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FORTHCOMING NMR MEETINGS

- Workshop "NMR and Structure of Biomolecules", March 4-9, 1990; Gainesville, Florida; Contact: W. S. Brey, University of Florida, - see Newsletter 375, 68.
- Workshop on In Vivo Magnetic Resonance Spectroscopy III, March 29 - April 1, 1990, San Francisco, California; Contact: Dr. M. W. Weiner or Dr. G. B. Matson, Magnetic Resonance Unit, Veterans Administration Medical Center, 4150 Clement Street (11D), San Francisco, CA 94121; (415) 750-2146.
- 31st ENC (Experimental NMR Conference), April 1-5, 1990; Asilomar Conference Center, Pacific Grove, California; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667; Attendance: 1,200.
- Frontiers of Polymer Characterization by NMR Spectroscopy, Symposium at the American Chemical Society National Meeting, April 22-27, 1990, Boston, Mass.; See Newsletter 372, 54.
- 10th European Experimental NMR Conference, May 28 - June 1, 1990; Veldhoven, The Netherlands. Contact: M. J. A. de Bie: see Newsletter 376, 26.
- Workshop of Special Topics in Medical Magnetic Resonance, sponsored by the Society of Magnetic Resonance in Medicine and the National Research Council of Canada, July 23-27, 1990; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.
- Expanding Frontiers in Polypeptide and Protein Structural Research, sponsored by the National Research Council of Canada, July 23-27, 1990; Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.
- Tenth International Biophysics Conference, sponsored by the International Union of Pure and Applied Biophysics and the National Research Council of Canada, July 29 - August 3, 1990; Vancouver, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Additional listings of meetings, etc., are invited.

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. 378 (March)----- 16 February 1990
No. 379 (April) ----- 16 March 1990
No. 380 (May)----- 20 April 1990
No. 381 (June)----- 18 May 1990

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(708) 972-4470November 16, 1989
(received 11/20/89)

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

RE: Availability of Tetrakis(trimethylsilyl)silane (TKS)

Dear Barry:

Last year we published a paper (1) on the use of tetrakis(trimethylsilyl)silane (TKS) as a suitable standard for solid-state ^1H , ^{13}C , and ^{29}Si NMR spectroscopy. This material exhibited narrow, distinct ^1H or ^{13}C resonances whose chemical shifts were independent of magnetic field, and possessed the desirable physical attributes and magnetic relaxation properties for its convenient use as a reference standard.

Since the time of publication, I have had numerous inquiries as to the availability of TKS. I would like to inform your readership that TKS is now available from Aldrich Chemical Co. at a reasonable price (Catalog No. 32,992-4; 1g, \$10.00; 10g, \$62.00).

Sincerely,



Robert E. Botto
Chemistry Division

1. J. V. Muntean, L. M. Stock, and R. E. Botto, J. Magn. Reson., 76, 540-542 (1988).



North Carolina State University

Department of Chemistry
College of Physical and Mathematical Sciences

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

December 6, 1989
(received 12/8/89)

Dr. Shapiro:

We wish to remind the users of ^{15}N probes that the most efficient method of initial testing is with the DEPT polarization transfer experiment (1, 2), conveniently on formamide. The traditional testing procedure has been to acquire with the proton decoupler on during acquisition, using an experiment such as 1PDNA on the General Electric OmegaTM system. However, the DEPT experiment can be set up fairly simply. The broad range of excitation precludes the requirement that the pulse width be set precisely. Thus very good enhancement is observed with $\theta = 135^\circ$, 90° , or 45° , with 45° giving the best results.

Figure 1 shows a spectrum of formamide in DMSO collected at 50.68MHz on our General Electric OmegaTM 500 using the pulse sequence 1PDNA. Four scans were collected with a 45 second relaxation delay. Figure 2 is a spectrum of the same sample taken with the DEPT experiment, $\theta = 45^\circ$. The same number of scans were taken, but the relaxation delay was only 2.5 seconds. Using the same number of acquisitions a large improvement in signal-to-noise ratio is observed with a concomitant decrease in collection time.

We would also like to describe an apparatus to aid in low-temperature work. On the OmegaTM system VT air is routed through metal cooling coils before reaching the probe. The coils are set into a dewar filled with some cooling medium--water, dry ice, or liquid nitrogen depending upon the minimum temperature desired (Figure 3). When working below room temperature it is necessary to switch the gas supply to nitrogen to prevent the moisture in the air supply from freezing out into the coils. While the start-up is straight-forward care is required in returning the system to room temperature. When the coils have been immersed in liquid nitrogen for an hour or so the glass connector to the probe becomes frozen, brittle and susceptible to breakage. Thus it is necessary to avoid moving the coils until the liquid nitrogen has evaporated. This means that the nitrogen gas must also be left on. It may be a matter of hours before a dewar which is even half full of liquid nitrogen evaporates. To avoid this waste of time and nitrogen gas, we modified the configuration of the heat-exchange system to make it possible to by-pass the coils (Figure 4). Then at the end of a low-temperature experiment the VT control is slowly ($\sim 5^\circ/5$ minute) raised to room temperature, and switch A is turned to by-pass the coils. Then the gas supply can be changed back to air safely, the VT turned off and the sample ejected.

Please apply this contribution to Ed Stejskal's subscription.

Charles Moreland

Billy Roberts

Hanna Gracz

Wanda Smith

REFERENCES

1. Morris, G, Freeman, R., J. Amer. Chem. Soc. (1979) 101: 760-762.
2. Schenker, K.V. and von Philipsborn, W., J. Magn. Res. (1985) 61: 294-305.

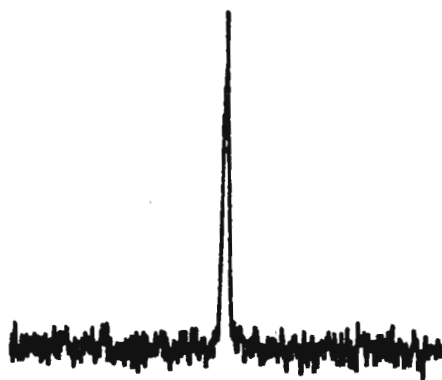


Figure 1



Figure 2

Figures 1,2: ^{15}N NMR of formamide in DMSO- d_6 . Fig. 1: 1PDNA, $70\ \mu\text{s}$ 90° pulse, 45 sec relaxation delay. Fig. 2: $70\ \mu\text{s}$ 90° pulse on transmitter, $91\ \mu\text{s}$ 90° pulse on decoupler, 2.6 sec relaxation delay.

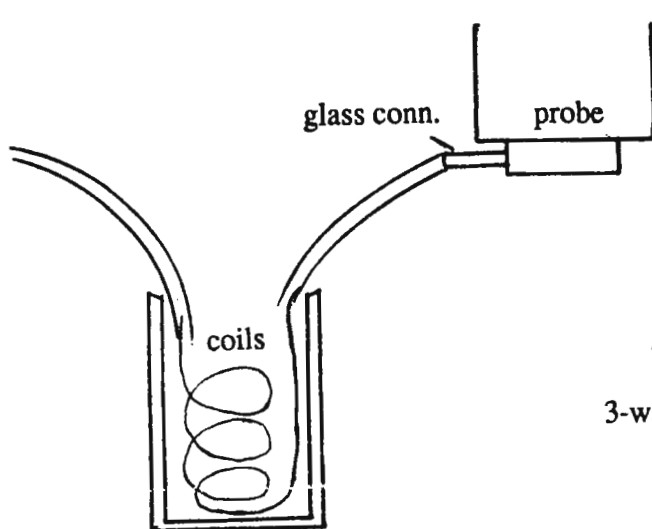


Figure 3

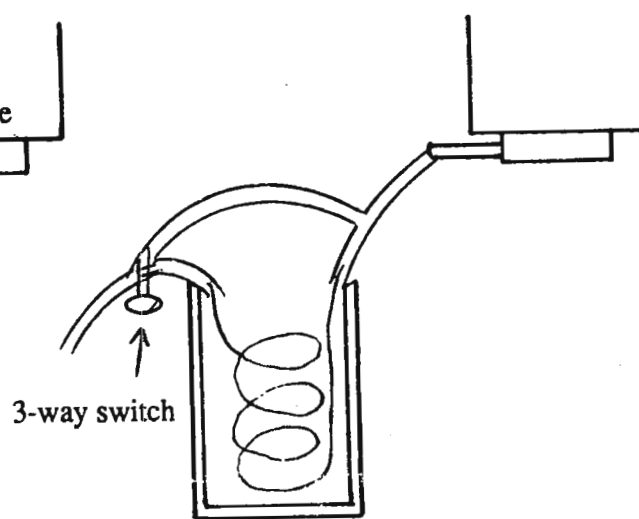


Figure 4

Figures 3,4: See description in text.

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November 22, 1989 (received 11/30/89)

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

Subject: ^{19}F In Vivo NMR of Psychiatric Drugs

Dear Barry:

The clinical efficacy of antipsychotic drugs is well established. However, there is a great deal of individual variation in clinical response in psychiatric patients given similar dosages. This suggests that differences in pharmacokinetics may account for different therapeutic effects. Hence a method to monitor tissue levels of drugs in vivo would be valuable. Fluorine-19 NMR is being used increasingly to follow the fate of fluorinated drugs in vivo in both animals and humans.

In collaboration with Joe Newton, M.D. and Craig Karson, M.D. of the Little Rock VA Medical Center and Dave Cardwell, Jay Sprigg and P. Mohanakrishnan of the University of Arkansas for Medical Sciences, we have observed the ^{19}F signal from the neuroleptic drug trifluoperazine (TFP) in the brain and calf muscle of a human. Spectra were acquired at 60.1 MHz on the General Electric Signa System equipped with a spectroscopy research package. A home-built single loop surface coil was used for excitation and detection. Quantitation was made relative to a phantom containing 0.055 mM trifluoperazine in 2% agarose. For internal calibration, a small vial of 2,2,2-trifluoroethyl-p-toluene sulfonate (TFEPTS) was incorporated into the coil assembly. The figure shows the structure of TFP and the spectra taken from both the front and rear of the head. The signal on the right is the internal standard, while the signal on the left is due to in vivo trifluoperazine and structurally similar metabolites. The bottom spectrum, which was obtained from the front of the head of a patient on 120 mg/day oral dose, comes from trifluoperazine primarily in brain. The much stronger signal from the back of the head probably comes from brain, muscle and fat in that region. By comparison to the TFP phantom, we estimate the concentration of TFP and its fluorinated metabolites in brain to be in the range 0.024 to 0.030 mM. This is similar to values determined in animals by ^{19}F NMR and by chromatography. We have

also detected the ^{19}F signal of the antidepressant drug fluoxetine.

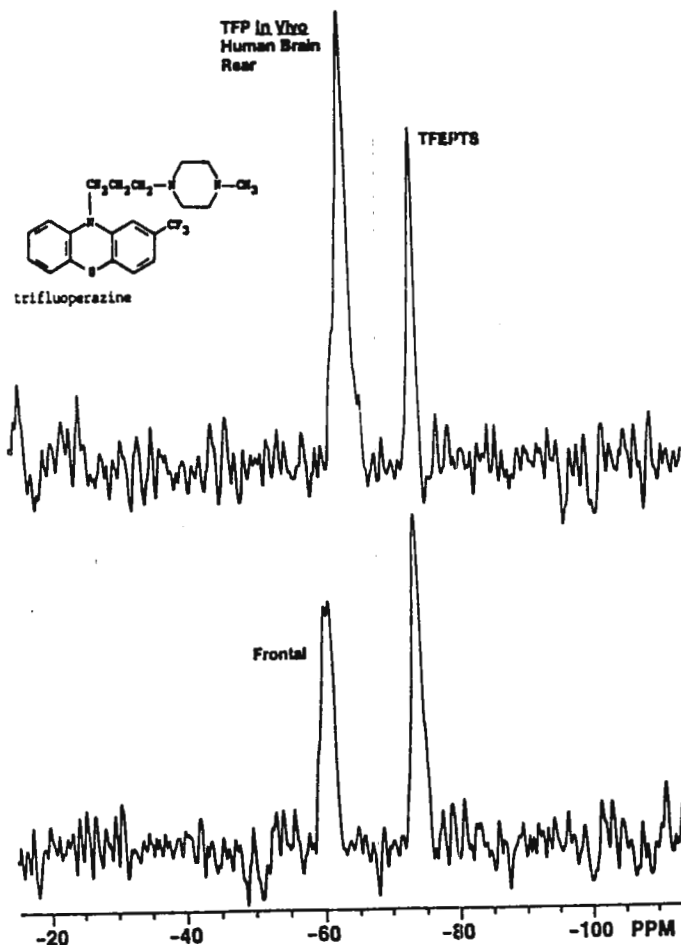
Our results suggest that in vivo ^{19}F NMR maybe a viable method for following the level of psychiatric drugs in human brain. Sensitivity improvements are necessary to permit monitoring at more typical therapeutic doses.

Sincerely,



Richard A. Komoroski
Associate Professor of
Radiology and Pathology

RAK/ms





Abbott Laboratories
One Abbott Park Road
Abbott Park, Illinois 60064-3500

November 14, 1989 (received 11/22/89)

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

RE: ^{13}C -Resolved 3D NMR of Proteins

Dear Dr. Shapiro:

Due to the low cost and ease of ^{15}N -labeling, most of the applications of heteronuclear three-dimensional NMR spectroscopy have involved proteins uniformly labeled with ^{15}N . Using these 3D techniques homonuclear COSY, TOCSY and NOESY spectra can be markedly simplified by editing with respect to the ^{15}N -chemical shifts in a third dimension (1,2). Even with the enhanced resolution offered by the ^{15}N -resolved 3D NMR experiments, however, it is difficult to assign the proton NMR signals in many cases because of the limited number of proton-proton scalar correlations that can be obtained. This is due to the rapid decay of proton magnetization during the time required for coherence transfers involving small proton-proton J-couplings. In addition, from the ^{15}N -resolved 3D data, it is difficult to identify long-range NOEs in the crowded aliphatic part of the NOESY spectra, important for determining the tertiary structures of proteins.

^{13}C -resolved 3D NMR experiments involving ^{13}C -labeled proteins can help solve some of the problems described above. For example, ^{13}C -resolved 3D NMR experiments have recently been described which rely on coherence transfer between ^{13}C nuclei by a ^{13}C COSY (3) or TOCSY (4) type mechanism. Using these techniques the aliphatic protons and carbons can be assigned by amino acid type. ^{13}C -resolved 3D NOESY experiments have also been described (5,6) which are extremely useful for identifying NOEs in the crowded aliphatic part of the NMR spectrum. Figure 1 illustrates the utility of these two complementary 3D NMR techniques.

Sincerely,


Hugh Eaton, Stephen Fesik, Robert Gampe, Ed Olejniczak, Andrew Petros,
Erik Zuiderweg

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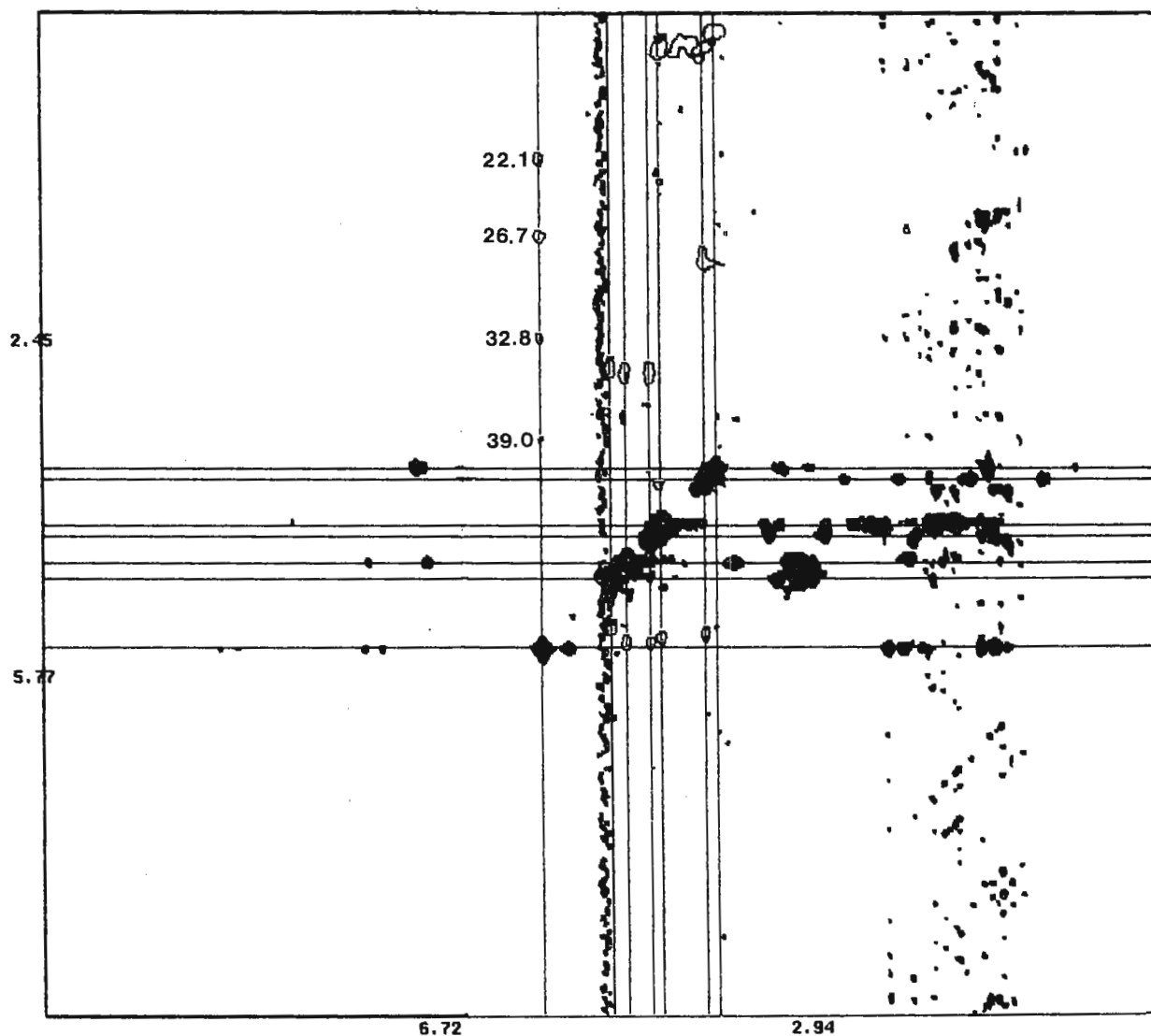
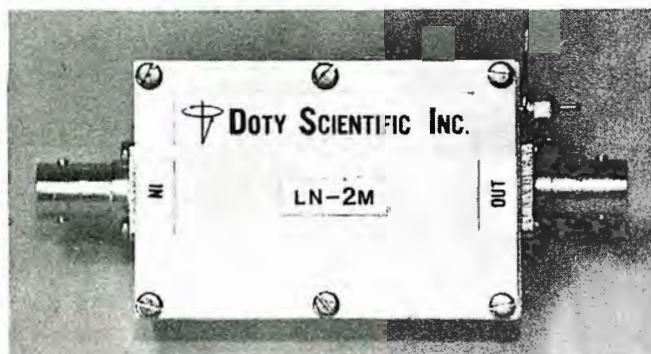


Figure 1. ω_1, ω_3 plane extracted from a 3D [^{13}C - ^{13}C - ^1H] TOCSY-Reverse INEPT spectrum (one contour level) at 50.2 ppm superimposed on the corresponding plane obtained from the 3D [^{13}C - ^1H - ^1H] NOESY spectrum of [^{13}C , ^{15}N] T4 lysozyme. The ^{13}C TOCSY experiment can be used to identify the type of amino acid residues from the characteristic ^{13}C chemical shifts (vertical lines), and the ^{13}C -resolved NOESY experiment is used to identify neighboring protons (horizontal lines).

