TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 416

May 1993

Continued on Page 44

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FORTHCOMING NMR MEETINGS

11th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, Swansea, Wales, U.K., **July 4 - 9, 1993**; 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.

Gordon Research Conference: Magnetic Resonance, Wolfeboro, NH, **July 11 - 16, 1993**; Contact: Dr. A. M. Cruickshank, Gordon Research Center. University of Rhode Island, Kingston, RI, 02881-0801; (401) 783-4011 or -3372; Fax: (401) 783-7644.

35th Rocky Mountain Conference on Analytical Chemistry, Denver, Colorado: **July 25-29, 1993**; Contact: Patricia L. Sulik, RML, Inc., 456 S. Link Ln., Ft. Collins, CO 80524; Phone: (303) 530-1169.

Science Innovation '93, Boston, Mass., **August 6 - 10, 1993**; "NMR Determination of Protein Structure", Discussion Leader - Ad Bax, *NIH*; : For information contact AAAS Meetings, 1333 H Street, NW, Washington, DC 20005; Phone: (202) 326-6450; Fax: (202) 289-4021; See *Science*, 260, 557-565 (23 April 1993).

12th Annual Scientific Meeting and Exhibition of the Society of Magnetic Resonance in Medicine, New York, NY, **August 14-20, 1993**; Contact: SMRM, 1918 University Ave., Suite 3C, Berkeley, CA 94704; Phone: (510) 841-1899; Fax: (510) 841-2340.

1993 FACSS Meeting, Detroit, Michigan, **October 17-22, 1993**; Contact: H. N. Cheng, Hercules, Inc., Research Center, 500 Hercules Road, Wilmington, DE 19808; Phone: (302) 995-3505; Fax:. (302) 995-4117. See TAMU NMR Newsletter 411, 10.

Pacific Conference, Pasadena, California, **October 19-23, 1993**; Contact: Ms. B. Belmont, Pacific Conference, 14934 S. Figueroa St., Gardena, CA 90248;Phone: (310) 538-9709.

12th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, Manchester, England, **July 2 - 7, 1995** [sic]; 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** [sic]; Contact: Dr. Wm. A. Bubb, Secretary, Univ. of Sydney, Dept. of Biochemistry, Sydney, NSW 2006, Australia. See TAMU NMR Newsletter 414, 8.

Additional listings of meetings, etc., are invited.

CENTRAL RESEARCH & DEVELOPMENT
Experimental Station
P.O. Box 80328
Wilmington, Delaware 19880-0328

March 29, 1993
(received 4/3/93)

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

New Uses for Dynamic NMR Simulations

Dear Barry,

We have recently observed hindered alkyl rotation on the surface of derivatized C_{60}. Dynamic ^1H NMR lineshapes were found for *t*-Bu-C_{60} anion in the temperature region -100 to -20°C, proceeding from a static spectrum consisting in the methyl region of a pair of signals in a ratio 2:1 (*gauche/trans*). Our computer program for general mutual exchange was specifically modified for this case according to the formalism of Johnson for intramolecular nonmutual exchange. Analysis of the temperature dependence of the estimated rate constants led to the activation parameters $\Delta H^{\ddagger}$= 3.6 kcal/mol and $\Delta S^{\ddagger}$= -26 e.u. The entropy of activation seems quite out of line for an intramolecular process, and it is supposed that this large negative value reflects some requirement for ion-pairing with the lithium counterion.

No such complications are anticipated for the corresponding *t*-Bu-C_{60} radical. EPR observation of this species again reveals dynamics due to hindered rotation. At 225°K we observe a quartet of septets with a(3H) = 0.335G and a(6H) = 0.085G consistent with strong coupling to a single *trans* methyl group and weaker coupling to the 2 *gauche* methyls; at 340°K the fast exchange spectrum consists of a 10-line pattern with an appropriately averaged coupling constant, a(9H) = 0.175G. In this case we might think of the electron as a surrogate "spy nucleus", perhaps even the most ephemeral of spies! In any event, first order analysis along the lines presented above reduces the problem to a series of 4 1x1, 12 2x2 and 4 3x3 submatrices which need to be diagonalized and the corresponding subspectra summed. Experimental and calculated spectra (shown in double derivative mode to enhance resolution) are presented in Figure 1. The activation parameters for alkyl rotation in *t*-Bu-C_{60} • are $\Delta H^{\ddagger}$= 7.3 kcal/mol and $\Delta S^{\ddagger}$= -3 e.u.

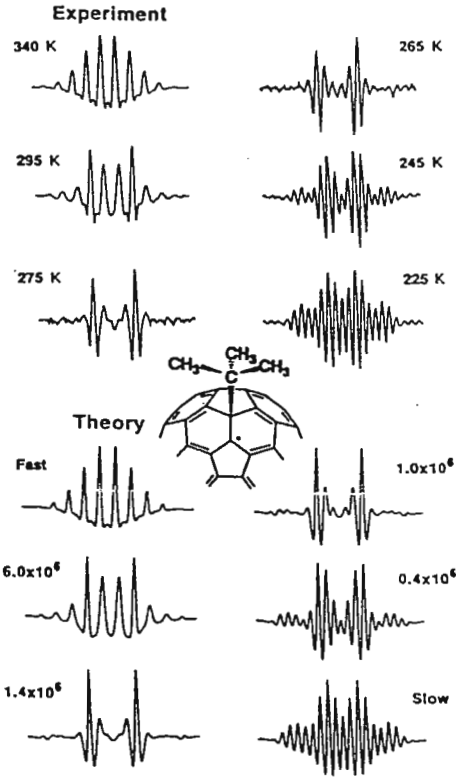

Figure 1.

Please credit this contribution to Patricia Watson's account.

References: 1. P.J. Fagan, P.J. Krusic, D.H. Evans, S.A. Lerke and E.R. Johnston, *J. Am. Chem. Soc.* **1992**, *114*, 9697. 2. P.J. Krusic, D.C. Roe, E.R. Johnston, J.R. Morton and K.F. Preston, *J. Phys. Chem.* **1993**, *97*, 1736.

Best regards,

Eric Chris

Eric R. Johnston D. Christopher Roe

Wesleyan UNIVERSITY

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FAX: (203) 344-7960

DEPARTMENT OF CHEMISTRY

750 MHz NMR of DNA

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

Monday, March 29, 1993
(received 4/5/93)

Dear Barry:

We have recently obtained 750 MHz NMR data on a single stranded DNA which exhibits a tertiary structure. The data was obtained using the Varian UNITYplus system with an Oxford magnet in a total time of about 30 hours by Dr. E. Hoffmann of Varian. The sample was at about 1.5 mM in 90% H_2O/10% ^2H_2O and a conventional one-dimensional spectrum was obtained as was a NOESY. Both data sets were obtained using shaped pulses to mimize excitation of the water resonance. This DNA is closely related to the DNA aptamer we recently reported on in *Biochemistry* **32**, 1899 (1993). However, this DNA contains 28 residues, of an undisclosed sequence, whereas the aptamer has 15 residues.

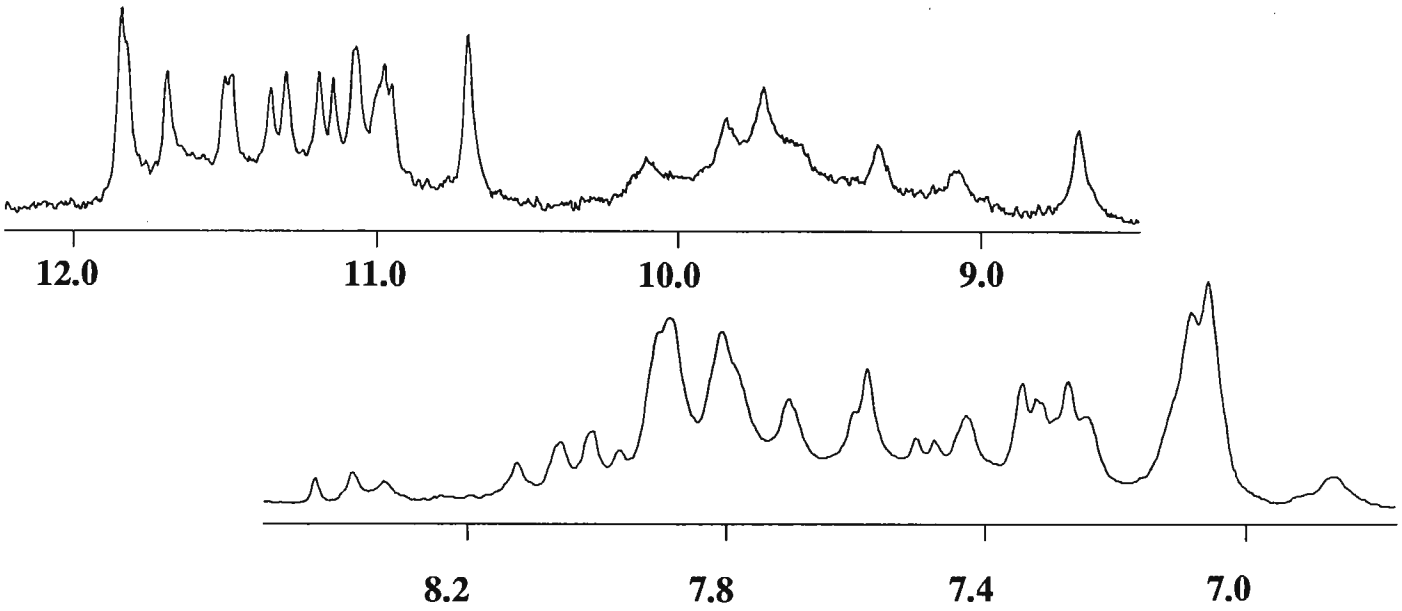
The one-dimensional data, part of which is shown below, demonstrates that the expected resolution is obtained at 750 MHz and that CSA broadening is minimal of both the sugar and aromatic protons. A remarkable feature of the aromatic-H2', H2" region, part of which is shown below, is that *all* 28 sets of connectivities can be determined from this single data set. The resolution in other regions of the spectrum is also very high. The two-dimensional data not only offers the expected resolution but the water suppression is extremely good and there is little in the way of t_1 noise or other artifactual signals. indicating a stable spectrometer system.

This sample provides an example of where going to 750 allows almost all of the assignment information to be obtained from a single experiment. In fact, we may be able to determine the tertiary structure from the single NOESY data set (but we are obtaining additional data at lower fields). Since isotopic labeling of DNA is not nearly as easy as that of proteins the high resolution of the 750 is most welcome.

