doug Memhart, Ph.D.  
NMR Applications Manager



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July 22, 1994  
(received 11/19/94)

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

## ATTENUATION BY PROGRAMMABLE PULSE MODULATION

Dear Barry:

Not too long ago, I was at a customer site with an older VNMR based Varian high resolution NMR instrument. The customer was interested in carrying out an nOe difference experiment. Unfortunately, the hardware set-up, with the standard 63 dB hardware attenuation, did not provide for the needed saturation pulse selectivity. Luckily, the spectrometer had a wave-form generator. The programmable pulse modulator (PPM) offers the ability to attenuate a selective excitation pulse without or in addition to hardware attenuators placed in line thereby providing for an additional level of control over selectivity. A pulse sequence "cyclenoePPM.c" is referred to which is currently available on-line from the Varian USERLIB (*Mag. Mom.* V(2), 14, 1992), along with a macro and set of parameters. This sequence was adapted (by yours truly) from cyclenoe.c. Certain elements of the sequence involving PPM pulse sequence statements are discussed along with some required calibrations.

The key pulse sequence element is the "shaped\_pulse" statement which takes five arguments. The first element is a shape from the "shapelib" directory. Two non-s2pul parameters are defined as character strings, "satshape" for the saturation shaped pulse and "conshape" for the control shaped pulse. The second argument is the width of the pulse. This is "tau" or "sattime", depending on whether frequency cycling over the multiplet pattern is used or not. The third argument is the phase of the shaped pulse, and the fourth and fifth arguments are, respectively, the receiver off and transmitter on gating times.

Figure 1A shows a spectrum of an expansion for 20 mM gramicidin S in DMSO. The "quartet" at 4.56 ppm was irradiated using a gamma-H2 of 11 Hz with the cyclenoePPM.c sequence using internal subtraction with the result shown in figure 1B. The relevant parameters include satpwr=20, spacing=7.2 Hz ( $^1J_{H-H}$ ), pattern=4, intsub='y', cycle='y', control=-21.3, tau=0.100, satshape='hard102', conshape='hard102', LB=1Hz and nt=128. Gamma-H2 was calibrated using the "shapecal.c" pulse sequence with the results given in figure 2.

Yours sincerely,

Fernando Commodari, Ph.D.

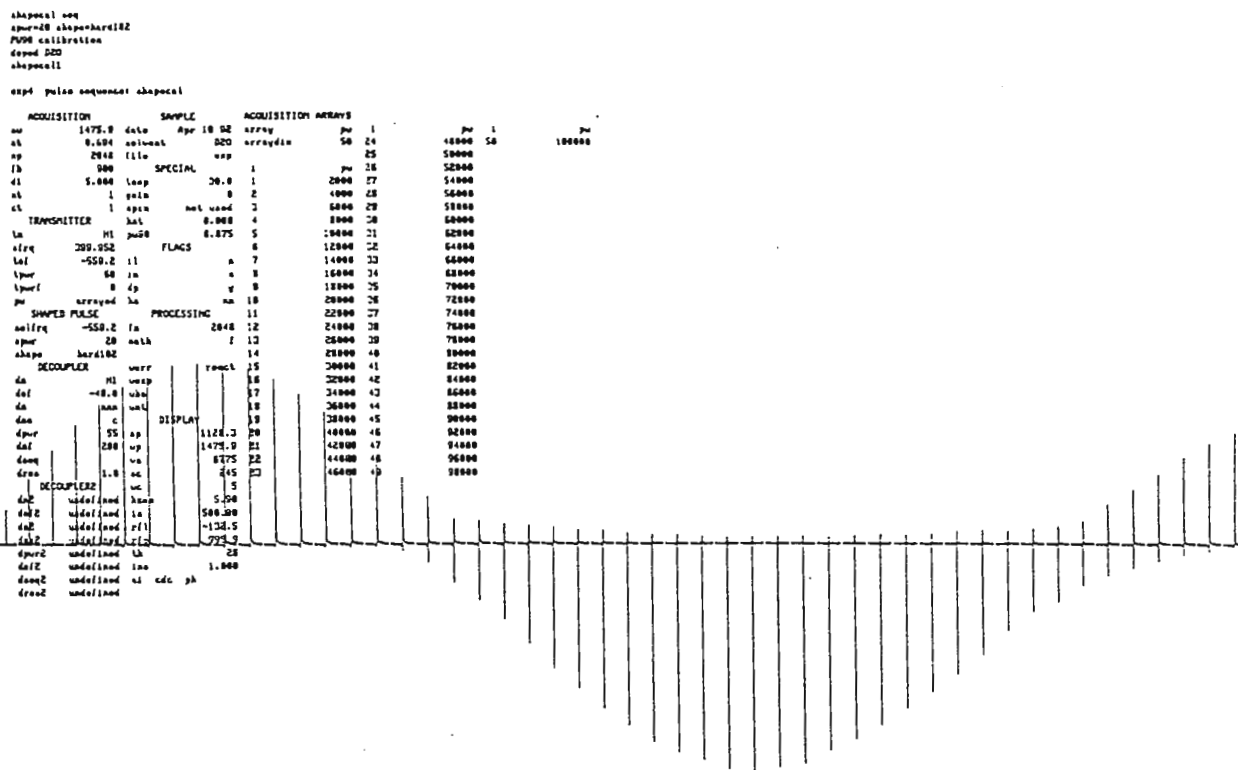
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 Probe: SuiB Operator: Fernando Gonzalez Verian-Florian Park