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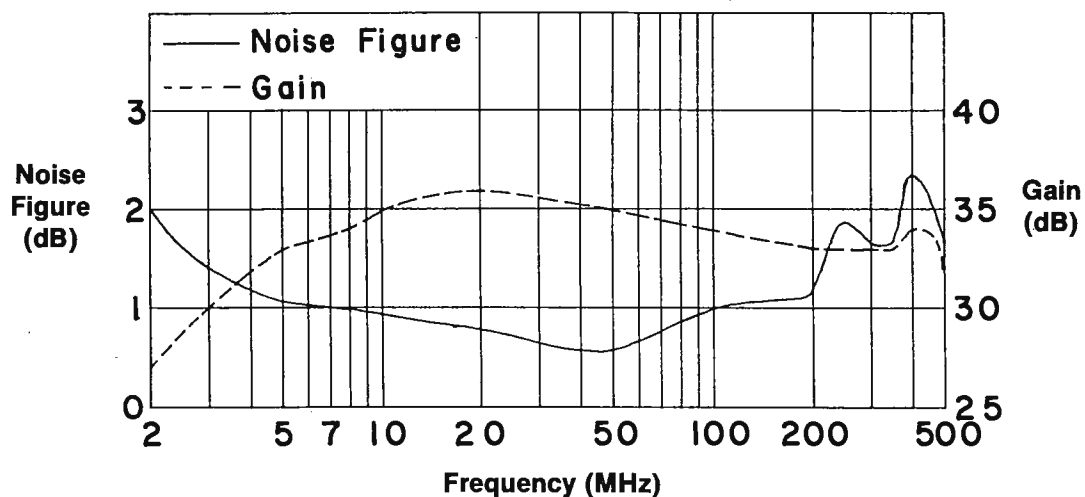
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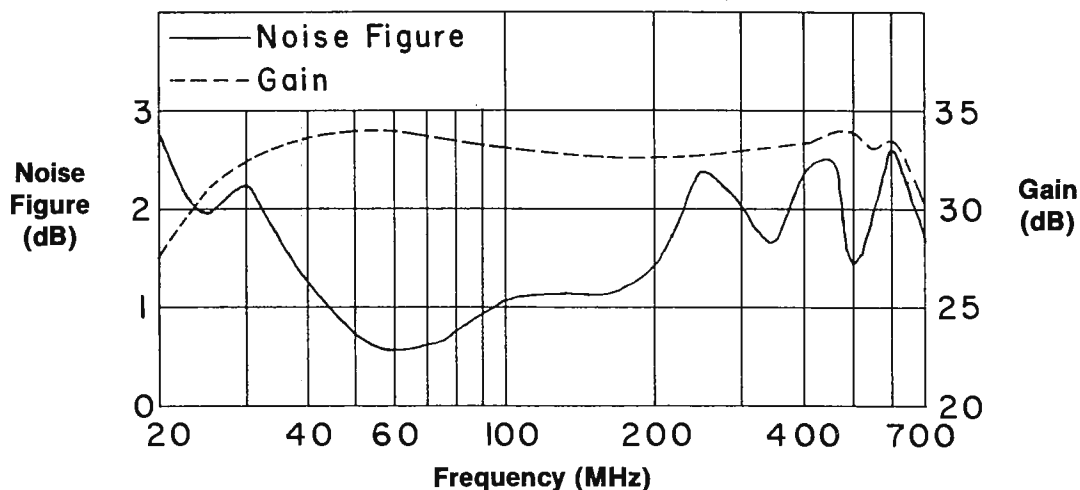
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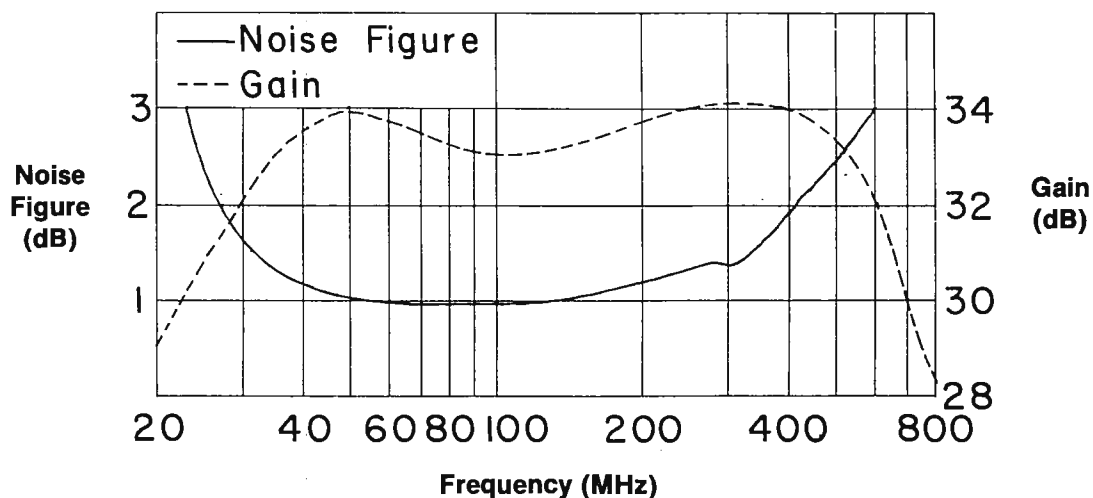
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Fax Number: (+15) 952-2714Dr. B. L. Shapiro
TAMU NMR NEWSLETTER
966 Elsinore Court,
Palo Alto, Calif., 94303Nov 20th, 1989
(received 11/21/89)

RE: Separation of perfusion and diffusion in the brain of a rat by MRI

Dear Dr. Shapiro:

Perfusion (microcirculation) of liquids and diffusion of molecules in biological tissues are of interest due to the fact that both phenomena carry different useful information. Diffusion⁽¹⁾ can provide information about restricted motions whereas perfusion represents the amount of liquid flow per unit volume. Due to the random character of the distribution of capillary vessels responsible for perfusion, the main effect on the MRI signal is to attenuate it in a way similar to diffusion. Using the fact that diffusion effects are not reversible but that perfusion ones are for times shorter than 100ms, we have implemented a set of imaging sequences to separate these two phenomena in the brain of a normal rat at 5T.

The attenuation of the MRI signal is modeled by the following expression:

$$e^{-k D [1-f + F f]}$$

where $k \propto (\text{gradient})^2$; f is the perfusion tissue ratio; D is the true diffusion coefficient and F depends on the imaging sequence. F is 1 when perfusing spins are rephased at the peak of the spin echo. By manipulating the value of F and the gradients one can obtain pure "f" and "D" images. Fig.1 shows a transaxial view of the brain of a normal rat acquired with resolution 0.6 mmX0.6 mmX4 mm and TE = 85 ms/ TR = 3.33 s. Fig. 2 shows a diffusion image with average $D = (0.48 \pm 0.05) \times 10^{-5} \text{ cm}^2/\text{s}$ and Fig. 3 shows a perfusion image with average $f = (3 \pm 1) \%$.

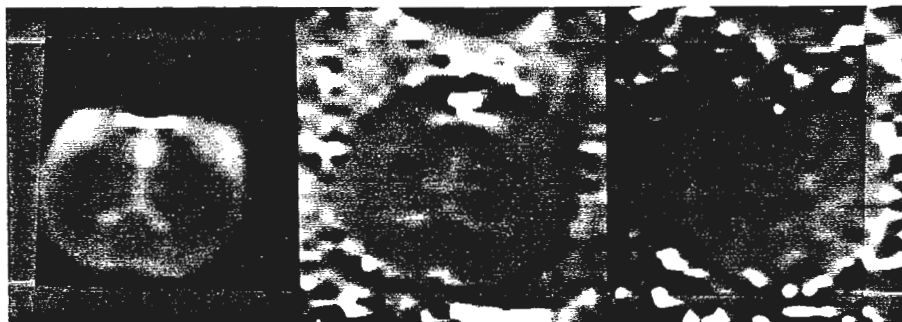



Fig. 1

Fig. 2

Fig. 3

1) R. F. Karlicek and I. J. Lowe, J Mag Reson, 37,75-91 (1980)

Please credit this letter to Dr. D. M. Kramer.


Dr. H. E. Avram (Diasonics MRI)


M.S./J.H. Chen


Dr. L. E. Crooks

The 31st ENC, April 1 - 5, 1990
The Asilomar Conference Center, Pacific Grove, California
Contact: ENC, 750 Audubon, East Lansing, MI 48823
(517) 332-3667

SUN **REGISTRATION and INFORMAL POSTER VIEWING**

MON **POSTER SESSION #1; C. G. Wade, Chair.**

1D, 2D, and 3D NMR IN LIQUIDS; G. Bodenhausen, Chair.

Band-Selective Pulses for Multidimensional Spectroscopy;
Ray Freeman, Cambridge University.

Physical Principles and Biophysical Applications of Proton NMR in
 H_2O ; *Maurice Gueron, Ecole Polytechnique, Palaiseau.*

2D and 3D Experiments for Measurements of Coupling Constants and
 Other Interesting Parameters;
Gerhard Wagner, University of Michigan.

Cross-Correlations and Spin-Diffusion in Biomolecules;
Anil Kumar, University of North Carolina.

Some Fancy Heteronuclear Multi-Dimensional NMR Experiments;
Ad Bax, National Institutes of Health.

CALCULATIONS, SIMULATIONS, AND NUMERICAL METHODS;
D. P. Weitekamp, Chair.

Superoperator Calculations of 2D Exchange Spectra;
Jean Jeener, Universite Libre de Bruxelles.

Linear Prediction Analysis in 1D and 2D Spectra;
James Norris, University of Chicago.

TUES **SOLID STATE NMR; R. Tycko, Chair.**

Sideband Symmetries and Rotational Resonance;
Malcolm H. Levitt, Massachusetts Institute of Technology.

Enhancement of Spin Diffusion by Rf Pulses and Sample Rotation;
Beat Meier, ETH - Zurich.

QUADRUPOLEAR NUCLEI - MOSTLY 2H ; B. Bluemich, Chair.

Solid State NMR of Quadrupolar Nuclides (^{27}Al and ^{95}Mo) in
 Catalytic Environments;
Paul D. Ellis, University of South Carolina.

2H NMR Studies of Orientational Order in Liquid Crystals and
 Binary Mixtures;
Gina L. Hoatson, College of William and Mary.

2D Exchange NMR of Powders: Data Processing and Spectra
 Simulation;
Claudia Schmidt, University of California at Berkeley.

The 31st ENC, April 1 - 5, 1990
The Asilomar Conference Center, Pacific Grove, California
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TUES **HARDWARE; K. W. Zilm, Chair.**

Con't

Experimental Strategies for Dynamic Angle Spinning and Double Rotation;

Alex Pines, University of California at Berkeley.

DNP Enhanced NMR and EPR at 140GHz;

David J. Singel, Harvard University.

WED **MISCELLANEOUS; A. N. Garroway, Chair.**

STM Observation of the Characteristic Frequency of Individual Spins;
Yishay Manassen, Weizmann Institute of Science.

A Portable Magnetic Resonance Microscopy Apparatus: Application to Interdisciplinary Fields; *Motoji Ikeya, Osaka University.*

MATERIALS AND MACROMOLECULES; M. W. Baum, Chair.

Application of NMR Methods to the Protein Folding Problem;
Jean Baum, Rutgers University.

Rotational Resonance: A New Approach to Structural Determination in Polycrystalline Solids;
Robert G. Griffin, Massachusetts Institute of Technology.

Determination of Internuclear Distances by Rotational-Echo Double-Resonance NMR;
Jacob Schaefer, Washington University.

POSTER SESSION #2; C. G. Wade, Chair.

THURS **IMAGING; J. J. Ackerman, Chair.**

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RADIODIAGNOSTICKÉ PRACOVISTĚ

PŘEDNOSTA: PROF. MUDr. ALFRED BELÁN, DrSc.

146 22 PRAHA 4 - KRČ
VÍDEŇSKÁ 800

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

Prague 1. 11. 1989
(received 11/22/89)

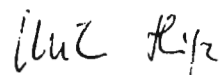
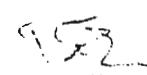
Temperature Probe for Whole Body Magnet

Dear Prof. Shapiro,

Whole body imagers enable spectroscopic examination of organs used for transplantation. The use of a wide bore magnet is necessary because of the transportation box which is in our case 50x32x32cm¹.

To study the behavior of organs during hypothermic preservation procedure, it is necessary to keep the temperature in the box constant and to be able to change it. It was for this reason that we designed and built a temperature probe for our MAGNETOM SH in which it is possible to measure kidney and other organs in original preservation bags. The system is based on evaporation of liquid nitrogen which is transported from the nitrogen container. The probe incorporating a spiral heat-exchanger is made from nonmagnetic material. A 12cm 31P coil was designed to be placed the center of the heat exchanger and a steril box containing the organ is put into the coil. A control unit as well as the nitrogen container are positioned 5m from the magnet, in separate room. The regulation works in the temperature range from laboratory temperature to -10°C. Cooling of the whole system from 24°C to 3°C take place during 25 minutes and the temperature is stable within 0.2°C. Measurement of small kidney spectra takes some of 20 minutes to complete.

Yours sincerely,

 
Milan Hájek and František Bohm
Institute for Clinical and Experimental Medicine, Prague

¹Eurotransplant manual pp. 26-33, 4th edition 1981

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Tel: (415) 229-3501 FAX (415) 229-1651

Department of Physics

November 21, 1989 (received 11/30/89)

Carr-Purcell Sequences Compensated for all Three Spin Components

Dear Barry,

We have been devising and testing Carr-Purcell¹ like sequences that are equally compensated for all 3 components of spin magnetization.² The traditional C-P sequence is compensated for M_x (assuming x phase π pulses); this is the Meiboom-Gill³ modification of C-P. But M_y and M_z are rapidly de-phased by H_1 inhomogeneity. For measuring T_2 , one is only interested in M_x , so the Meiboom-Gill modification works well.

However, Carr-Purcell-like sequences are useful in solids for re-focusing chemical shift, leaving only like-spin dipolar couplings.⁴ The resulting echo envelope is just the FID that would obtain if all chemical shifts were absent. The dipole couplings, in turn, yield *distances*. Perfect π pulses leave the dipolar Hamiltonian unchanged:

$$H_D \equiv I_{z_1} I_{z_2} - \frac{1}{2} I_{x_1} I_{x_2} - \frac{1}{2} I_{y_1} I_{y_2} .$$

Imperfect π pulses (about x) mix I_y and I_z and change H_D . To avoid this, one needs either very high H_1 homogeneity⁴ or a pulse sequence that avoids cumulative errors from the π pulses, on all 3 components of spin.²

Alternation of the phase of the π pulses between x and y yields a sequence

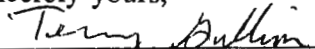
$$\frac{\pi}{2} - \tau - \pi_x - 2\tau - \pi_y - 2\tau - \pi_x - 2\tau - \pi_y - \text{etc.}$$


that we call *xyxy*. Clearly, it will treat M_x and M_y nearly the same. An 8-pulse version is *xyxyxyxy*; this gives rise to the 16-pulse sequence *xyxyxyxyxyxyxyxy*. Experimentally, such sequences *do* handle M_x and M_y equally well. The figure below shows 700 echoes with a spacing $2\tau = 50 \mu\text{s}$. The liquid sample was far from the center of the field, leading to a FID $1/e$ decay time of $40 \mu\text{s}$. The π pulses are $9.5 \mu\text{s}$ long. The observed decay is only slightly faster than T_2^{-1} , as measured by CPMG.

One of us (TG) is using these sequences in REDOR experiments⁵ that use pulses on both ^{15}N and ^{13}C to re-introduce the N-C dipole coupling while magic-angle spinning.

Please credit R. E. Norberg's account.