Sincerely,

Philip Bolton

Ke Yu Wang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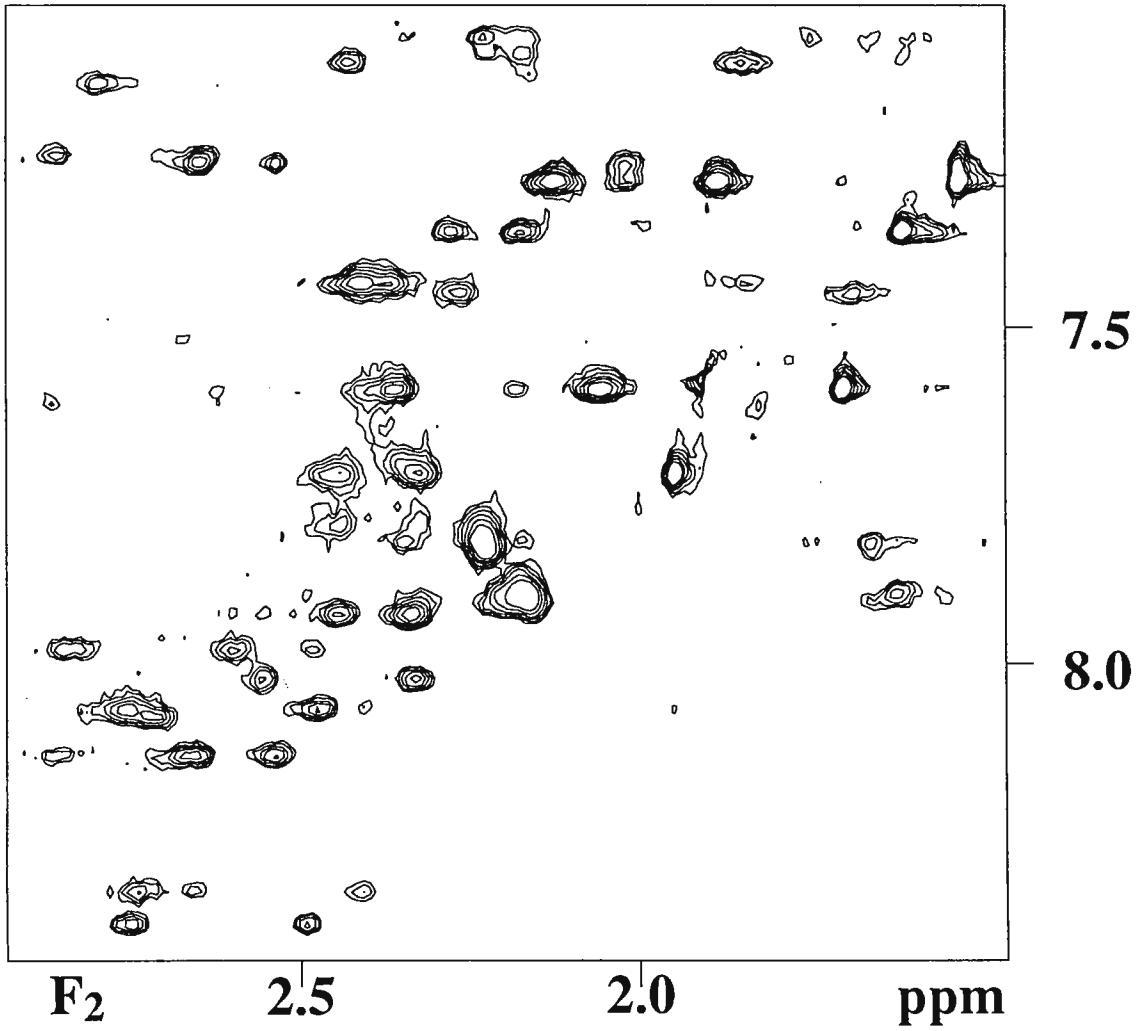
7.5
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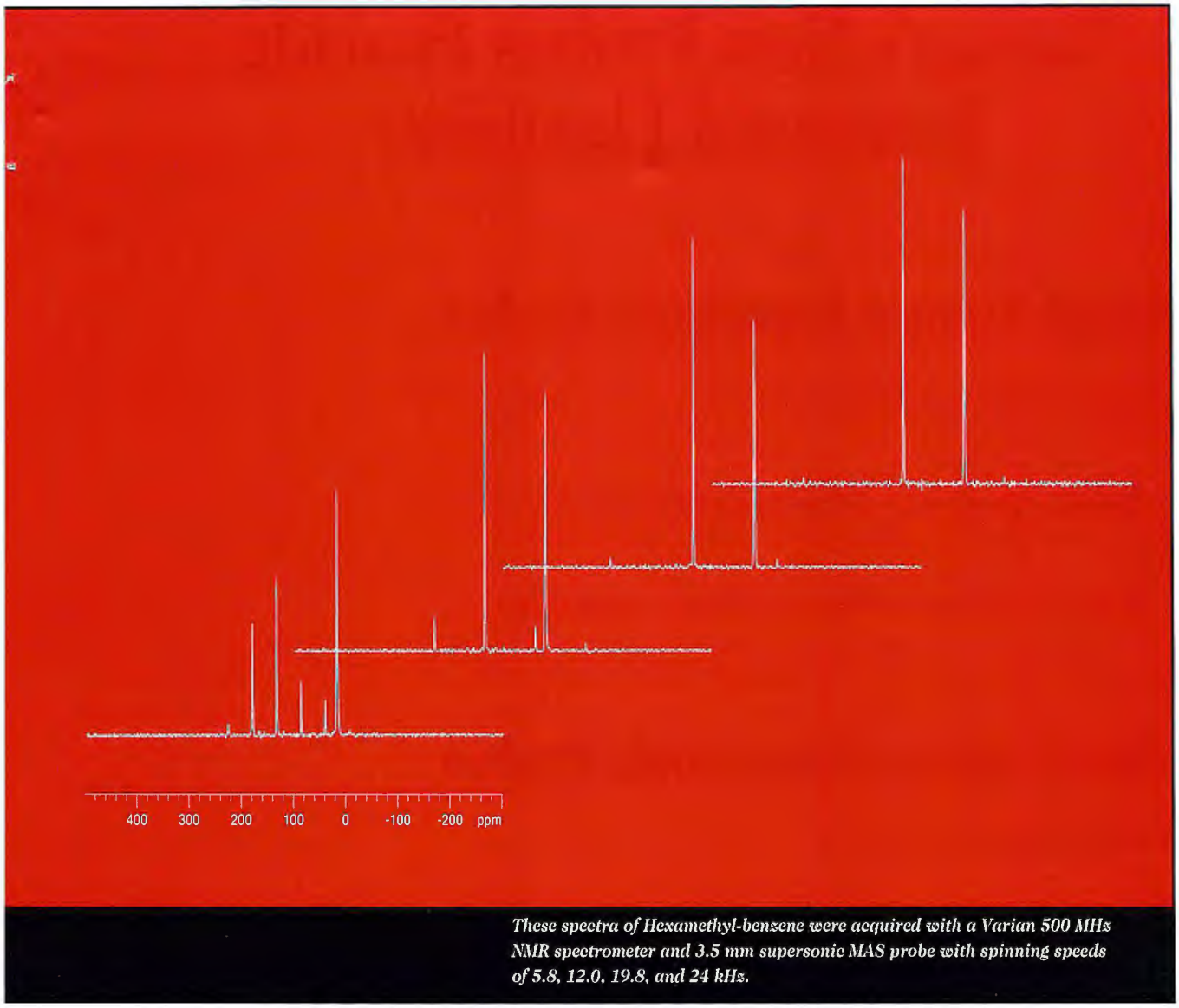
Supersonic Spinning for Solid Samples



These spectra of Hexamethyl-benzene were acquired with a Varian 500 MHz NMR spectrometer and 3.5 mm supersonic MAS probe with spinning speeds of 5.8, 12.0, 19.8, and 24 kHz.

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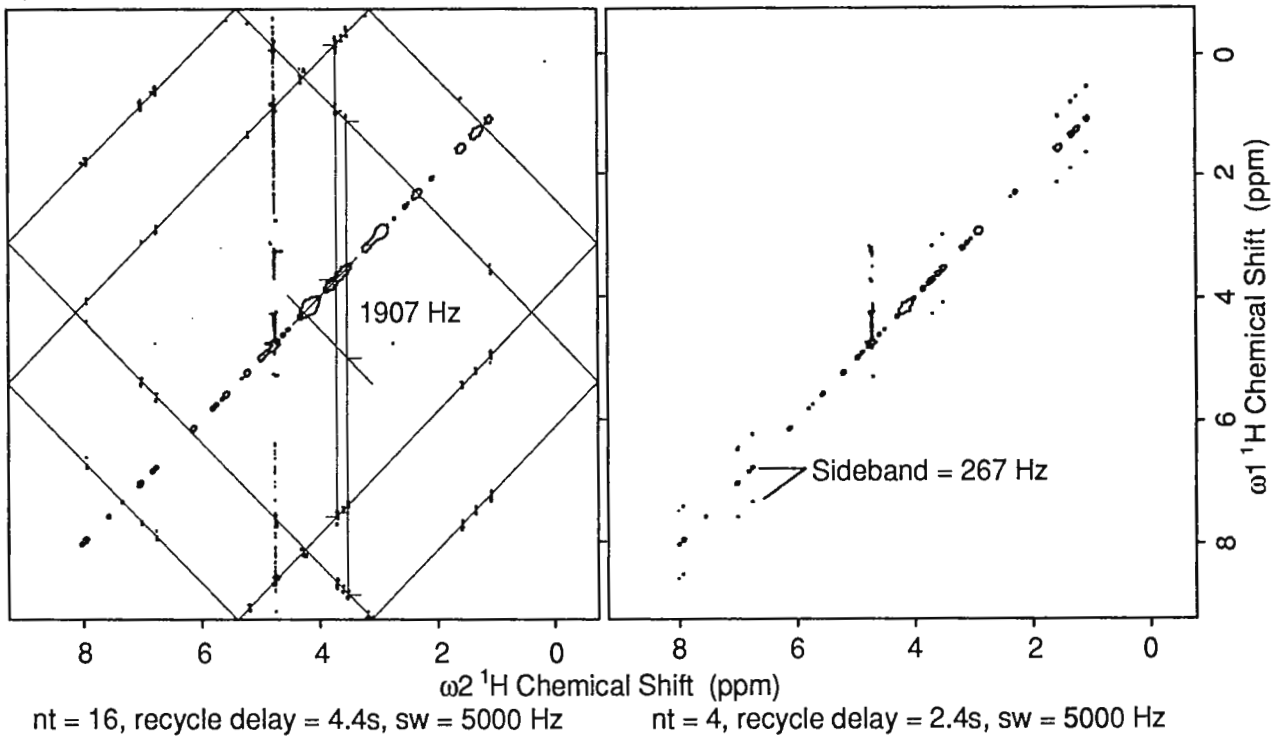
School of Chemical Sciences
1209 West California Street
Urbana, IL 61801

(received 4/3/93)

Artifacts in 2D Spectra from VT Oscillations

Instability of the sample temperature is notorious for causing "t1" noise and in the case of slow stable oscillations, the appearance of "sideband" like signals in f1 (see Magnetic Moments Vol. IV No. 4 "Temperature Control in NMR" by Steven Patt). The frequency of the sideband is related to the frequency of the t1 increment and the temperature oscillation:

$$\text{cycle time of oscillation} = \frac{2*nt*(d1+at)*sw}{\text{"sideband freq."}}$$



The two 2D NOESY spectra above were taken on a VXR-500 with Nalorac ID probe. All off-diagonal signals are negative contours so that the "sideband" artifacts can be easily distinguished from the positive NOESY crosspeaks (the diagonals are at a very high positive contour level). The sample temperature appears to oscillate with a period of 360 seconds. The temperature oscillation period is observed to be independent of the VT temperature differential (the difference between the incoming gas and the set temperature) for the range of 2° to 20° overhead. The VT flow rate also does not alter the "sideband" period over the range of 4 to 10 lpm. By replacing the VT air source with compressed nitrogen, we eliminated fluctuations in the air supply pressure as the problem. Turning off the room air conditioning, however, does completely eliminate the "sidebands". The problem appears to be the 1.4°C oscillation of air flowing directly onto the magnet from the air conditioner.

(Supported by NIH GM-41612, CA-52506, BTRG-P41RR05964-01, SIG-1-S10-RR06243-01).

Sincerely

Zhehong Gan Howard Robinson

email: gan@aries.scs.uiuc.edu or h-robinson@uiuc.edu

credit to: Dr. Vera Mainz.

University of Illinois
at Urbana-Champaign

School of Chemical Sciences
142B RAL, Box 34-1
1209 West California Street
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March 23, 1993
(received 3/29/93)

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

Dear Dr. Shapiro:

I would like to inform your readership of the existence of the Association of Managers in Magnetic Resonance Laboratories (**AMMRL**). This organization came into existence at the 1992 ENC in Asilomar through the efforts of the steering committee (Ben Bangerter, Yale University; Ron Garber, Univ. of South Carolina; Shaw Huang, Harvard Univ.; Vera Mainz, Univ. of Illinois, Urbana-Champaign; Rudi Nunlist, Univ. of California, Berkeley; Jane Strouse, UCLA) and the interest of a large number of ENC participants. The AMMRL exists to provide a channel of communication for managers and directors of magnetic resonance laboratories. This is accomplished mainly through an email forwarding system maintained by Rudi Nunlist and Vera Mainz. The email address for the organization is: ammrl@bloch.cchem.berkeley.edu. Any message sent to this address will be forwarded to all members of AMMRL on email - currently approximately 100 people. Those members who do not have an email address receive summaries of the email traffic about four times a year, as well as a newsletter.

This organization is very informal - there are no dues, and the membership information becomes part of the membership directory which is sent out yearly. It has been successful in providing a format for questions, requests for help in solving specific problems, job postings, etc. At this year's ENC in St. Louis another meeting was held, where Tom Farrar of NSF spoke about the current funding situation, summaries were given of the last years activities by Ron Garber (Instrument Rate Survey), Vera Mainz (organizational questions), Gerry Pearson (Email Summary), and the group broke into two groups to discuss user training, instrument performance checks, instrument maintenance, and upgrading vs. console replacement.

Anyone having responsibilities for managing NMR and EPR instruments is invited to participate. If you are interested in joining the AMMRL, write to me, or email to:

mainzv@aries.scs.uiuc.edu
or ammrl@bloch.cchem.berkeley.edu.

Sincerely,

Vera V. Mainz, Director
Molecular Spectroscopy Laboratory

Monsanto

PHYSICAL SCIENCES CENTER

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700 Chesterfield Village Parkway
St. Louis, Missouri 63198
Phone: (314) 694-1000

April 3, 1993
(received 4/8/93)

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

Estimating Parameters With Bayesian Probability Theory

Dear Barry:

For the past few years we have been exploring the use of Bayesian probability Theory (BPT) in lieu of the traditional discreet Fourier Transform for estimating parameters from NMR data.[1] In this letter we'd like to share some of our most recent results using CPMAS ^{13}C spectra. Figure 1 contains a comparison of non-linear least-squares curve deconvolution (a frequency-domain method) with BPT spectral simulations (a time-domain method). Figure 1a shows the DFT of an experimental FID and the DFT of a *modeled* interferogram constructed from BPT parameter estimates for this FID. The bottom trace is a direct overlay of the two spectra. The top trace is an overlay of each resonance from the BPT analysis. The middle trace contains the residual from their difference. Figure 1b is a similar display for our best effort at non-linear least-squares curve deconvolution.

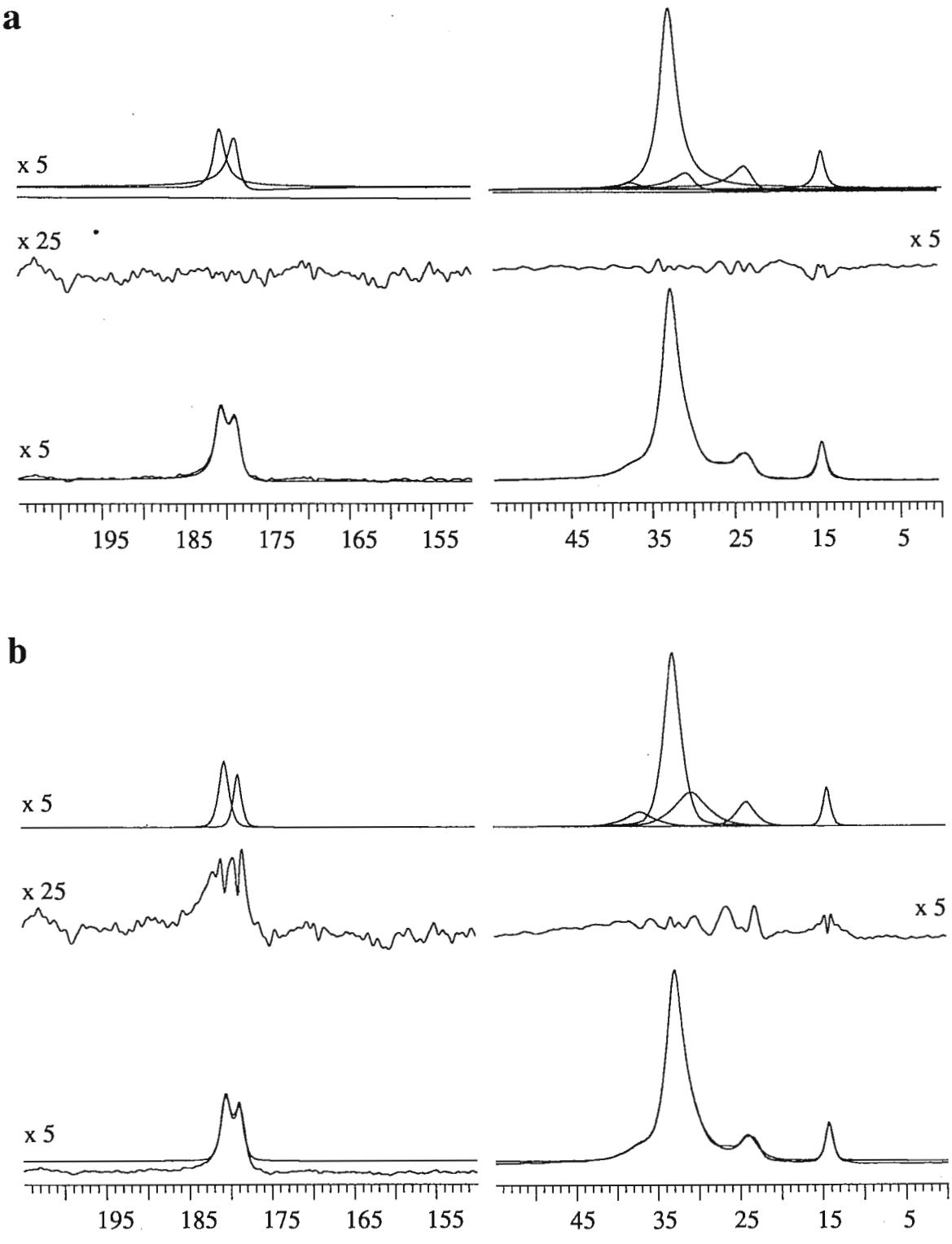
The modeled data shown in Fig. 1a were computed from BPT estimates of the number of resonances present, and the frequency, amplitude, and decay rate of each resonance. The BPT and deconvolution results clearly conflict and are due to limitations of the deconvolution method. Curve deconvolution is vulnerable to baseline distortions and/or extremely broad lines. Also, spectra with several severely overlapped resonances can be fitted by more than one reasonable solution. BPT analysis is immune to both problems. BPT analysis is unaffected by DC offset, and extremely fast decaying components of the FID can be either modeled or removed. The BPT analysis generates a set of probability distribution functions which are used to evaluate the quality of the final model. For instance, in this spectrum the BPT model containing seven resonances is calculated to be 10^{28} more likely than the corresponding six-resonance model.