Figure 2. Shapcal.c sequence used to calibrate  $\gamma\text{H}_2$ . The null at 360 is 88000  $\mu\text{s}$  leading to a  $\gamma\text{H}_2$  ( $=1/360$ ) of 11 Hz with spwr=20 and shape='hard102'. Sample: Doped  $^2\text{H}_2\text{O}$

As figure 2 shows, setting spwr=20 and shape='hard102' provides about 60 dB of attenuation ( $\gamma\text{H}_2=11\text{Hz}$ ). This leaves about 20 dB more just in the "spwr" parameter; setting spwr=0 should give a  $\gamma\text{H}_2$  of about 1-2 Hz. One may also use a 'hard10' or a smaller amplitude, shaped rectangular pulse, for finer control of selectivity of the saturation pulse.

All of this is possible on a standard system, equipped with programmable pulse modulators, without any hardware "fiddling" or upgrades to 73 dB attenuators.

The "hard102.RF" and "hard.RF" ASCII files for the shapelib are shown below:

```

#Hard:          Simple Rectangular Pulse
                0.0      1023.0  1.0

#Hard102:       0.1 the amplitude of a simple rectangular pulse
                0.0      102.0   1.0

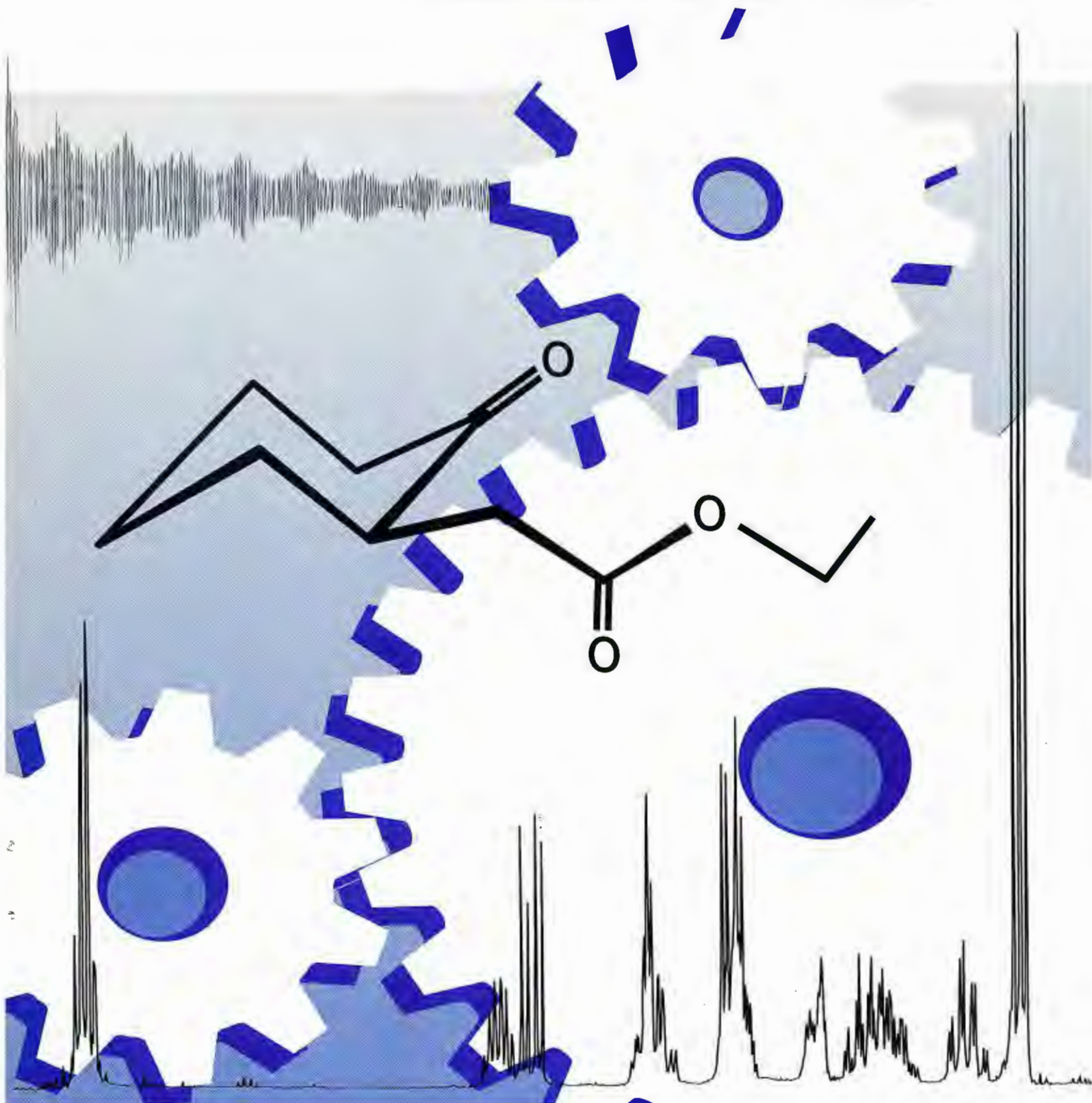
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The second column represents the amplitude of the shaped pulse.



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RE: **Deriving Distance Restraints from Single Mixing Time NOESY Dataset**

Dear Barry,

The increased spectral dispersion afforded by multidimensional heteronuclear NOESY has greatly contributed to the number and quality of protein solution structures that have been solved. Central to the structure determination process is the extraction of distance restraints from the NOESY data. It has long been established that the distance between two protons,  $r_{ij}$ , may be determined from Equation 1.

$$\sigma_{ij} = \frac{A}{r_{ij}^n} \quad (1)$$

where  $n = 6$  for rigid structures,  $A$  is a function of the correlation time,  $\tau_c$  and  $\sigma_{ij}$  is the cross relaxation rate. A cross relaxation rate can be derived under a two spin assumption by fitting NOE intensities from multiple mixing times.

In general, it is not practical to collect 3D or 4D NOESY data at several mixing times due to the acquisition time of the experiments and perhaps from sample instability and finite disk space. Instead, the measured NOE intensities,  $I$ , from one mixing time dataset may be used to determine the distance between the protons pairs. Assuming  $I_{ij}$  is linearly proportional to  $\sigma_{ij}$ , the intensity is substituted for the cross relaxation constant,  $\sigma_{ij}$ , as shown in Equation 2.

