THE NMR NEWSLETTER

No. 461

February 1997

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

2 For applicable digits, see detailed instrument specifications.

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FORTHCOMING NMR MEETINGS

30th New Mexico Regional NMR Meeting Lovelace Respiratory Research Inst., Albuquerque, NM, **March 8, 1997**; Contact: E. Fukushima at <eiichi@audrey.tli.org>, 505-262-7155, or 505-262-7043 (fax). See Newsletter 461, 2.

5th Annual "Advances in NMR Applications" Symposium, Orlando, FL, **March 23, 1997**; Contact: Ms. Chris Tierney, Nalorac, 841-A Arnold Drive, Martinez, CA 94553;)510) 229-3501; Fax: (510) 229-1651; Email: christierney@nalorac.com. See Newsletter 460, 42.

38th ENC (Experimental NMR Conference), Orlando, FL, **March 23 - 27, 1997**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073. See Newsletter 460, 41.

International Society for Magnetic Resonance in Medicine, Fifth Scientific Meeting and Exhibition, Vancouver, BC, Canada, **April 12-18, 1997**; Contact: ISMRM, 2118 Milvia St., Suite 201, Berkeley, CA 94704, USA; (510) 841-1899; Fax (510) 841-2340; Email: info@ismrm.org.

Symposium on NMR Spectroscopy of Synthetic Macromolcules, ACS National Meeting, San Francisco, **April 13-17,** 1997; Contact: H. N. Cheng or English, A. D. See Newsletter 456, 20.

International School of Structural Biology and Magnetic Resonance, 3rd Course: Protein Dynamics, Function and Design; Erice, Sicily, Italy; **April 18-28, 1997**; Contact: Ms. Robin Holbrook, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, CA 94305-5055; (415) 723-6270; Fax: (415) 723-2253; Email: holbrook@smi.stanford.edu. See Newsletter 460, 30.

6th Meeting of AUREMN (NMR Users Association of Brazil), Rio de Janeiro, Brazil, **12 - 16 May, 1977**; Contact: Snia Maria C. de Menezes, Petrobás/Cenpes/Diquim/Radial 2, Quadra 07 - Ilha do Fundão, 21949-900 Rio de Janeiro, Brazil; Tel. +55 21 598-6171 and 598-6914; Fax. +55 21 598-6296; Email; sonia@cenpes.petrobas.gov.br.

The 30th New Mexico Regional NMR Meeting (otherwise known as the NMR^2 Meeting) will be held at the Lovelace Respiratory Research Institute (formerly the Lovelace Institutes and, before that, the Lovelace Medical Foundation), 2425 Ridgecrest Drive, SE, Albuquerque, NM, on Saturday, March 8th, 1997.

This is an informal meeting of anyone (even those outside New Mexico) interested in any kind of magnetic resonance, including electron resonance. **Mark Conradi** of Washington University, St. Louis, will be our guest, following a long list of past guest speakers including Richard Ernst, Paul Lauterbur, Irv Lowe, Ad Bax, Paul Callaghan, Alex Pines, Al Garroway, and Al Redfield. Conradi might (you are warned - this is an informal meeting) talk on how to do traditional high resolution NMR in an inhomogeneous field, how to transfer large amounts of Xe-129 polarization to something else, and/or a new take on SEDOR as an alternative to MQ magnetic resonance to count coupled spins in solids.

Over the years, we have received consistent financial assistance from several sources including Bruker, JEOL, CIL, NCC, and Isotec. All inquiries should be directed to **Eiichi Fukushima** at <eiichi@audrey.tli.org>, 505-262-7155, or 505-262-7043 (fax).

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NORTHWESTERN UNIVERSITY

January 17, 1997
(received 1/22/97)

Joseph B. Lambert
Clare Hamilton Hall Professor of Chemistry

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2145 Sheridan Road
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Telephone (847) 491-5437
Internet lambert@casbah.acns.nwu.edu
Facsimile (847) 491-7713

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

Dear Barry:

This letter is to describe two NMR-related results from our recent work in organosilicon chemistry. Silicon-29 NMR has proved to be the critical structural tool in this field when X-ray structures cannot be obtained. Resonance positions often are diagnostic for specific structural classes. A class that has been lacking in silicon chemistry has been the analogue of the carbocation, R_3Si^+. We have recently prepared the first example of this species and proved its existence by its ^{29}Si chemical shift. Because silicon is prone to form strong bonds with electronegative elements and to assume high oxidation states, the chemical problem is to avoid reaction of tricoordinate silicon with nucleophiles. Consequently, we reduced the nucleophilicity of the medium by the use of hydrocarbon solvents (arenes such as benzene or toluene), and we used a remarkably unreactive anion, tetrakis-(pentafluorophenyl)borate ($(C_6F_5)_4B^-$). Further protection even from these weak nucleophiles was obtained by the use of mesityl (2,4,6-trimethylphenyl) substituents: Mes_3Si^+. The species was generated from Mes_3Si-allyl by reaction with various electrophiles to expel the allyl group. The proof of the success of the reaction was the ^{29}Si chemical shift of δ 225. This very high frequency position, indicative of low coordination, was corroborated by ab initio calculations carried out by Thomas Müller of the Humboldt University in Berlin.

We also have found ^{29}Si NMR to be invaluable in structural analysis of branched and dendritic polysilanes. The ^1H and ^{13}C chemical shifts for the permethylated molecules tend to be very uninformative, but the ^{29}Si chemical shifts are diagnostic for the number of Si-Si bonds to a given atom. Of even more use is 2D silicon-silicon INADEQUATE, which can provide connectivity information on the skeleton of the species. This technique to our knowledge has not previously been used, probably because it would not be of use for polydisperse linear polysilanes. Monodisperse dendritic polysilanes, however, are natural substrates for the technique. We have applied it to the system $[(Me_3Si)_2SiMeSiMe_2]_3SiMe$, which contains a core silicon attached to three wings. The wings are composed of a spacer dimethylsilyl group between the core and a branchpoint silicon, which in turn is attached to two trimethylsilyl groups. When we originally reported this material, we were unable to obtain crystals for X-ray analysis. Thus the INADEQUATE method proved to be ideal for a structure proof. The 2D spectrum is shown on the next page and provides all the connectivities expected for this dendritic species. The method should be of general use with dendritic and branched polysilanes, particularly in the absence of crystal structures.

Sincerely,

Joseph B. Lambert Ernani Basso Yan Zhao

Short title: New results in silicon-29 NMR

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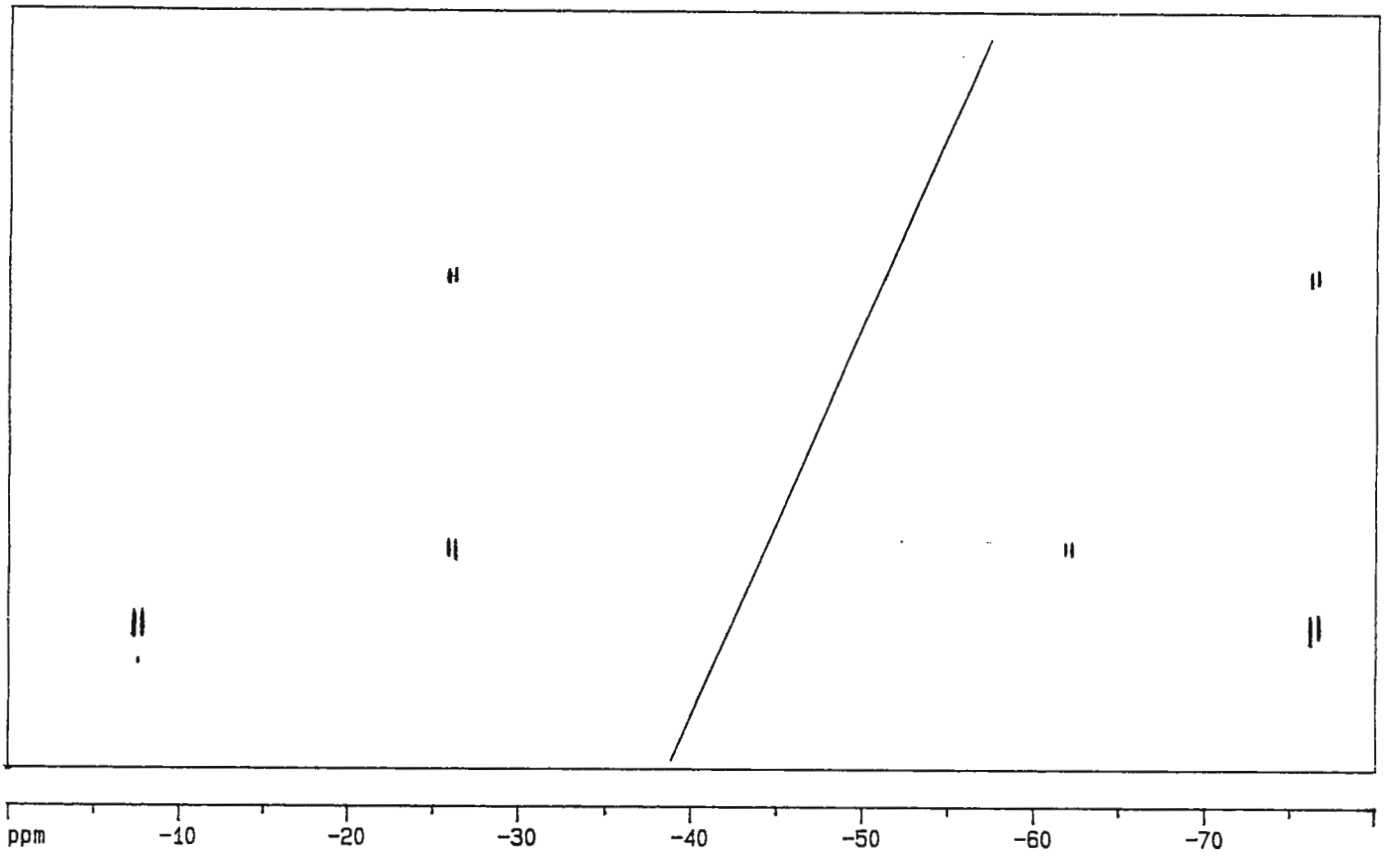

Postdoctoral Position
NMR Studies of Protein Structure and Protein-Water Interactions

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Dr. William S. Price, Chief Scientist
wprice@wri.co.jp

Professor Yoji Arata, Director
arata@wri.co.jp

Water Research Institute
Sengen 2-1-6,
Tsukuba
Ibaraki 305
Japan
Ph: (81-298) 58 6183, FAX: (81-298) 58 6166

The University of Akron

Department of Chemistry
Buchtel College of Arts and Sciences
Akron, OH 44325-3601
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January 15, 1997
(received 1/16/96)

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

Subject: Heteronuclear 3D-NMR of Polymers

Dear Barry,

Over the past couple of years, we have had tremendous success in applying $^{1}H/^{13}C/X$ heteronuclear 3D-NMR experiments to the study of polymer structure. These experiments have all succeeded without the benefit of isotopic labeling. However, we have been cheating; most of our published work to date has involved X-isotopes which are present in 100 % natural abundance (e.g. ^{19}F and ^{31}P).

We have recently started to study linear and dendrimeric polycarbosilanes. In these systems the three nuclei of interest are $^{1}H/^{13}C/^{29}Si$, which are present in 100%, 1.1% and 4.7% natural abundance, respectively. Detection of this three spin system is equivalent to doing NMR spectroscopy on 0.05% of the molecules in solution. Sensitivity is achieved by using ^{1}H detection and relatively high concentrations of polymer (typically 20-200 mg/mL). In indirect detection experiments we must be able to suppress signals from the 99.95% of the molecules which don't contain the necessary combination of NMR active isotopes. Pulsed field gradients (PFG's) are absolutely essential for this purpose. If the undergird signals are suppressed by PFG's, they do not reach the receiver system of the instrument and more than 10^3 improvement in dynamic range is achieved. For this purpose, a variation of the pulse sequence described in *J. Magn. Reson., Ser. A*, **120**, 125 (1996) was used.

Figure 1a shows the $^{1}H/^{29}Si$ HMQC spectrum of poly(1-phenyl-1-silabutane) (PPSB), using three bond $^{1}H/^{29}Si$ couplings. The three main resonances (A-C) are from Si in mm, mr/rm, and rr triads. The three weaker signals (D-F) are from the penultimate Si in the chain. Figure 1b-c are slices from the $^{1}H/^{13}C/^{29}Si$ 3D-NMR spectrum at the shifts of Si A, B, and C, respectively. The spectra are interpreted in much the same way as the $^{1}H/^{13}C/^{19}F$ spectra of fluoropolymers reported in *Macromolecules*, **29**, 4808 (1996). We are using $^{1}J_{CH}$ and $^{2}J_{CSi}$ to relate the ^{29}Si shifts with the ^{1}H and ^{13}C shifts of methylene groups two bonds away. This permits us to unambiguously assign the resonances of Si centered in mm, mr and rr triads.