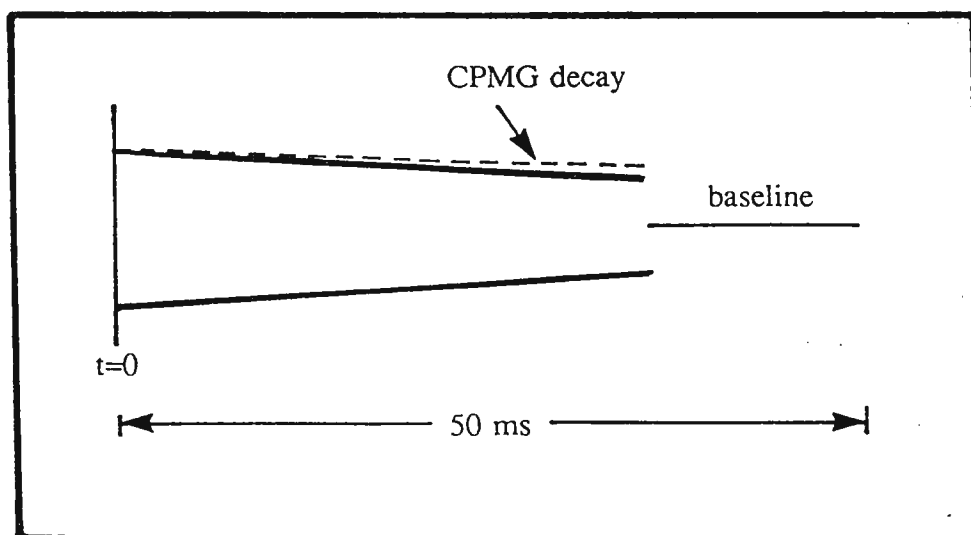
Sincerely yours,


Terry Gullion


David Baker


Mark S. Conradi

1. H. Y. Carr and E. M. Purcell, Phys. Rev. **94**, 630 (1954).
2. A. J. Shaka, S. P. Rucker, and A. Pines, J. Magn. Reson. **77**, 606 (1988).
3. S. Meiboom and D. Gill, Rev. Sci. Instrum. **29**, 688 (1958).
4. M. Engelsberg and C. S. Yannoni, preprint.
5. T. Gullion and J. Schaefer, J. Magn. Reson. **81**, 196 (1989) and Adv. Mag. Res. **13**, edited by W. S. Warren, to appear 1989.



700 echoes generated from 16-pulse Carr-Purcell-like sequence.
Unlike CPMG, this sequence is equally effective in refocusing M_x and M_y (and M_z).



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Biotechnology Research
Institute

Référence File

6100, avenue Royalmount, Montréal, Québec, Canada H4P 2R2

Tel.: (514) 496-6100

November 28, 1989 (received 12/7/89)

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

Phase-sensitive NOESY w/o suppression of zero-quantum coherence in
the study of protein-peptide interactions

Dear Dr. Shapiro,

In the acquisition of NOESY spectra of proteins or peptides, it is a common practice to suppress crosspeaks arising from zero-quantum coherence transfers between *J*-coupled protons (Wider et al, 1984, *J. Magn. Reson.* 56, 207). Otherwise, there would be antiphase multiplets for each pair of coupled protons in a phase-sensitive NOESY (Macura et al, 1981, *J. Magn. Reson.* 43, 259) similar to crosspeaks from *zz*-coherence transfers (Bodenhausen et al, 1984, *J. Magn. Reson.* 59, 542). Retaining zero-quantum coherence transfers can, however, be very useful in some applications of NOESY, as we found out in our recent studies of the interaction of thrombin with peptide inhibitors.

Due to enzyme-induced line broadening (via exchange from bound protons), these antiphase crosspeaks in the NOESY spectra of peptides are markedly attenuated in the presence of a binding enzyme, a phenomenon that was used to identify amino acid residues involved in the interaction of thrombin with fibrinogen-like peptides (Ni et al, 1989, *Biochemistry*, 28, 3082) and with hirudin-derived peptide inhibitors (Ni et al, 1989, submitted to *Biochemistry*). This is illustrated in Figure 1 with the NOESY spectrum (mixing time: 200 ms) of a peptide derived from the C-terminal region of hirudin, H₂N-Asn(52)-Asp(53)-Gly(54)-Asp(55)-Phe-Glu-Glu-Ile(59)-Pro-Glu-Glu-Tyr-Leu-Gln(65)-COOH. Figure 1A displays the spectral region that contains all of the α CH- β CH crosspeaks (NOE + zero-quantum) of the peptide (4 mM) in the presence of thrombin (0.3 mM). Note that only residues Asn(52) and Asp(53) exhibit multiplet crosspeaks characteristic of zero-quantum coherence between their α CH and β CH protons. The α CH- β CH crosspeaks of residues Asp(55) and Ile(59), for example, are dominated by the transferred NOEs between these protons from the bound peptide. When the NOESY spectrum was acquired with a 10% variation of the mixing time, zero-quantum crosspeaks were suppressed as shown in Figure 1B. Random variation of the mixing time may also have caused the increased level of *t*₁-noise seen in this spectrum.

Yours sincerely,

Feng Ni
Feng Ni, Ph.D
Protein Engineering

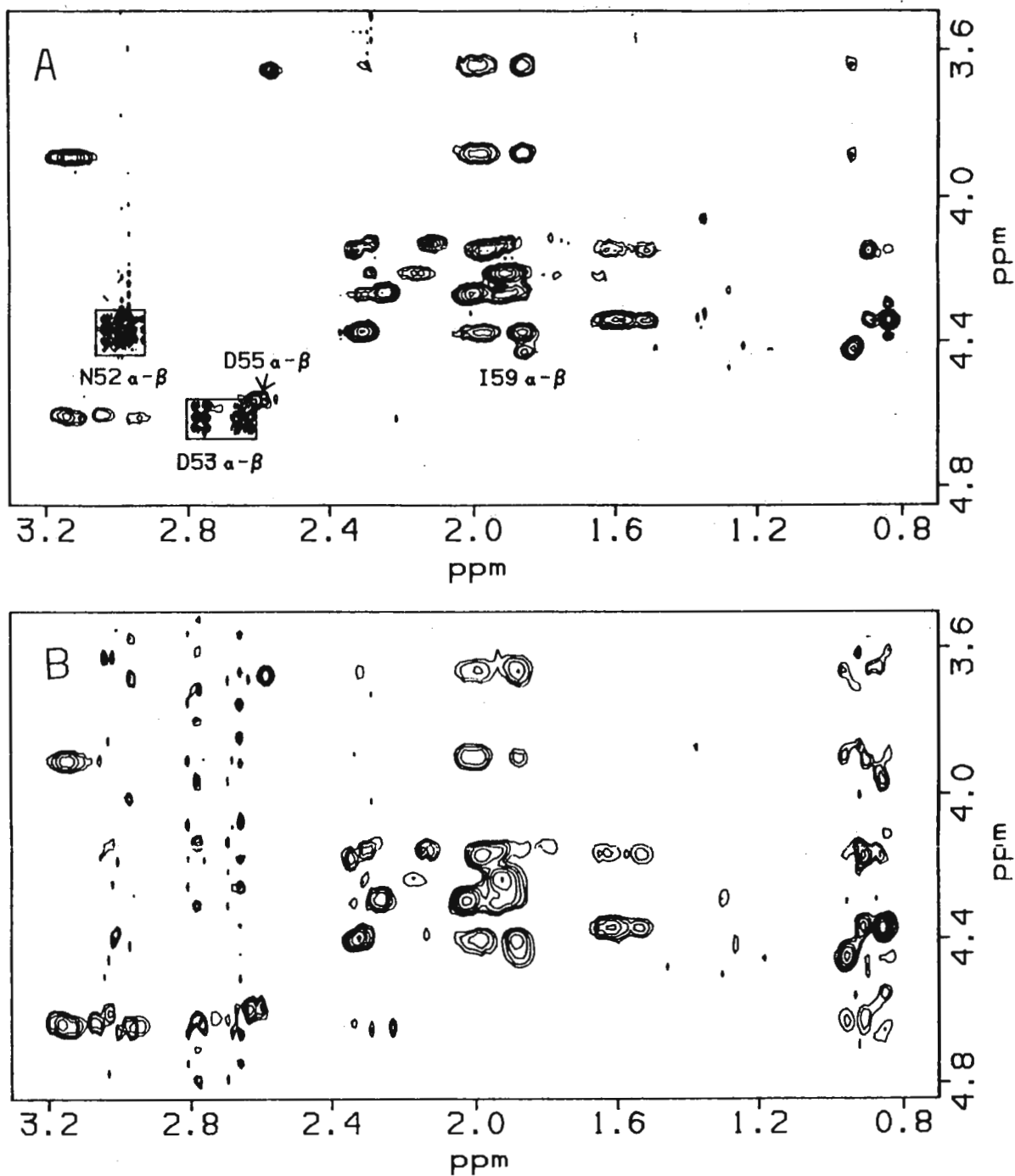


Fig. 1

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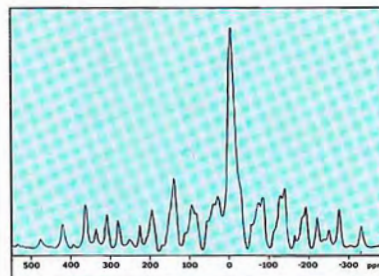
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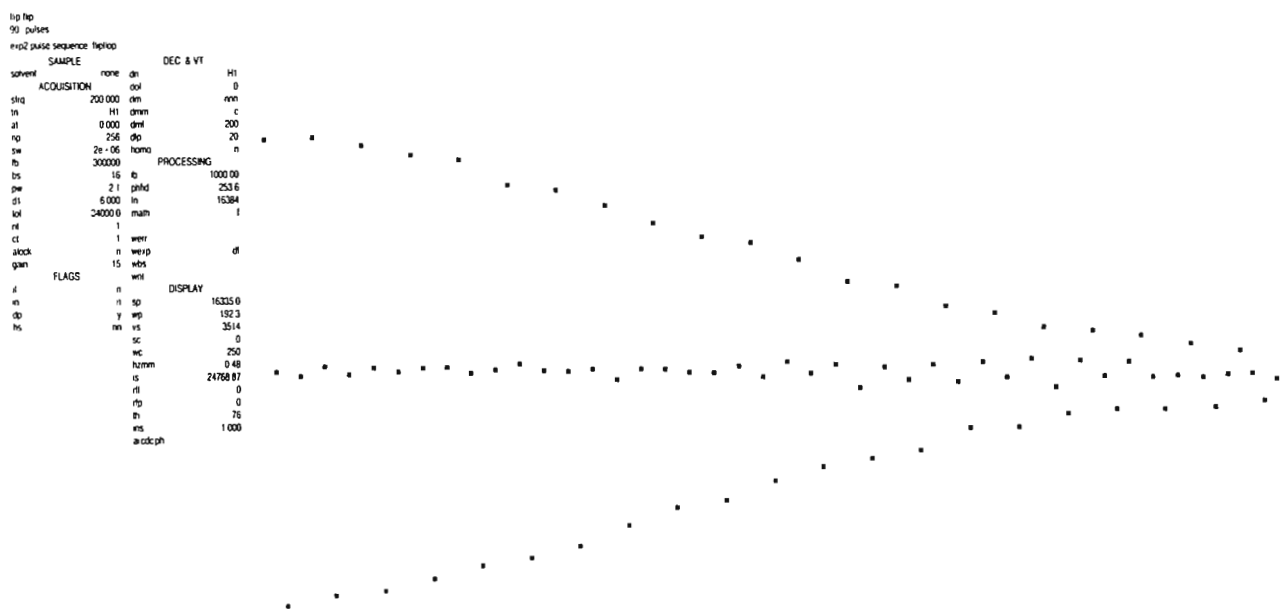
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TUNE-UP SEQUENCES: FLIPFLIP WITH 90° PULSES

The pulse sequence used is $(90^\circ - \text{acq})_n$. When the pulse is exactly 90° , the following pattern is observed. This experiment is used to set the 90° pulse width. This and the following sequences exactly reproduce those shown in figure 2 of D. P. Burum, M. Linder and R. R. Ernst, Journal of Magnetic Resonance, 43, 463, (1981). This is figure 2a of that paper.



MAC 50304V9

TUNE-UP SEQUENCES: FLIPFLOP

This is the sequence $(90(x) - \text{acq} - 90(-x) - \text{acq})_n$, and is used to tune out "phase glitch" in probe and pulse amplifier. It is the second sequence normally used in setup. Phase glitch is manifest as an inphase oscillation in the data. This sequence is used by other manufacturers to set exact 180° phase shifts, a procedure that is unnecessary in UNITY. (Burum, Linder and Ernst, figure 2h)





Rm. 1A-355
600 Mountain Avenue
Murray Hill, New Jersey 07974

December 12, 1989
(received 12/15/89)

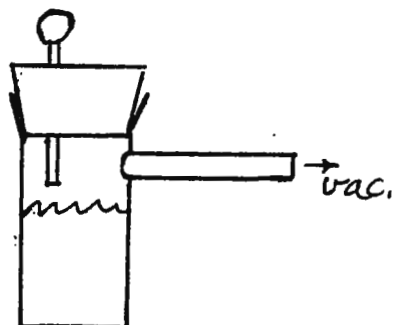
Dr. Barry L. Shapiro
TAMU Newsletter
966 Elsinor Court
Palo Alto, CA 94303

RE: Getting Viscous Materials into Small NMR Sample Tubes, etc.

Dear Barry,

On occasion everyone who works with polymers is faced with the problem of enticing a viscous fluid into a small sample container, such a 4mm sphere, through an even smaller entry tube. The sketch in the figure below shows a trivial device for doing this that is easy to make and has served well in our lab on numerous occasions. The elemental device is a flat-bottomed glass tube fitted with a side-arm, for evacuation, and a fore-shortened rubber stopper, pierced to accommodate the stem of, say, a spherical sample tube. If one can afford to be profligate with the sample, it may be placed directly in this container, with the entry of the stem poised above the surface of the fluid. After the air-space is evacuated and the stem depressed into the fluid, pressurization of the tube forces bubble-free sample into the sphere. When only small quantities of sample are available, an auxiliary reservoir may be conveniently handled by placing it within a teflon support that slides into the body of the tube. By replacement of the simple pierced stopper with one bored for a rod threaded to fit the end caps of o-ring equipped MAS rotors, one may easily evacuate and seal bubble-free material into rotors and substantially reduce problems arising from ejection of end caps on heating of partially filled rotors, a particularly messy accident with some low molecular weight polymers. Clearly this elemental device may be made more elegant by addition of a stopper equipped with o-ring seals and made to act as a portable dry-box but we have not been obliged to do this in our uses of it.

Please credit this submission to the account of Lynn Jelinski.



Sincerely yours,

Dean Douglass

Tel: (201)-582-6950
FAX: (201)-582-3958
E-mail: arpa!allwise!ded



376-26

10th European Experimental NMR Conference

VELDHOVEN - THE NETHERLANDS - MAY 28/JUNE 1, 1990

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Prof. B.L. Shapiro

~~Department of Chemistry~~ 966 Elsinore Court
~~Texas A & M University~~ Palo Alto, CA 94303
~~College Station, TX 77843~~
USA

Date: 27 November 1989
Our ref.: DB 11/19
Your ref.:
Subject: Forthcoming 10th EENC

Dear Prof. Shapiro,

I would greatly appreciate if you could publish the following announcement in the NMR Newsletter.

The 10th European Experimental NMR Conference will take place at the Conference Centre "Koningshof" at Veldhoven from Monday, May 28th to Friday, June 1st 1990. The scientific programme will deal with selected topics in NMR spectroscopy, with the emphasis being on the **new** experimental aspects of the techniques. The programme consists mainly of invited lectures, with keynote lecturers outlining the new developments in their topic and contributing lecturers, who broaden the overall scope of the topic. In addition to the lecture programme two poster sessions are planned (the deadline for submitting poster abstracts is February 15, 1990). The subjects of the selected topics are:

- New experimental NMR techniques
- Data analysis
- NMR in solids
- Pulse sequences and tailored excitation
- In-vivo NMR spectroscopy/localized spectroscopy
- Multidimensional NMR
- Biomolecular structural analysis

The fees for the attendance of the meeting have not yet been finalized, but it seems likely that the following fee schedule will be in effect:

Registration fee, active participants:

Dfl 300,--

This registration fee is inclusive of the conference dinner, the excursion and the refreshments during the conference breaks. Student grants are available for part or all of the registration fee.

Conference arrangement (room and all meals):

Dfl 510,-- - Dfl 625,--

For further information, please contact the Secretariat of the 10th EENC:

Miss S.H. de Bie - Diefsteeg 14 - NL-2311 TS Leiden, The Netherlands. Tel.: ..31-71-133617

Yours Sincerely,

Dr. M.J.A. de Bie
Conference Director

Mailing Addresses: Secretariat 10th EENC - Miss S.H. de Bie - Diefsteeg 14 - NL-2311 TS Leiden - The Netherlands. Tel.: ..31-71-133617
M.J.A. de Bie - Bijvoet Center - Utrecht University - Padualaan 8 - NL-3584 CH Utrecht. Tel.: ..31-30-533801. Telefax: ..31-30-540980.
H. Angad Gaur - AKZO Corporate Research CRL - P.O. Box 9300 - NL-6800 SB Arnhem. Tel.: ..31-85-662256 Telefax: ..31-85-665280.

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November 20, 1989
(received 11/25/89)

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

Dear Dr. Shapiro,

To improve the throughput of routine NMR runs and to relieve some of the routine sample load from our higher field instruments (Varian XL-300, Bruker WH-400 and AMX-500), the High-Resolution NMR Service Lab at UBC has recently acquired a Bruker AC-200E quad-nuclear cryospectrometer. The system came with automatic sample changer, barpen barcode reader, computer-switchable quad-nuclear QNP probe, auto-lock, auto-shim and auto-receiver gain adjust, etc. The Bruker supplied software (DISRVK06 and BARPEN) support the automatic sample changer and barcode reader separately. However, a completely integrated automatic timesharing system to use both the barcode reader and automatic sample changer simultaneously is not currently available commercially. We, therefore, developed in house our own macro-software system combining the capabilities of the separate Bruker software, viz. DISRVK06 (the NMR FT program), BARPEN.EXE (the barpen/barcode macro program), PASCALL (the call utility program) and BARPEN (the PASCAL-based colour monitor display program) to produce a powerful and easy to use automatic timesharing system with password access security and protection against keyboard mistakes. It should be noted that this software obviates the need for a barcode reader on the sample changer and corresponding barcodes on the spinners (rotors). This is the suggested configuration by Bruker but it is too cumbersome for walk up use.

The system can be used in four different modes, viz.:

- 1) The usual manual operation.
- 2) Auto sample changer operation with dialogue-driven experiment setup.
- 3) Bruker barpen/barcode operation (user attendance required), and
- 4) The UBC completely automatic timesharing operation.

To access the different modes, the following commands were implemented:

EXE AUTOSAMP.CHGR brings one into the 2nd mode.
EXE BARPEN.BKR brings one into the 3rd mode, and
EXE BARPEN.UBC brings one into the final 4th mode.