In spectra containing multi-resonance degeneracy in combination with an ill-defined baseline, our experience is that BPT yields superior results compared to conventional frequency domain methods.

Sincerely,

Bill Hutton Joel Garbow Norm Hoffman Larry Bretthorst

1. J.J Koytk et al *J. Magn.Reson.* **98**, 483-500, 1992 and references therein.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These spectra of AlPO-11 below illustrate these effects. The top spectrum shows single resonance DOR at 625 Hz spinning. The bottom trace shows significant improvement at 980 Hz; note especially the appearance of the octahedral peak at 30 ppm. The middle trace shows 625 Hz DOR with proton decoupling; the peaks are narrower than at 980 Hz, especially the octahedral signal.

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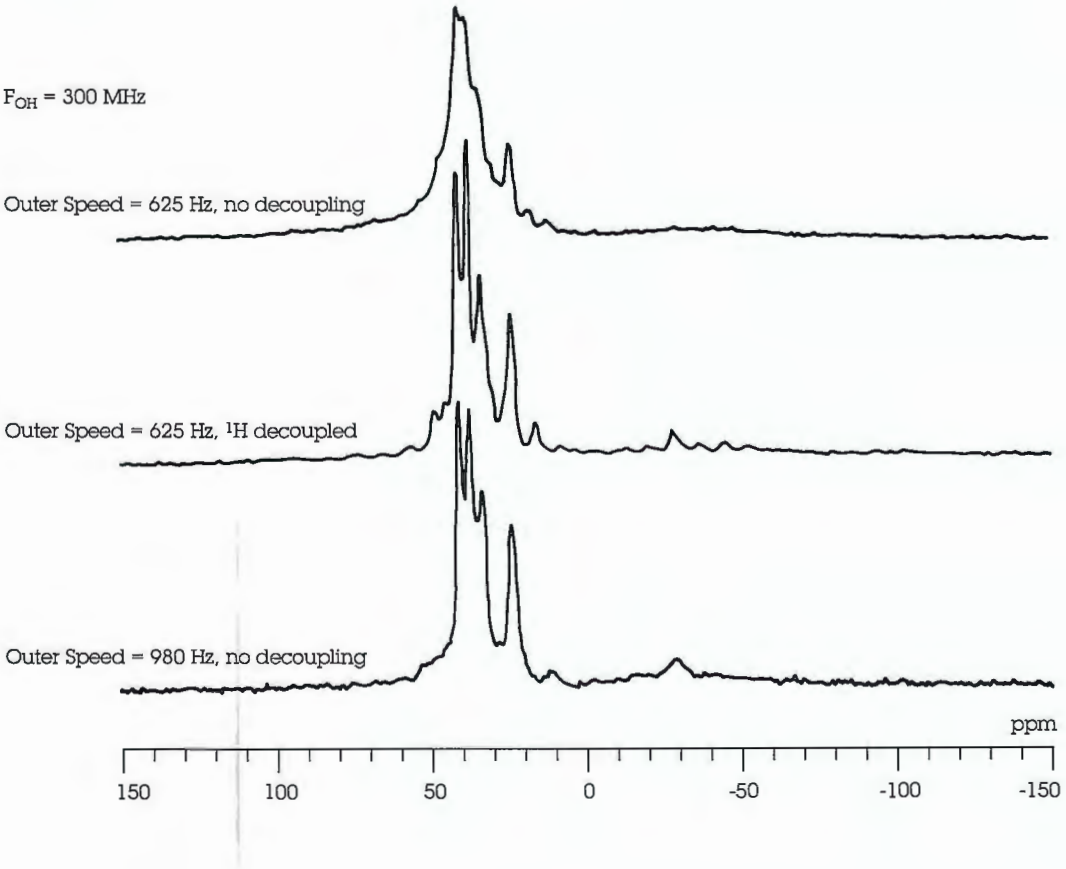

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Internet: jerry@nmr-r.mgh.harvard.edu

April 1, 1993
(received 4/17/93)

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

Login, Accounting and Electronic Logbook for Ancient Computers

—— or ——

Bean Quantitation by NMR

Dear Barry:

We are required to keep a strict accounting of all time used on each of our NMR instruments, and to apply time charges against project fund numbers. On Unix-based computers, this is a relatively easy task to accomplish, since a username may be assigned to each project. Use of each account is password protected, and the login and logout time of every session may be recorded in an accounting file. The keeping of the accounting file is transparent to the user (except, of course, when the P.I. gets the bill at the end of the month).

Older computers such as the Aspect on our Bruker MSL–400 do not support logins or passwords. The software on our General Electric Signa clinical imager has been implemented in such a way as to preclude using separate accounts on its Data General Eclipse computer. We have rectified these shortcomings with an accounting system in which the login is accomplished on one of our SparcStations. The DTR line of a serial port on the Sparc is used to enable or disable the display of each controlled NMR instrument.

The user logs into the controlling workstation—which may be performed remotely from any workstation or personal computer on our network—and executes a startup procedure. This procedure prompts for a spectrometer username, password and project. Accounting information including the start time is recorded, and the NMR instrument's display is enabled. The user may then log off the controlling workstation if desired.

When the spectrometer session is completed, a spectrometer log off procedure is similarly executed. This records the stop time, disables the display, and prompts the user for responses to questions which formerly were answered in writing (younger folks may look this up in the encyclopedia) in a paper logbook. In this way the user is forced to look at, and enter at least a minimal response to, each logbook question such as what probe did you use, were there

any instrument problems, did you clean up your mess (your mother doesn't work here...), etc.

The entire logbook is now in machine readable form, which offers a number of advantages. It may be searched electronically for users of a particular probe, entries use the ASCII character set instead of Egyptian hieroglyphics, and the entire document may be duplicated, sorted, backed up, and lost in disk crashes with all the conveniences normally associated with disk files.

The composite video monitor on the Signa and the MSL–400 imaging accessory are disabled by interrupting the video signal with a reed relay. The 500 ohm coil of the relay is directly operable by the DTR signal. This arrangement provides electrical isolation between the video circuitry and the controlling computer. The MSL–400 color display RGB signals are gated off with TTL circuits. We chose disabling the displays as the least invasive way of performing the intervention. Should a user be inadvertently logged off, or the controlling computer or network fail, any data acquisition in progress is unaffected. The logic is designed to be fail safe, so that disconnection of the cables allows the instruments to operate (a deference to scientific interests as opposed to those of the bean counters). The connections are inconveniently located to at least mildly discourage amateur hackers.

Best regards,

Mike Vevea Marty Lizak Jerry Ackerman

Post-Doctoral Position Available

The NMR group at the Schering-Plough Research Institute currently has an opening for a Post-doctoral scientist. The successful candidate will use the newer 3 and 4 dimensional heteronuclear experiments to study proteins istopically enriched with ^{15}N and ^{13}C as well as complexes of these proteins with small molecules. The lab is equipped with an Omega-PSG 500 MHz spectrometer, a Varian Unity-Plus 600 MHz spectrometer, and Sun and Silicon Graphics work-stations running FELIX processing software and DGII, IRMA and DISCOVER software for structure calculation and refinement.

The initial appointment is for one year and is renewable for a second year by mutual agreement. For more information contact Dr. Hugh Eaton, K15-0450, Schering-Plough Research Institute, 2015 Galloping Hill Rd., Kenilworth, NJ 07033.

Université de Lausanne - Faculté des Sciences
INSTITUT DE CHIMIE MINÉRALE ET ANALYTIQUE
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Prof. Bernard L.Shapiro
966 Elsinore Court
Palo Alto, CA 94303
U S A

(received 4/12/93)
24 March, 1993

WATER EXCHANGE ON $Gd(DTPA)(H_2O)^{2-}$ AND $Gd(DOTA)(H_2O)^{-}$

Dear Prof. Shapiro,

Gadolinium(III) ion complexes as for example $Gd(DTPA)^{2-}$ and $Gd(DOTA)^{-}$ are now widely used in MRI as contrast agents. A number of studies concerning complex formation constants and ^{1}H-NMR relaxivity enhancement can be found in literature[1]. According to X-ray[2] diffraction as well as luminescence studies there is one water molecule directly bound to the ion. But there is no information on the water exchange on these complexes in aqueous solution. To get more insight we studied the pressure dependence of the ^{17}O NMR transverse relaxation rates of both complexes using our home built high-pressure probe. The evaluation of the experimental data follows Swift and Connicks treatment. The pressure dependence of the water exchange rate is given by

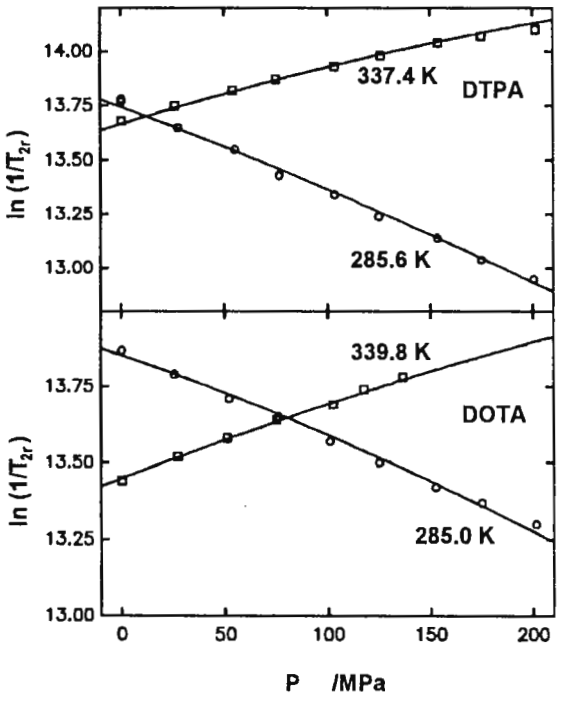

Figure 1 Pressure variation of the relaxation enhancment of the ^{17}O-NMR bulk water resonance in presence of Gd(III)-complexes measured at 54.2 MHz.

$$\ln(k_P) = \ln(k_0) - P\Delta V_0^* / RT$$

where k_0 and k_P are the rate constants at zero pressure and P, respectively. ΔV^* is the volume of activation for the exchange reaction, a very useful quantity in the elucidation of the mechanism of the reaction.[3].

Figure 1 shows the pressure variation, measured at two temperatures, of the transverse relaxation enhancement observed on the bulk water signal. The data were analysed using a non-linear least

squares fitting routine and the results are given in Table I. The exchange rates are one to two orders of magnitude slower than the one found for the octa aqua Gadolinium(III).[4] The volumes of activation ΔV^{*} are very positive (compared for example to those of the aqua ions of Al^{3+} and Ga^{3+} [5]) and can be related to a dissociative mode of activation for the water exchange process. For water exchange reactions on octa-hydrates of heavy lanthanide(III) ions we found earlier[6] negative activation volumes ranging from -5.7 to - 6.9 cm^3 mol^{-1}, indicating an associative activation mode.

Table I. Kinetic Parameters for Water Exchange on $[Gd(DTPA)(H_2O)]^{2-}$ and $[Gd(DOTA)(H_2O)]^{-}$ as Derived from Variable Pressure ^{17}O NMR

		$[Gd(DTPA)(H_2O)]^{2-}$		$[Gd(DOTA)(H_2O)]^{-}$	
10^{-6} k_{ex}	s^{-1}	1.4	(285.6 K)	1.8	(285.0 K)
10^{-7} k_{ex}	s^{-1}	5.7	(337.4 K)	6.2	(339.8 K)
ΔV^{*}	cm^3 mol^{-1}	+12.5	± 0.2	+10.5	± 0.2

K. Micskei[a)] E. Brücher[a)] L. Helm[b)] A.E. Merbach[b)]

This contribution is part of a collaboration of the Institute of Inorganic and Analytical Chemistry, Kossuth University, Debrecen (Hungary)[a)] and the Institut de Chimie Minérale et Analytique, Lausanne (Switzerland)[b)].