Best regards,

Peter L.Rinaldi
Professor of Chemistry
Director of the Molecular Spectroscopy Laboratory

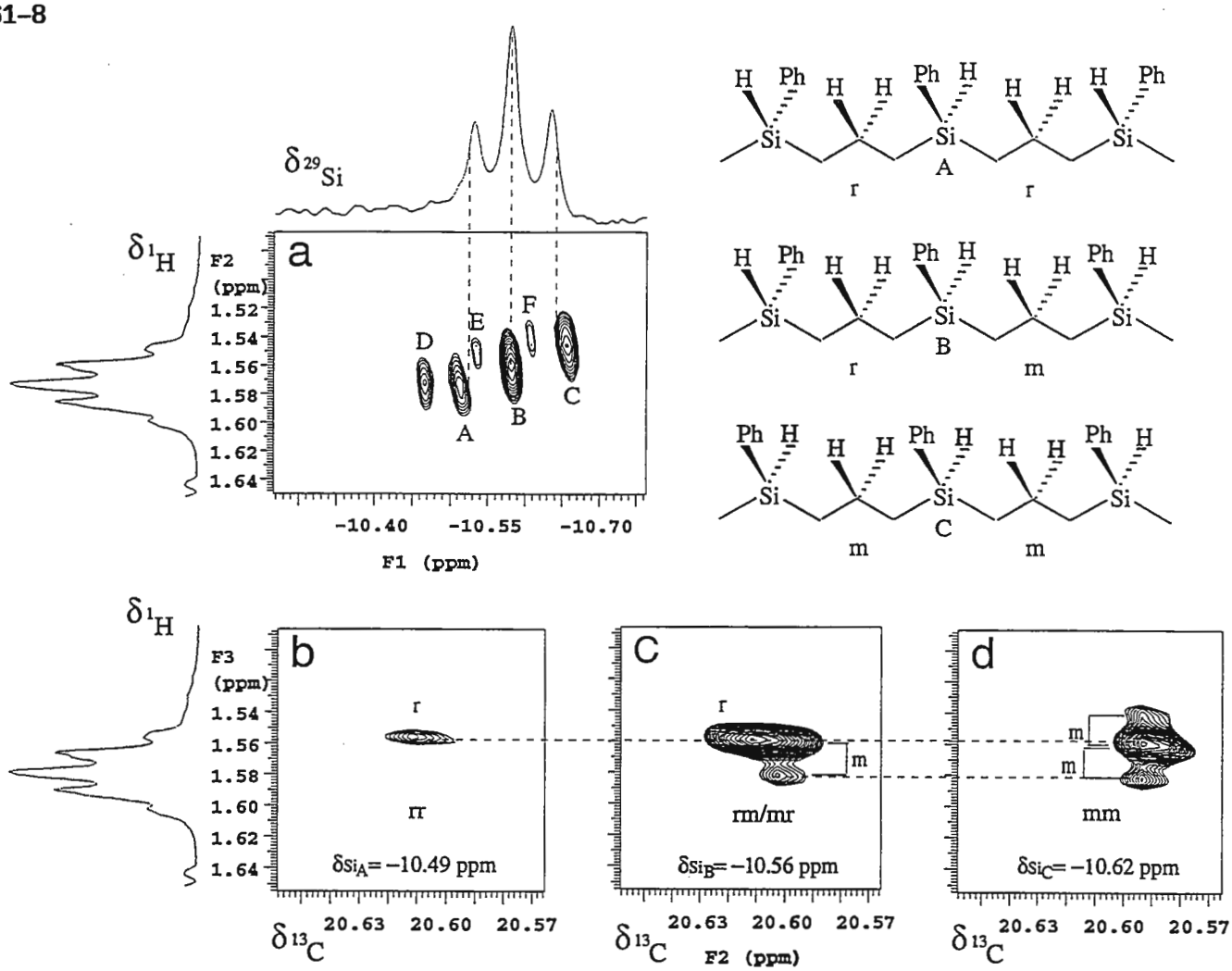

Figure 1. $^{1}H/^{13}C/^{29}Si$ Triple resonance NMR spectra of PPSB (250 mg) obtained in C_6D_6: (a) long-range ($^{3}J_{SiH}$ = 9 Hz) ^{1}H-^{29}Si HMQC 2D-NMR spectrum showing the ^{1}H and ^{29}Si 1D- spectra along the f_2 and f_1 axes, (b-c) f_2f_3 slices at different ^{29}Si chemical shifts from the $^{1}H/^{13}C/^{29}Si$ 3D-NMR; b) f_2f_3 slice at δ^{29}_{Si} = -10.49 ppm; c) f_2f_3 slice at δ^{29}_{Si} = -10.56 ppm; and d) f_2f_3 slice at δ^{29}_{Si} = -10.62 ppm. The 3D-NMR spectrum was obtained on a Varian Unityplus 600 MHz spectrometer with a Nalorac 5 mm $^{1}H/^{2}H/^{13}C/X$ gradient probe, at 30 °C, with 90° pulses for ^{1}H, ^{13}C and ^{29}Si of 10.2 μs, 22.0 μs and 14.0 μs, respectively, relaxation delay 1 s, Δ = 1.78 ms ($1/(4x^{1}J_{CH})$), τ = 10 ms ($1/(4x^{2}J_{CSi})$, $^{2}J_{CSi}$ = 5 Hz), acquisition time = 0.117 s (with simultaneous ^{13}C Wurst and ^{29}Si Waltz-16 decoupling); 4 transients were averaged for each of 2 x 32 increments during t_1 and 2 x 27 increments during t_2, 1909.6 Hz spectral window in f_3, 357.0 Hz spectral window in f_1, and a 100 Hz spectral window in f_2 dimensions. The durations and amplitudes of the gradient pulses were 2, 1 and 1 ms; and 0.364, 0.243, and 0.064 T/m, respectively. The first gradient pulse serves as a homospoil pulse, so its value relative to the other two is not critical. The total experiment time was 14 hours.

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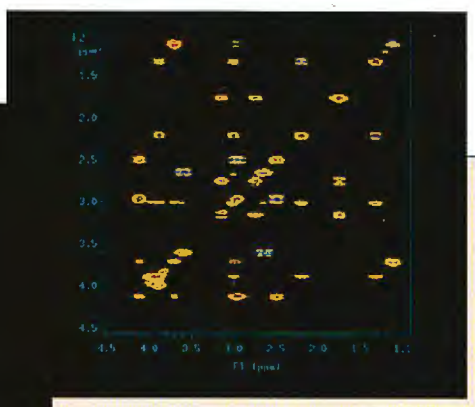
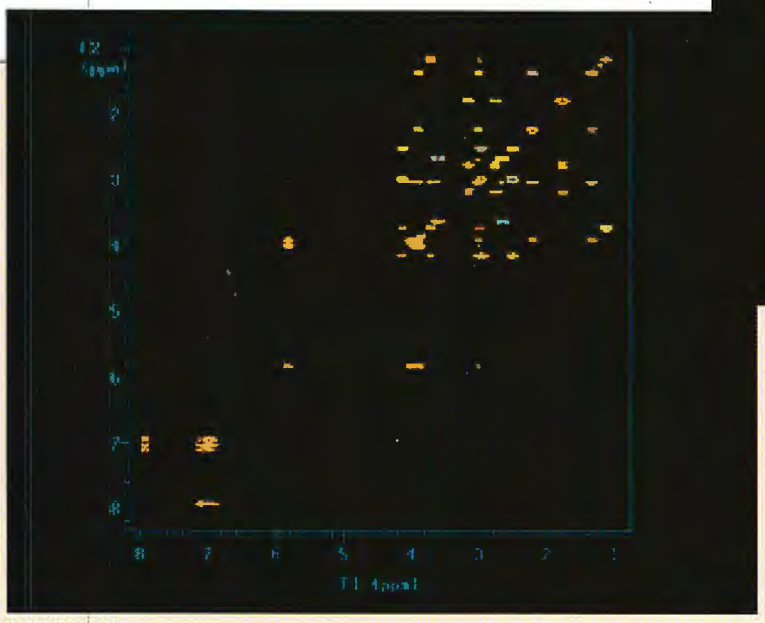
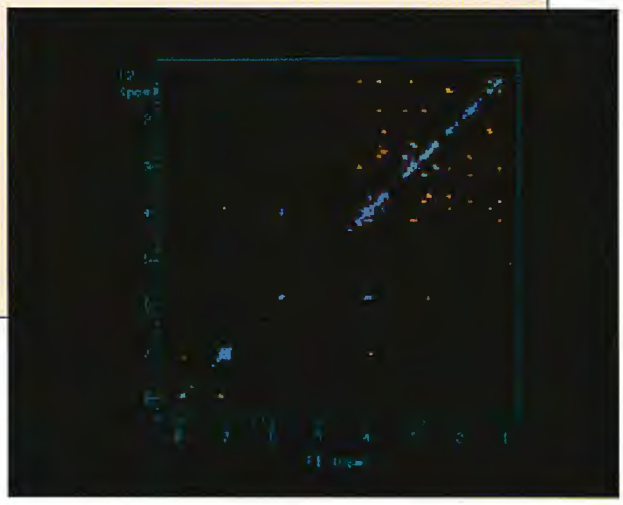


Expanded region of the TOCSY spectrum.



Spinlock experiments, such as this TOCSY spectrum of strychnine, are routinely performed with *MERCURY*.



MERCURY features advanced experiment capability, as is demonstrated in this high-quality ROESY spectrum.

Manufacturing Facilities Varian NMR Instruments, Building 4, 3120 Hansen Way, Palo Alto, California 94304-1030, Tel 415.493.4000 • **Australia** Mulgrave, Victoria, Tel 3.9.566.1133 • **Austria** Vösendorf, Tel 1.695.5450 • **Belgium** Brussels, Tel 2.721.4850 • **Brazil** São Paulo, Tel 11.820.0444 • **Canada** Mississauga, Ontario, Tel 1.800.387.2216 • **China** Beijing, Tel 1.256.4360 • **France** Les Ulis, Tel 1.6986.3838 • **Germany** Darmstadt, Tel 06151.7030 • **Italy** Milan, Tel 2.921351 • **Japan** Tokyo, Tel 3.5232.1211 • **Korea** Seoul, Tel 2.3452.2452 • **Mexico** Mexico City, Tel 5.514.9882 • **Netherlands** Houten, Tel 3063.50909 • **Russian Federation** Moscow, Tel 095.290.7905 • **Spain** Madrid, Tel 91.472.7612 • **Sweden** Solna, Tel 8.445.16.20 • **Switzerland** Basel, Tel 295.8000 • **Taiwan** Taipei, Tel 2.705.3300 • **United Kingdom** Walton-on-Thames, Tel 01932.898.000 • **United States** California, Tel 800.356.4437 • **Other sales offices and dealers throughout the world**

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

January 13, 1997
(received 1/17/97)

Water Suppression with (almost) no effort

Dear Barry,

Simple is best ! The WEFT pulse sequence (1-3) for water suppression in protein solutions is about as simple as it gets. WEFT is perhaps the only method that can be used if resonances under the water resonances (e.g., protein α-protons) are to be observed. Unfortunately, due to the increasing effects of radiation damping at high fields and in more sensitive probes, the WEFT method fails since radiation damping results in the water resonance having an effective relaxation time close to that of the protein resonances. We have recently developed an extremely simple variant of WEFT which we have termed Water-PRESS (4) (see Figure 1) which circumvents the problems of radiation damping.

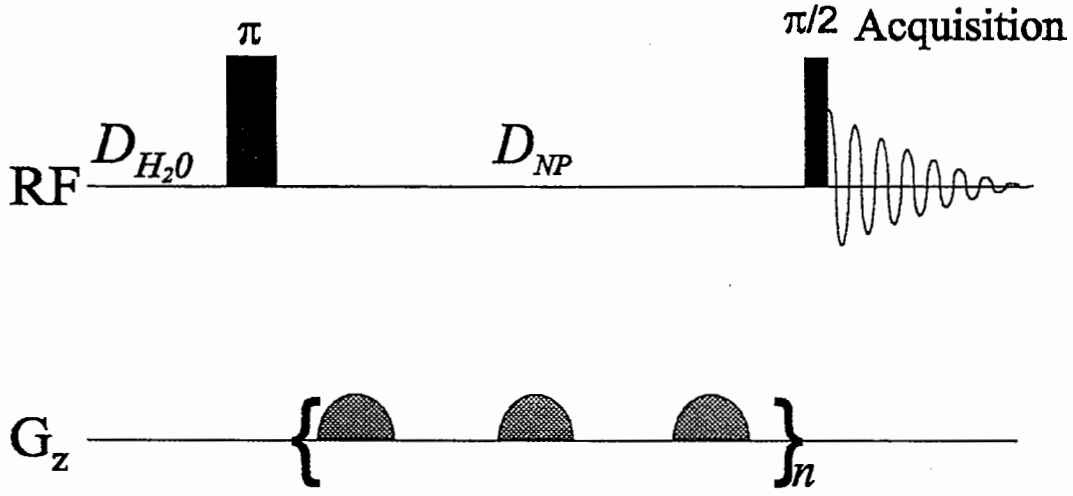

Figure 1 The Water-PRESS subunit consists of a relaxation delay (D_{H_2O}) to allow the water magnetization to reach thermal equilibrium. Next a π pulse is applied to rotate the magnetization to the -z axis. A delay (D_{NP}) of sufficient length is used to allow the water magnetization to relax to the origin. During D_{NP} a series of n very weak homospoil pulses (0.5 ms, ~ 1 G cm^{-1}) are applied so as to inhibit the effects of radiation damping. At the end of the D_{NP} the protein resonances, by virtue of their faster relaxation rate, have achieved thermal equilibrium. If, as in the present example, a non-selective π/2 pulse is applied an almost pure protein spectrum will be acquired (see Fig. 2).