With the automated system, our users can set up sample(s) to be run automatically in the timesharing mode. This is accomplished with a few strokes of the barpen over barcodes generated for the various experiments to be performed. The keyboard is deactivated automatically to avoid typing accidental entries. The user can enter samples one at a time or can request that more than one sample be run. A maximum of 4 experiments (different nuclei, COSY etc.) can be run per sample at a single submission. The general system environment is that all three jobs of the NMR FT program are active with Job 1 displaying the Welcome menu up front in the waiting mode for more samples to be set up, Job 2 performing data acquisition and Job 3 processing and plotting in Region 1 of ADAKOS. The system manager or experienced users with approval may access Region 2 with the keyboard active and perform other work without interrupting the automatic operation in Region 1. It is intended that LIGHTNET will be run in Region 2 to communicate with a separate workstation in the Lab.

The automation system is designed in such a way to allow easy setup for detection of common nuclei ($H^1/C^{13}/F^{19}/P^{31}$) and frequently performed experiments for users with minimal NMR technical experience. Experienced users can, (with approval), exit the completely automatic mode without interrupting the automatic timesharing operation, and use in a semi-manual mode in Job 1. In this mode, the user can set up new samples for more sophisticated and complicated experiments to be added to the auto sample series and, if necessary, make changes to those previously set up but not yet run. One can also go to Job 2 to check the current data acquisition status and to Job 3 to check the processing and/or plotting status. While in the waiting mode of the automatic operation, one has the option of checking the Job 2 or Job 3 status by a stroke of the barpen over the status barcode.

We find the automatic system described provides easy access to the 200 MHz spectrometer for users with little experience in NMR spectroscopy. Those interested may obtain further information from the undersigned. Better still, those who happen to be in Vancouver, B.C. are very welcome to drop in to see it in operation.

Kindly credit this to Dr. F. G. Herring's account. Thank you very much.

Yours sincerely,

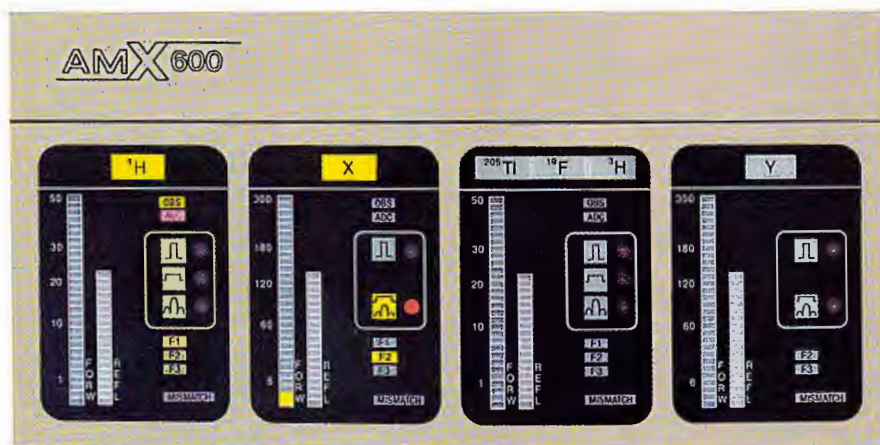


S. Orson Chan
N.M.R. Spectroscopist

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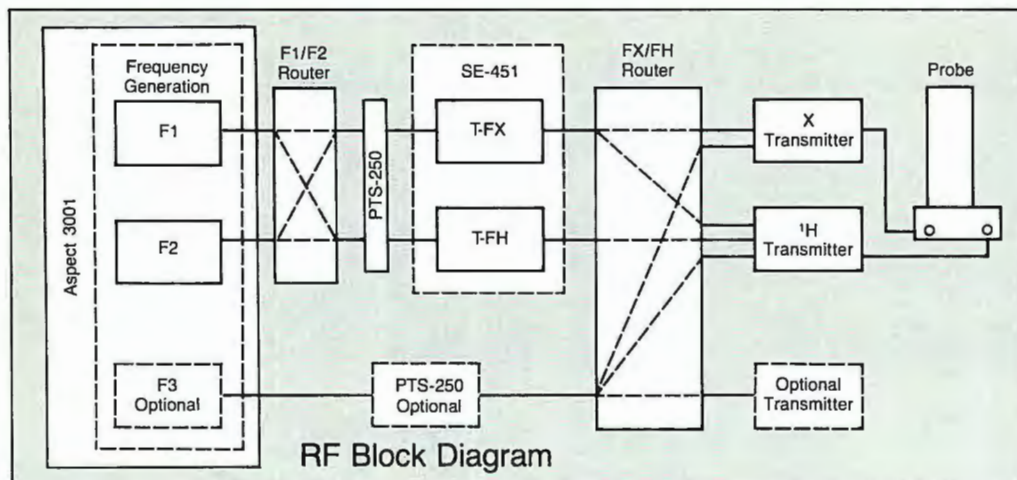
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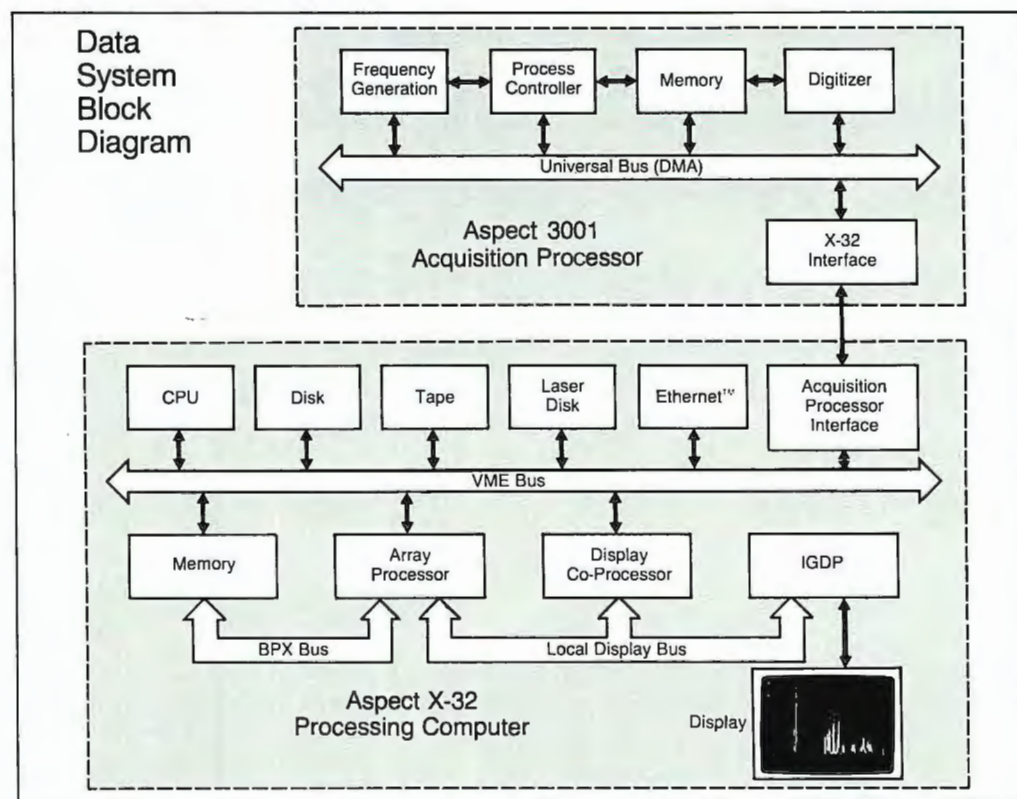


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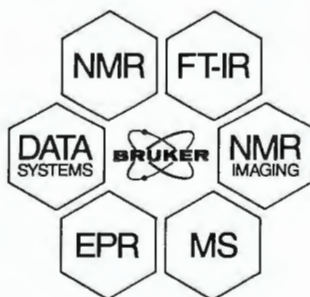


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November 28, 1989 (received 12/1/89)

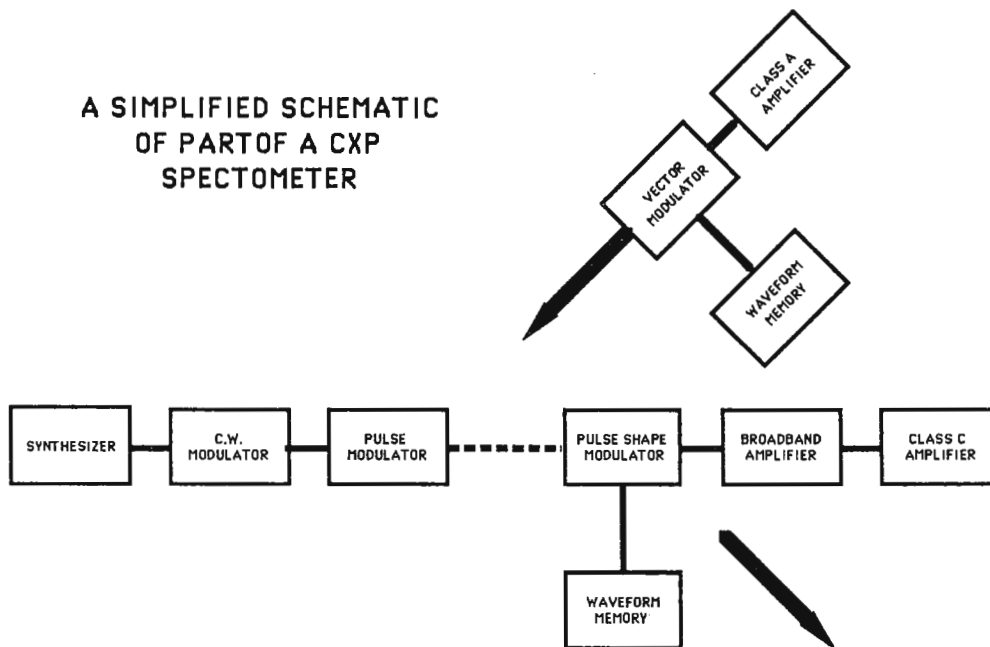
Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303
U.S.A.

Dear Barry,

In-vivo NMR spectroscopy laboratories that were reasonably quick off the mark in buying their hardware at the beginning of the Eighties are now finding that this hardware can be very restricting, because it is incapable of implementing simultaneous phase and amplitude modulation of selective pulses. Such a capability is very important for in-vivo work, where one wishes to manipulate the spins over a very large volume in a way that is insensitive to large B_1 variations.^{1,2} This note is a description of how one might cheaply upgrade a venerable spectrometer to the standards of current practice.

Our animal unit is based on a BRUKER CXP spectrometer, originally furnished with Class C power amplifiers, that facilitated amplitude modulation, but only if the RF waveform was superimposed on a voltage pedestal that was required to switch on the Class C amplifiers. Although the replacement of the Class C amplifiers by Class A is a necessary condition for the upgrade, it is by no means sufficient. The figure shows a simplified schematic of part of the CXP spectrometer. To facilitate phase and amplitude modulation, both the pulse shape modulator and the waveform memory also need to be replaced. The pulse shape modulator has been replaced by a commercial vector modulator (U.S. \$2,500) whose frequency range we have transposed down to our own (15MHz to 100MHz) by means of conventional superheterodyne techniques (parts and fabrication costs U.S. \$750). The waveform memory has been replaced by a new board, designed and constructed in house, that fits in the same slot in the Aspect 3000; that responds to the same control signals as the original waveform memory; but which interfaces with the vector modulator to communicate both amplitude and phase information. At the same time we increased the memory capacities from 1K on the old board to 4K. The total cost for components and fabrication was less than U.S. \$500. The output of the vector modulator is capable of driving either the new Class A amplifier or the old Bruker amplifier chain. The latter is still useful for variable amplitude spin-locked $T_{1\rho}$ measurements. The cost of the Class A amplifier, the only potentially costly part of exercise, depends on the needs of the consumer. We have a predilection for power (Amplifier Research Model, 2000LM29, U.S. \$52,000).

A SIMPLIFIED SCHEMATIC
OF PART OF A CXP
SPECTROMETER



The new upgrade permits phase and amplitude modulation of the RF pulses with an amplitude resolution of 0.07dB, a phase resolution of 0.45° and up to 4096 points per waveform with a minimum time resolution of 1μs per point.

- References
1. Silver, M.S., Joseph, R.I., and Hoult, D.I. Phys. Rev. A. 31, 2753 (1985).
 2. Ugurbil, K., Garwood, M., and Rath, A. J. Magn. Reson. 80, 448 (1988).

Yours sincerely,

Peter S. Allen

David C. Ellinger

Dan Gheorghiu

PSA/sm

THE UNIVERSITY OF MICHIGAN
INSTITUTE OF SCIENCE AND TECHNOLOGY
BIOPHYSICS RESEARCH DIVISION

November 21, 1989 (received 11/25/89)

2200 BONISTILL BOULEVARD
ANN ARBOR, MICHIGAN 48109-2099

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

Simulation of NOESY Spectra

Dear Barry:

We have recently developed software (1) to simulate NOESY spectra of proteins. We have used the program CORMA (2) that creates a list of NOE cross peak intensities from atomic coordinates with the assumption of a single correlation time. CORMA was provided by T. James. We have written a Pascal routine to simulate multiplet structures from the dihedral angles of the atomic model. The calibration of the coupling constants was obtained from standard Karplus-type curves. A mixture of Lorentzian, $L(\omega)$, and Gaussian, $G(\omega)$, line shapes was used. We found the best agreement with the experimental spectra with:

$$S(\omega) = 0.2 L(\omega) + 0.8 G(\omega)$$

The resolution in both dimensions was matched to that of the experimental spectra. The program package requires as input a set of atomic coordinates, a list of chemical shifts of assigned proton resonances, line widths, the mixing time, and the relative contributions of Lorentzian vs. Gaussian line shape.

We have applied the program to simulate NOESY spectra of the elastase inhibitor eglin c for which we have complete assignments (3). Fig. 1 shows a comparison of a small part of an experimental 200 ms spectrum with the simulated spectrum obtained computed with the atomic coordinates of the X-ray structure of the protein in the complex with subtilisin Carlsberg (4). A single correlation time of 4 ns was assumed. In the crystal structure, the first 7 residues are disordered, and no coordinates are available. Therefore, their resonances do not appear in the simulated spectrum (the coordinates of the X-ray structure were kindly provided by Dr. Wolfram Bode). While most parts of the simulated spectrum are nearly identical to the experimental spectrum there are some signals that are significantly different, indicating differences between the structure of the free inhibitor in solution and the X-ray structure of the inhibitor in the complex with subtilisin Carlsberg (5).

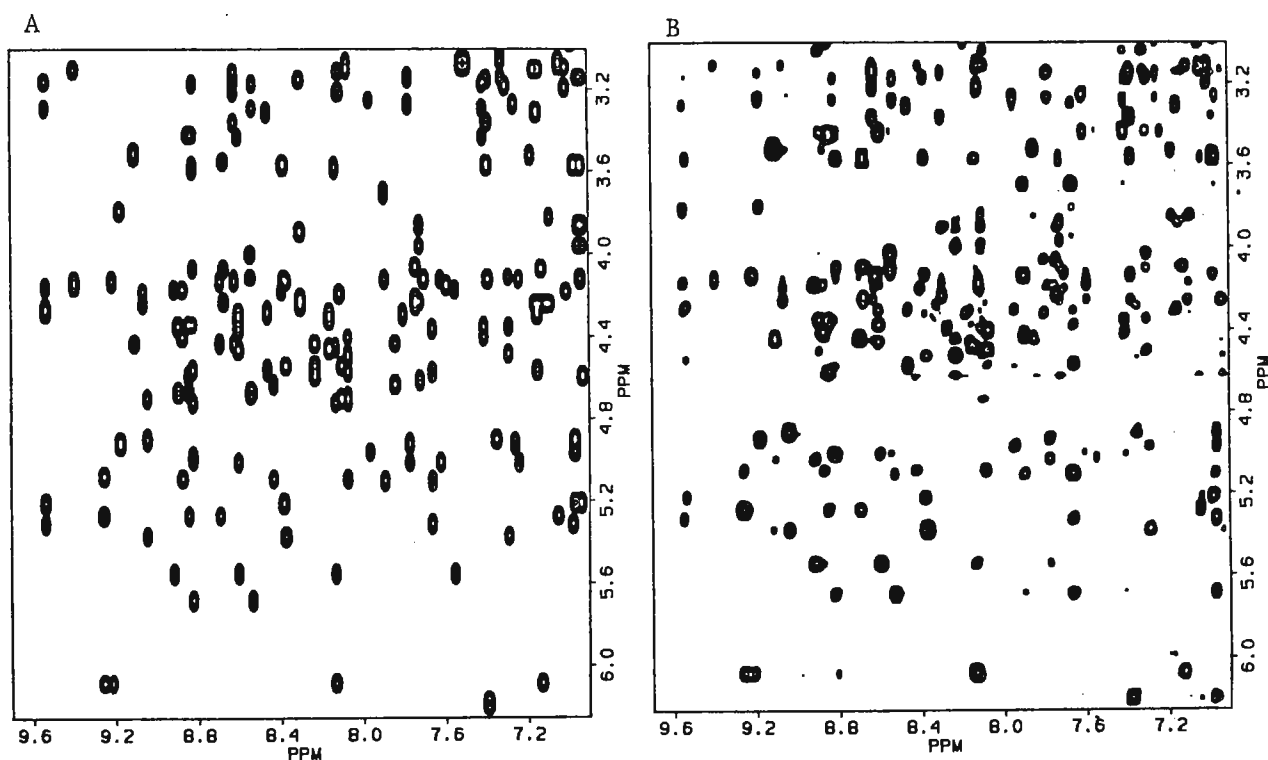
Sincerely


Nicholas Beeson Sven G. Hyberts Terry Weymouth Gerhard Wagner

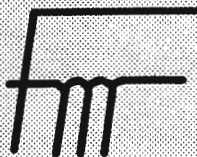
1. N. Beeson, S. G. Hyberts, T. Weymouth and G. Wagner, manuscript in preparation.
2. J. W. Keepers and T.L. James, J. Magn. Reson. 404-426 (1984).

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4. W. Bode, E. Papamokos and D. Musil, *Eur. J. Biochem.* 166, 673-692 (1987).
5. N. Beeson, S.G. Hyberts, T. Weymouth and G. Wagner, to be published.

Fig. 1: Comparison of a NOESY spectrum simulated from the X-ray coordinates at a mixing time of 200 ms (A) and an experimental NOESY spectrum (B). To show the quality of the simulation, a blow-up of a small region is shown.