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

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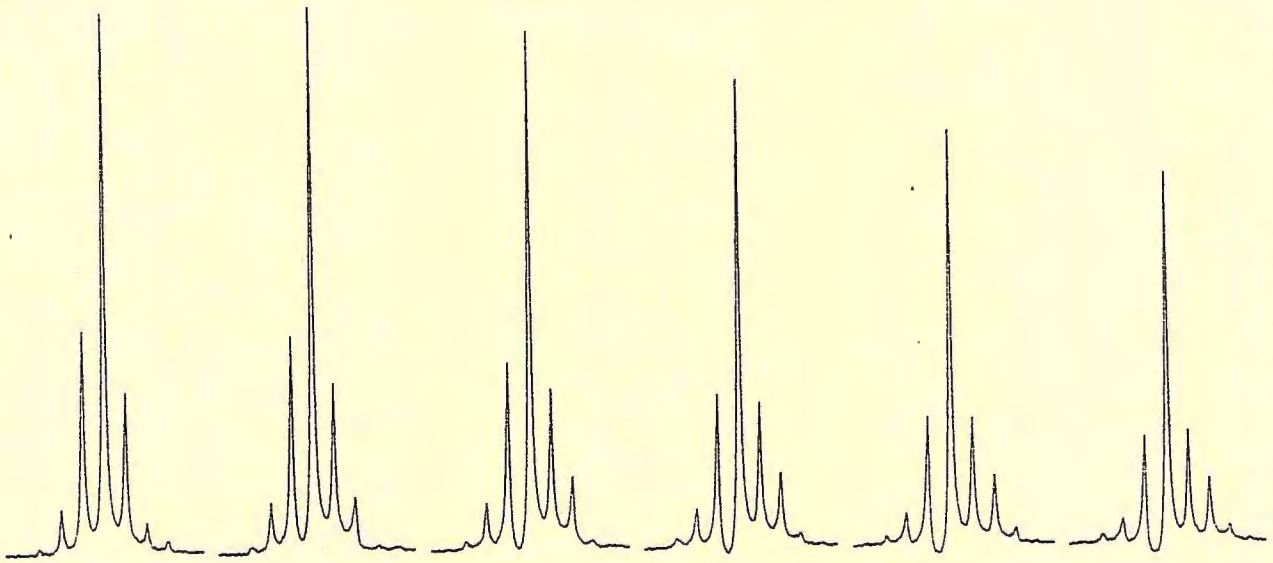

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Spectra provided by Varian Applications Lab, Palo Alto, CA and Dr. Laima Baltusis

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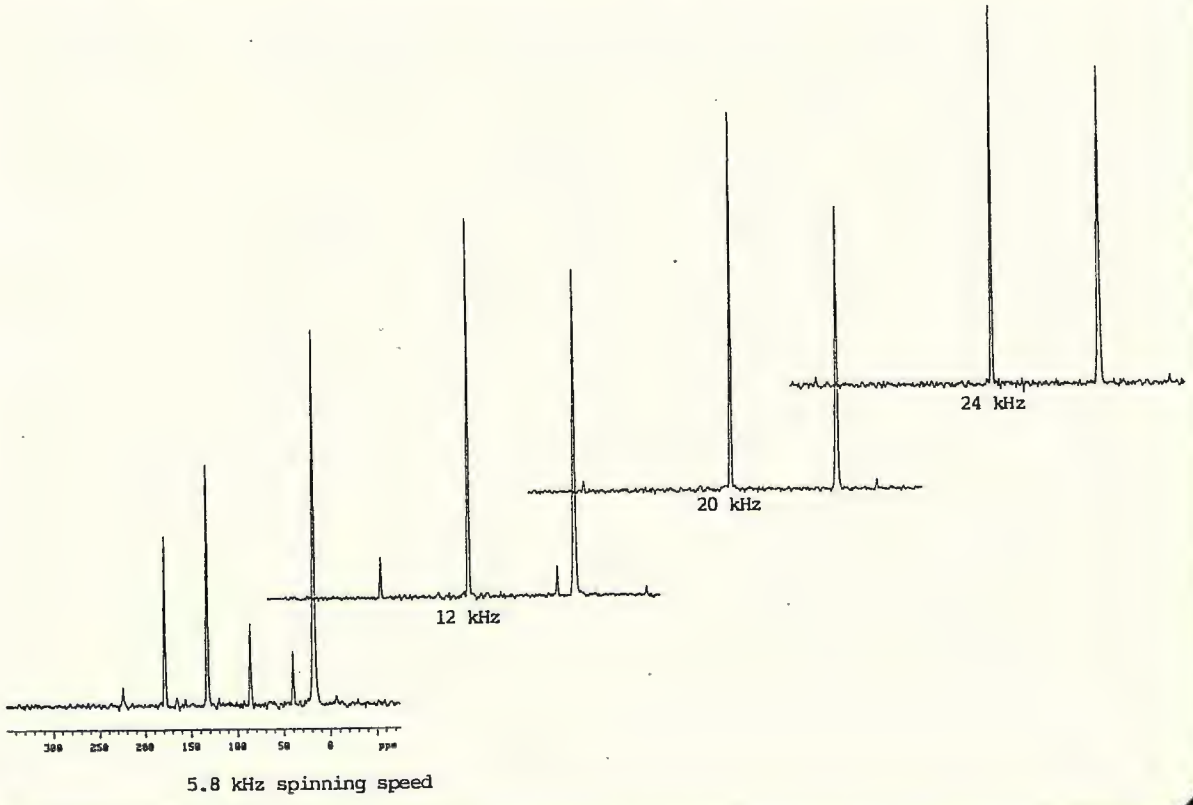
The spectra was provided by the Varian applications lab in Palo Alto and Dr. Laima Baltusis.



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March 23, 1993
(received 3/27/93)

Colorado State University

Solid-Sample Imaging with MAS and Multiple-Pulse Line Narrowing

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

Department of Chemistry
Fort Collins, Colorado 80523
Tel. (303) 491-6480
Fax (303) 491-1801

Dear Barry:

We hope that some of your readers will be interested in recent ^{13}C and ^{1}H NMR imaging results we have obtained on solids, using MAS and multiple-pulse line narrowing. For several years, our working hypothesis in solid-state NMR imaging has been that this approach would be most valuable if chemical shift information were included. With the introduction of the concept of combining MAS with rotating gradients by Wind and Yannoni (U.S. Patent No. 4,301,410, Nov. 17, 1981), followed by the experimental demonstration of the concept by Cory, Reichwein, Van Os and Veeman (*Chem. Phys. Lett.* **143**, 467 (1988)), as refined by Schauss, Blümich and Spiess (*J. Magn. Reson.* **95**, 437 (1991)), the overall approach for utilizing the isotropic chemical shift in solid-sample imaging has been available.

Graduate students, Herman Lock and Yahong Sun, have designed, constructed and tested a ^{13}C MAS imaging probe. Figure 1 shows a plot of ^{13}C chemical shift vs. one spatial dimension; we have also done the analogous experiment in two spatial dimensions. The chemical shift can be selected or displayed in a third dimension. Experiments on seeds, coal and heterogeneous polymer blends are underway.

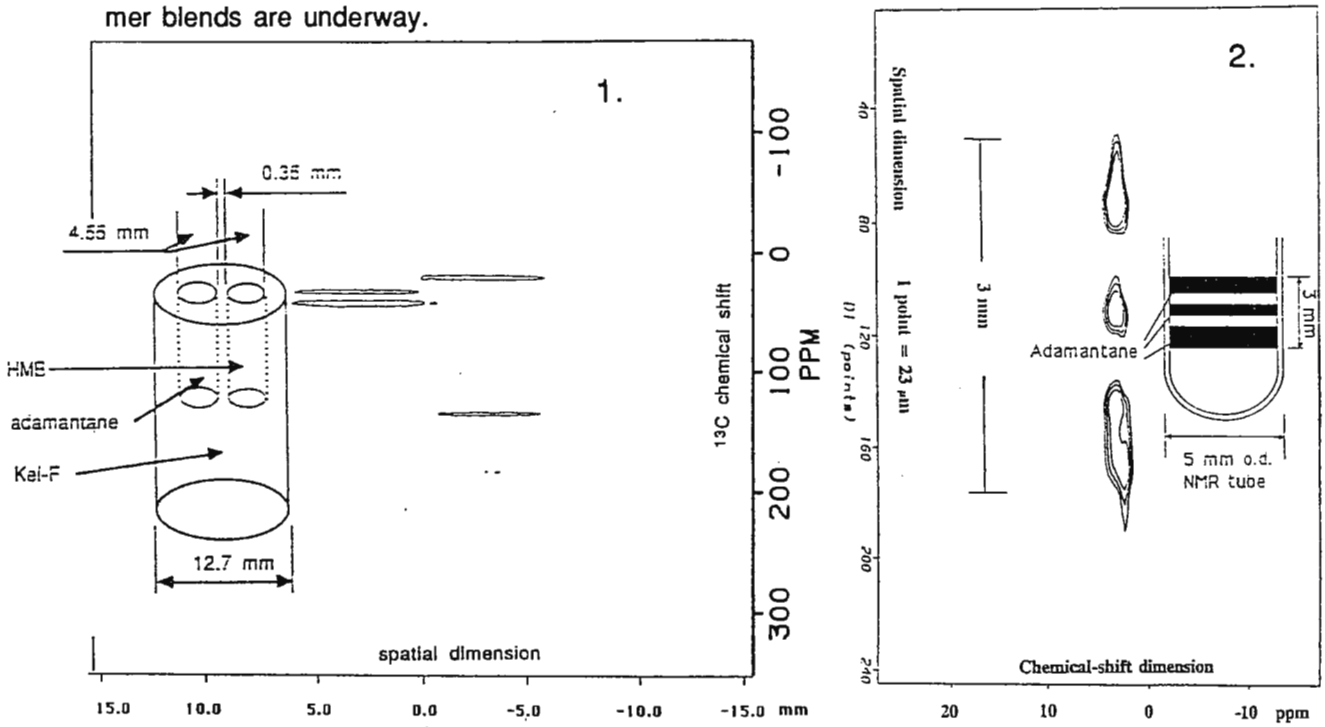

Figure 2 shows a ^{1}H CRAMPS image obtained by postdoc, Marian Buszko (now at the Univ. of Florida), who designed and built the ^{1}H apparatus. This approach employs TREV (Takegoshi and McDowell, *Chem. Phys. Lett.* **116**, 100 (1985)) for ^{1}H-^{1}H dipolar line narrowing, which Marian found advantageous because of favorable offset characteristics (Buszko and Maciel, *J. Magn. Reson.*, in press). ^{1}H CRAMPS and MAS-only imaging studies of seeds are underway.

Sincerely,

Gary E. Maciel
Professor

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FOREST RESEARCH INSTITUTE

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5 April 1993 (received 4/9/93)

Dr B Shapiro
TAMU NMR Newsletter
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UNITED STATES OF AMERICA

RE: Multivariate Analysis of NMR Spectra for Chemical Composition of Wood

Here at the NZ FRI our geneticists are very keen to screen large numbers of trees for carbohydrates, acid insoluble lignin and extractives along with various physical properties (density, strength etc.). This translates to considerable effort on behalf of the chemists performing lengthy, mundane and repetitive bench analyses. How then can a NMR spectroscopist help?

We are in the midst of developing and validating a method which will provide rapid screening for the above properties from ^{13}C CP-MAS spectra. Spectral data is transferred from the Bruker Aspect 3000 to WIN NMR where the data is processed and exported as a text file for principal component analysis. Principal component analysis of the spectra and the results of the classical techniques produces a regression equation to which the spectra of unknown samples can be fitted. Hence from a single CP-MAS spectrum it is possible to obtain both chemical and physical data of *Pinus radiata* timber. Regression coefficients (R^2) obtained for the correlation of the CP-MAS data with classical techniques are shown in Table 1.

Table 1. Regression coefficients for principal component analysis of CP-MAS spectra.

	Lignin	Extractives	Total CHO	Density
R^2	0.921	0.955	0.935	0.906

At this stage we have only looked at a very limited sample set for only a few properties. The current stage of research is building on the sample size to improve the statistical validity and extending the properties to include further physical properties such as strength, shrinkage and even to include properties of pulps produced from the timber (Kappa number, beatability, brightness etc.).

Yours sincerely

Roger Meder

MCDONNELL DOUGLAS

McDonnell Douglas Research Laboratories

March 14, 1992
(received 3/25/93)

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

Phase Shift Problem in Chemical Shift and Multislice Imaging Experiments

Dear Dr. Shapiro,

Last year, we performed some NMR micro-imaging studies of the phase separation of a binary mixture following a temperature quench. A Chemical Shift Imaging (CSI) experiment was best suited for the study, as LT and coworkers already had obtained a substantial amount of chemical shift data without imaging in S. Lacelle's laboratory in Sherbrooke. We had some problems implementing the technique on our Bruker MSL-300 spectrometer with the microimaging accessory. These problems appear to derive directly from our PTS synthesizer during frequency switching. The synthesizer performs as specified, giving phase continuous frequency switching within 10 kHz decades. Switching outside that region causes problems we, and at least some other MSL users, were not previously aware of.

In one dimension, we implemented the CSI experiment shown in Fig. 1. The slice is selected by application of a G_z gradient with selective rf pulses centered about the appropriate transmitter offset, which we define as ν_{sl}. The transmitter has to be switched back during the acquisition to ν_o to provide reasonable acquisition sweep widths and digital resolution. The experiment worked fine as long as we kept $\nu_{sl} < \pm 10$ kHz. As soon as we went to ± 10.000 kHz or larger (9.999 kHz was OK), we saw degradation of the S/N and artifacts appeared in the spectra. CGF has observed similar problems with multislice 2D imaging on the MSL.

We then set up another experiment that was quite informative: do a simple FID on water, but switch the offset on– and off–resonance during the acquisition. When the offset was $< \pm 10$ kHz, the phase stayed coherent during the switch, as shown in the first switch in Fig. 2. But as soon as we switched ± 10.000 kHz or more, the phase would change randomly following the switch (Fig. 2). On our equipment, the phase in this case stayed coherent ~80% of the time. The resulting spectra thus showed evidence of the desired results, but artifacts and degraded S/N are present.