The inclusion of a series of very weak gradient pulses in the D_{NP} delay has the effect of preventing the initiation of radiation damping. We demonstrated that the Water-PRESS sequence work extremely well when $D_{H_2O} \geq 5 \times T_1^{H_2O}$ (4) but this requirement makes the sequence unacceptably long if it is to be used in combination with multi-dimensional NMR experiments. We have now investigated the use of the Water-PRESS method when the $D_{H_2O} << 5 \times T_1^{H_2O}$ such that the water magnetization is kept in a steady state instead of being allowed to fully relax. We found that it works surprisingly well when run in the steady state (see Figure 2).

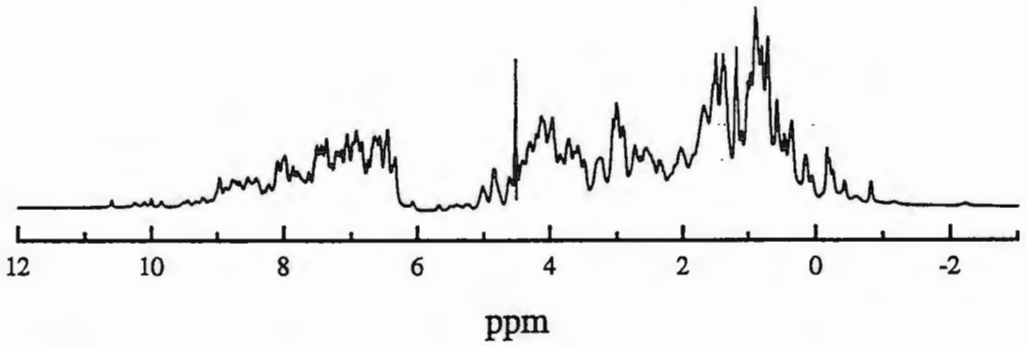

Figure 2 An example of water suppression in lysozyme solution (10 mM in 10:90 ^2H_2O:H_2O, pH 3.66) using the Water-PRESS sequence under steady state conditions at 310 K. The relevant experimental parameters were D_{H_2O} = 2.3 s (N.B. this includes the acquisition time of 0.8 s) and D_{NP} = 1.363 s. Because the Water-PRESS sequence was run in the steady state the effects of radiation damping are largely eliminated throughout the entire sequence including the acquisition time and the only remaining evidence of the water (including a contribution from the transmitter spike) is the small narrow spike at ~4.5 ppm.

References

1. F. W. Benz, J. Feeney, and G. C. K. Roberts *J. Magn. Reson.* **8**, 114 (1972).
2. S. L. Patt and B. D. Sykes *J. Chem. Phys.* **56**, 3182 (1972).
3. R. K. Gupta *J. Magn. Reson.* **24**, 461 (1976).
4. W. S. Price and Y. Arata *J. Magn. Reson.* **B 112**, 190 (1996).

Please credit this to the account of Y. Arata.

Yours sincerely

W. S. Price

William S. Price

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ARGONNE NATIONAL LABORATORY
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December 18, 1996
(received 12/24/96)

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

Re: **NMR Imaging of Polymer Dissolution**

Dear Barry,

In recent years there has been an ongoing effort in our lab to study solvent transport behavior in polymer and coal samples. Time-resolved NMR imaging methods have been employed to monitor solvent uptake in polymers (*Macromolecules,* **1994,** 27, 2607-2614.) Data obtained from imaging experiments has been used to construct a theoretical model for solvent transport. Very recently, we have had to opportunity to observe the opposite effect: dissolution of a polymer into a solvent.

Poly(ethyl methacrylate) with average M_W=850,000 was purchased from Aldrich. This was hot pressed to form a clear solid pellet. A 2 x 2 x 10 mm specimen was placed in a 5mm NMR tube with methanol and allowed to dissolve. Spin-echo 2-D images were obtained using a slice selection technique. The slice was 2mm thick and taken through the center of the long axis of the sample.

The Figure presents time-resolved proton images of the methanol. The dark regions in the center represent the PEMA sample. The first image (a) shows the sample in the initial stages of the dissolution process. The non-uniform signal intensity in the image may be due to colloidal particles of PEMA which have separated from the main sample but which have not yet fully dissolved. The second (b) and third (c) parts of the Figure show later stages of dissolution. Here, the methanol signal has become more uniform. It is interesting to note the effect of molecular weight on this process. PEMA with a molecular weight of M_W=515,000 or less does not dissolve in methanol, but instead, undergoes a phase transition to become a rubber.

Studies such as these have potential for the study of physical and chemical properties of polymers. Also, NMR imaging may be used in this way as a method for the study of drug delivery systems.

Sincerely

David M. Gregory
Chemistry Division

Robert E. Botto
Chemistry Division

*Work performed under the auspices of the Office of Basic Energy Sciences, Division of Chemical Sciences, U. S. Department of Energy, under contract no. W-31-109-ENG-38.

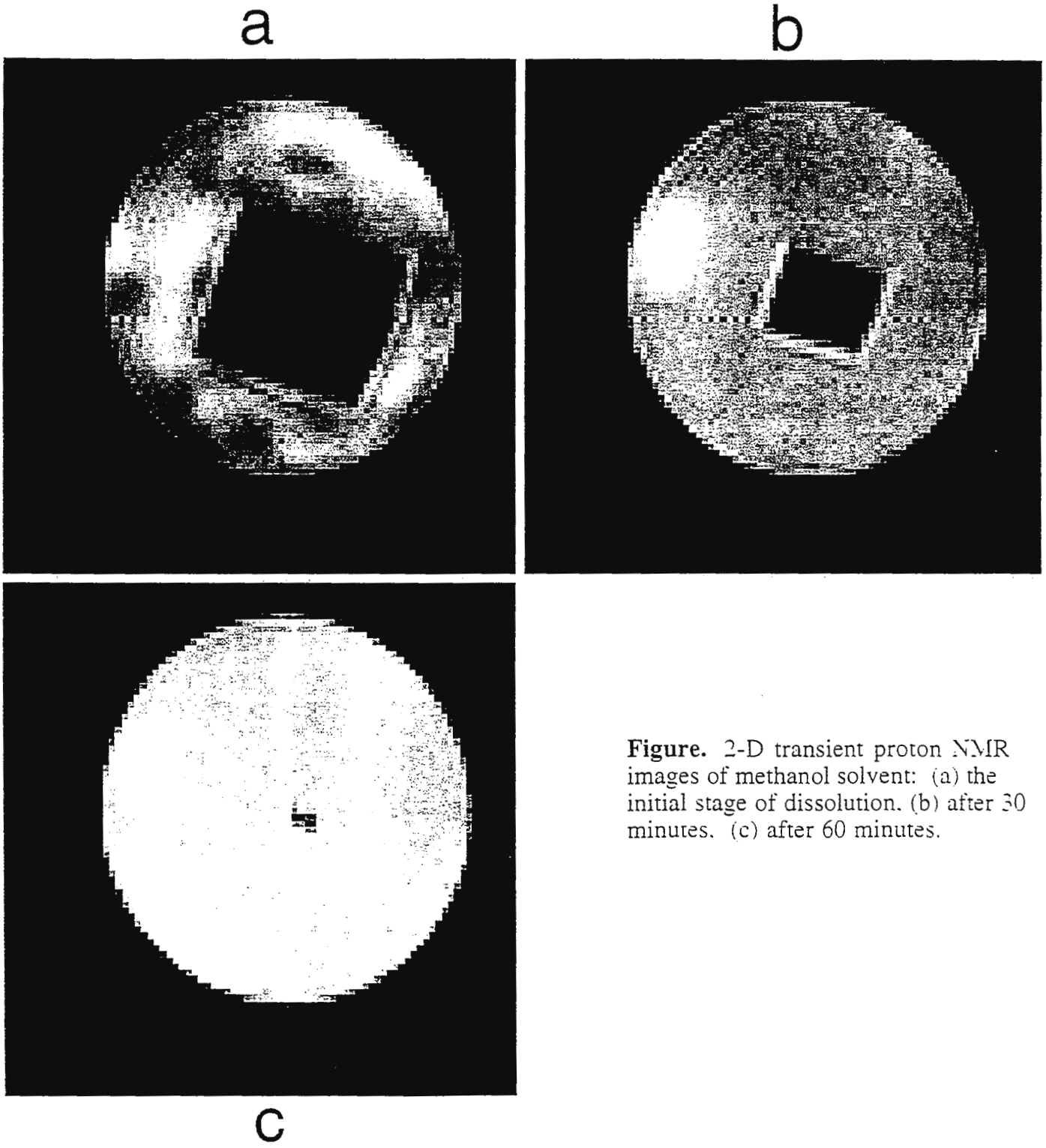

Figure. 2-D transient proton NMR images of methanol solvent: (a) the initial stage of dissolution. (b) after 30 minutes. (c) after 60 minutes.

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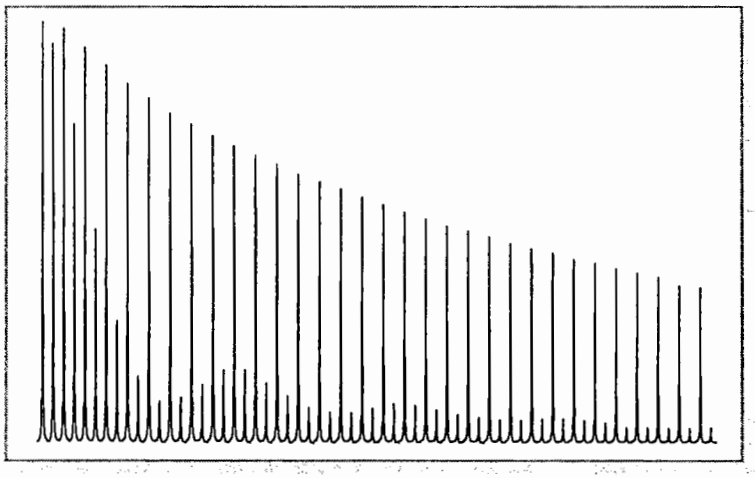


$^{13}C/^{15}N$ REDOR with ^{1}H decoupling, obtained on [2-^{13}C,^{15}N]-glycine.

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

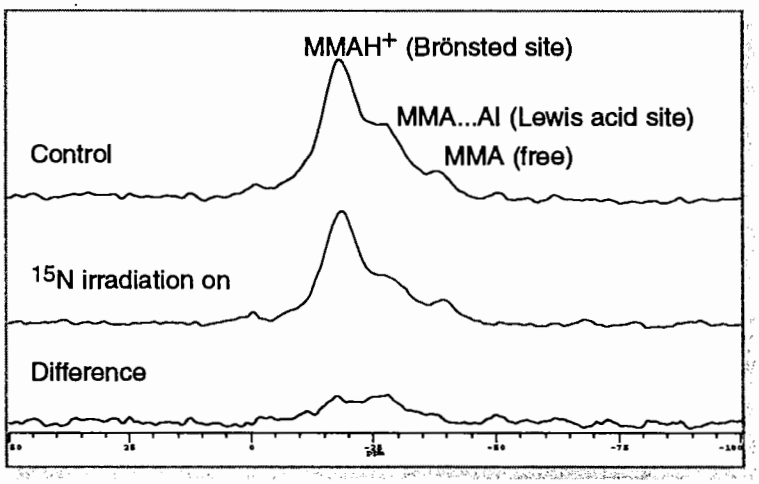
Control experiments (1st, 3rd, etc. peaks) demonstrate high decoupling powers. Significant signal remains after 64 rotor periods, as seen in the next to last peak.

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

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

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

data courtesy of C. Grey, SUNY, Stony Brook.



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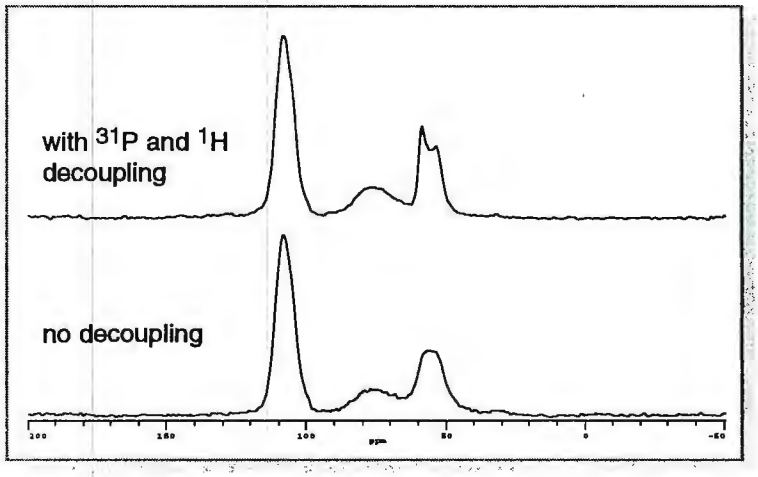


^{27}Al Observation with X/H decoupling, of $AlPO_4$-H2.