Available: Varian 12" diameter pole tip, 4" gap electromagnet model V7300. Operated for many years at UCSF to image rats at 0.35 Tesla. Field uniformity is 18 milliGauss (5ppm) over 1" DSV. Has set of imaging gradient coils. Includes type V7700 power supply with a V7570 Field Dial Regulator Mark II and a V7772 heat exchanger. Purchase price in 1976 was \$40,000. Asking \$4,000 and purchaser must pay transportation. Contact Larry Crooks, University of California, 400 Grandview Drive, South San Francisco, CA 94080. Phone voice 415-952-1369, FAX 415-952-2714.



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FMR has combined its operations into a new facility. The move was made reduce our delivery times and

expand our services. The process has, to date, reduced our new probe and probe rework delivery times from an average of 180 days to 90 days. Repairs have been reduced from 60 days to less than 30 days. Our goal is to be able to deliver all probes and custom rebuilds within 30 days and do repairs in less than 15 days. This effort is a response to customer feedback which has consistently indicated that they want faster repairs and new order deliveries.

FMR is in the process of expanding its product line to give the NMR marketplace options to upgrade existing NMR systems. Our goal will be to have similar delivery times for all our product lines.

Probe Services

FMR provides probe services designed to give the NMR marketplace the capability for repairing and upgrading their existing NMR spectrometers.

- Fast and cost effective probe repairs.
- Probe upgrades. Do you have a probe laying around the laboratory which hasn't been used in years? If so why not upgrade it? This can be done for a lot less than a new probe. Existing or broken probes can be repaired and upgraded to different insert sizes and nuclei. The following areas are prime reasons for upgrading an existing probe:

- ☛ New technology to give better performance. Some probes can be upgraded give more than a 100 % better sensitivity.
- ☛ Repair or upgrade and older unused probe to serve as a backup for existing used probes.
- ☛ Upgrade an unused probe to be a ^1H biological (salt) probe. This will give shorter 90° pulses and better sensitivity in salty water solutions.
- ☛ Upgrade an unused or broken probe to be able to do new experiments. For example FMR can upgrade an older, little used probe to be a ^1H $\{^3\text{P}-^{15}\text{N}\}$ probe for today's powerful 2DFT experiments. FMR can provide an X-Nucleus decoupler with its own internal phase shifts and decoupling modulation schemes to go with this new probe. Special adapters are available allowing Bruker AM systems without reverse mode to run pulse sequence programs with decoupler phase equations.

Do You Have Enough Sensitivity ?

Corollaries of Murphy's Law:

- All horizontal surfaces tend to be occupied.
- All computer disks tend to be full.
- All NMR instrument needs more sensitivity to do the desired experiment.

With this last corollary in mind, it is surprising how few operators know their system's noise figure. Measurement of this parameter separates the console performance from the probe performance and magnet shim status. This prevents blaming the probe and/or shimming for hours to test the NMR system performance. There are many procedures for determining this important parameter, but most of them are either very expensive, take a lot of time, are difficult to perform and/or are not reproducible. To solve this problem, FMR supplies an inexpensive test instrument and procedure for NMR instruments to do a complete noise figure measurement to be reproducibly performed in less than 5 minutes. Do you know your system's noise figure?

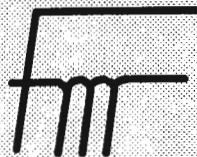
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No Magic in Probes !

Instrumentation Note 15

Many people believe that probe construction and performance is black magic. While at times their design and performance may seem like magic, there is no magic. There are just many details requiring close attention. Some of the "details" can be in conflict with other important "details". When this happens creative compromise is necessary. Since many of the "details" are VERY important, getting any one of them wrong may make the probe's performance suboptimal. Listed in the table below are important probe performance parameters.

Performance Parameters

Sensitivity.
90° Pulse Width.
Resolution and Lineshape.
RF Pulse Homogeneity.
VT Range and Regulation.
Decoupler Gamma H₂.

Sensitivity

The most important details which affect a probes sensitivity are:

Sensitivity Parameters

Coil Length.
Coil Filling Factor
Probe Q.
Dielectric value of sample.
Percentage of the overall coils inductance in the coil leads.

The first and second items in the Sensitivity Parameters list reduce to how much sample can the probe coil see. It is not the first thing that comes to an NMR operators mind when thinking of sensitivity, but it is one of the most commonly varied probe parameters in the vendors war of sensitivity numbers. For a 5mm ¹H probe the typical coil is between 10 and 20 mm long. Coils as long as 25 mm have been produced. At least three things limit the possible coil length:

1. Length of magnet's high homogeneity magnetic field. In general the higher the magnetic field the shorter the useful magnetic length. Also the more narrow the bore the shorter the useful magnetic field. A perfect probe can never perform better than the magnet homogeneity allows. Many vendors use long coil lengths. These long coils make magnet to magnet variations more important. Some vendors optimize probe coil lengths based on each

magnet. If you are in the market to buy an instrument, this can be one of the potential reasons why the sales demonstrations can be better than the performance you get on delivery.

2. Length of the room temperature shims active region. When the main magnetic field is long enough, the probe designer still needs to be concerned with the useful areas over which the RT shims operate. If the main field is long enough and the shims don't operate with the necessary purity over the coil's length, the probe's performance will be suboptimal. The first symptom that the coil is too long for the magnet and shim set is a tendency for the NMR signals to "split" or become doublets with VERY small changes in Z² or with small changes in room temperature.
3. RF Homogeneity. Solenoid coils can have good RF homogeneity over long lengths. However, most superconducting magnet probes must use some type of Helmholtz coils. As these coils get longer they have lower RF homogeneity. One way to see this low RF homogeneity is to measure the ratio of the signal from a 90° pulse and a 180° or 360° pulse. The larger the ratio the better the probe's RF homogeneity. This parameter can be very important in many 2DFT experiments even with compensated pulses.

The other dimension determining coil filling factor is off axis. The filling factor in this dimension is limited by how close to the sample the coil can be placed. As the sample insert is brought in closer to the sample tube the tolerances on probe construction become tighter. The first symptom of these tolerances being wrong is trouble with sample spinning. As these tolerances get tighter, higher quality tubes are required. Another way to get a better filling factor is to use thinner wall tubes. The thinner walls allow more sample to be inside the probe's coil and therefore the probe gives higher sensitivity numbers. The sensitivity difference from very thin wall tubes is much larger with 5mm probes than larger diameter probes. When looking at probe performance specifications from NMR vendors be sure to know what sample tubes they are using.

The third item in the Sensitivity Parameters list (Probe Q) is determined by:

Q Factors

RF resistance of the coil materials.
Geometry of the coil
Q of the coils capacitors.

The factors affecting probe Q and their interaction with the sample dielectric will be explored in the next instrumentation note.

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Telex 017-72826

DEPARTAMENTO DE QUIMICA

Fax 754-8707

December 6, 1989
(received 12/15/89)

Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Ct
Palo Alto, CA 94303
U. S. A.

Four Parameters or Four Errors

Dear Dr. Shapiro:

I enjoyed reading the letter of Professor Freeman published in TAMU NMR Newsletter, 374, 35 (1989).

There he states that "one can describe an elephant with four parameters" as his footnote (2), which reads:

(2) *mit vier parameteren kann man ein elephant beschreiben*

He probably wanted to say:

(2) *mit vier Parametern kann man einen Elephant beschreiben*

I am interested in knowing if the four errors he made are in some way related to the four required parameters.

Sincerely yours,



Pedro Joseph-Nathan

'mdv.



The University of Georgia

Complex Carbohydrate Research Center

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

November 30, 1989
HvH 89-A-92
(received 12/5/89)

1D HOHAHA with double-DANTE inversion and DIPSI-2 mixing applied to carbohydrates

Dear Dr. Shapiro:

Recently we have been using various schemes of 1D HOHAHA [1] experiments for spectral assignments of complex sialyl-oligosaccharides. In accordance with the results obtained by Bax and coworkers [2], we found that magnetization transfer is accomplished quite efficiently in a relayed HOHAHA experiment. This technique enables, for instance, the localization of H5 protons of galactose residues ($J_{45} < 1.5$ Hz), and also H7 protons of sialic acids ($J_{67} < 1.5$ Hz). However, we found an alternative way for locating these resonances, namely, by simultaneous inversion of two resonances belonging to the same spin system. In the case of galactose units, usually protons H1 and H4 are uncovered, and the same is true for the H3ax and H3eq protons of sialic acid residues. The simultaneous inversion of two protons was achieved by a double-DANTE [3] pulse sequence. Examples of 1D HOHAHA spectra, obtained through double-DANTE inversion, of an *N*-acetylgalactosamine and sialic acid residue in a complex heptasaccharide are shown in Fig. 1. The spectra were recorded on a Bruker AM-500 spectrometer in the "reverse mode." We used the DIPSI-2 pulse sequence [4] for isotropic mixing, and the mixing time was incremented every eight scans to obtain better lineshapes. Although this method gives satisfactory results for localizing resonances following the small-*J* "bottleneck" in a given spinsystem, it is not possible to obtain accurate values for the coupling constants, which especially in the case of the sialic acid side chain are of interest to us. However, those coupling constants can be measured from a 1D COSY spectrum with chemical shift selective filter [5], which allowed us to obtain high selectivity irrespective of partial overlap of multiplets. Trace g in Fig. 1 shows the expanded multiplet of the sialic acid H8 proton in a 1D COSY experiment where the chemical shift filter was adjusted to the H7 resonance. The combination of the modified 1D HOHAHA and 1D COSY techniques allows fast spectral analysis of the target multiplets in complex spectra.

Sincerely,

Herman van Halbeek

Leszek Poppe

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- [2] F. Inagaki, I. Shimada, D. Kohda, A. Suzuki and A. Bax; *J. Magn. Reson.* **81**, 186 (1989)
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- [4] S. P. Rucker and A. J. Shaka; *Mol. Phys.* **68**, 509 (1989)
- [5] L. D. Hall and T. J. Norwood; *J. Magn. Reson.* **76**, 548 (1988)

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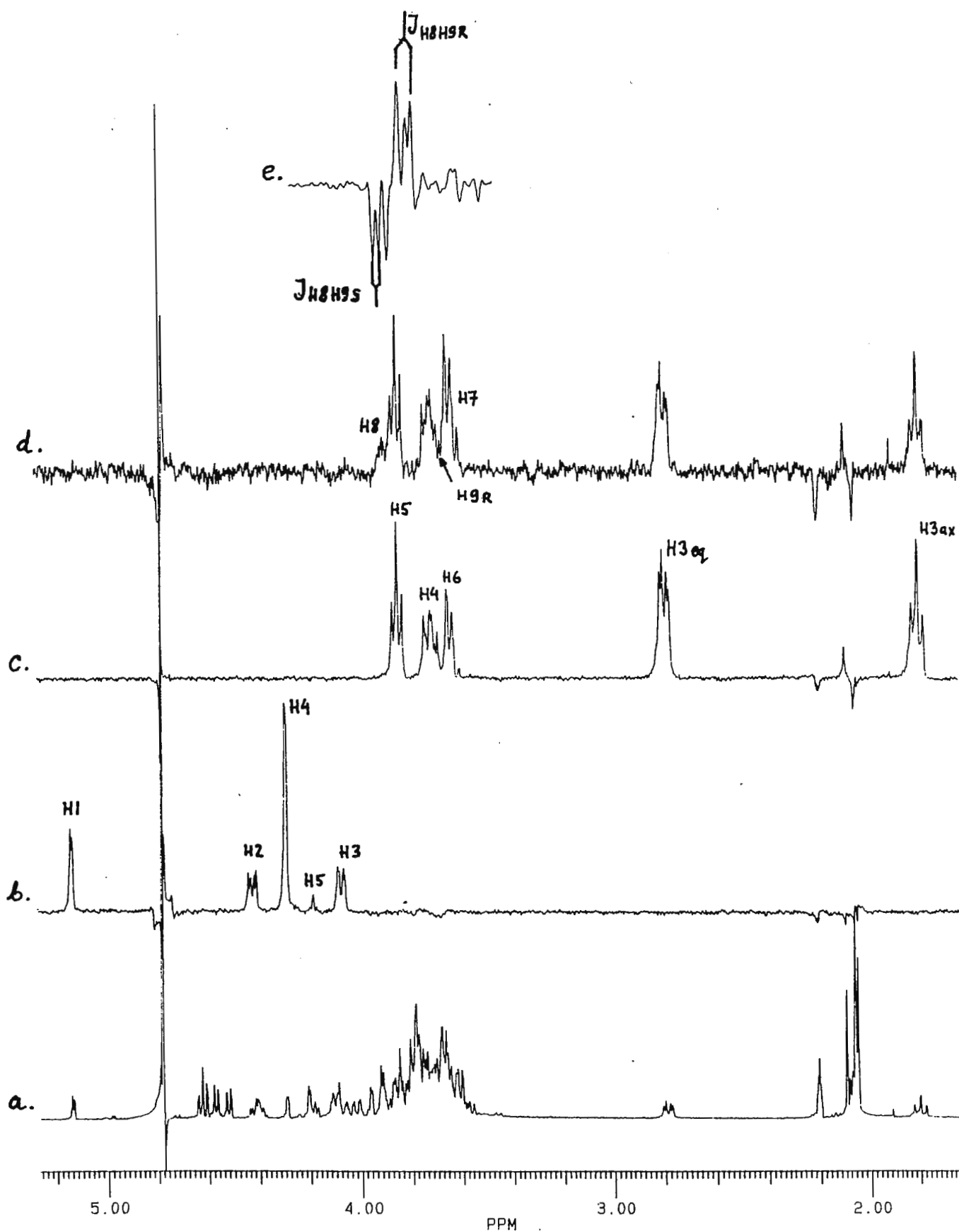
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Fig. 1. **a.** 500-MHz ^1H -NMR spectrum of a sialyl-hexasaccharide (D_2O , 20°C , pD 6).

b. Subspectrum of the α -GalNAc residue in this oligosaccharide, obtained by 1D HOHAHA with double-DANTE inversion of H1 and H4 (DIPSI-2 τ_{mix} 150-210 ms).

c and d. Subspectra of the α -NeuAc residue in this oligosaccharide, obtained by 1D HOHAHA with double-DANTE inversion of H3 $_{ax}$ and H3 $_{eq}$ (DIPSI-2 τ_{mix} 90-150 and 280-340 ms, resp.).

e. 1D COSY spectrum with chemical shift filter on NeuAc H7.

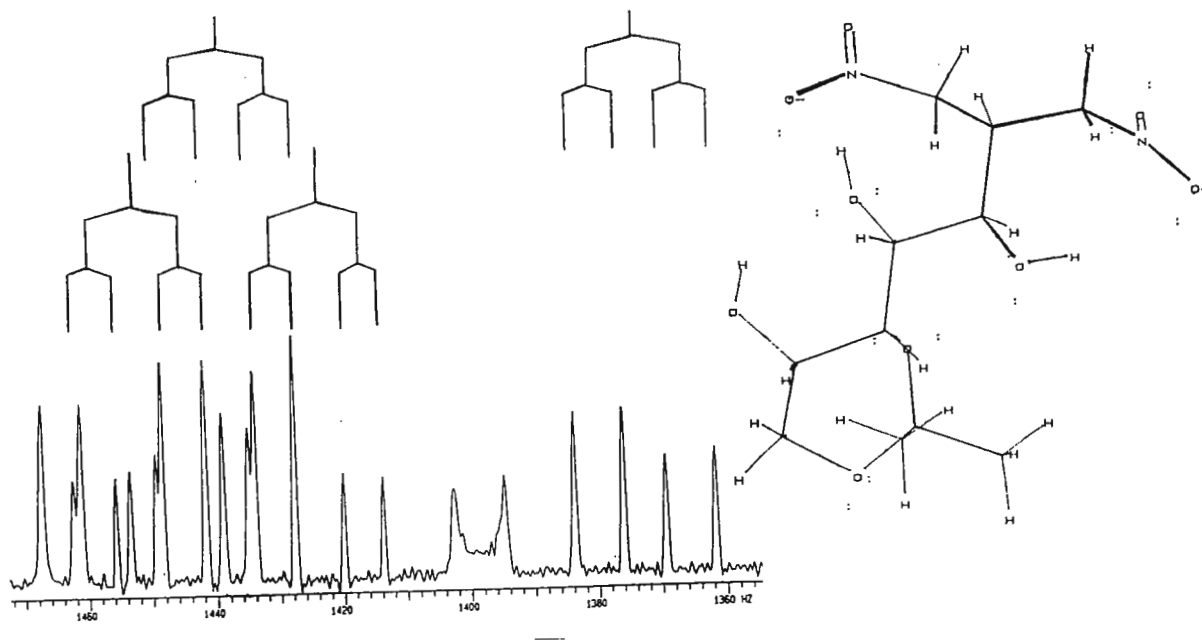


Nonequivalent Methylenes in 1-Deoxy-1,1-di(nitromethyl) hexitols

M. J. Minch, P. Gross and K. Drew

Department of Chemistry
University of the Pacific
Stockton, CA 95211

As part of our ongoing study of proton coupling in conformationally constrained carbohydrates, we have determined the 300 MHz ^1H NMR spectra of 1-Deoxy-4,6-O-isopropylidene-1,1-di(nitromethyl)-D-mannitol and 4,6-O-Benzylidene-1-deoxy-1,1-di(nitromethyl)-D-glucitol. The spectrum of the N-methylene proton region of the former (shown below) indicates that all four N-methylene protons are not equivalent and appear as partly superimposed four line patterns (16 lines total). Hindered rotation about the C(1)-C(2) and C(2)-C(3) bonds and different average orientations of the two nitro group planes with respect to their methylene proton pairs account for this nonequivalence. Molecular mechanics modeling (MM2) predicts an optimal conformation with these features if hydrogen bonding between sugar hydroxyls and the nitro groups is involved. The spectrum and a view of an MM2 optimized geometry are given below.



(received 12/12/89)

December, 1989

To All NMRI Customers:

Two events have occurred this month reflecting the importance of you, our customers, in the development thus far of New Methods Research, Inc., and also anticipating the next levels of development for **NMRI**, as a technology leader in spectroscopic and MRI data processing and analysis.

The first announcement appears in the December issue of INC magazine: **NMRI** is named to the **INC 500** list of fastest growing private corporations in the U.S.A., **NMRI** is listed at #140 for 1989. This achievement reflects **NMRI's** strong commitment to develop and support its growing International user community.

The second announcement which we can make at this time, is that there has been a major investment in New Methods Research, Inc., with a majority of the company's equity now being held by Digital Design Inc., the U.S. subsidiary of Digital Design S.A., a French company with offices also in several western European countries.