We have discussed these problems with other users, and at least some MSL users observe similar (although perhaps not identical) problems. On some medical imagers, multislice experiments are implemented by phase modulating the shaped rf to provide the frequency offset:

$$\text{Inv } \mathcal{F}\left[f(\omega-\omega_o)\right] = \exp(-i\omega_o t)\frac{\sin t}{t} . \qquad (1)$$

where $f(\omega-\omega_o)$ is a square excitation profile in frequency, and Inv $\mathcal{F}$ represent the inverse Fourier Transform. This method was implemented apparently to avoid problems with phase continuity during large frequency switches. Unfortunately, the MSL imaging accessory provides only amplitude shaping, so we cannot implement eq. 1. Newer PTS's have continuous phase switching to 100 kHz (optional on PTS-250's; older units can be upgraded).

Best Regards,

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(314) 233-2512

Luc Tremblay
ltrembla@avogadro.chimie.usherb.ca
(819) 821-7000 ext. 3099 (CANADA)

(Please credit this contribution to Serge Lacelle's account).

Figure 1. Schematic of 1D Chemical Shift Imaging Experiment

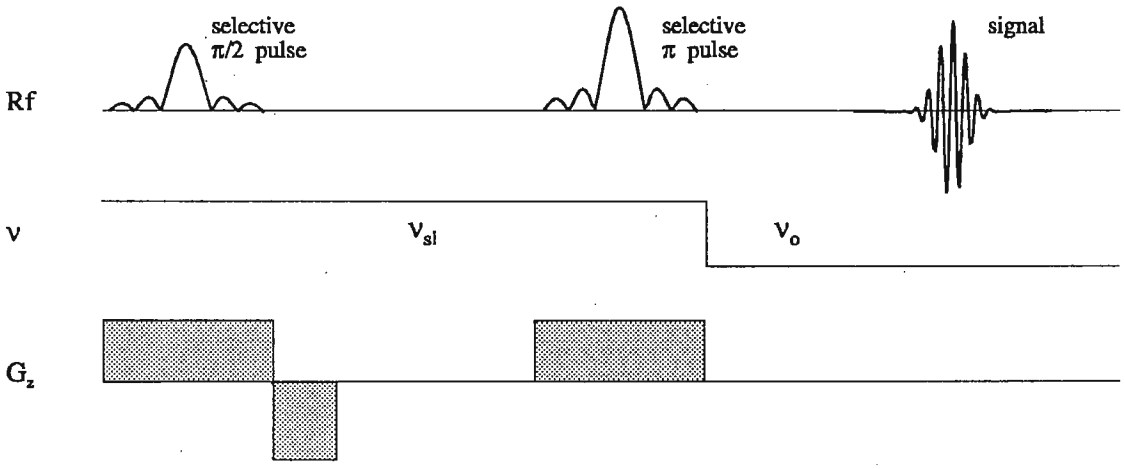
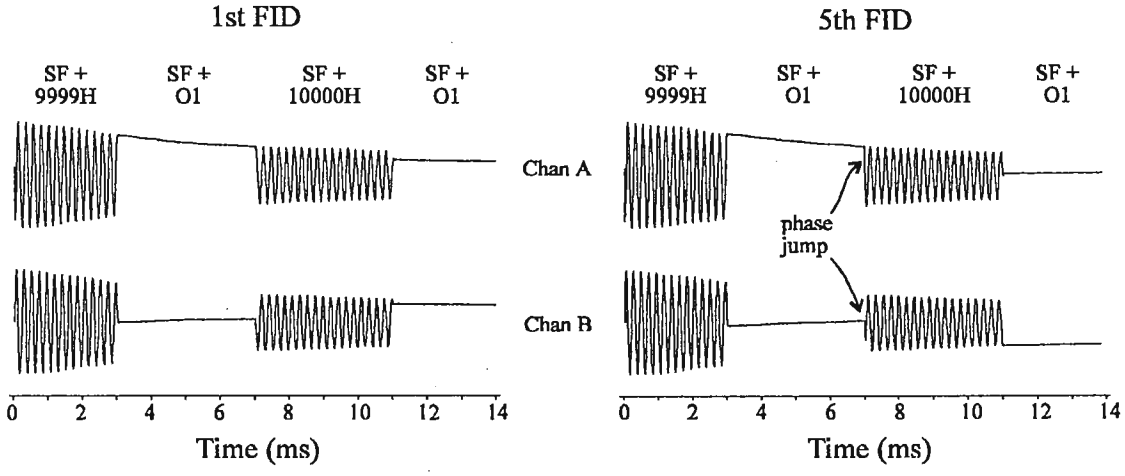
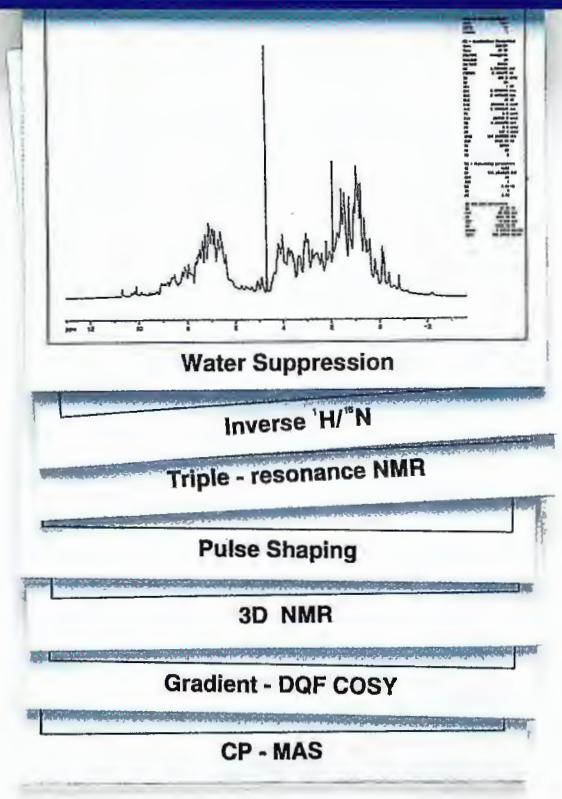


Figure 2. Water FID signals with frequency switches during acquisition (contact CGF to obtain a copy of the pulse program).



ARX



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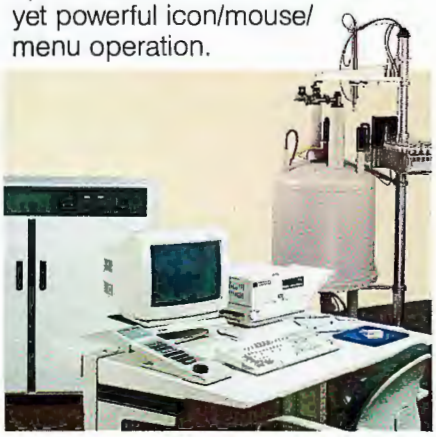

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All 5 mm ^{1}H 5+10 mm ^{13}C	ODCB/C_6H_6	0.2	0.2	0.2	
Lineshape Test					**SSB %**
All 5 mm ^{1}H	10% $CHCl_3$	6/12	7/15	7/15	<1
All 5 (10) mm ^{13}C	80% C_6H_6	3/7 (3/7)	3/7 (3/8)	4/8 (4/8)	<0.5 (<1)
Sensitivity Test					
5 mm ^{1}H Selective	0.1% EB	175	250	450	<10
5 mm ^{1}H Inverse Detection	0.1% EB	135	190	350	<15
5 mm ^{1}H Dual, QNP, VSP	0.1% EB	100	140	200	<15
5/10 mm ^{13}C QNP, Dual	ASTM	100/320	160/450	180/600	<15/<20
5/10 mm ^{13}C QNP, Dual	10% EB	70/200	100/300	150/400	
5/10 mm ^{13}C VSP multinuc.	ASTM	100/320	160/450	180/600	<15/<20
5/10 mm ^{13}C VSP multinuc.	10% EB	70/200	100/260	150/320	
5/10 mm ^{15}N VSP multinuc.	90% Form.	10/35	15/55	20/70	<25/<30

EB = ethylbenzene (for ^{13}C with ^{1}H-dec.);
ASTM = 60% C_6D_6 in dioxane
Form. = formamide (^{1}H-dec. without NOE)
lineshape: ^{1}H = $CHCl_3$ linewidth at ht. of ^{13}C-satellites/at 2^{6} this level
^{13}C = C_6H_6 linewidth at 0.55% / 0.11 level (^{1}H-dec.)
SSB = Spinning sidebands measured with 8 transients
QNP: 5 or 10 mm ^{1}H, ^{31}P, ^{13}C, ^{15}N
VSP: 5 mm ^{15}N – ^{13}P; 10 mm ^{109}Ag – ^{31}P.

Console	Dimensions and weights are approximate; voltage +10/ – 5% max. variation; other line freq. and voltage upon request
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Department of Medicinal Chemistry
Central Research Division
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Eastern Point Road
Groton, CT 06340

Central Research
NMR Spectroscopy

March 24, 1993
(received 3/27/93)

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

Temperature Stability of the AMX Revisited

Dear Dr. Shapiro,

We reported on the temperature stability of our Bruker AMX-600 spectrometer (TAMU NMR Newsletter, September 1992, pg. 7). Since that time we have received our BTO-2000 (thermocouple junction oven) and BCU-05 (cold air source) and would like to report on their performance. The combination of this new hardware with the BVT-2000 and a stable air source indeed produces extremely good temperature stability.

The BTO-2000 is a matchbox-sized oven which sits at the base of the probe and keeps the thermocouple junction at constant temperature. We have previously observed temperature fluctuations in the sample which could be traced to room-temperature fluctuations, and had devised a magnet skirt to partially offset this effect. The BTO-2000 improves on this idea in a far more convenient and elegant way (in fact, we devised our magnet skirt after discussions with Bruker Instruments about the source of temperature instability in real-world systems):

	Stability*	
	Short-term (minutes)	Long-term (hours/days)
Previous Results		
No skirt	16 mdeg	91 mdeg
With skirt	7 mdeg	22 mdeg
Current Performance	**<5 mdeg**	**11 mdeg**

(*Room air temperature is maintained constant to approximately +/-0.5°C with an incremental heat, constant-flow air-conditioner system. Nitrogen gas is provided from a gas-withdrawal liquid N_2 tank at a rate of 400L/H.)

The BCU-05 operates without coolant and is reasonably quiet. Therefore, icing of the low-temperature bath is a thing of the past (we had used 50-70% antifreeze in the Haake bath), and there is no discernible vibration which is transmitted to the probe. The lighter insulated trunk is much easier to position near the probe than the Haake bath heat exchanger.

All in all, this temperature control system provides exceptional stability and our multi-dimensional NMR data is better as a result.

Sincerely,

Walt

Walter Massefski, Jr.

Position Available

SOLID STATE NMR SPECTROSCOPIST

Oklahoma State University is setting up a university facility for solids NMR spectroscopy and seeks a spectroscopist with solids experience to assist users with experiments, maintain the equipment, and collaborate with faculty on research. A 300 MHz widebore spectrometer, equipped for MAS solids experiments including 1H multipulse, triple resonance, and extended range variable temperature, will be installed during the summer of 1993. Materials under investigation by the principal faculty users include polymer composites, polymer liquid crystals, ceramics, inorganic catalysts, minerals and clays, composites based on sol-gel silica, and plant cell walls.

The spectroscopist should have extensive hands-on experience in MAS NMR spectroscopy, preferably a Ph.D. degree, ability to maintain the equipment, and a spirit of helpful cooperation and communication skills to assist and instruct users and collaborate on research. The annual salary will be $30,000 or more, depending on qualifications. Ideally, the spectroscopist would start work two to four weeks prior to installation. (The anticipated starting date is in the May through July time frame).

Applicants should send resumes, publications, and names and phone numbers of three references to Dr. Warren T. Ford or Dr. Corinna L. Czekaj, Department of Chemistry, Oklahoma State University, Stillwater, OK 74078.

OSU is an affirmative action, equal opportunity employer. Applications from women and underrepresented minorities are particularly welcome.

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DEPARTMENT OF CHEMISTRY

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

March 9, 1993 (received 3/23/93)
^{7}Li Quadrupole
induced relaxation of
^{13}C in an aryllithium

Dear Barry:

Schleyer et al have reported that in ^{13}C NMR of 2,4,6-tri-t-butylphenyllithium (96% ^{7}Li, 4% ^{6}Li) complexed to THF-d_8 at 200 K the resonance for ^{13}C bonded to ^{6}Li is a clean 1:1:1 triplet $J(^{6}Li, ^{13}C) = 16.4$ Hz, superposed on a strong broadened doublet absorption, separation 70 Hz, due to ^{13}C bonded to ^{7}Li in the major isotopomer, see Figure A. Clearly, the well resolved triplet resonance indicates the lithium compound is a monomer and that intermolecular carbon-lithium bond exchange is too slow to perturb the NMR line-shape.

Under conditions of slow ^{7}Li relaxation the ^{13}C-^{7}Li absorption should consist of a 1:1:1:1 quartet $J(^{13}C, ^{7}Li) = 42.8$ Hz. That the resonance is a broad doublet, separation 70 Hz, implies that ^{7}Li relaxation influences the ^{13}C line-shape.

We derived relaxation matrices for species "^{13}C ^{6}Li," needed to calculate the ^{13}C line-shape due to anisotropy of the chemical shift, dipolar and quadrupole induced, respectively, relaxation mechanisms, by taking elements of the appropriate operators. Carbon-13 line-shape calculations show that none of these mechanisms influence the ^{13}C resonance of "^{13}C ^{6}Li." Even slow relaxation $k_1 \sim 0.35$ s^{-1} would result in ^{13}C triplets with outer lines of equal width but different from that of the central line; yet the intensities are essentially the same. Using the diagonal elements of the relaxation matrices for pseudo-species "^{13}C ^{6}Li" and "^{13}C ^{7}Li" together with the known spectral density ratios for the three mechanisms $j_\alpha(^{7}Li)/j_\alpha(^{6}Li)$ at extreme narrowing[2] it works out that for dipolar and chemical shift anisotropy relaxation the ^{7}Li induced ^{13}C relaxation rate (seen as line broadening) is never more than 10 times that for ^{6}Li. However, in the case of quadrupole induced relaxation the value is 5555.7 and that has to be the mechanism responsible for the ^{13}C line-shape of "^{13}C ^{7}Li." A calculation of these line-shapes, Figure B shows that in the Schleyer sample $j_{\pm1,\pm2}$ at 200 K is between 0.55 s^{-1} and 0.65 s^{-1}.