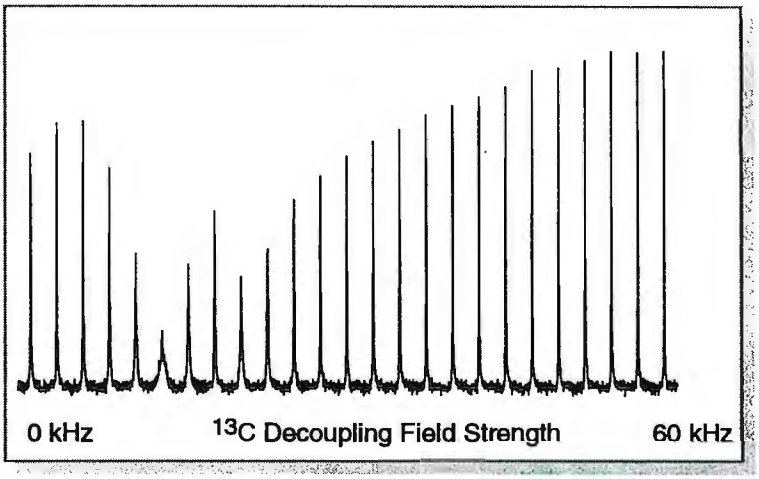
Decoupling of the ^{31}P and ^{1}H nuclei provides enhanced resolution in the ^{27}Al spectrum.

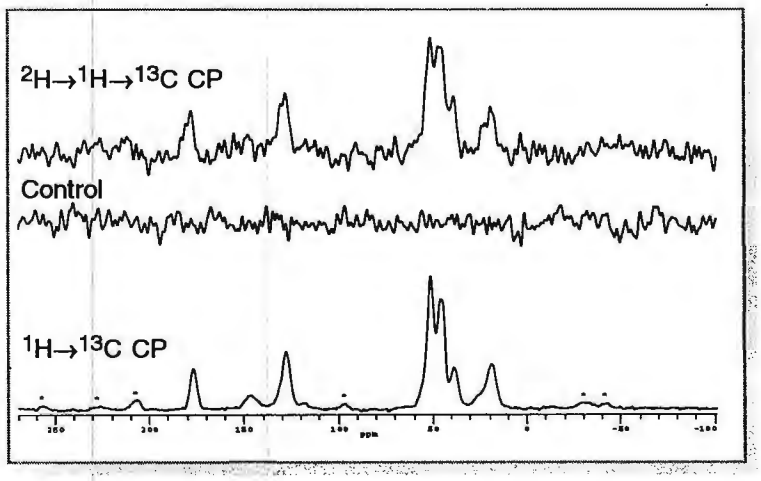
The signal-to-noise does not degrade with addition of ^{1}H and ^{31}P decoupling. This is the result of good X/Y channel isolation in the probe and filtering between the probe and receiver.

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

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

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





^{13}C CP and Double-Cross Spectra of d_8-PS/PMMA copolymer.

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

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

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E-mail: jerrym@itsa.ucsf.edu

January 21, 1997
(received 1/25/97)

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

Dear Dr. Shapiro:

RE: New Version of MATPULSE

A new version of MATPULSE is available from the Internet (http://nmrsg.bioiphys.upenn.edu/). As described in reference 1, this menu-driven, GUI program uses the Shinnar - Le Roux scheme (2) for generation of computer optimized frequency selective RF pulses. A separate menu is provided for examination of the gradient refocusing needed for excitation pulses. The program includes Bloch equations menus which enable the excitation bandwidths to be examined in the presence and absence of nuclear relaxation, and also features the ability to generate shaped pulses with modulated gradients (1,3). The program does require that the user have MATLAB (The Math Works, Inc.) version 3.x or higher, as well as the MATLAB Signal Processing Toolbox.

The program produces numerous kinds of frequency selective RF pulses which are of interest to high resolution NMR researchers, including computer optimized saturation, inversion, spin echo, and water suppression pulses (1). The rapid generation and display of these pulses and profiles facilitates examination of the effects of the various pulse parameters.

Features added to the new version of MATPULSE include the ability observe the effects of cascaded pulses, and a new menu to generate frequency selective self-refocused pulses via root reflection as described by James B. Murdoch in the Society of Magnetic Resonance Abstracts, pg. 552, 1995. In this scheme, a conjugate pair of roots from the A polynomial (one of the pair of polynomials representing the rotation produced by the pulse (2)) are reflected about the unit circle. The procedure for generation of a z-magnetization excitation pulse (4) is illustrated below in Fig. 1. Although several steps are involved, MATPULSE requires only a few mouse clicks to step through the procedure.

Typically, some adjustment of the pulse length is required to achieve the desired refocusing. Generation of a general rotation self-refocused pulse (4) is achieved by starting with a linear phase (symmetrical) pulse and following the scheme illustrated in Fig. 1. The advantage of using MATPULSE is that the tradeoffs of various parameters may be readily examined and adjusted, and the pulse shapes tailored for particular uses.

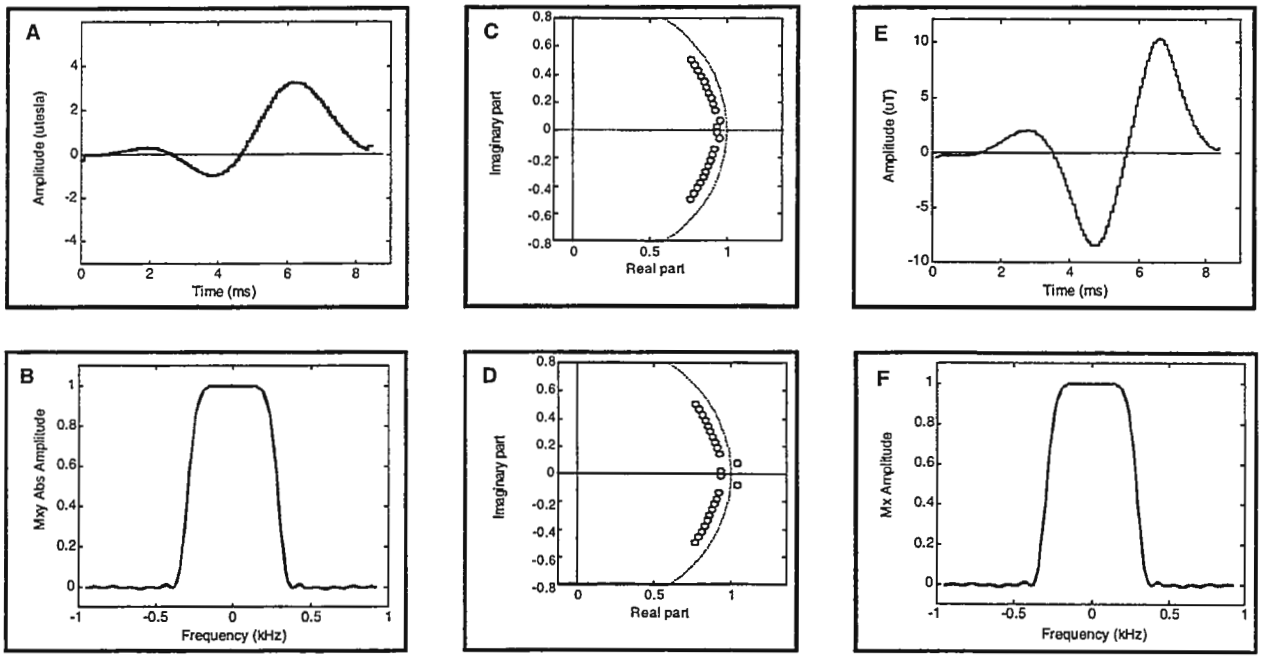


Fig. 1. The steps required for generation of self-refocused z-magnetization excitation pulse.
A. Initial minimum phase pulse: Width, 500 Hz, duration 8.02 ms, 128 points, passband ripple selection .05%, rejectionband ripple selection .005%.
B. Resulting excitation magnitude profile (magnetization is not refocused).
C. A portion of the A polynomial roots shown around the unit circle.
D. Reflection of the selected roots about the unit circle.
E. The resulting self-refocused RF pulse shape.
F. The resulting pulse profile, depicting the Mx magnetization.

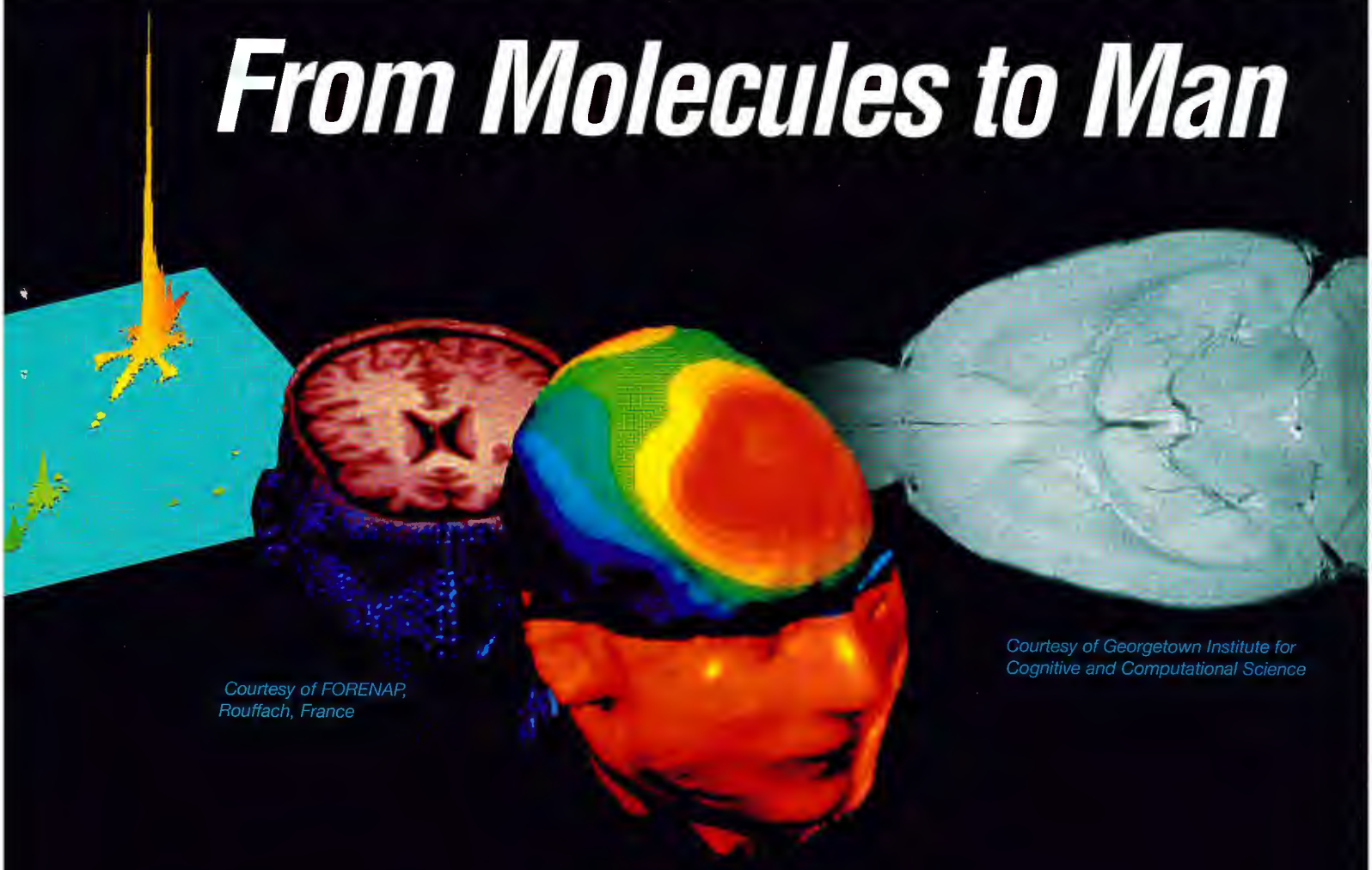
Please credit this to Michael W. Weiner's account.

Sincerely,

Gerald B. Matson
Adjunct Prof., Pharm. Chem., and
Facilities Manager, MR Unit, DVAMC

1. G.B. Matson, "An integrated program for amplitude-modulated RF pulse generation and re-mapping with shaped gradients", *Magn Reson Imaging* 12: 1205-1225 (1994).
2. J. Pauly, P. Le Roux, D. Nishimura, and A. Macovski, "Parameter Relations for the Shinnar-Le Roux Selective Excitation Pulse Design Algorithm", *IEEE Trans Med Imaging* 10: 53 - 65, (1991).
3. S. Conolly, D. Nishimura, A. Macovski, and G. Glover, "Variable-Rate Selective Excitation", *J Magn Reson* 78: 440 - 458, (1988).
4. H. Geen and R. Freeman, "Band-Selective Radiofrequency Pulses", *J Magn Reson* 93: 93-141, (1991).