The investment and acquisition of a controlling interest of **NMRI** stock by Digital Design, will provide our company with the financial and management resources to move forward to the next levels of corporate development. This will include significant advances in our technological and support base in the U.S.A., and especially Europe, and in **NMRI's** ability to stay at the forefront of laboratory data processing for the chemical and biomedical communities. Digital Design, S.A. is a European company with two main product lines: computer workstation peripherals, and image processing systems for biomedical and other applications.

No major changes are envisioned for **NMRI's** near-term product plans; the acquisition by the Digital Design group should strengthen product development across **NMRI's** current markets in advanced analytical and biomedical data processing and laboratory networking. We do anticipate an increased emphasis, already underway this last year at **NMRI**, in the area of MRI data analysis. We also expect accelerated software development in areas such as multi-dimensional NMR spectroscopy and the interface of this technique with biomolecular modeling.

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(800 Customer Support number remains the same)



The University of Alabama at Birmingham
Department of Chemistry
205/934-4747

November 27, 1989
(received 12/7/89)

Professor B. L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 90303

Subject: Design of an Adapter to Use with Jay Young VNMR Valve-equipped NMR Tubes to Monitor Low Temperature Reaction Systems

Dear Dr. Shapiro:

For the past several years we have been utilizing multinuclear NMR to follow the course of reaction systems over a wide temperature range (-95°C to room temperature). This research involves the reaction of extremely air sensitive compounds that must be handled in an inert atmosphere dry-lab and a high vacuum line. Since the reactions are carried out in the NMR tube and make use of precisely measured stoichiometric amounts of reactants, nonconventional experimental protocols had to be developed. For some time, the typical procedure involved loading in the dry-lab the NMR tube, which was custom-equipped with a greaseless stopcock, with the measured quantity of the less volatile reactant and toluene- d_8 as the solvent. The tube was attached to the vacuum line and degassed, the stoichiometric quantity of the more volatile reactant was distilled into the tube, which was maintained at -115°C , and the NMR tube was then sealed with a torch. This was usually a very time consuming procedure due to the variation in the volatility of the more volatile reactant. Due to the thin walls of the NMR tubes sealing was difficult and some experiments were lost due to breakage of the seals. Furthermore, the NMR tubes had to be refitted with greaseless stopcocks and when the seals were not rotationally symmetric sample spinning was adversely affected.

With the advent of the J. Young VNMR valve-equipped tubes, the manipulation of the air and moisture sensitive reaction systems became much easier. The vacuum line transfer of the more volatile reactant component still consumed considerable time until a septum adapter (See Figure) was designed for use with the J. Young valve. The greaseless joint (A) attaches directly to the high vacuum line and the standard glass tube (B), which is supplied with each valve, allows the valve to be plugged directly into the adapter. The sidearm is equipped with a rubber serum stopper (C) [Fisher 03 225 5] for direct injection of a premeasured quantity of a reactant solution.

Once attached to the vacuum line, the adapter and the NMR tube

that is loaded with a toluene- d_8 solution of one of the reactants is degassed at -115°C (ethanol/liquid nitrogen slush). The valve is then closed and the adapter region is brought to atmospheric pressure by backfilling with prepurified nitrogen. The stoichiometric amount of the other reactant solution, which is contained in a hypodermic syringe with its needle point plunged into a rubber stopper, is then injected through the serum stopper into the void region of the adapter. After removing the syringe needle, the valve is slowly opened to a position that will permit a trickle of solution to run down the -115°C cooled walls of the tube and into the other reactant solution to give a reaction system that can be monitored with time and selected temperature intervals above -115°C . After the addition is complete, the tube is again degassed and the valve is closed. The tube is detached from the vacuum line and transferred to the pre-cooled probe of the spectrometer in a Dewar.

The entire time from dri-lab to spectrometer is now only about 30 minutes as compared to 2 to 4 hours with the previous transfer system. We have found that this protocol works extremely well for reactant systems containing aluminum and gallium alkyls, boranes, and aminoarsines. This protocol can be readily modified to other solvent systems and temperature ranges.

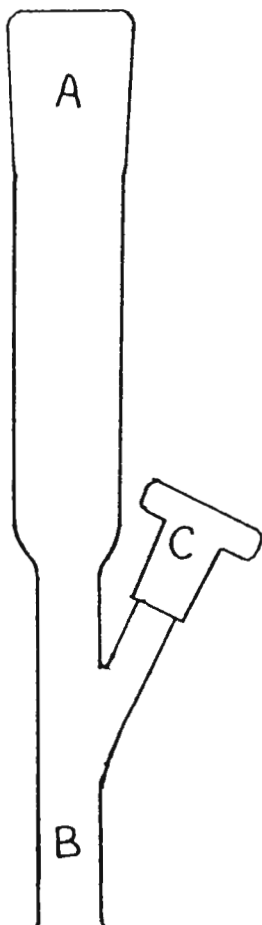
Sincerely yours,

Charles L. Watkins

Charles L. Watkins
Professor

Larry K. Krannich

Larry K. Krannich
Professor and Chairman



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SANTA BARBARA • SANTA CRUZ

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

DEPARTMENT OF CHEMISTRY AND BIOCHEMISTRY
 405 HILGARD AVENUE
 LOS ANGELES, CALIFORNIA 90024-1569

December 8, 1989
 (received 12/11/89)

Intramolecular Transverse Cross-Relaxation in Nearly-Like Spins

Dear Barry:

Non-Lorentzian line shapes resulting from *intramolecular transverse cross-relaxation* between two spins in the liquid state are evident in the ^1H NMR spectra of sodium *p*-chlorobenzenesulfonate in viscoelastic micellar solutions. This appears to be the first experimental confirmation of line-shape effects predicted by R. M. Lynden-Bell [*Prog. NMR Spectrosc.* **2**, 163 (1964)] and by K. V. Vasavada and J. I. Kaplan [*J. Magn. Reson.* **62**, 37 (1985)]. The latter workers made a theoretical investigation of the effects of cross relaxation in a two-spin system (like, nearly-like and unlike) relaxed only by intramolecular dipole-dipole relaxation. They calculated line shapes for a physically realizable two-spin system undergoing slow molecular tumbling, i.e., for $\omega\tau \gg 1$, but with $1/\tau \ll$ dipolar line width, but they did not present any experimental data. When the lines strongly overlap a curious sharp dip occurs in the center of the calculated spectrum. Such a line shape effect has not previously been reported in an experimental spectrum to our knowledge (a more general analysis has been presented in a very recent paper by N. P. Benéfits, D. J. Schneider, and J. H. Freed [*J. Magn. Reson.* **85**, 275-293 (1989)]).

We have taken advantage of the unusual properties of viscoelastic micellar solutions, as described previously by us [*J. Am. Chem. Soc.* **108**, 7102 (1986)]. Small molecules and ions, such as the *p*-chlorobenzenesulfonate ion, induce the normally spherical micelles produced by 5 mM hexadecyltrimethylammonium bromide to change into extremely long cylindrical micelles by being incorporated into the surface of the micelle, where they undergo highly anisotropic motion, as shown in the Figure below. Rotation about the 1,4 carbons of the aromatic ring is expected to be quite fast, as is probably also lateral diffusion in the micelle. On the other hand, rotation perpendicular to the 1,4 direction is expected to be very slow and thus the pairs of protons that are ortho to one another on each side of the ring, which are unaffected by the fast motion along the 1,4 axis, give rise to very broad lines by virtue of intramolecular dipole-dipole relaxation. This is the case even though these nuclei are relatively far (2.4 Å) apart and would normally be relaxed largely by intermolecular dipole-dipole relaxation via the protons of the surrounding medium. The four protons in I can be considered to a first approximation to form two independent proton pairs as far as relaxation effects are concerned. Because dipole-dipole relaxation is proportional to the inverse sixth power of the distance and the separation between ortho and meta proton pairs are 2.4 and 4.2 Å, respectively, the relaxation rate between ortho proton pairs is about thirty times greater than that between meta proton pairs, a ratio that is made even higher by the fast rotation of the aromatic ring about the 1-4 direction. The relaxation rate arising from para proton pairs is therefore clearly negligible.

The 200 MHz ^1H NMR spectrum of I shows a characteristic dip between the overlapped broad lines of the ortho and meta protons. Theoretical calculations, based on the parameters obtained from the analysis of a 500 MHz spectrum of I in the same solution, gives a calculated line shape that is superposable on the experimental line shape. Details will appear in *J. Magn. Reson.*

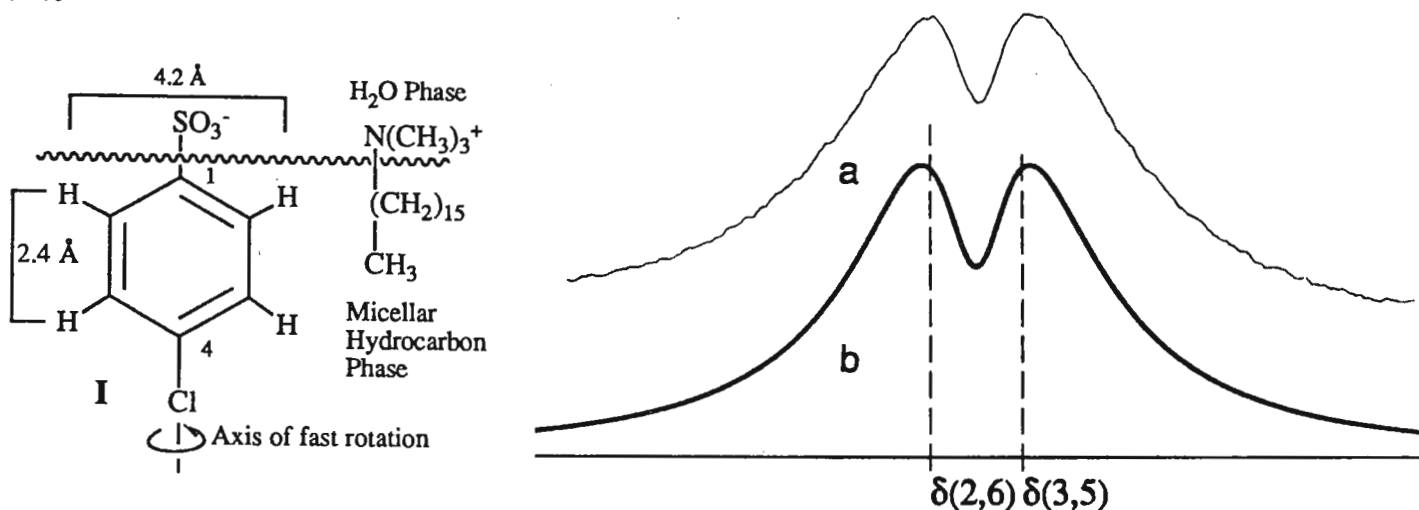


Figure Experimental (a) and calculated (b) 200 MHz ^1H NMR spectrum of the aromatic protons of the *p*-chlorobenzenesulfonate ion present in rod-like micelles in a viscoelastic solution. The parameters for the calculated spectrum are derived from a fitting of the 500 MHz spectrum, which consists of two well separated broad lines) and are as follows: $\delta(2,6) - \delta(3,5) = 0.420$ ppm, $1/T_2^I = 1/T_2^S = 487 \text{ s}^{-1}$, $1/T_2^{IS} = 390 \text{ s}^{-1}$, $1/T_2^{\text{other}} = 40 \text{ s}^{-1}$. The experimental spectrum was obtained with a line broadening of 5 Hz, which gives a contribution to $1/T_2^{\text{other}}$ of 15.7 s^{-1} .

Please consider this letter as meeting Jane Strouse's contribution requirements.

Sincerely yours

Frank

Dan

Frank A. L. Anet and Dan J. O'Leary

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Half Length (mm)	350	275	570	396	460	735	575
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December 8, 1989
(received 12/12/89)

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

Speciation of Quadrupolar Nuclei in Fuels; Position Available

Dear Barry:

Differentiating nitrogen compounds by direct NMR observation and quantitation is potentially useful for following hydrodenitrification (HDN) and fluid catalytic cracking processes, catalyst poisoning, storage stability of fuels, etc. Direct observation of sulfur and oxygen would also be useful.

Direct ^{15}N spectroscopy of these complicated mixtures, using chromatographic concentration, relaxation reagents, and large-volume, sideways probe design, was not sufficiently sensitive to be of practical use on our 7 Tesla instrument. Instead, we have found that careful attention to minimizing microviscosity by choice of solvent and temperature, combined with probe design and

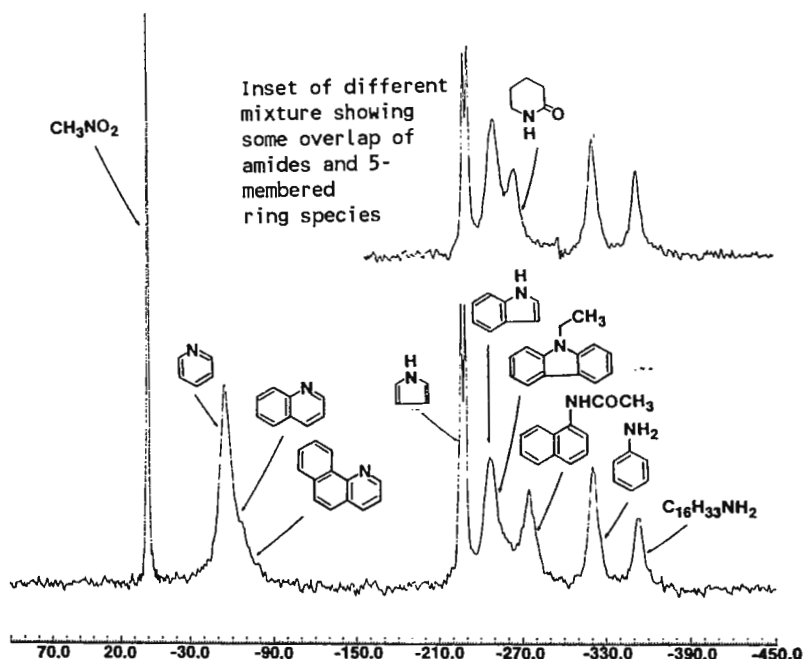
pulse techniques to limit spectral artifacts, can provide ^{14}N spectra which

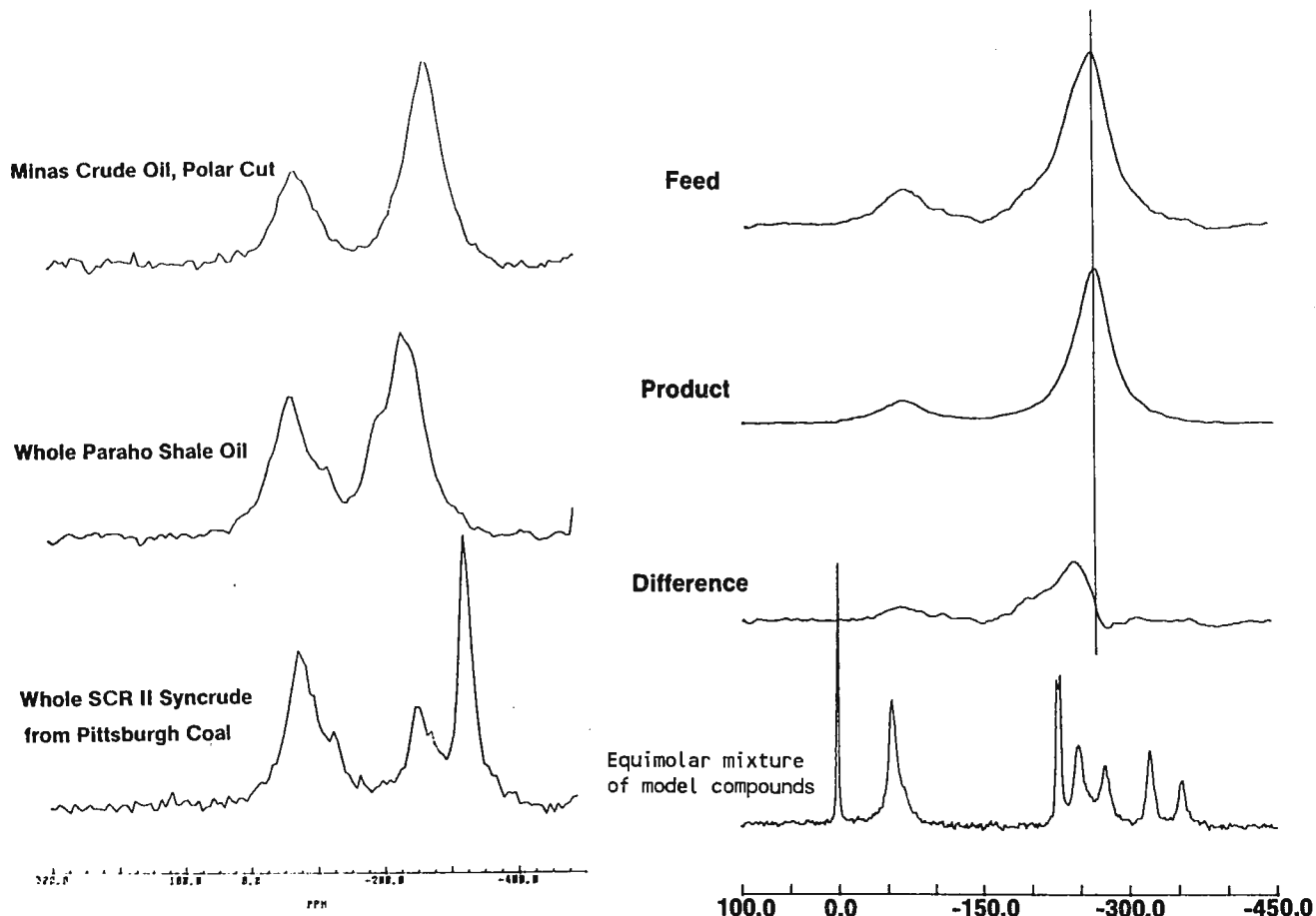
differentiate and quantify nitrogen types. Figure 1 is of a mixture of model compounds. Note especially the separation of six- and five-membered ring nitrogen species, and the further differentiation of these according to number of attached aromatic rings (planar sheet size).