$$I_{ij} = \frac{A'}{r_{ij}^n} \quad (2)$$

However, even in the absence of spin diffusion, the problem of using a single mixing time dataset to represent the cross relaxation constant is that the NOE intensities are not linearly proportional to the mixing time. In particular, for molecules having longer correlation times, the nonlinear terms affect the larger intensities (short interproton distance) at longer mixing times. Therefore, the use of a short mixing time to minimize the nonlinear effects is often employed, but this will provide less signal in the dataset.

A common way of deriving distances from a single mixing time NOESY dataset is to group the NOE intensities into three bins: strong/medium/weak e.g. 2.7/3.5/5.0 Å. The loose bounds of the bin method tend to compensate for nonlinearity as well as spin diffusion. One drawback of the bin method is that the fine resolution of each intensity may be neglected. For example, an NOE cross peak whose intensity is slightly below the cut off intensity value may be categorized in a weaker bin and be given a looser restraint. On the other hand, an NOE of a similar

magnitude but slightly larger than the cut off value would be categorized in a stronger bin and be given a tighter restraint. Five bin distance restraints have been used to reduce the discontinuity of the three bin method [1]. A second drawback is that the subjective nature of establishing the intensity limits between bins often becomes a trial and error process that is solved by minimizing the individual NOE term penalty during the structure calculation.

The method presented here alleviates the nonlinear effect and derives from a single mixing time NOESY dataset each upper boundary distance restraint using a continuous function. To correct this nonlinearity,  $A$  in Equation 2 varied smoothly, for each of the intensities, between the values calculated for the two intensity extremes. Each NOE intensity is thereby treated independently along the distance range and not subjected to strict boundary limits. This method has been used to derive distance restraints from 4D  $^{13}\text{C}/^{13}\text{C}$  NOESY ( $t_m = 75$  msec), 4D  $^{13}\text{C}/^{15}\text{N}$  NOESY ( $t_m = 65$  msec) and 3D  $^{13}\text{N}$  NOESY-HSQC ( $t_m = 65$  msec) datasets recorded for structure determination on a complex of an SH2 domain with a pentapeptide [2].