From Molecules to Man



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Magnet Flange to Field Center (mm)	550	605	550	550	758	825	550	825	735
Max. Drift rate (ppm/hr.)	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
5 Gauss Fringe Field from magnet center:									
– radially (m)	3.7	4.8	4.6	5.1	6.8	8.2	5.3	7.0	6.7
– axially (m)	4.7	5.9	5.8	6.3	8.5	10.4	6.6	8.9	8.4
Homogeneous Volume (mm dsv)* shimmed to linewidth less than:									
± 10 ppm	160	180	120	160	220	250	120	160	120
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Cryogens (helium) Typical boil-off (refrigerated)	100 ml/h		100 ml/h		100 ml/h	
Refill interval:	125 days		160 days		160 days	
Magnetic field:	2.94 T		2.94 T		4.0 T	
1H-Frequency:	125 MHz		125 MHz		170 MHz	
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Stray field (0.5 mT): axial:	8.5 m[1]		14.5 m		14.5 m	
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Homogeneity	dsv (mm)	ppm	dsv (mm)	ppm	dsv (mm)	ppm
*half-height line-width	220	0.15*	220	0.1*	220	0.1*
** peak-peak variation in 12-plane-plot	300	2.5**	300	0.5**	300	1.5**
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Max. gradient (mT/m)	950	200	100	50
at given current (A)	100	100	100	100
Voltage (V)	150	150	150	150
Switching (µs)	50	80	200	180
Effective Volume, dsv (mm)	40	80	120	180
Linearity (%) over dsv	5	4	4	4

**Shielding not necessary due to small diameter*

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Gradient System	Feature	Data
B-Ga63	outer diameter	890 mm
	inner diameter	630 mm
	linearity	± 7% at dsv 450 mm
	max. gradient	30 mT/m at 600A
	switching	150 µs at 600A/1000V
		430 µs at 600A/350V
B-GA55	outer diameter	780 mm
	inner diameter	550 mm
	linearity	± 7% at dsv 300 mm
	max. gradient	45 mT/m at 600A
	switching	150 µs at 250A/300V
		300 µs at 600A/350V
		110 µs at 600A/1000V
B-GA38	outer diameter	610 mm
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Nuclear Magnetic Resonance
and Medical Spectroscopy

7701 Burholme Avenue
Philadelphia, Pennsylvania 19111
215 728 5353/3049
FAX 215 728 2822

(received 1/17/97)

Intracellular Magnesium in Human Brain

Dear Dr. Shapiro:

Intracellular magnesium concentration ($[Mg]_i$) has been measured under *in vivo* conditions through the pH-dependent complexation of the ion with the phosphates of nucleotides tri- and diphosphates (NTP and NDP) which affects the chemical shift (δ) of the nucleotides' signals in ^{31}P magnetic resonance spectroscopy (^{31}P MRS). In order to ensure reliable brain-localized ^{31}P MRS to quantify $[Mg]_i$ a number of improvements to a 3-dimensional chemical shift imaging protocol (3D-CSI) were made resulting in faster collection times, better spectral baselines, higher spectral resolution, and optimized spectral localization (Fig.1). A dual-tuned ^{1}H-^{31}P birdcage resonator[1] minimized moving the subject's head by using the same coil for imaging and spectroscopy and allowed the study of the whole brain volume; shimming was done using an automated procedure[2] giving optimal values in 3-5 min; ^{1}H-decoupling and NOE increased the s/n ratio of the spectra[3]; and presaturation of the broad spectral components plus shorter gradient ramping times improved the baseline of the localized spectra[4]. The utilization of 3D-CSI to localize the ^{31}P signals has the advantage of lacking chemical shift artifacts however there is contamination due to point spread function effects. To minimize contamination, the coil geometry and subject's positioning are such that excitation of the strong neck muscles signals is kept to a minimum and a tight selection of brain spectra is done in postprocessing.

To obtain the δ of βNTP ($\delta_{\beta\text{-NTP}}$) the original methods were modified to use the phosphocreatine (PCr) signal as reference, because it is a strong, well resolved brain signal and its δ is insensitive to physiological changes and because the original reference signal (α-NTP) is contaminated with other signals in brain spectra (diphosphodiesters). The brain $\delta_{\beta\text{-NTP}}$ determined in this way was -18.92 ppm $\pm$ 0.009 (mean $\pm$ S.E. in 5 volunteers with an average of 20 voxels per volunteer). pH was determined from the δ of inorganic phosphate, giving a constant brain pH value of 6.99 $\pm$ 0.012 (mean $\pm$ S.E.; in 30 voxels total from 5 volunteers). Brain $\delta_{\beta\text{-NTP}}$ values were transformed to $[Mg]_i$ using the complexation endpoints (free and totally $[Mg]_i$ -complexed) at the corresponding cellular $[Mg]_i$/NTP mole ratio, and a dissociation constant of 0.055 mM at pH 6.99. The calculated brain $[Mg]_i$ value was 0.162 mM $\pm$ 0.003 (mean $\pm$ S.E.,

n = 5). This value is smaller compared to previously reported values (≈ 0.350 mM). This discrepancy is due in part to the lack of consideration in previous reports, of the correct cellular $[Mg]_i$ complexation endpoints.

The tight value of $[Mg]_i$ in normal human brain reported here suggests a strong regulation of $[Mg]_i$ in brain cells and it is in full agreement with the possible active transport of $[Mg]_i$ out from the cells and with the regulatory aspects that have been proposed for the ion on neural metabolism. It also suggests that small variations of $[Mg]_i$ could account for some of the physiopathological alterations in diseased brain.

References:

1. J. Murphy-Boesch, *et al. J. Magn. Reson.* **103**, 103-114 (1994).
2. J. Hu, *et al. J. Magn. Reson.* **108**, 213-219 (1995).
3. J. Murphy-Boesch, *et al. NMR Biomed.* **6**,173-180 (1993).
4. R. McNamara, *et al. NMR Biomed.* **7**, 237-242 (1994).

Fernando Arias-Mendoza Radka Stoyanova Truman R. Brown

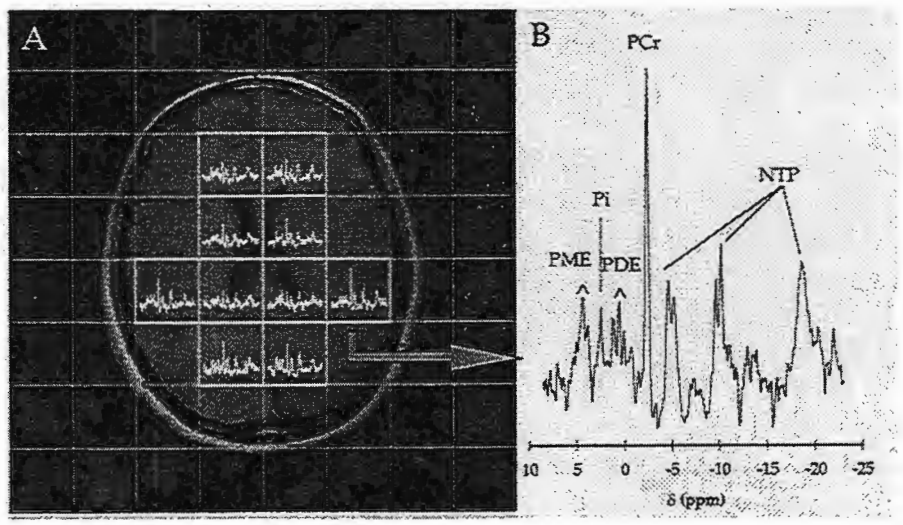

Figure 1: Panel A: Localized, ^1H decoupled-^{31}P CSI spectra of a volunteer overlaid on the corresponding axial proton image, filtered with a Lorentzian function of 5 Hz. The grid on the proton image correlates with the position of the CSI array. The voxels with spectra correspond to volumes (30 ml each) entirely included in the brain tissue and far away from possible muscle contamination. Panel B: Magnification of the rightmost spectrum in panel A to demonstrate the quality of the localized data. The assignments in the spectrum are: PME, phosphomonoesters; Pi, inorganic phosphate; PDE phosphodiesters; PCr, phosphocreatine; NTP mainly the γ, α and β-phosphate of NTP respectively but also the α and β-phosphate of NDP, and the phosphodiester moiety of diphosphodiesters. PCr is the reference at -2.52 ppm.

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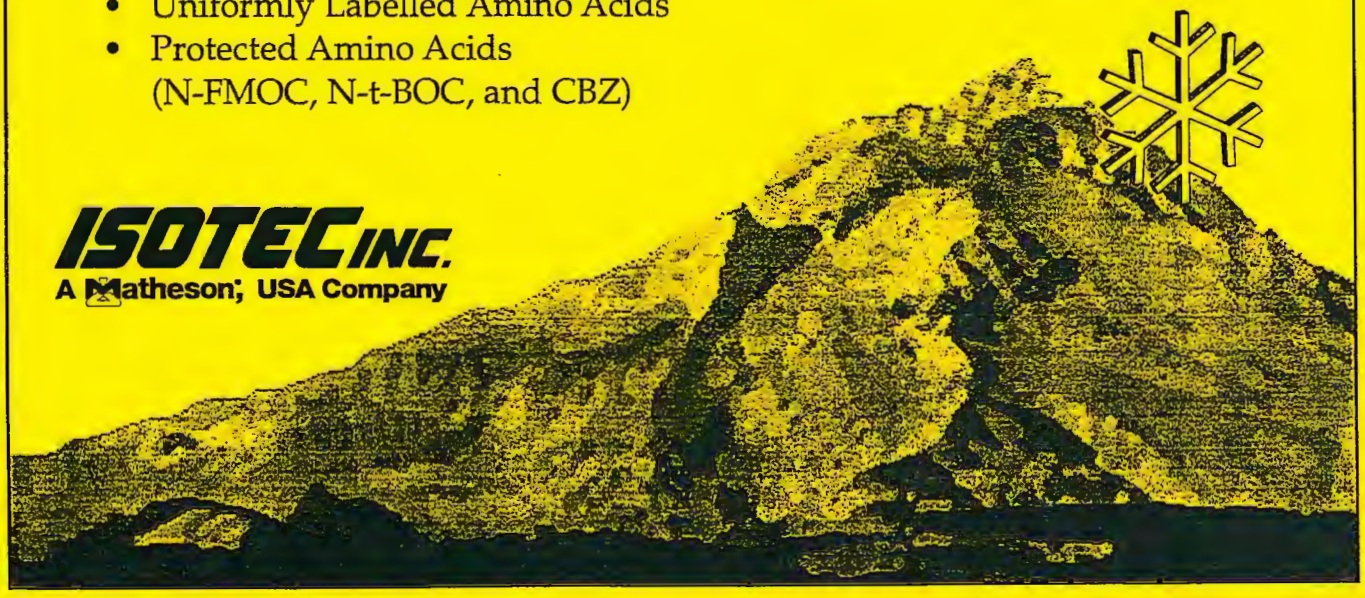

and...

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HERCULES

December 30, 1996
(received 1/2/97)

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

Dear Dr. Shapiro:

Tacticity of Polystyrene

Owing to its industrial importance, polystyrene has often been studied by NMR. Since tacticity is a major determinant of polymer properties, it has attracted a fair amount of attention[1-2]. Through the efforts of many research groups, a consensus has emerged on the ^{13}C NMR assignments of the aromatic C_1 carbon at the triad level and even for most of the pentads. However, there is a greater divergence of opinion on the assignments of the backbone methylene carbon. As for the other carbons (backbone methine and aromatic C_2, C_3, and C_4), complete ^{13}C assignments have not been made.

Recently, G. H. Lee (at Sun Refining and Manufacturing) and I reviewed and re-examined the NMR spectral assignments of polystyrene. We used 2D NMR, curve deconvolution, and spectral simulation approaches and obtained revised ^{13}C NMR assignments of the tacticity of the backbone methylene carbon. Furthermore, we provided tentative tacticity assignments for the backbone methine carbon.

For example, the deconvoluted spectrum and the assignments of the backbone CH_2 carbon are shown in Figure 1 and Table 1. The observed intensities agree well with a Bernoullian model. Further details on this work may be found in a recent publication[3].

Yours very truly,

H. N. Cheng

References:

1. ^{1}H NMR studies of polystyrene include: (a) H. J. Harwood, T. K. Chen, A. DasGupta, J. F. Kinstle, E. R. Santee, L. Shepherd, F.-T. Lin, *ACS Polym. Prepr.*, **26**(1), 39 (1985). (b) G. J. Ray, R. E. Pauls, J. J. Lewis, L. B. Rogers, *Makromol. Chem.*, **186**, 1135 (1985). (c) N. Ishihara, T. Seimiya, M. Kuramoto, M. Uoi, *Macromolecules*, **19**, 2465 (1986).
2. ^{13}C NMR studies of polystyrene include: (a) H. Sato, Y. Tanaka, *ACS Symp. Ser.*, **247**, 181 (1984). (b) H. J. Harwood, T. K. Chen, F.-T. Lin, *ACS Symp. Ser.*, **247**, 197 (1984). (c) A. E. Tonelli, *Macromolecules*, **16**, 604 (1983). (d) T. Kawamura, N. Toshima, K. Matsuzaki, *Macromol. Rapid Comm.*, **15**, 479 (1994). (e) D. R. Hensley, S. D. Goodrich, H. J. Harwood, P. L. Rinaldi, *Macromolecules*, **27**, 2351 (1994).
3. H. N. Cheng, G. H. Lee, *International Journal of Polymer Analysis and Characterization*, Vol. 2, pp. 439-455 (1996).