Figure 2 shows how a variety of nitrogen-containing fuel liquids give spectra showing unique nitrogen types. Note especially the difference in carbazoles in crude oil vs. shale oil, and the

large amount of amine nitrogen in SCR II Coal Oil, not seen in the other two materials. Figure 3 demonstrates the method's use in process modelling. The difference spectrum resulting from the subtraction of product from feed spectra (ASTM D2007 polar cuts, intensities adjusted using total nitrogen by elemental analysis) shows that primarily five-membered single-ring types (pyrroles) are removed, with little effect of the HDN process on carbazoles.

^{14}N NMR OF EQUIMOLAR MIXTURE OF MODEL COMPOUNDS





Similar efforts with ^{33}S proved impractical at this field strength, but we expect improvement of all aspects of this work with installation of a 12 Tesla instrument in a few months.

Sincerely,

D. M. Wilson
D. M. Wilson

POSITION AVAILABLE - NMR SPECTROSCOPIST

Chevron Research Company has an immediate opening for an NMR spectroscopist in our Process Research Division at our research center in Richmond, California.

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Candidates should be U.S. citizens or permanent residents. Chevron is an equal opportunity employer. Interested parties should contact: Jack A. Duisman, Chevron Research Company, 100 Chevron Way, Richmond, California 94802, (415) 620-4318

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December 12, 1989
(received 12/16/89)

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

Dear Barry:

NMR Spectroscopy of Perfused Cells

In order to monitor metabolism of cells and their response to drug treatment we have set up a perfusion system for spectroscopic measurements. The cell perfusion system is similar in design to that developed by Dr. Jack Cohen and his coworkers at NIH (1). In this system, cells embedded in an agarose matrix are perfused in a 10 mm NMR tube with appropriate medium. A schematic drawing of our agarose thread making apparatus is shown in Figure 1. In our system the cells are mixed with low temperature agarose in a glass tube with ball and socket joint to ensure perfect seal necessary for extruding the threads. The whole apparatus, including the teflon tubings and the glass tube with joints, is autoclaved each time before the experiment. In addition pumped air passes through a sterile filter before reaching the cell-gel mixture. We would like to thank Jack Cohen and Pat Faustino of his laboratory for their help during the set up of our perfusion apparatus.

Using this system we have collected ^{31}P NMR spectra of human colon carcinoma (HT29) cells on our JEOL 400 MHz spectrometer using a 10 mm probe ($^1\text{H}[^{15}\text{N}-^{31}\text{P}]$) built by Dr. Craig Bradley of Cryomagnets. A typical ^{31}P NMR spectrum (1500 scans with 60° pulse and 3.0 s pulse delay) obtained with 3×10^7 cells at 37° is shown in Figure 2. The characteristic ^{31}P peaks are labeled on the spectrum. The higher P_i peak is due to the presence of inorganic phosphate in the medium. A tripan blue check performed immediately after data collection shows 90-97% cell viability.

Please change our subscription of TAMU newsletter from Luciano Mueller to Susanta Sarkar's account.

Yours sincerely,


Robert Rycyna


Robert Clark


Susanta Sarkar

Reference:

1. D.L. Foxall, J.S. Cohen and J.B. Mitchell, Exp. Cell, Res., 154, 521-529, 1984.

SK&F

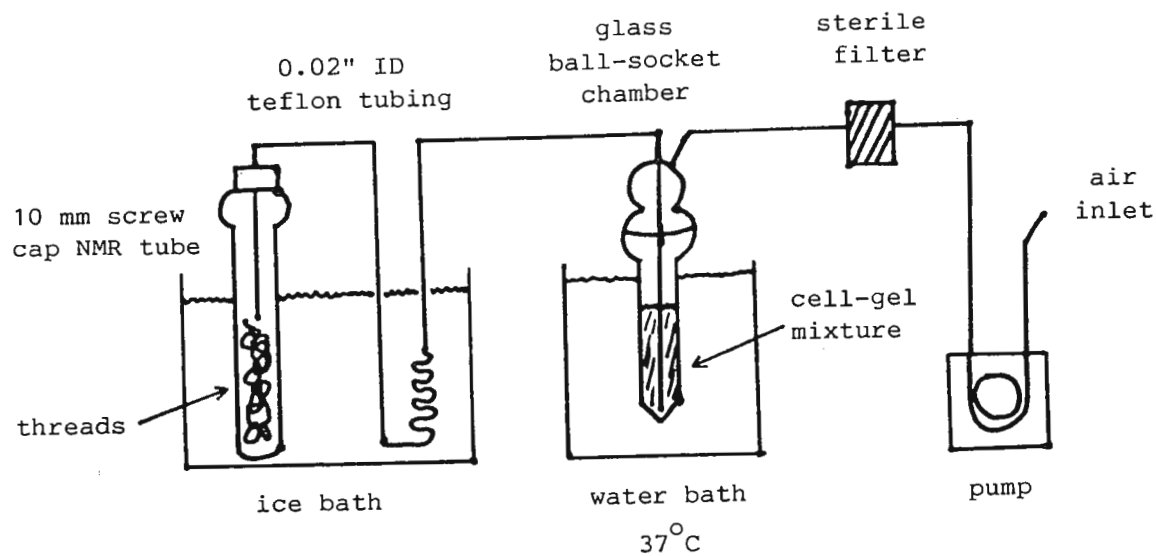


Figure 1. Agarose thread-making apparatus

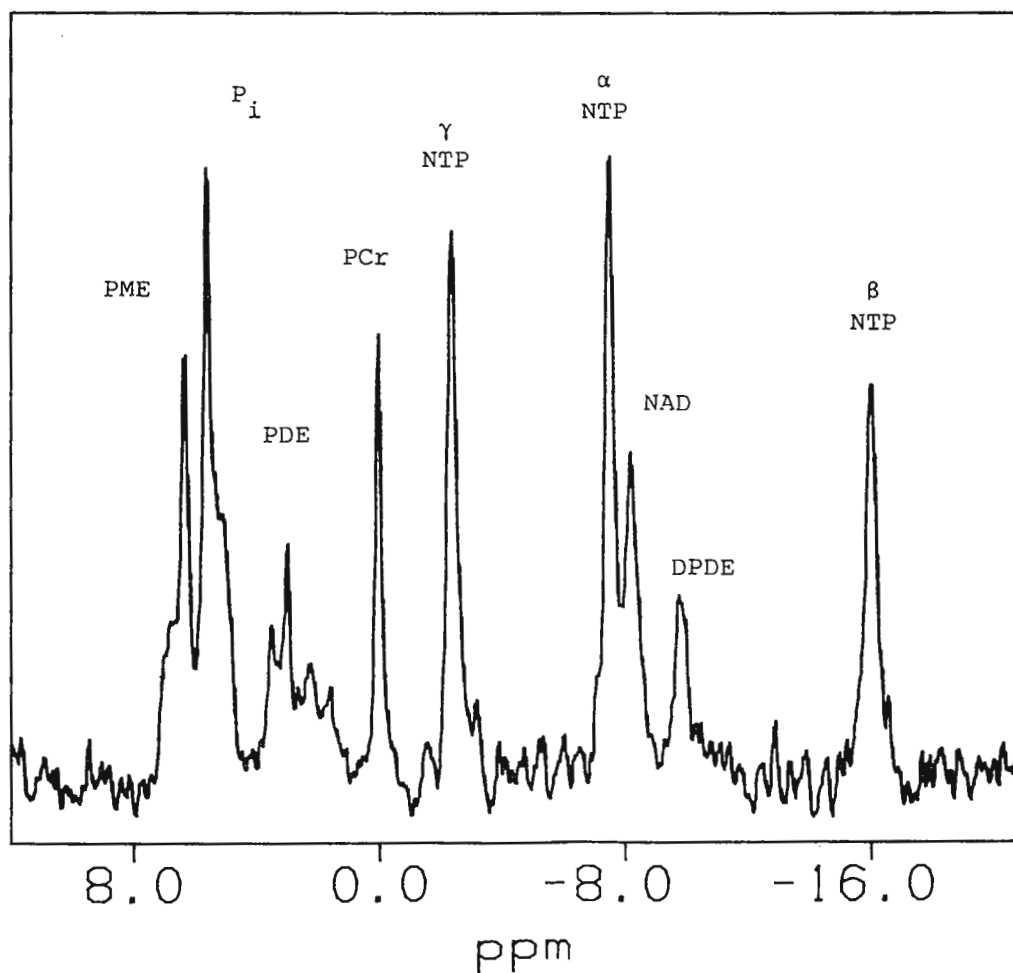


Figure 2. 161.92 MHz ^{31}P NMR spectrum of HT29 cells.

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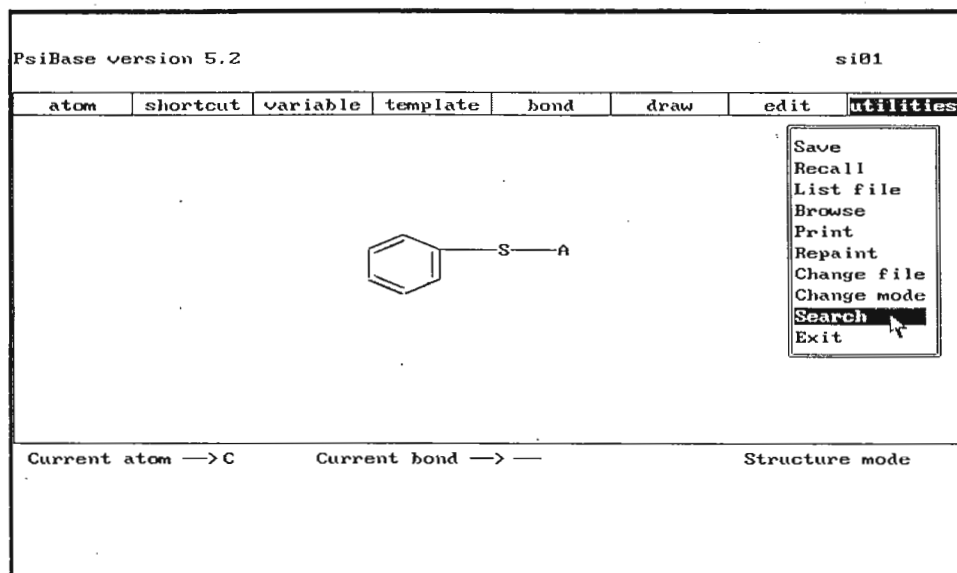
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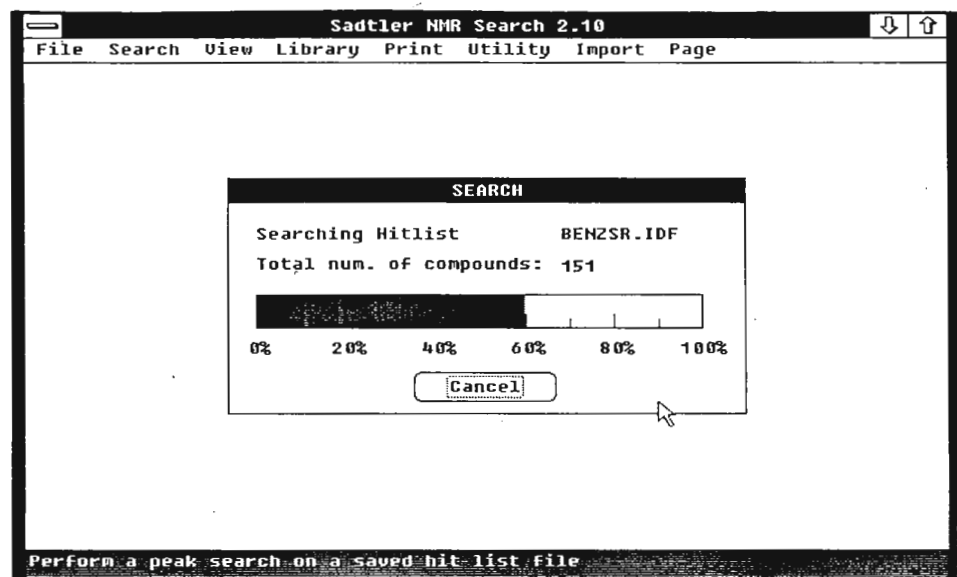
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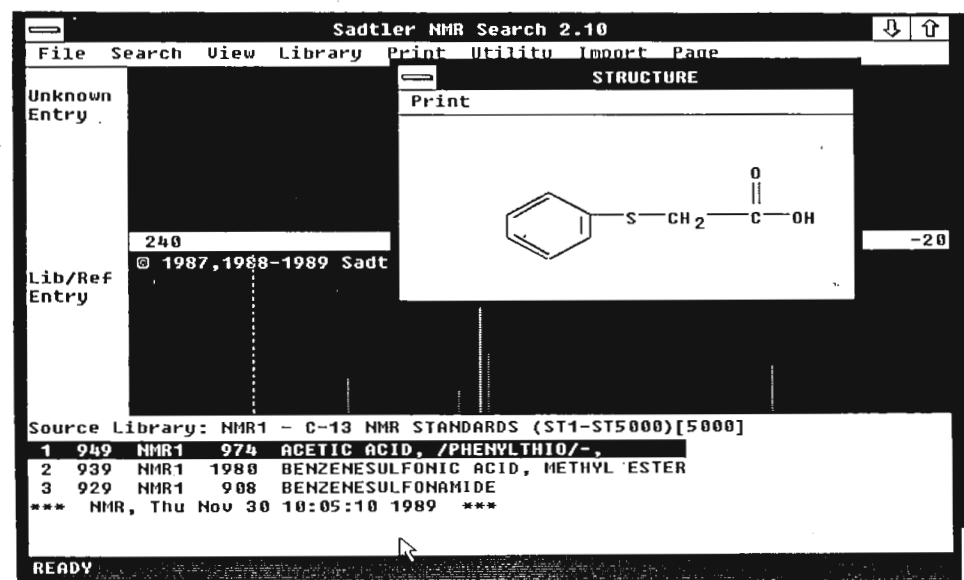
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**Shell Research B.V.**

Professor B.L. Shapiro
TAMU NMR Newsletter
966 Elsenore Court
PALO ALTO, CA94203
U.S.A.

Uw ref.:

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IDC

Amsterdam, 24 November 1989
Badhuisweg 3 (received 11/27/89)
Tel. via telefoniste (020) 309111
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Hr/Mw

QUANTITATIVE NMR IMAGING

(or, 'exactly how many worms have you got in that can?')

Dear Barry,

Here, at KSLA, applications of imaging spectroscopy demand quantitatively rigorous methods. Having taken great pains to produce data sets with 'guaranteed' integrity. We are now turning our attention to post-acquisition process methodology.

Common to all fields of imaging spectroscopy, the extraction of meaningful information from a competent data set depends largely upon the interrogative methods available to the analyst (1). Commonly, the data is composed of a 2-, 3-, or 4-dimensional matrix, where one or more dimension(s) provide contrast, designed by the experiment to be sensitive to a changing physical property. The subsequent manipulations which create access to the data (i.e. transform functions, linear regression, exponential multiplication, etc.) can be general in nature, irrespective of the data source. However, interrogation of the contrast function is specific (i.e. mathematical morphology programs, deconvolution of exponential time constants, etc.), since this gives physical meaning to the contents of the voxel.

All of this is obvious, and would lead one to believe that currently available software routines may provide all the required capabilities, with only small modification to recognise intrinsic NMR parameters. The object, then, would seem to be one of two paths:

- a) find the most versatile 'image analysis' package, in which an NMR 'tool kit' can be installed, or
- b) find the best 'NMR-dedicated' package, in which an image analysis 'tool kit' can be installed.

/2

Correspondentie:
Koninklijke/Shell-Laboratorium, Amsterdam
Postbus 3003
1003 AA Amsterdam

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d.d.

24 November 1989

Pag. 2

and, of course, convince someone(else) to do it.

Now, before a number of good people get their baseball (or cricket?) bats out of the closet, it should be acknowledged that there are some excellent packages being developed at the moment. The purpose here (in the spirit of enlightenment) is to open a forum in which other newsletter contributors can relate their experiences (and needs), and in so doing alleviate their own 'DREADED PINK REMINDER' problems. (Does this qualify as a public service announcement?).

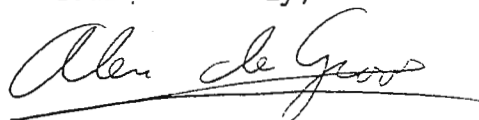
The technical content of this note comes in the form of the image shown below in figure 1. It represents a simple example of the type of problem requiring the interrogative capabilities described above. What one sees is a proton NMR image of a porous bed, with two fluids resident in the pores and the respective reference cells. The task is not only to extract the relative areas of each constituent and their NMR characteristics, but also, as the engineers say, to have the numbers add up to '1.0'. (After all, it always does in the simulations!).

We have been attacking this sort of problem with a number of packages, which we will show in a future installment, and we would be interested to hear from other users with similar quantitative experiences.

Yours sincerely,



(G.J. Nesbitt),



(A. de Groot)

Reference:

1. MULTIDIMENSIONAL BIOMEDICAL IMAGE DISPLAY AND ANALYSIS IN THE BIOTECHNOLOGY COMPUTER RESOURCE AT THE MAYO CLINIC, R.A. Robb, Machine vision and application (1988) 1; 75-96.

Figure 1: Proton image of two fluids on porous media with reference cells.