These effects discussed here are not uncommon. Usually ^{7}Li relaxation in organolithium compounds is too fast to obtain from the ^{13}C NMR line-shape.

Best wishes from the entire group.

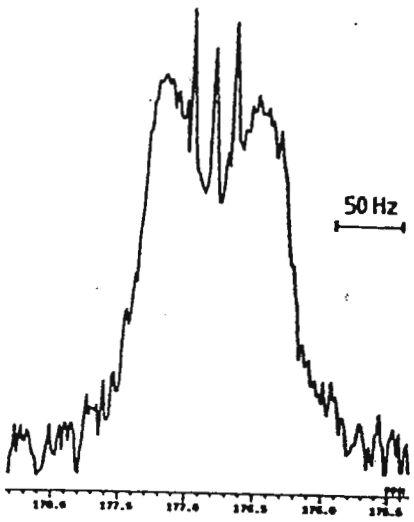
Yours sincerely,

Gideon Fraenkel Sheela Subramanian* Albert Chow

*out of country

1. Bauer, W.; Winchester, W. R.; Schleyer, P.v.R. Organometallics **1987**, 6, 2371.

2. Kaplan, J.I.; Fraenkel, G. NMR of Chemically Exchanging Systems; Academic Press: New York, 1980; Chap. 4.



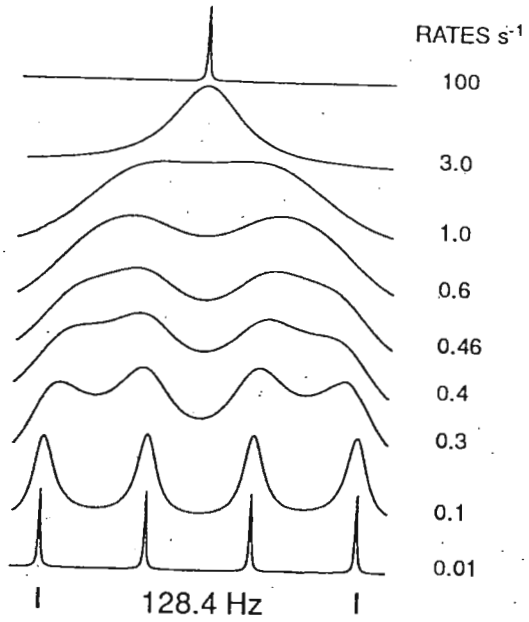


Figure 8. A stack plot of calculated line-shapes for quadrupole relaxation at extreme narrowing approximation.

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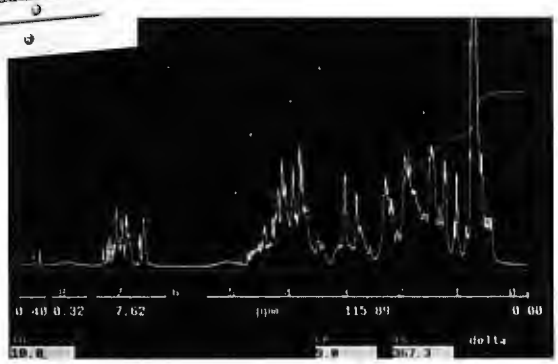

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Professor Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

February 23, 1993
(received 3/23/93)

"Comparison of the Solution Conformations of cyclo(-DTrp-DAsp-Pro-DVal-Leu-) in DMSO and Water"

Dear Professor Shapiro:

Endothelin, a potent 21 amino acid vasoconstrictor, has been associated with impairment of cardiovascular and renal function. This polypeptide exists in three distinct forms (Et-1, Et-2 and Et-3) and has been shown to cause a significant decrease in coronary blood flow, leading to myocardial ischemia. One of the endothelin receptors (ETA) has been found to be specific for Et-1 and to cause vasoconstriction when bound to endothelin in vascular smooth muscle. Substances that inhibit the binding of endothelin to its receptor are believed to have potential as therapeutic agents in the treatment of angina, acute renal failure and hypertension. Recently cyclo(-DTrp-DAsp-Pro-DVal-Leu-) (CPP-1) was identified as a potent Et-1 receptor antagonist which competes with endothelin for receptor binding .

We recently obtained the NMR solution structures of cyclo(-DTrp-DAsp-Pro-DVal-Leu-) (CPP-1) and the corresponding sodium salt (CPP-2) in DMSO and water, respectively. All sequential ^1H resonance assignments were made and well over 60 distance constraints were derived from the ROESY spectra. Both distance geometry and restrained molecular dynamics calculations were used to generate an ensemble of tertiary structures. Hydrogen bonding information was obtained from a deuterium exchange experiment in which the NH protons of Val and Asp were found to experience slow hydrogen exchange. Distance geometry calculations (DG) were performed using DSPACE (Hare Research, Woodinville, WA). Restrained molecular dynamics (RMD) calculations were obtained using the X-PLOR program (Polygen Corp., Waltham MA).

An ensemble of initial structures of CCP-1 in DMSO and CCP-2 in 90% H2O/10% D2O were generated. Each of the initial structures was first refined with the distance geometry algorithm with NOE distance constraints only. These DG refined structures implied possible hydrogen bonding between (Asp-2 NH)-(Val-4 C=O) and (Val-4 NH)-(Asp-2 C=O). This result was consistent with the deuterium exchange experiment which showed slowly exchanging amide protons for Asp-2 and Val-4. The hydrogen bonding constraints, together with the NOE distance constraints were thus used for further structural refinement with distance geometry and restrained molecular dynamics calculations. The final refined structures of CCP-1 and CCP-2 are shown in Figure 1 and 2, respectively. No significant conformational changes were observed in the backbone on going from DMSO to water.

Recently a paper was published (R.A. Atkinson and J.T. Pelton, FEBS, 296, 1-6, 1992) describing the structure of CPP-1 in 20% CD_3CN/80% H_2O. The backbone of the Atkinson structure appears to be identical to the structures we obtained in DMSO and water however significant variation in the side chain orientation was reported. Our data confirms that the backbone conformation of cyclo(-DTrp-DAsp-Pro-DVal-Leu-) does not differ significantly in different solvents but also shows differences in side chain orientation in DMSO vs. water. Further analysis of this finding is in progress.

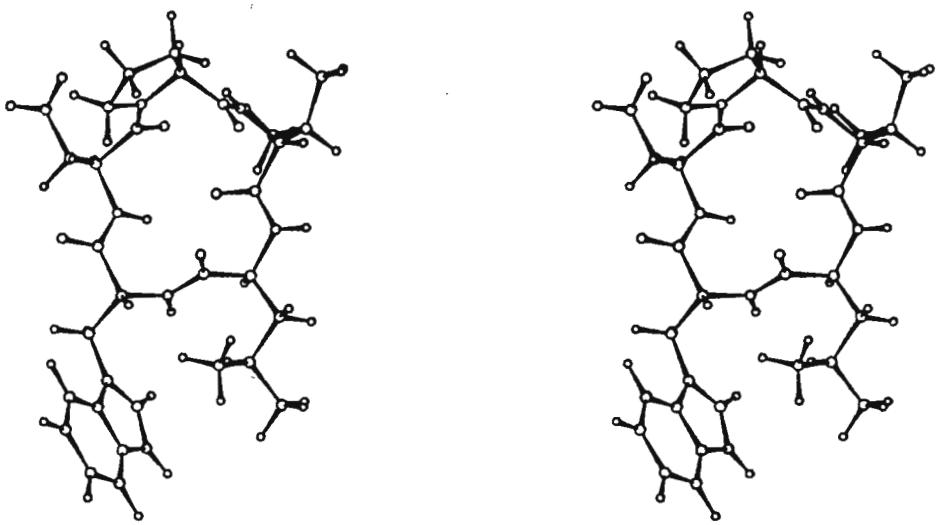

Figure 1. Stereoview of a DG/RMD calculated structure of CPP-1 in DMSO.

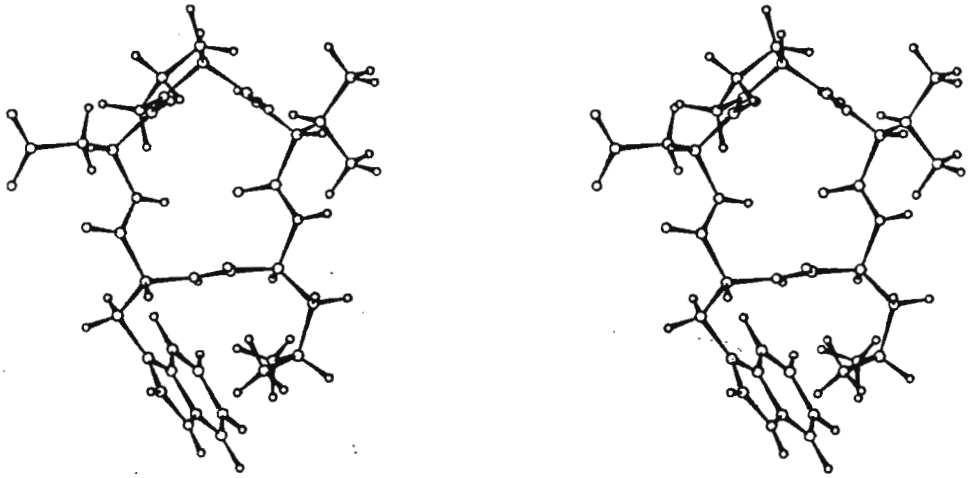

Figure 2. Stereoview of a DG/RMD calculated structure of CPP-2 in 90% H2O/10% D2O.

Sincerely,

Nina C. Gonnella, Ph.D.

Xiaolu Zhang, Ph.D.

Yong Jin

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

2 April 1993. (received 4/7/93)

The use of composite pulses in the Hahn echo spectra of biomolecules in water

Dear Dr. Shapiro:

Frequently the distortion of spectra by residual coherence from the solvent resonance and other baseline distortions can inhibit the observation or quantification of vital information. This is particularly the case in the accurate measurement of cross-peak intensities from NOESY spectra of proteins in aqueous solution. We routinely run many such experiments employing the Hahn echo method[1,2] which results in flat baselines in the acquisition dimension and assists in removing broad components at the bottom of the residual water peak. The quality of the data when using such a spin-echo based sequence is of course dependent on the quality of the spin-echo pulse. We have investigated whether the use of different composite 180° pulses in the pre-acquisition echo affects spectral quality.

Using the simple composite pulse $90_x180_y90_x$ in the Hahn echo portion of a NOESY experiment, results in the generation of large dispersive components from the residual water as shown in Fig. 1, a 2D NOESY spectrum of BPTI in water. The dispersive components result from the fact that the composite pulse employed with the EXORCYCLE[3] refocusses both absorptive and dispersive components. The EXORCYCLE phase cycle comprises of the incrementation in phase of the pulses that constitute the composite pulse in steps of 90° while the receiver alternates plus/minus, over a four step cycle. As an alternative phase cycle, we can employ the procedure of Hetherington and Rothman[4] which removes the dispersive component from the EXORCYCLE approach. This produces a much less disruptive residual water resonance in the 2D NOESY spectrum of BPTI, as shown in Fig. 2, allowing the observation of peaks otherwise obscured by the extended dispersive tails from the water. The Hetherington and Rothman cycle can be written: $90_x180_y90_x$, $90_y180_x90_y$, $90\text{-}_x180\text{-}_y90\text{-}_x$, $90\text{-}_y180\text{-}_x90\text{-}_y$, with the receiver alternating plus and minus.

The improvement not only leads to less disturbance in the spectrum but allows the more successful application of time-domain convolution filters for elimination of the water resonance in processing[5]. Since this method uses a Hahn echo which limits the magnitude of the extended tails of the solvent, we are currently referring to this procedure as the HELMET procedure. This is not only because it is more convenient for us when discussing

it between ourselves, but moreso because we have always wanted to use this acronym for something and have never really had the opportunity before.

Please credit this contribution to the account of Peter Wright.

Sincerely

John Cavanagh
Scripps, La Jolla

Jon Waltho
Sheffield, UK

(1) M. Rance & R.A. Byrd, J. Magn. Reson. 52, 221 (1983).
(2) D.G. Davis, J. Magn. Reson. 81, 603 (1989).
(3) G. Bodenhausen, R. Freeman & D.L. Turner, J. Magn. Reson. 27, 511 (1977).
(4) H.P. Hetherington & D.L. Rothman, J. Magn. Reson. 65, 348 (1985).
(5) J. P. Waltho & J. Cavanagh, J. Magn. Reson. (submitted).

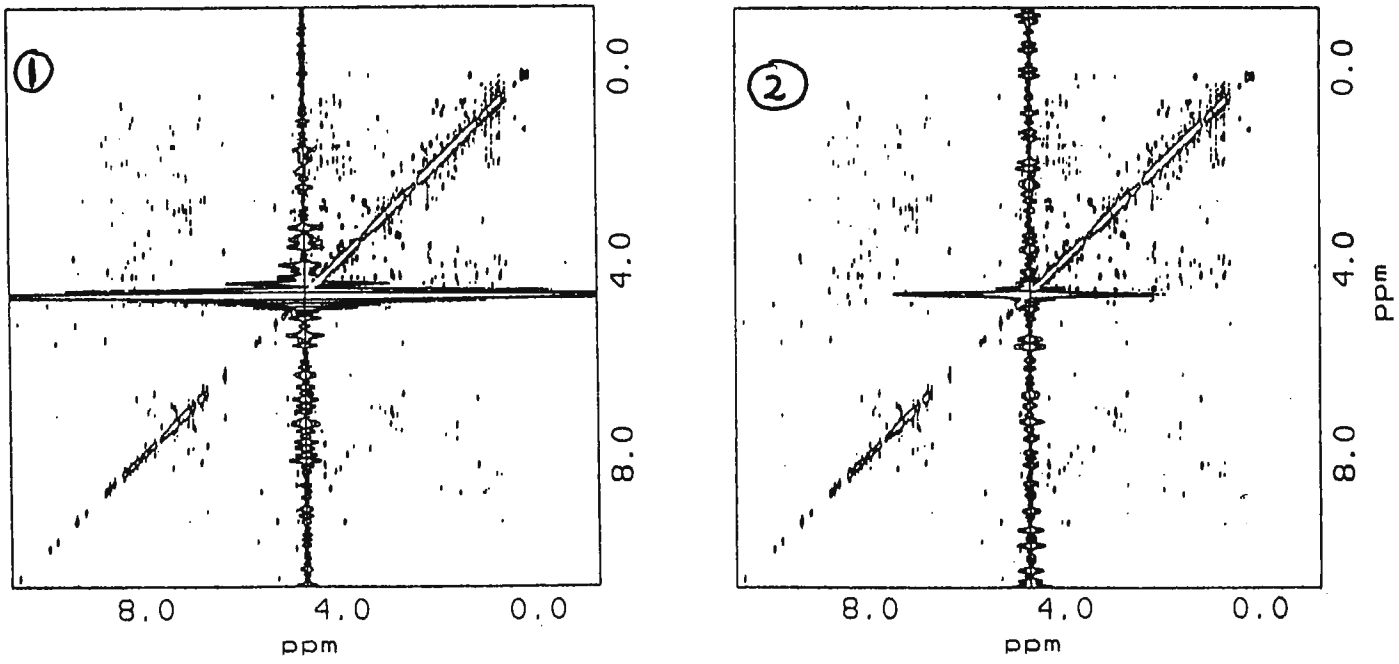

Figure 1 : 2D NOESY spectrum of BPTI in water recorded using a Hahn echo prior to acquisition consisting of the composite pulse described and the EXORCYCLE phase cycle.