Table 1. Assignments of backbone CH_2 carbon of atactic polystyrene: first-order Markovian probabilities and the observed versus calculated intensities

No	^{13}C Shift	Assignment	Probability	Observed	Calculated (P_m = 0.48)
1	46.8	mrmrm	$P_{mr}^2P_{rm}^3$	3.5	3.0
2	46.5	rrmrm	$2P_{mr}^2P_{rm}^2P_{rr}$	7.1	6.5
3	46.2	rrmrr	$P_{mr}^2P_{rm}P_{rr}^2$	4.6	3.5
4	45.9	mrrrm	$P_{mr}P_{rm}^2P_{rr}^2$	4.9	3.2
5	45.4	mrrrr	$2P_{mr}P_{rm}P_{rr}^3$	6.6	7.0
6	45.2				
7	44.9	mrmmr	$2P_{mr}^2P_{rm}^2P_{mm}$		
8	44.8	r(mrmmm)x	$2P_{mr}^2P_{rm}^2P_{mm}^2$	11.6	11.5
	44.8	m(mrmmm)x	$2P_{mr}P_{rm}^2P_{mm}^3$		
9	44.7	rrrrr,rrmmm	$P_{mr}P_{rr}^4+2P_{mr}P_{rm}P_{mm}^2P_{rr}$		
	44.6	m(rmmrr)x	$2P_{mr}^2P_{rm}^2P_{mm}P_{rr}$	16.4	15.8
10	44.5	r(rmmrr)x	$2P_{mr}^2P_{rm}P_{mm}P_{rr}^2$		
11	44.3	rmmmr	$P_{mr}^2P_{rm}P_{mm}^2$	3.6	3.0
12	44.1	rmrrm	$2P_{mr}^2P_{rm}^2P_{rr}$	7.1	6.5
13	43.8	mmmmr	$2P_{mr}P_{rm}P_{mm}^3$	2.4	5.5
14	43.7	mmmmm	$P_{rm}P_{mm}^4$	2.2	2.5
15	43.5	rmrrr	$2P_{mr}^2P_{rm}P_{rr}^2$	11.7	13.0
16	43.4	mmrrm	$2P_{mr}P_{rm}^2P_{mm}P_{rr}$		
17	43.2	mmrrr	$2P_{mr}P_{rm}P_{mm}P_{rr}^2$	6.7	6.5
18	42.8	rmrmr	$P_{mr}^3P_{rm}^2$	2.0	3.2
19	42.7	rmrmm	$2P_{mr}^2P_{rm}^2P_{mm}$	5.9	6.0
20	42.5	mmrmm	$P_{mr}P_{rm}^2P_{mm}^2$	3.7	2.8

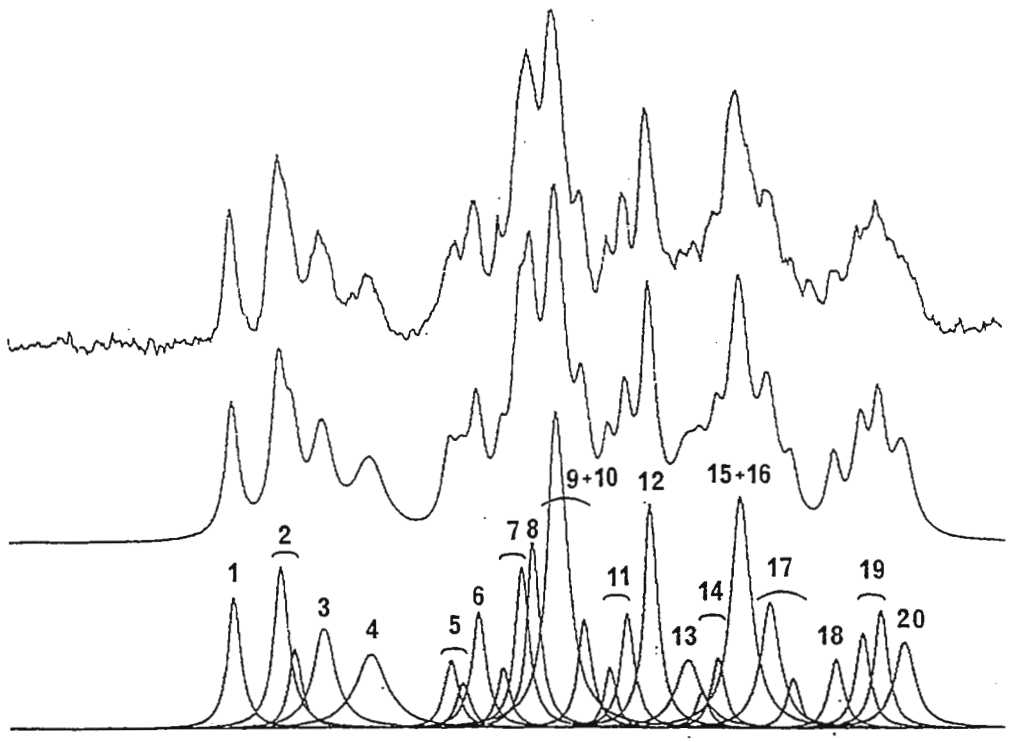
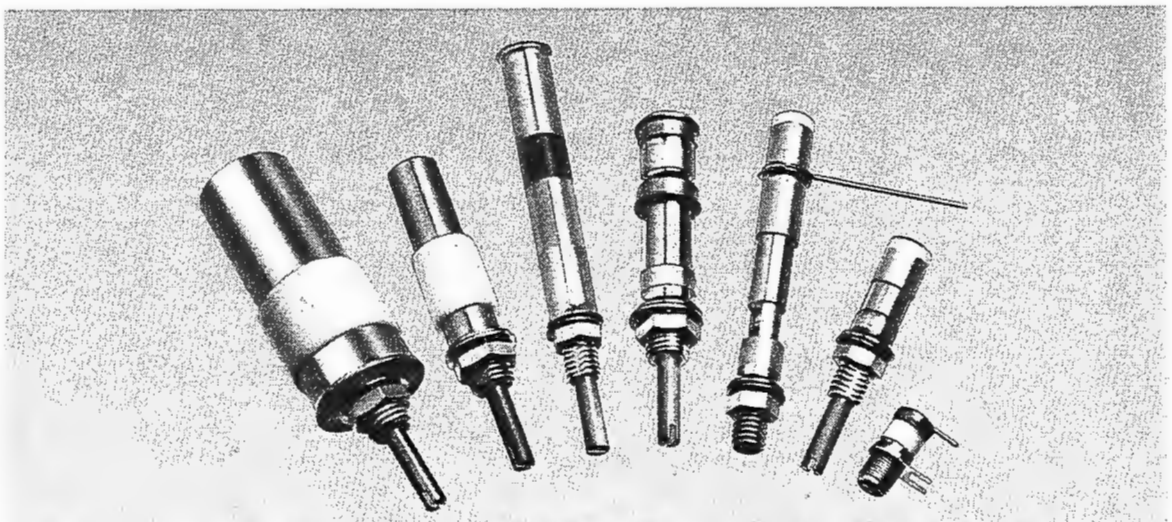


Figure 1. Curve deconvolution of the CH_2 carbon: top trace, observed spectrum; middle trace, fitted spectrum; lower trace, individual components.



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5th ANNUAL
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Omni Rosen Hotel, Ballroom D & E
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(located next to the Clarion Plaza Hotel, site of the 38th ENC)

Sunday, March 23, 1:15 to 6:00 p.m.

Agenda includes:

The Role of NMR in the Study of Drug Metabolism.
John Shockcor, John Lindon and Jeremy Nicholson, Glaxo Wellcome

Carbon 13, A Renaissance?
Andy Roberts, Duncan Farrant and Philip Sidebottom, Glaxo Wellcome

Liquid Phase Combinatorial Chemistry and Characterization of Intermediates by Routine Solution State NMR.
Ron Kim, Mahua Manna, Steve Hutchins and Kevin Chapman, Merck & Company

Experimental Aspects of Advanced Diffusion Measurements with PFG NMR.
Donghui Wu, Aidi Chem and Charles S. Johnson, Jr., University of North Carolina

Evaluation of HTS Probes and Preliminary Results for Biomacromolecular, Metabolite and Natural Product Compounds.
R. Andrew Byrd and Siddhartha Sarma, NCI-FCRDC
John Shockcor, Glaxo Wellcome
Gary Martin, Pharmacia & Upjohn, Inc.
Ron Crouch and Toby Zens, Nalorac Corporation

Structure Determination of Proteins in the 30 kD Range and New Methods for Determining Long Range Order.
G. Marius Clore, National Institutes of Health

High Field Offers More Than High Resolution and Sensitivity.
Ad Bax, National Institutes of Health

Quadruple Resonance Probes - New Tools for Biological NMR.
Arthur Pardi, University of Colorado
Gershon Wolfe and Brian Marsden, Nalorac Corporation

Advances in Solution State NMR Probe Performance.
Toby Zens and Gershon Wolfe, Nalorac Corporation

Application of $^{1}H/^{31}P/X$ Triple Resonance Experiments in Organometallic Chemistry.
Emilio Bunel, E.I. DuPont

$^{1}H/^{13}C/^{29}Si$ Triple Resonance Heteronuclear 3D NMR of Organosilicon Compounds at Natural Abundance.
Peter Rinaldi, University of Akron

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- CW mode
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- Thermal
- Blanking

Interface functions
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F= Flag output
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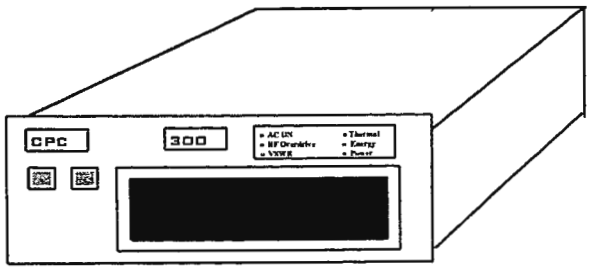
- VSWR (F)
- Energy (F)
- Power limit select Po or Po/2 (C/F)
- CW mode (C/F)
- Power Supply (F)
- Thermal (F)



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December 17, 1996
(received 12/24/96)

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

SCHOOL OF SCIENCE

NMR & x-ray Measurements of Adenosine Conformation: Position Available

IUPUI

Dear Barry,

Thank you for your reminder, I hope we meet the Dec. 20 deadline.

NMR and x-ray crystallography are clearly the two most extensively used techniques for macromolecular structural investigation. Our results on the adenosine conformations of a number of nucleotides bound to ATP-utilizing enzymes have led us to compare the results on glycosidic orientation obtained by the two techniques. The table (on the next page) lists these results for a group of these enzymes. The NMR results were obtained by us in recent years on the basis of TRNOESY measurements. The x-ray data given are from the Protein Data Bank (PDB). The interesting point is that the glycosidic orientations obtained by NMR fall in a narrow range of about ±8° around 51°. Note that the group of enzyme include those that catalyze phosphoryl transfer (kinases), adenylyl transfer (amino-acyl tRNA synthetase), and pyrophosphoryl transfer (PRPP synthetase). The rather narrow range for these orientations suggests a protein structural motif for the recognition and binding of the adenosine nucleotides. On the other hand, the orientations in the x-ray data vary all over the place. Since the x-ray data were obtained in the solid state in which the enzymatic reaction does not take place, it may well be that the substrate is trapped during the crystallization process in a conformation that may not be productive. In contrast, the NMR data are obtained in an active state of the complex, although the amino-acid environment cannot be easily investigated by NMR for some of these enzymes (with the exception of adenylate kinase). Thus, while the crystallographic data is the best source for the protein structure information, NMR results offer the best means to obtain substrate conformation in enzyme-bound reaction complexes. It makes sense to put the results together by appropriate modeling methods to gain insight into the mechanism. That is what we are planning to do.

With best regards,

Sincerely yours,

B. D. Nageswara Rao

NMR CENTER
DEPARTMENT OF PHYSICS
402 North Blackford Street
Indianapolis, Indiana
46202-3273

317-274-6900
Fax: 317-274-2393

NMR studied Complexes[£]	glycosidic angle (deg)	x-ray studied Complexes[£]	PDB entry id	glycosidic angle (deg)
Adenylate K•AMP[†]	43	Adenylate K•AP_5A (AMP site)	1ake	7-14
Adenylate K•MgATP[†]	44	Adenylate K•AP_5A (MgATP site)	1ake	99-107
Arginine K•MgATP[*]	50	3-Phospho-glycerate K •MgATP•3-PG	3pgk	- 8
Creatine K•MgATP[‡]	51	3-phospho-glycerate K •MgAMPPNP	1qpg	96
3-phospho-glycerate K •MgATP•3-PG[¥]	46	aspartyl-TRS•ATP	1asy	72
Pyruvate K•MgATP[§]	44	glutaminyl-TRS•ATP	1gtr	60
PRPPS[#]•MgATP[‖]	50	seryl-TRS•AMP•AHX	1ses	32(AMP)
Methionyl-TRS•MgATP[¶]	54	glutamine synthetase•Mn•AMP	1lgr	63
Free ATP[*]	5			

[£]. K represents kinase, TRS represents tRNA synthetase; [#]. PRPPS represents 5-phospho-α-D-ribose 1-diphosphate synthetase; [*]. Murali et al., 1994, Biochemistry, 33:14227-14236; [‡]. Murali et al., 1993, *ibid*, 32:12941-12948; [§]. Jarori et al., 1994, *ibid*, 33:6784-6791; [‖]. Jarori et al., 1995, Eur J. Biochemistry, 230:517-524; [¶]. Murali et al., submitted; [†]. Lin et al., unpublished results; [¥]. Murali et al., unpublished results.

Position Available: Application are invited for the position of a postdoctoral Research Associate on an NIH project entitled "Active-Site Structures of ATP-utilizing Enzymes". Experience in structural computations/molecular biology methods pertinent to NMR will be a real plus. Appointment will be initially for a year; extension is possible for several years on the basis of progress and mutual satisfaction. Interested applicants may send their *c.v.* and at least two letters of recommendation to the above address (on the previous page). IUPUI is an equal opportunity/affirmative action employer. For further information contact by email: brao@iupui.edu, or phone: (317)274-6901.