After peak intensity normalization [1], the 10 weakest ( $I_w$ ) and the 10 strongest ( $I_s$ ) cross peaks were averaged and assumed to correspond to protons separated by the distance extremes of 5 Å or 1.8 Å, respectively. The conversion factors,  $A(I_w)$  and  $A(I_s)$ , were then calculated with Equation 3.

$$A(I) = r_{ij}^6 I \quad (3)$$

For the more intense cross peaks, the nonlinear relationship between intensity and mixing time becomes more apparent so that  $A(I)$  was smaller than  $A(I_w)$ . The ratio of  $A(I_s)/A(I_w)$ , from Equation 3, was about 0.32 in the  $^{15}\text{N}$  edited 3D NOESY-HSQC and 4D  $^{13}\text{C}/^{15}\text{N}$  NOESY data. In the 4D  $^{13}\text{C}/^{13}\text{C}$  NOESY,  $A(I_w)$  was equal to  $A(I_s)$  suggesting the nonlinearity associated with the side chain proton NOE intensities was not significant at a 75 msec mixing time.

In the absence of a gauge that predetermines the distances for the intensities in the intermediate range, a linear function was derived for  $A(I)$  with  $A(I_s)$  and  $A(I_w)$  at the two extremes (Equation 4).

$$A(I) = \frac{(I_s - I)}{(I_s - I_w)} [A(I_w) - A(I_s)] + A(I_s) \quad (4)$$

The distance corresponding to the NOE intensity was then calculated by Equation 5:

$$distance = \left( \frac{A(I)}{I} \right)^{1/6} \quad (5)$$

The coordinates from a distance geometry(DG)/simulated annealing (SA) structure calculation using X-PLOR were used to compare the continuous upper boundary to the three bin restraints method. In both X-PLOR calculations the same 1751 NOE intensities were used to generate the two restraint tables. The continuous upper boundary limit was 10% greater than the calculated distance and was used to compensate for the possible enhancement of the NOE caused by spin diffusion. When the calculated distance from Equations 4 & 5 was shorter than 2.1 Å, the distance was limited to 2.1 Å to prevent excessive restraint. All lower distance boundaries were set to 1.8 Å in order to compensate for the possible loss of NOE intensity due