Figure 2: 2D NOESY spectrum of BPTI in water recorded using a Hahn echo prior to acquisition consisting of the composite pulse described and the Hetherington and Rothman phase cycle noted.

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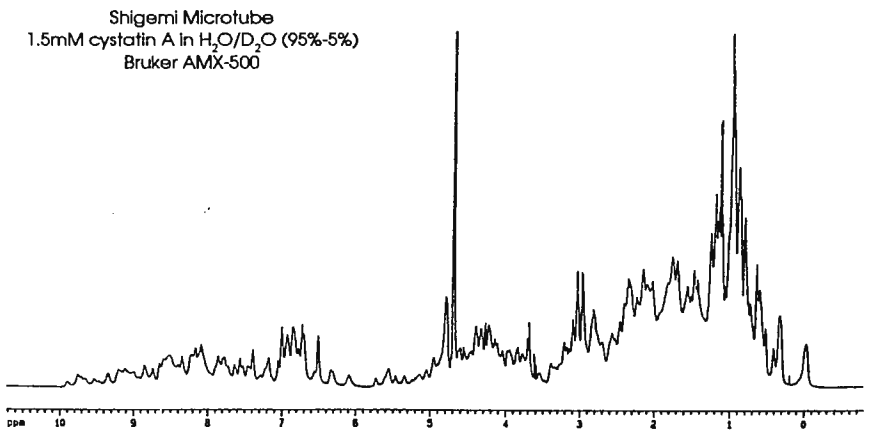

Figure 1: 1D spectrum of 1.5 mM protein dissolved in 250µl H_2O solution.

A protein, 11kDa, was dissolved in 250µl H_2O solution (pH 3.8) containing 5% D_2O for frequency lock and packed into the Shigemi Microtube. This spectrum was measured at 37°C on a Bruker AMX500 spectrometer with scan times 64. The solvent signal was suppressed by using low power RF irradiation.

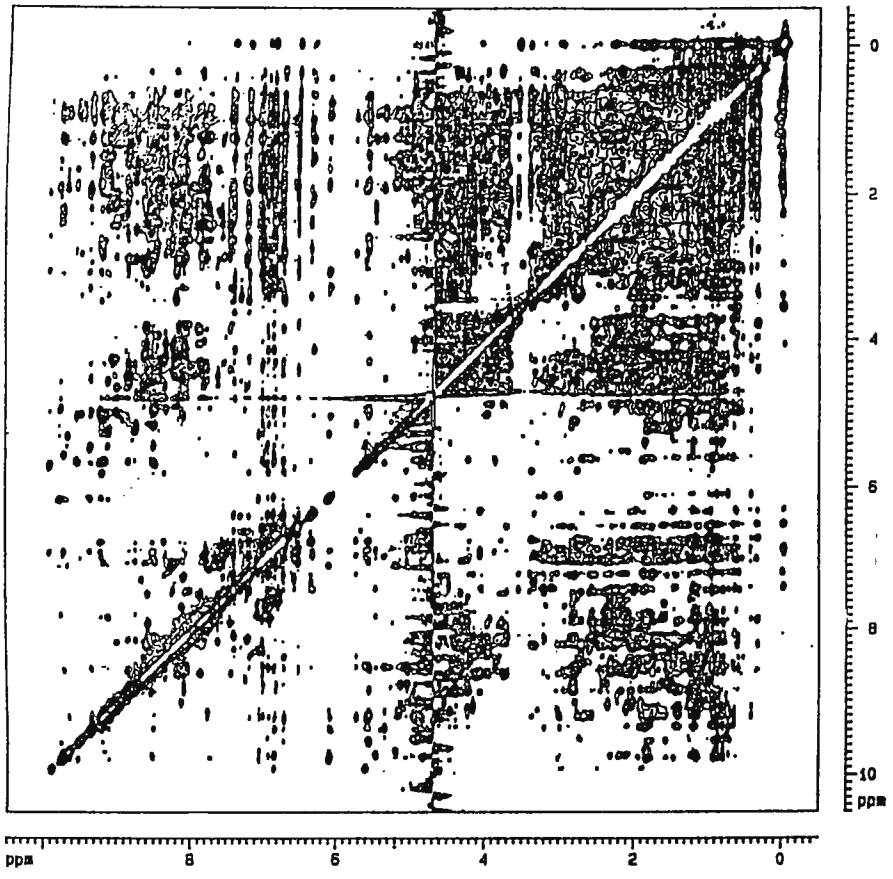

Figure 2: 2D NOESY spectrum of the same sample in Shigemi Microtube.

This 2D NOESY spectrum (τm=150 msec) of the protein in 250µl H_2O solution was measured with scan times 32 on a Bruker AMX500 spectrometer. The observed data matrix size was 1024 (t_2) x 200 (t_1) complex points. This matrix was processed with zero filling along t_2 dimension and resulted in a final data matrix of 1024 (F2) x 512 (F1) real points. It should be noted that baseline correction and digital processing were **not** applied to remove the water signal.

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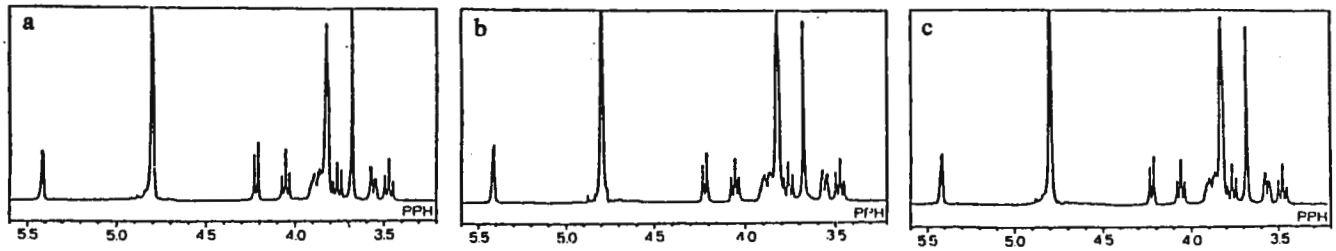
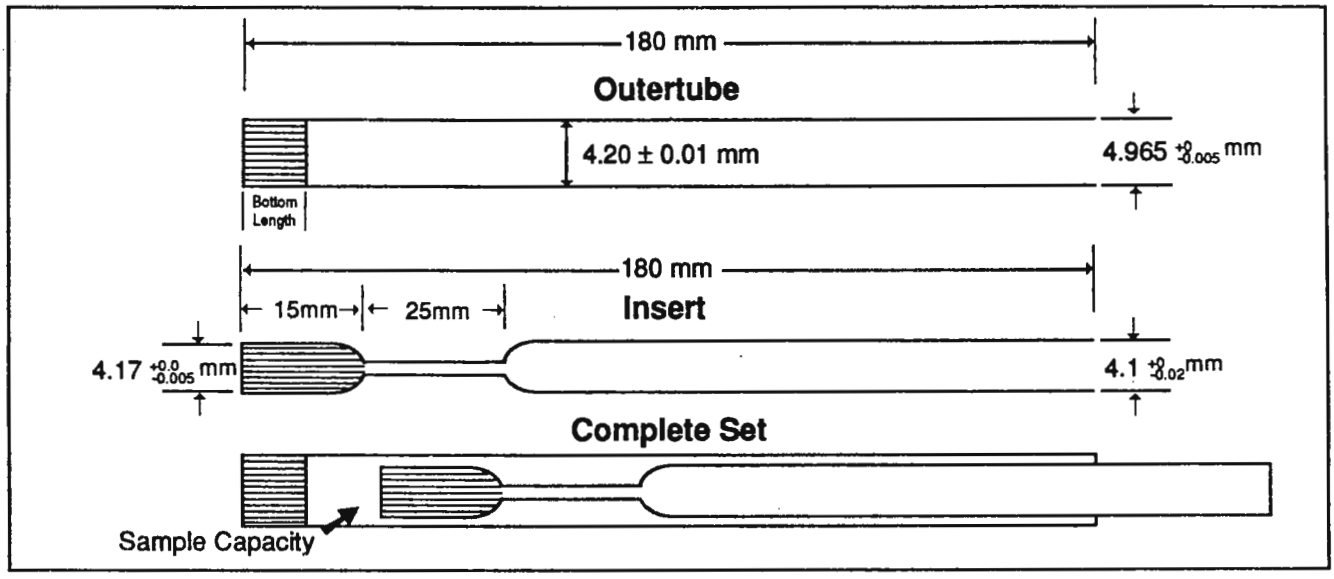


The 400-MHz 1H-NMR spectra of 10mM Sucrose in D_2O with varying sample height in the Symmetrical microtube. a) 5mm (49 µl); b) 3mm (41 µl); c) 2mm (28 µl)



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

March 16, 1993
(received 3/22/93)

Title: Osmotic Regulation of PCCG Snythesis in Nitrogen Fixing Bacteria.

Dear Dr. Shapiro,

Following our recent report[1] on the isolation and characterization of a phosphocholine substituted macrocyclic 1,3 ; 1,6 β-glucan (PCCG) in Bradyrhizobium japonicum USDA 110 (BRJ 110) and derived bacteroids we have embarked on studies of PCCG production and function. By examining *in vivo* ^{31}P spectra of BRJ 110 cells grown under different environmental conditions we observed that PCCG was preferentially produced in low osmotic (LO, 65mOsm/Kg H_2O) growth medium, (conditions that are present when BRJ 110 cells are free living in the soil). Airlift perfused [2] solutions of BRJ 110 cells grown on agar under high osmotic conditions (HO, 650mOsm/Kg H_2O), (conditions similar to those found for the symbiotic, cell encapsulated bacteroids) produced relatively minor amounts of PCCG. To evaluate the kinetics of this osmoregulated process we examined the ^{31}P spectra of 2g fresh wt. cells grown under HO conditions (Fig. 1-1) for 2h, and subsequently transfered them to an LO solution. Figure 1-2 illustrates the rapid metabolic turnover of the phosphomonoesters(δ 4.8) to Pi (δ 2.0) following the first 2h of adaptation. PCCG production appears to begin within 2h after the change of medium Fig. 1-3. After 14h the concentration of PCCG has doubled.

Although the airlift suggested by Fox et. al[2] works well in this study it limits us because we cannot provide a constant supply of nutrients for the cells during our *in vivo* experiments. Therefore, the production of PCCG can never reach its full potential. Future studies will require liquid perfusion (growth medium) of agarose immobilized cells. With this kind of approach we will be able to accurately evaluate the metabolic events regulated by osmotic pressure changes without having the present limitation on the availability of nutrient substrates.

Philip E. Pfeffer

Guillaume Becard

---

[1] Rolin, D.B., Pfeffer,P.E., Osman, S.F., Szwergold, B.S., Kappler, F. and Benesi, A.J. Structural studies of a choline-phosphate substituted b-(1,3);(1,6) macrocyclic glucan from *Bradyrhizobium japonicum* USDA 110 Biochimia et Biophysica Acta 1992 1116: 21-225.

[2] Fox, G.G., Ratcliffe, R.G. , and Southon, T.E. Airlift systems for *in vivo* NMR spectroscopy of plant tissues. J. Mag. Reson. 1989 82: 360-366.