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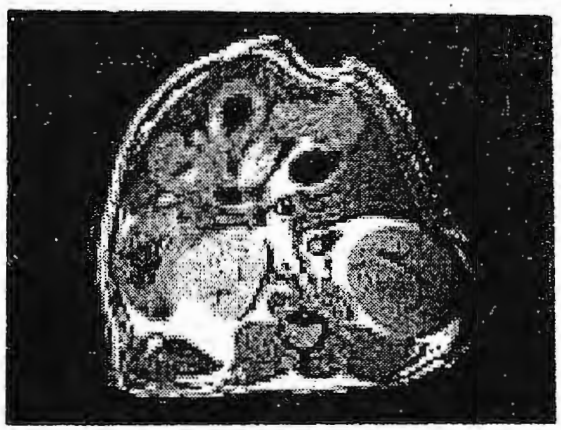
The live mouse image, to the right, was obtained with a Doty micro-imaging probe furnished with a 41 mm *rf* volume coil and 50-72 gradients in a wide bore 500 MHz Varian Unity+.



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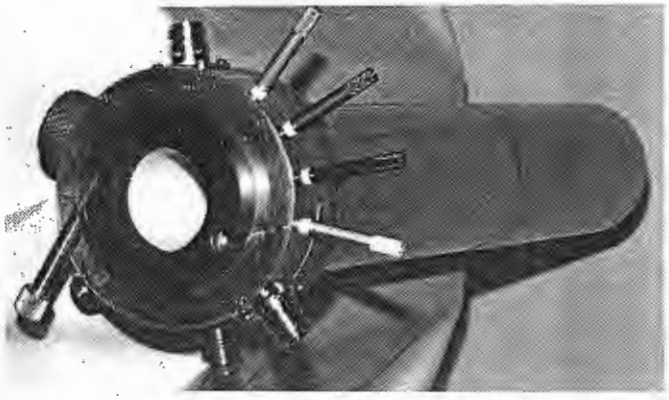

40 mm Clear Bore $^1H/^{19}F$ imaging probe for WB

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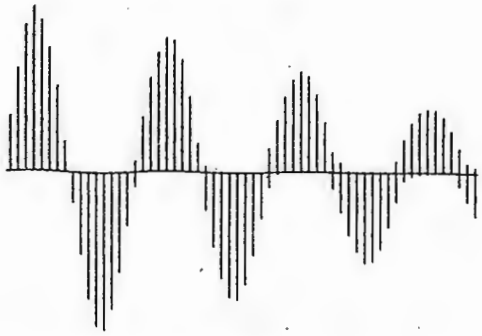
Model 85-120 gradient coils
14 G/cm 25% duty cycle, air cooled

This model is ideal for rat research in the new 150-mm vertical-bore 300 MHz magnets. It uses interchangeable rf modules (up to 60-mm ^1H-X), allowing the widest range of multi-nuclear applications from 3 to 60 mm. The extra space in these coils allows a substantial increase in sensitivity for samples larger than 24 mm.



B1 homogeneity for 40-mm MRI. Above: Pulse-width array in 300-MHz 89-mm vertical-bore magnet with 50-mm 120 G/cm gradients, 41-mm rf coil diameter, 36-mm diameter x 25-mm length water phantom.

Doty Scientific, Inc. 700 Clemson Road, Columbia, SC 29229 USA
Phone (803) 788-6497 Fax: (803) 736-5495 Email: sales@doty.com.usa

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December 18, 1996
(received 12/27/96)

Silent Field Mapping

Dear Barry,

Greetings from the frozen prairie!

Shimming used to be an art, and many a spectroscopist was understandably proud of being able to beat any machine at getting a good lineshape. Lately, though, the machines are doing a lot better. This is largely due to use of various field mapping techniques. Rather than using a single response (lock level or FID area) to characterize the field, the map uses many simultaneous responses to simultaneously (and hopefully independently) determine all of the shim currents.

Those of you who have heard the high-tech field mapping robots whirring through their routine, or listened to the ticking (or pounding, depending on magnet size) of a modern PFG system imaging the field, may be interested to know that the same thing can be done on an older spectrometer, without making any noise at all, and indeed without investing in much new hardware (more about that later).

"Silent field mapping" is based on projection-reconstruction spectroscopic imaging (PRSI). Like its cousin, pulsed-gradient, phase-encoded, fourier spectroscopic imaging (FSI) (employed in one form or another in most of the recently published methods), PRSI generates an (N+1)-dimensional data set: N spatial dimensions, and one spectral (frequency) dimension. (1) Unlike its cousin, PRSI uses static gradients, not pulsed ones, and therefore doesn't make any noise. You could even do this by hand on a CW instrument, if you had enough patience. [A variation uses a quasi-static gradient to produce a phase map (2) which is, of course, proportional to the field/frequency map, but is complicated by "phase wrapping" effects and multiple peaks.]

We decided to try resurrecting this technique for use with large sample tubes (20mm) on our GN-300. This instrument has computer-controlled shim coils. We measured gradient strengths of: Z, 105 Hz/mm; Y, 24 Hz/mm; X, 23 Hz/mm, corresponding to the maximum DAC values available, so it seemed to be possible to map a small range of frequency shifts in 3 dimensions, and a larger range in 1 dimension (Z). Settling times of the shim currents are on the order of a second for large changes, less for small ones.

The procedure (1a) involves selecting a set of projection angles and computing the required gradients. The spectral dimension involves a pseudo-angle, and a change in the spectral width which must also be calculated for each projection. These calculations, and the automation of the procedure, are carried out on our system by an NMRScript™ controlling a Tecmag Libra system (the aforementioned new hardware) which replaced the venerable 1280 computer a few years ago.

After the data are acquired, the spectroscopic image is reconstructed using *viewit* (3), our all-purpose software package (available in the public domain). Peak-picking in the spectral dimension produces the N-dimensional field map. With this technique in hand, one can map each shim coil in the usual way, and then fit an arbitrary field to a sum of coil fields using least squares.

So far, we have produced 1D and 2D field maps, and we are getting our courage up to go all the way to 3D after the first of the year. Some 1D examples are shown to illustrate the principle.

May all your fields be homogeneous,

Carl D. Gregory, Clint S. Potter, H. Douglas Morris, and Paul C. Lauterbur*

(*) to whose account this should be credited.

1. (a) M.L.Bernardo, Jr., P.C.Lauterbur, K.L.Hedges, "Experimental Example of NMR Spectroscopic Imaging by Projection Reconstruction Involving an Intrinsic Frequency Dimension", J. Magn. Reson., 61, 168 (1985). (b) P.C.Lauterbur, D.N.Levin, R.B.Marr, "Theory and Simulation of NMR Spectroscopic Imaging and Field Plotting by Projection Reconstruction Involving an Intrinsic Frequency Dimension", J. Magn. Reson., 59, 536 (1984).
2. S.Sukumar, M. O'Neil-Johnson, J.A.B.Lohman, "An Image Based Autoshimming Procedure Using Unshielded Gradient Systems", 37th ENC, Abstract WP 206 (1996)
3. http://kepler.ncsa.uiuc.edu/viewit.html; C.S. Potter and P.J. Moran, "Viewit: A Software System for Multi-Dimensional Biomedical Image Processing, Analysis, and Visualization", SPIE Conference on Biomedical Image Processing III and Three-Dimensional Microscopy, 1660, 767-773, (February 1992)

(Thanks to: National Institutes of Health-Center for Research Resources; National Center for Supercomputing Applications; and Illinois Department of Commerce and Community Affairs)

Figure 1: 1D spectroscopic image (along the Z axis) of a layer of toluene (two peaks) floating on a layer of water (1 peak). 32 projections reconstructed to 128x128 image. In addition to the spectra of the two layers, one can see some bending of the field, especially at the bottom of the tube. A: sample geometry; B: spectroscopic image (note ray artifacts from limited sampling); C: spectra of individual layers from SI experiment compared with conventional 1D spectrum; D: stacked plot of central region of SI image.

Figure 2: 1D spectroscopic images of a tube of water showing field along the Z axis: A: properly shimmed; B: Z1 error (deliberate misadjustment of Z1); C: Z2 error; D: Z3 error; E: Z4 error; F: Z5 error (note additional transmit frequency offset).

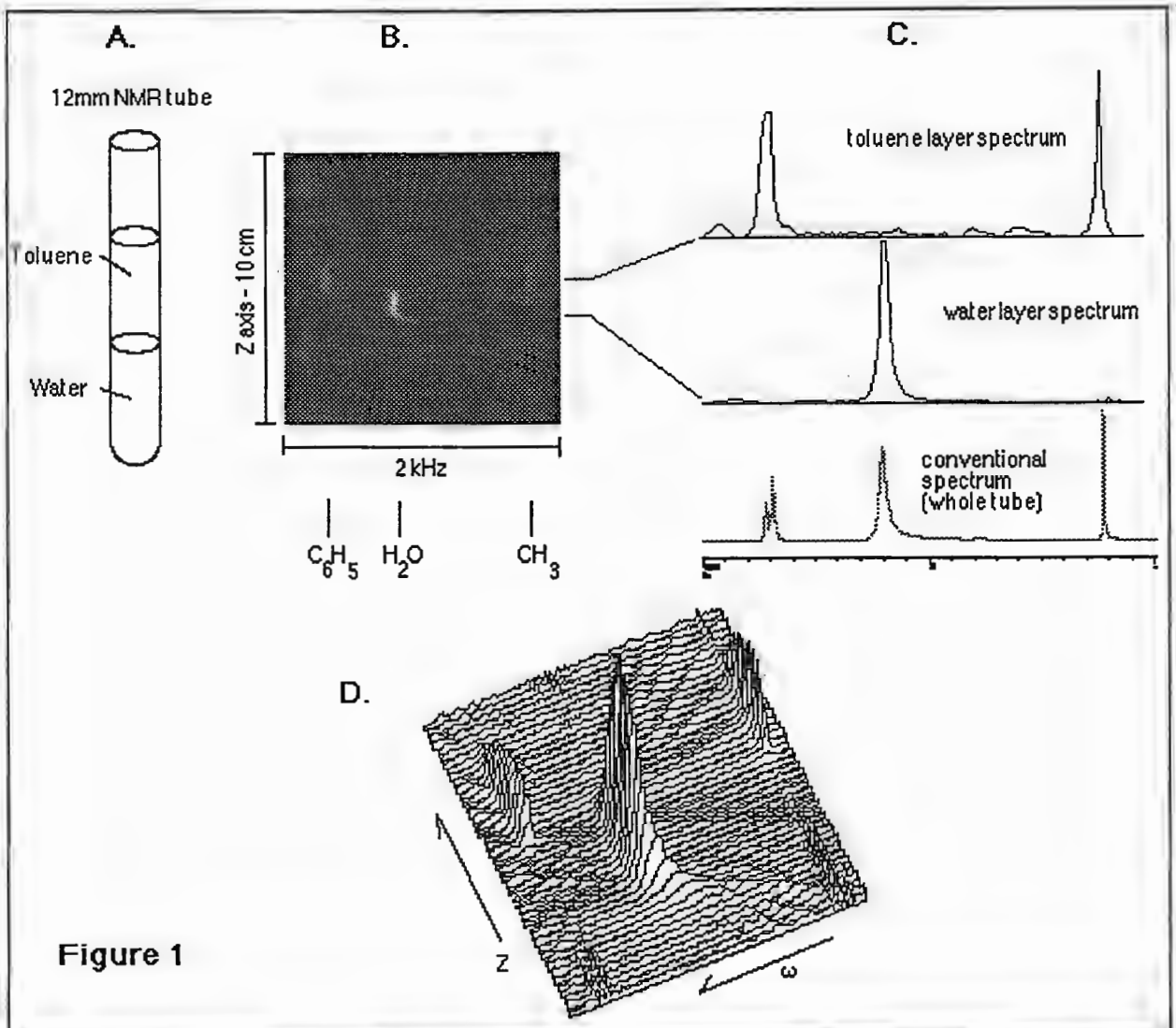

Figure 1

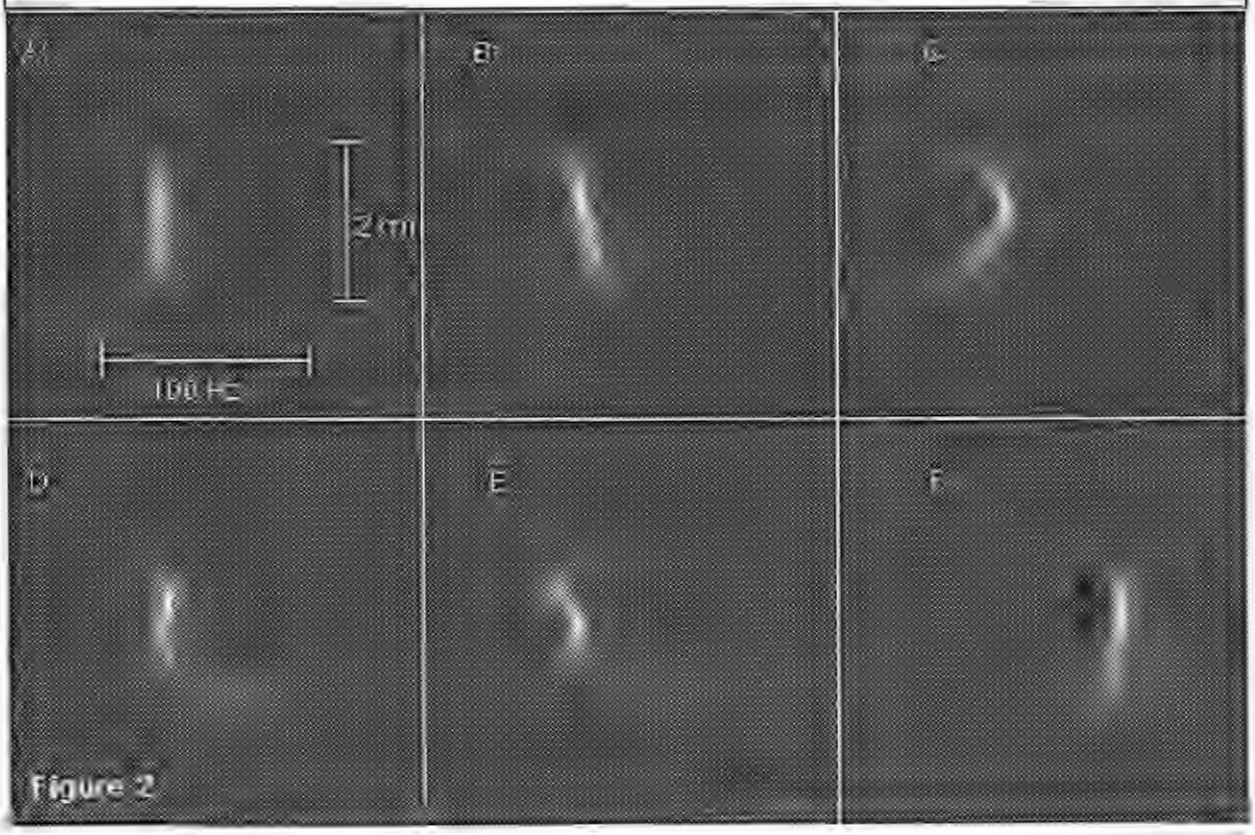

Figure 2

461-40

Brandeis University

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December 16, 1996
(received 12/27/96)