to different relaxation properties of the system or spin diffusion. The discontinuous three bin restraints used were strong (1.8 - 2.7 Å), medium (1.8 - 3.5 Å) and weak (1.8 - 5.0 Å). To insure a fair comparison, the bin intensity limit values were calculated from Equations 4 & 5. In both calculations the three bin method was employed for the intrapeptide (65) and peptide-protein (55) restraints. Each X-PLOR calculation yielded 23 structures and are compared. Slight improvements for most of the structural statistics resulted from the use of continuous distance restraints. This was especially true for the  $E_{\text{Noe}}$  and the Cartesian coordinate rmsd.

More detailed theoretical analysis of Equation 4 can be found elsewhere[3].

Best regards,



Robert Xu,



Mike Word,



Robert Gampe Jr.,



Don Davis,



Steve Brown

#### References:

1. Meadows, R. P., Nettesheim, D. G., Xu, R. X., Olejniczak, E. O., Petros, A. M., Holzman, T. F., Severin, J., Gubbins, E., Smith, H., and Fesik, S. W. (1993) *Biochemistry*, **32**, 754-765.
2. Xu, R. X., Word, J. M., Davis, D. G., Rink, M. J., Willard, D. H. Jr., and Gampe, Jr. R. T. *Biochemistry*, accepted.
3. Xu, R. X. submitted to *J. Biomol. NMR*.

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Applicants should send resumes, publications, and names and phone numbers of three references to Dr. Warren T. Ford, Department of Chemistry, Oklahoma State University, Stillwater, OK 74078. Fax 405-744-6007. Review of applications will begin December 15, 1994 and continue until the position is filled.

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Both appointments are for 2-3 years. Please encourage anyone interested to contact Robert Wind at the address given below.

***Robert A. Wind***  
***Environmental Molecular Sciences Laboratory***  
***Pacific Northwest Laboratory***  
***P.O. Box 999, MSIN P7-55***  
***Richland, WA 99352***

***Phone: (509) 376-1115***  
***Fax: (509) 376-2303***  
***E-mail: ra\_wind@pnl.gov***



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### All Newsletter correspondence should be addressed to

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966 Elsinore Court  
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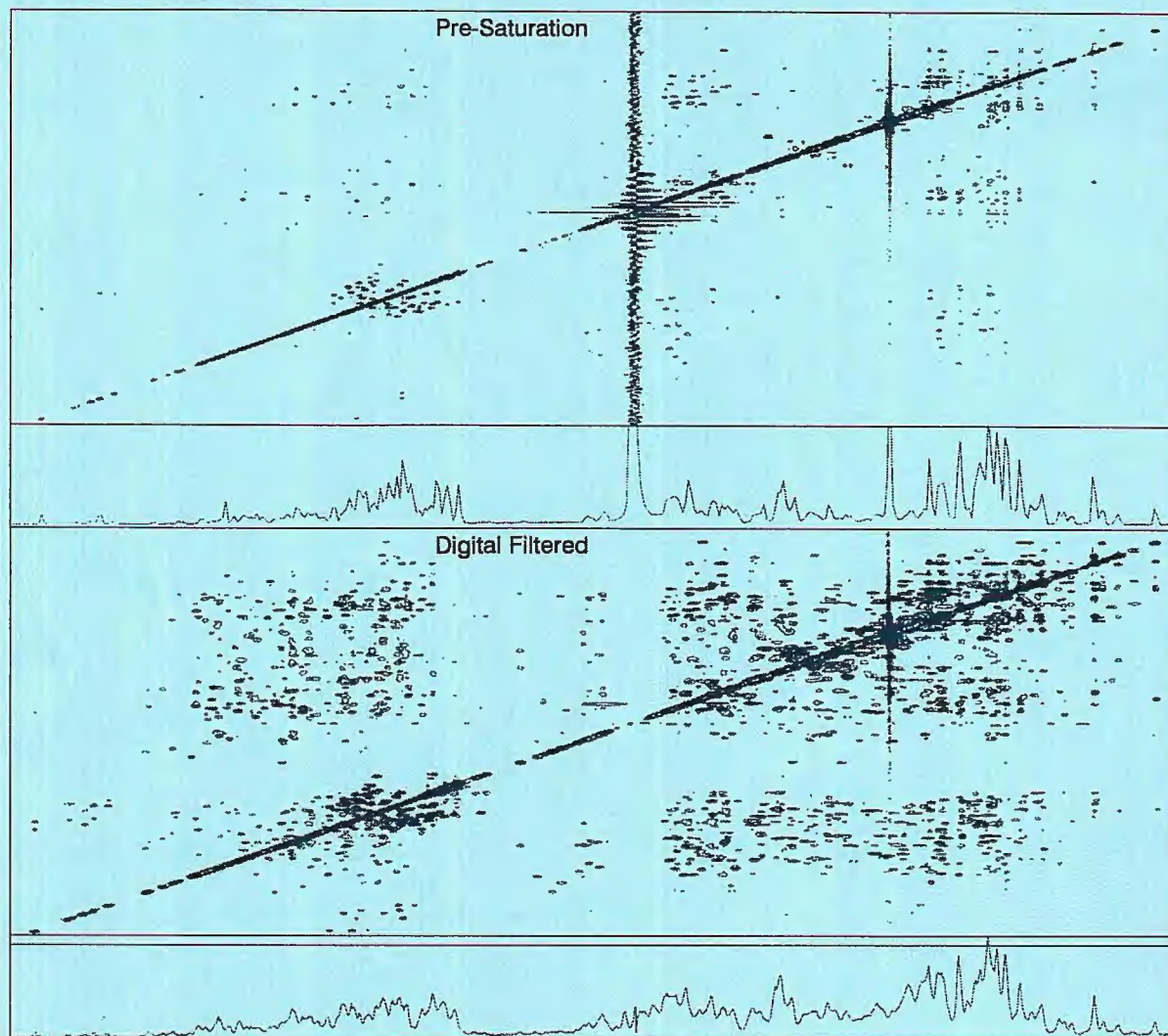
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