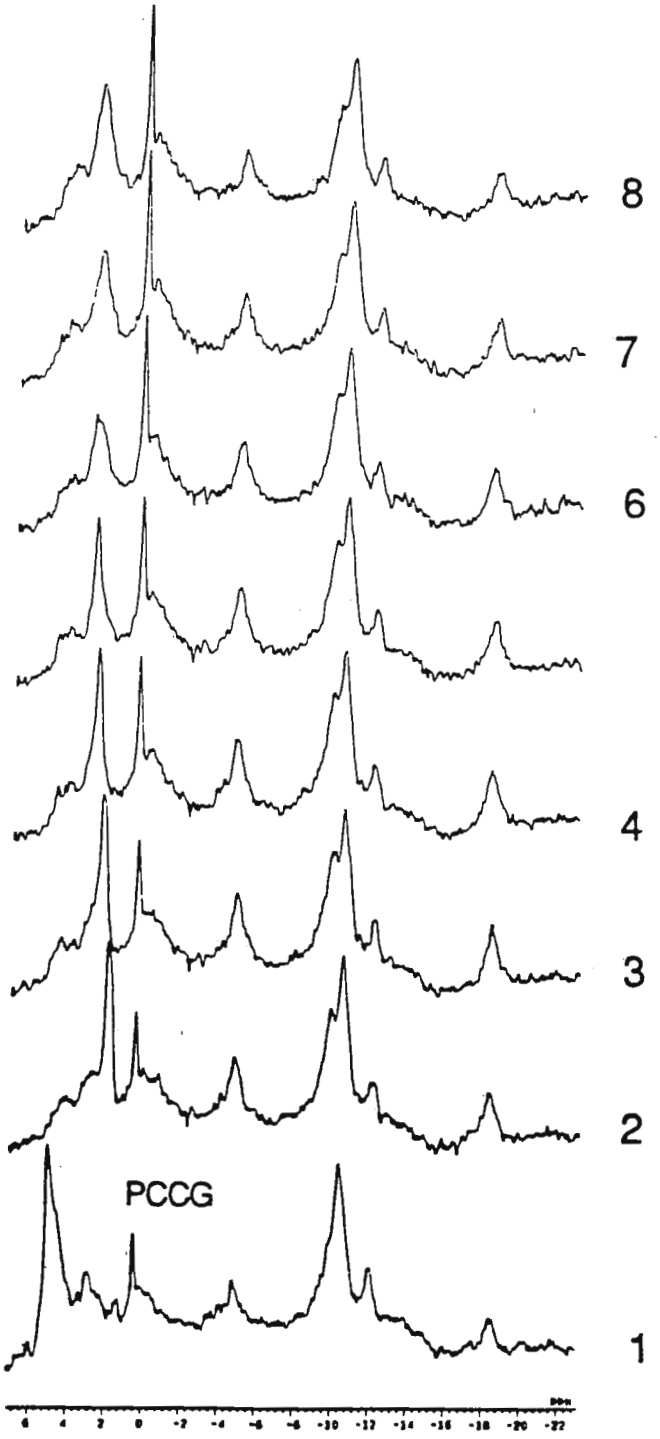

Figure1: 161.7 MHz *in vivo* ^{31}P NMR time course spectra of BRJ 110 cells (1 g FW) grown on HO Bacto-Agar medium(1) and sequentially placed in a LO (Mes/Hepes buffer pH=6.8 17mOsm/kg H_2O) environment (2-8). Each spectrum represents a two hr accumulation. Two hrs elapsed between spectrum 1 and 2.

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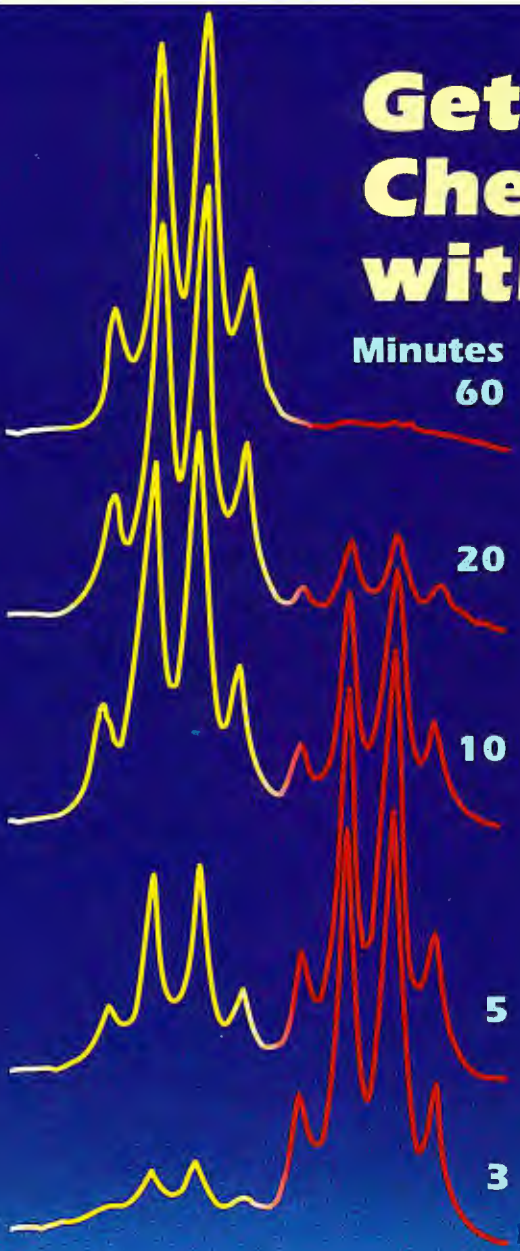

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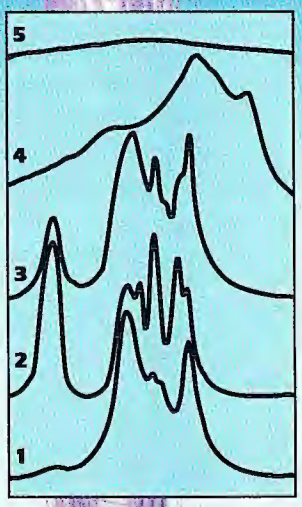

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By integrating the areas across the two quartets, the concentration of each component can be determind and rate constants for the reaction can even be calculated. This reaction was followed in a closed NMR tube, but could as easily have been followed in a flowing system. In a similar manner, many reactions of industrial interest can be readily monitored in the flow mode.

In the series of spectra in the right column, the curing of a five-minute, two-component epoxy was followed. The top two spectra are of the separate components; the bottom three are of the mixed adhesive. The last, broad spectrum is actually twice the width of the other four.

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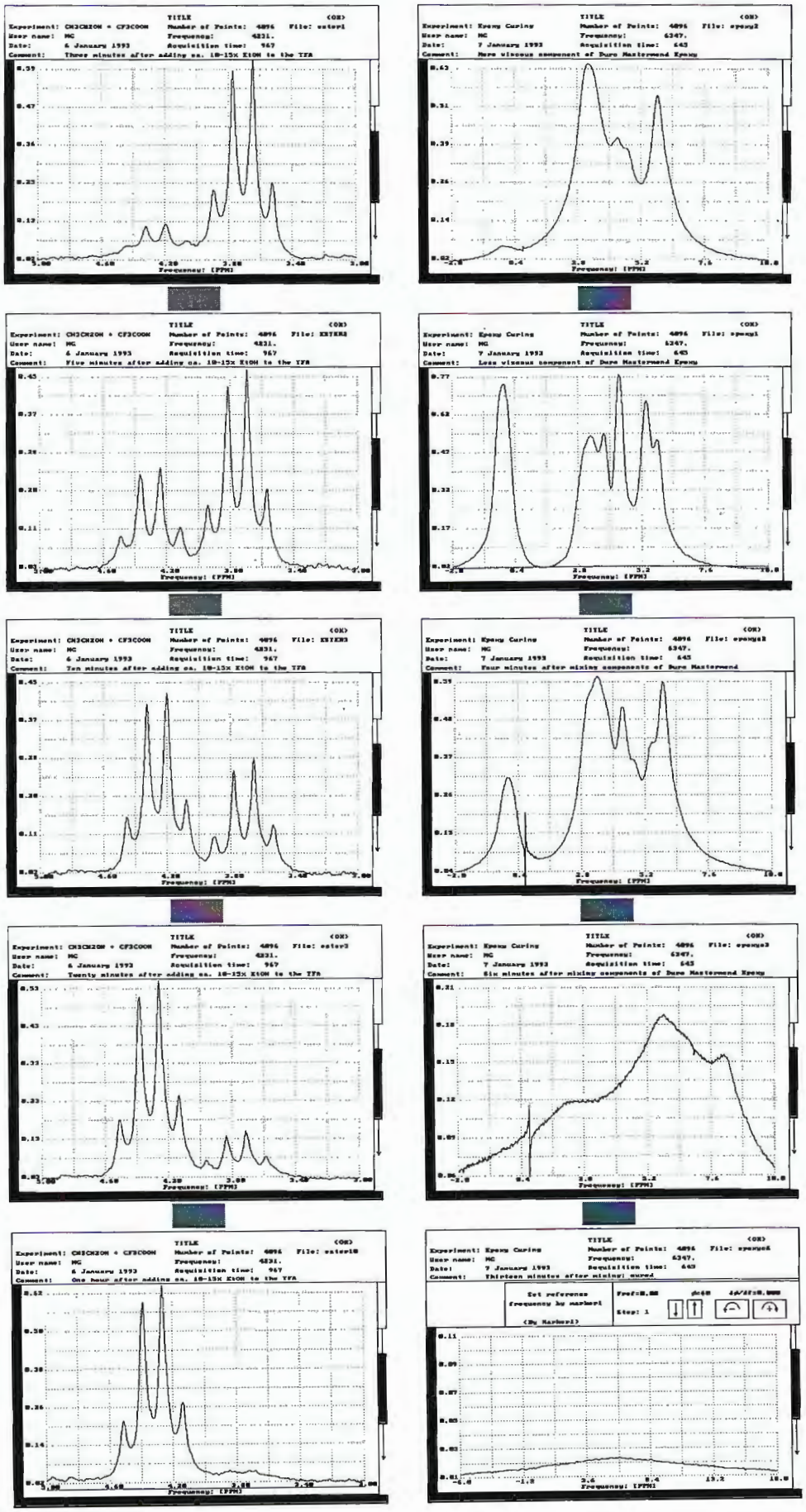

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TAMU NMR Newsletter

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Notice re 1993-94 Invoices and Subscription Rates

Subscription renewal invoices for the October 1993 - September 1994 year will be mailed out at the beginning of July. If you ought to receive such an invoice, and do not have it in your hands by July 15, please call or write me promptly. *Payment* of these invoices *must be received* by me *no later than September 10, 1993* to ensure uninterrupted mailing of the Newsletter issues. Please do not delay execution of any necessary paperwork!

Also, please be sure that the instructions on the invoice are followed precisely. In particular, *overseas subscribers* should be careful to see that their name and invoice number appear on the payment (or, better, that the extra invoice copy which is provided is returned to me with the payment check or money order). Anonymous checks, while otherwise useful, cannot always be credited to the correct account.

The subscription rate for the October 1993 - September 1994 year will remain as it is for the present year, *viz.*, US$170.00 for the twelve monthly issues, postpaid. Personal or academic subscriptions will continue to be offered at a 50% discount, at US$85.00.

The decision not to raise the subscription rates is predicated on the continuing generosity of our Sponsors and on Advertising revenues holding up at least at their present levels. I hope I'm right! Please do what you can to help by reacting to the ads which appear in the Newsletter, and by making sure to mention the Newsletter when you deal with the Advertisers. More ads are very much needed.

The new invoices contain an entry for an *optional* surcharge as follows: (1) for First Class mailing, available for US and Canadian addresses *only*, or (2) for Air Mail Printed Matter (AMPM) mailing *to all other countries*. (There will no longer be the possibility of sending overseas mailings by First Class *surface* mail, which turns out to be more costly and slower than AMPM.) Please adjust the amount you pay accordingly.

Thank you for your understanding and cooperation.

B. L. Shapiro
1 May 1993

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All Newsletter Correspondence

Should Be Addressed To:

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

(415) 493-5971

DEADLINE DATES

No. 418 (July)---------- 25 June 1993
No. 419 (August)---------- 23 July 1993
No. 420 (September)-------- 20 August 1993
No. 421 (October) -------24 September 1993

The Newsletter's fiscal viability depends very heavily on the funds provided by our Advertisers and Sponsors. Please do whatever you can to let them know that their support is noted and appreciated.

Mailing Label Adornment: Is Your Dot Red ?

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

Page Length Instruction

Attention overseas subscribers: If you must use paper which is longer that 11", *please* take care that all material (including signatures, addresses - everything!) ends no more than 10" from the top of each of your pages. It is costly to make reductions, and henceforth I reserve the right to chop the excess length off any page, no matter what the result. Beware of the dreaded guillotine! Your cooperation in this matter will be greatly appreciated.

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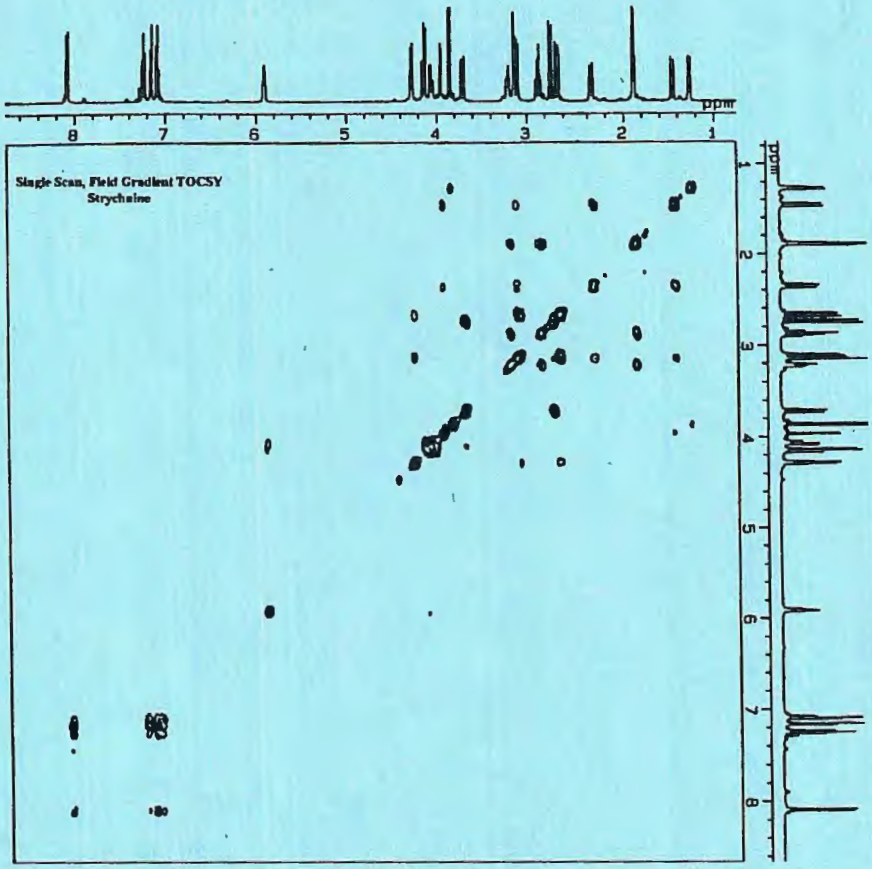

The above data is a 512 by 512 *TOCSY* spectrum and was obtained on an **Alpha**- 500 in less than 5 minutes with **JEOL**'s gradient probe. This same probe is also designed to do reverse and water suppression experiments. Pulse shaping, three channel operation, dynamic Rf control, matrix shims, as well as unsurpassed field reliability are all standard with the **Alpha**.

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