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

Title: **Saved by the Flip-Back and a Dangerous Computer Virus**

Dear Barry;

I have been meaning to write up a short note about how much difference the use of water flip-back pulses has made in our NMR analysis of a gallium-reconstituted ferredoxin. I sat down to the computer and wrote an absolutely stunning little vignette describing our work for the NMR Newsletter. Little did I know that my Macintosh's hard drive had been infected by the deadly virus BAD POETRY, which I had inadvertently down-loaded with a mail message warning me against good times. This truly hideous piece of code converted my high-minded (nay, deathless) scientific prose to a mishmash of mindless drivel, as you can see. I could rewrite the whole thing for you, but I would rather drink beer and watch *Babylon 5* reruns, so I will just send along what the cruel software demons left me, to wit:

We have a redoxin (two Fe, two S)
and because of line broadening, we have to guess
what the metal site looks like.[1]
Oy, vey! What a mess.[2]

Until young Sophia looked up and said "Gee...
if we replace it with gallium, then we can see
all the cross-peaks and
then I can get my degree!"

She worked very hard, and the story's been told[3]
how the new ferredoxin proceeded to fold.
but as for rejoicing,
we put it on hold.

Our NOESYs looked empty, our inverses bare;
of problems it seems we had more than our share;
for despite our best efforts and all of our care,
Not all of the peaks we expected were there.

RF we'd been using to flatten the water
Our signal-to-noise spin diffusion did slaughter.
And then to our rescue came Grzesiek and Bax
I forget in which journal (I think it was JACS).[4]

They spoke of the flip-back and WATERGATE[5] too,
so I asked for some help from my loving spouse Sue.
She took us to Bruker (U.S., not AG),
and ran the pulse programs in order to see
if it made any change in our HSQC.

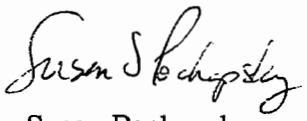
Oh wonder of wonders, the sight to behold
of the many new cross peaks
our new spectra showed.
They liked it at Bruker and chose this to be
their Web Page display for the next year or three.

(if you want to look at it type http:
//bruker.com and there it will be).
We quickly adapted these pulse schemes brand new
into many a pulprog, ZG and AU.

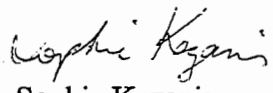
Now this work is finished and we are content,
and manuscripts off to the journals we sent.
Please count this towards Brandeis's debt to your book,
and I hope it lets Redfield off of the hook.

1) Pochapsky et al., *Biochemistry 33*, 6424 (1994).
2) A background in Yiddish idiom is not a requirement at Brandeis, but it can't hurt.
3) Kazanis et al., *J. Am. Chem. Soc. 117*, 6625 (1995).
4) It was. Grzesiek and Bax, *J. Am. Chem. Soc. 115*, 12593 (1993).
5) Piotto et al., *J. Biomol. NMR 2*, 661 (1992).

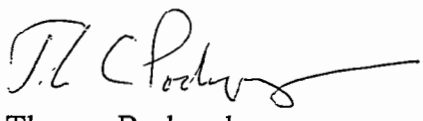
Sincerely,



Susan Pochapsky
Bruker Instruments, Inc.
Billerica, MA



Sophia Kazanis
Brandeis University
Waltham, MA



Thomas Pochapsky
Brandeis University
Waltham, MA

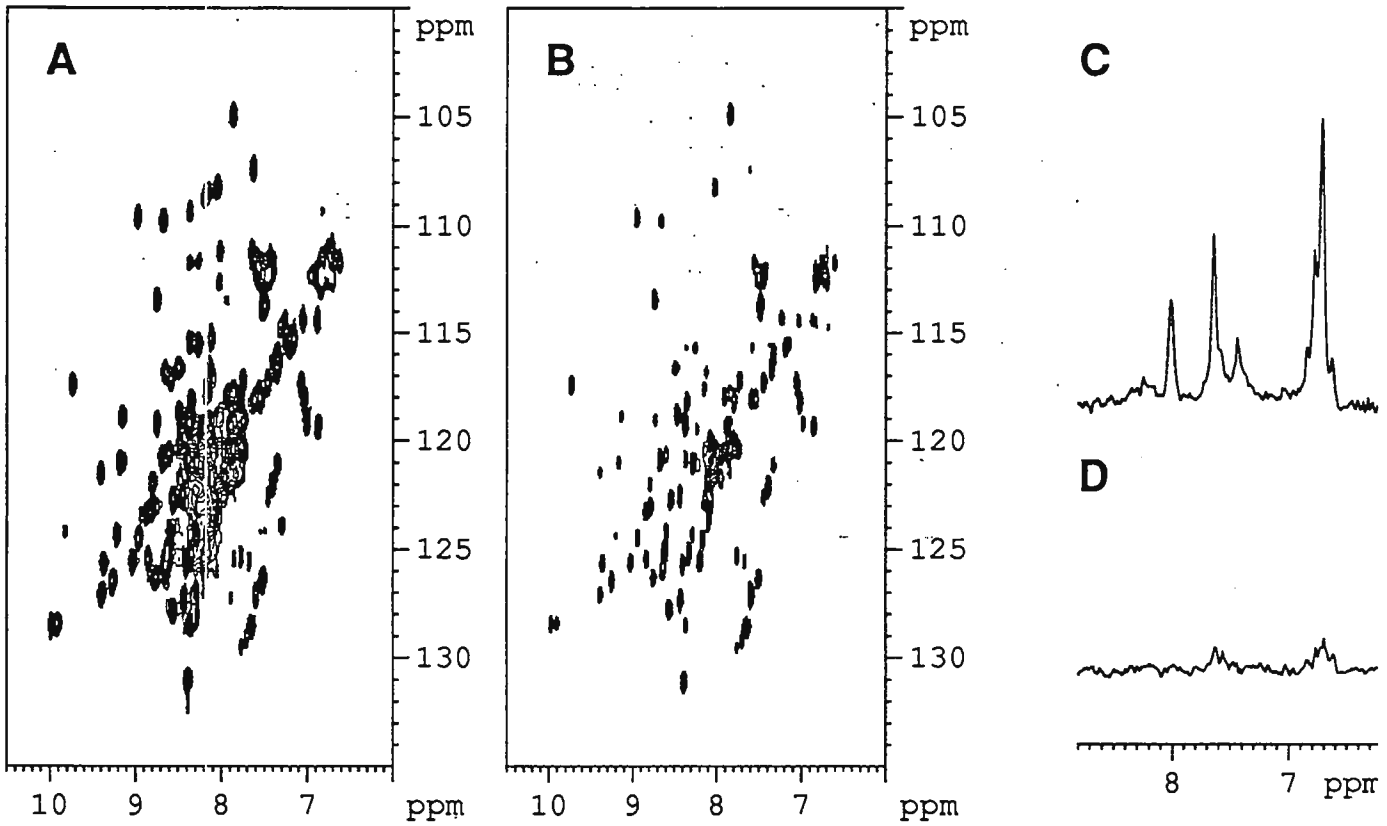


Figure 1: Comparison of Gallium Ferredoxin spectra. (A) HSQC with WATERGATE and water flip–back pulse. (B) HSQC with presaturation. (C) Row from flip–back HSQC. (D) Row from presat HSQC.

Aksel A Bothner-By
6317 Darlington Rd.
Pittsburgh, PA 15217

8 Jan 1997

(received 1/13/97)

Dr. B. L. Shapiro,
966 Elsinore Ct.
Palo Alto, CA 94303

COSINE DIVISION

Dear Barry:

Stupid error in my letter to you of 7 December 1996 reproduced in Newsletter No. 460. Everywhere in letter, read πJt for $\pi Jt/2$. Sorry about that.

Sincerely
Aksel

Questions? Call me at 412 521 6734, or email ab6d@andrew.cmu.edu.

Address all Newsletter correspondence to:

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

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

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No. 466 (July)	27 June 1997

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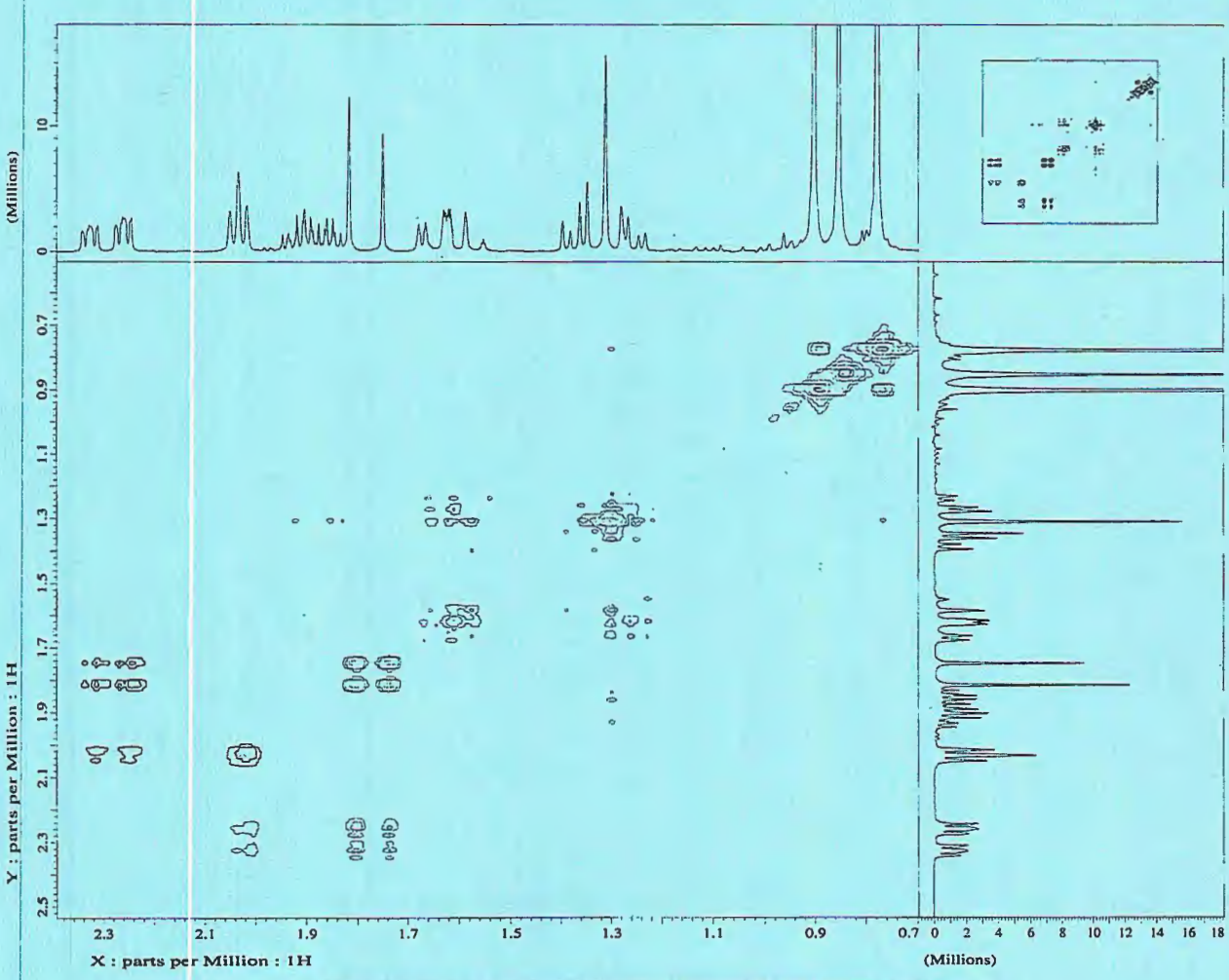

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