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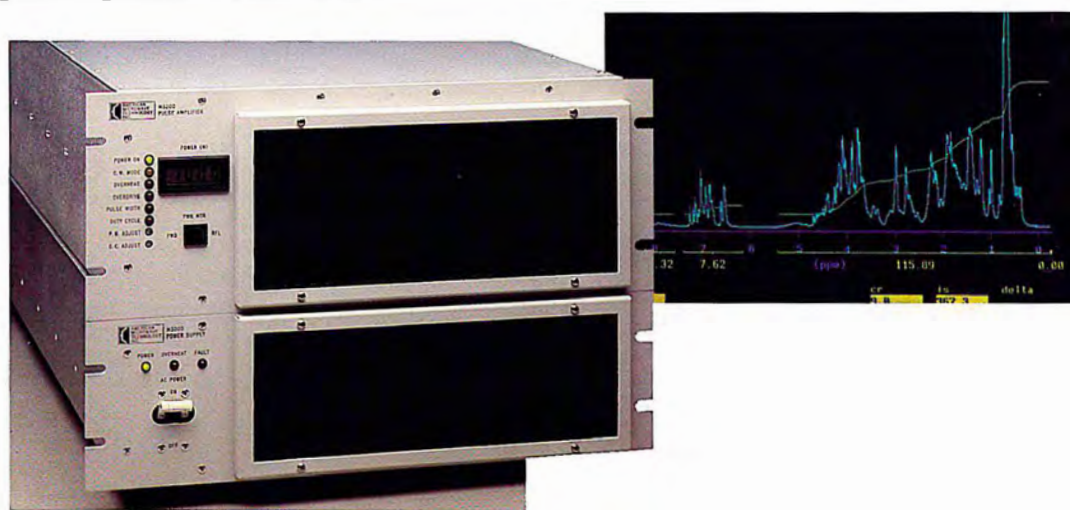
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BITNET: N130006@UNIVSCVM
December 12, 1989
(received 12/15/89)Title: "Solid State ^{95}Mo NMR with Cross Polarization"

Dear Barry;

I would like to communicate some of our recent work on the solid state nmr of ^{95}Mo . This work is being performed by a graduate student, Mr. John Edwards. Our group has been interested in various aspects of surface chemistry for several years and in collaboration with Professor Rick Adams of our department, we have begun to investigate the molybdate/ γ -alumina systems. These systems form the basis for hydrodesulfurization (HDS) catalysts. Our initial efforts have been submitted to JACS and Inorg. Chem. As a corollary to this work and our previous work on ^{27}Al , we wanted to establish the feasibility of performing cross polarization experiments with ^{95}Mo . The enclosed figure summarizes our work on ammonium heptamolybdate. The first figure (top) illustrates the static Hartmann-Hahn match spectrum. Note that the width is ~ 5 kHz wide. This is comparable to the width we obtained for ^{27}Al (H.D. Morris, S. Bank, and P.D. Ellis, J. Phys. Chem., in press). The next figure (lower left) demonstrates the buildup of magnetization vs. contact time. The growth rate is slow, with a $T_{1\rho}^{\text{H}}$ of ~ 22 ms. This is matched with a relatively long $T_{1\rho}^{\text{H}}$ of ~ 23 ms. The corresponding value of $T_{1\rho}^{\text{Mo}}$ is nearly 100 ms as depicted in the third figure (lower right). This is longest $T_{1\rho}$ we have measured. The $T_{1\rho}^{\text{Mo}}$ in an analogous compound, $[(\text{nBu})_4\text{N}]_2\text{Mo}_2\text{O}_7$, is ~ 50 ms.

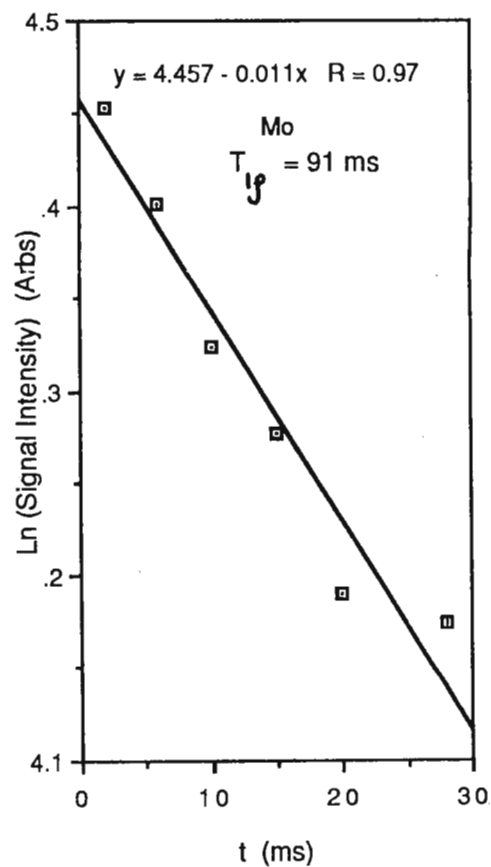
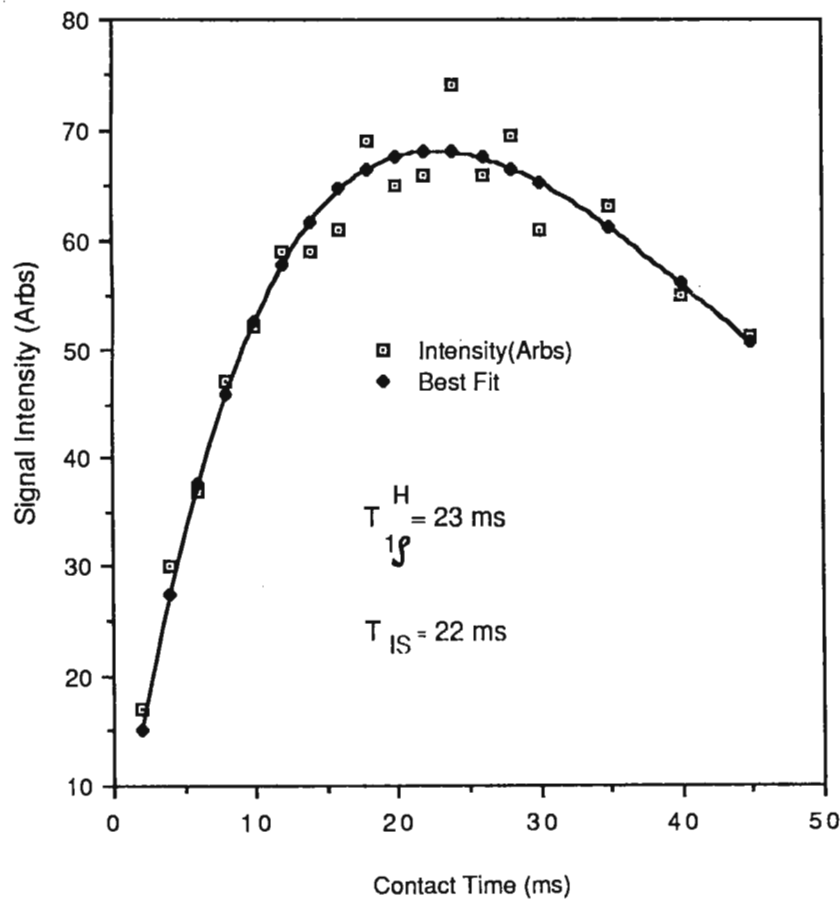
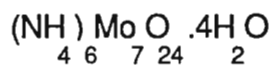
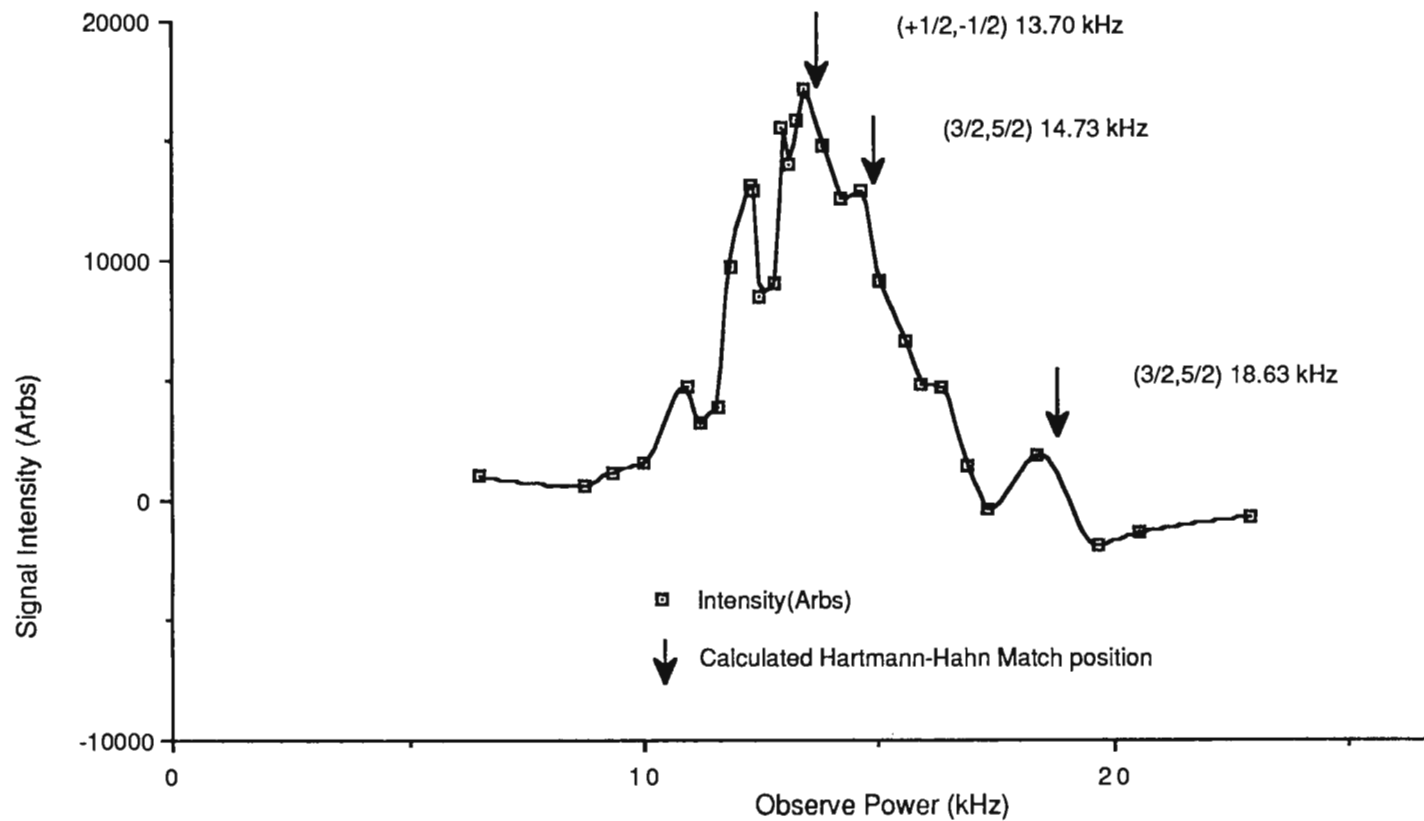
Clearly, the fact that we are observing the cross polarization signals at all is due to the long values of $T_{1\rho}$ for ^1H and ^{95}Mo . Paramagnetic impurities would have a tendency to short circuit $T_{1\rho}^{\text{H}}$. Therefore, we do not expect this approach to work in HDS systems, unless there is a very low concentration of surface paramagnetics. Details of this work will be submitted for publication.

Sincerely,

J.C. Edwards
John C. Edwards

Paul D. Ellis
Paul D. Ellis

376-60





CENTRE FOR NUCLEAR MAGNETIC RESONANCE

UNIVERSITY OF WARWICK
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RD/pmk

December 1, 1989
(received 12/12/89)

Dr. B.L. Shapiro,
TAMU NMR Newsletter,
966 Elsinore Court,
Palo Alto CA94303, USA.

Dear Dr. Shapiro,

We have been investigating low γ spin $I > \frac{1}{2}$ nuclei in the perovskite (ABO_3) structure. Several perovskites are closely cubic giving narrow NMR lines even on normally "difficult" nuclei. Examples are shown in Fig.1 for ^{47}Ti , ^{49}Ti and ^{89}Sr in SrTiO_3 and for ^{91}Zr and ^{135}Ba in BaZrO_3 . The normal reference for Ti is TiCl_4 which is not a very pleasant material to work with. We suggest that SrTiO_3 is used as a secondary reference for titanium since the static ^{49}Ti linewidth is ~ 150 Hz (decreasing to ~ 50 Hz upon spinning at ≥ 50 Hz), Ti is short and strantium titanate is readily available. The shift is 1765.7 ± 0.4 ppm relative to TiCl_4 . Similarly BaZrO_3 would be a good secondary reference for ^{91}Zr ($E = 9.299187$ MHz).

Please credit this note to Dr. O W Howarth's account.

Yours sincerely,

R Dupree and A Varanasi

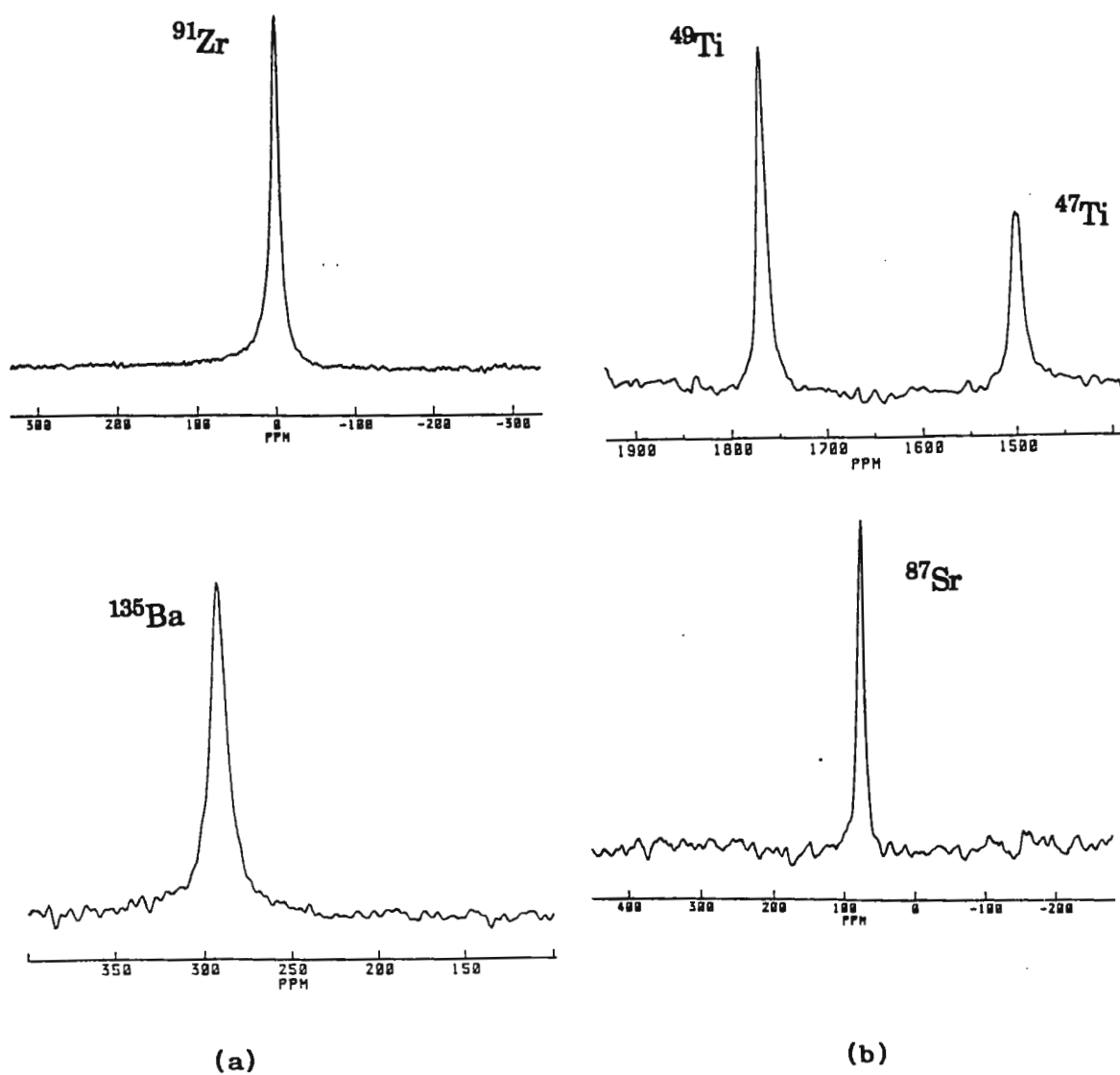


Figure 1 (a) ^{91}Zr (upper trace) and ^{135}Ba (lower trace) in BaZrO_3
 (b) ^{49}Ti and ^{47}Ti (upper trace) and ^{87}Sr (lower trace) in SrTiO_3

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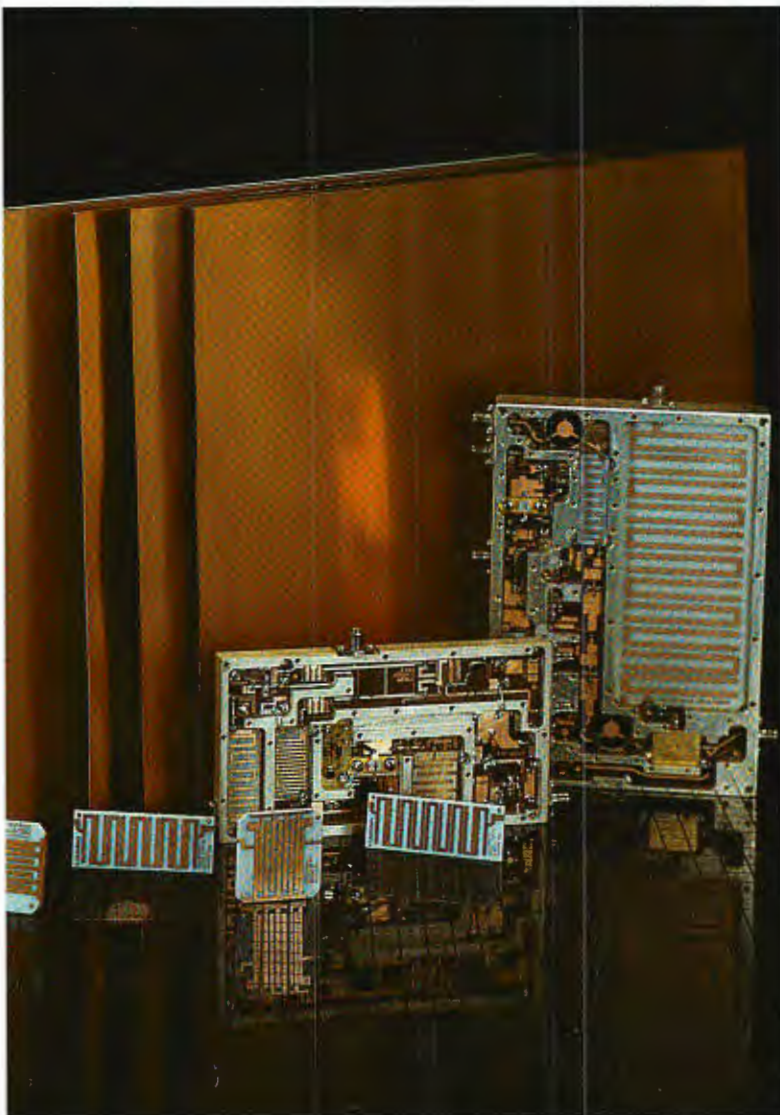
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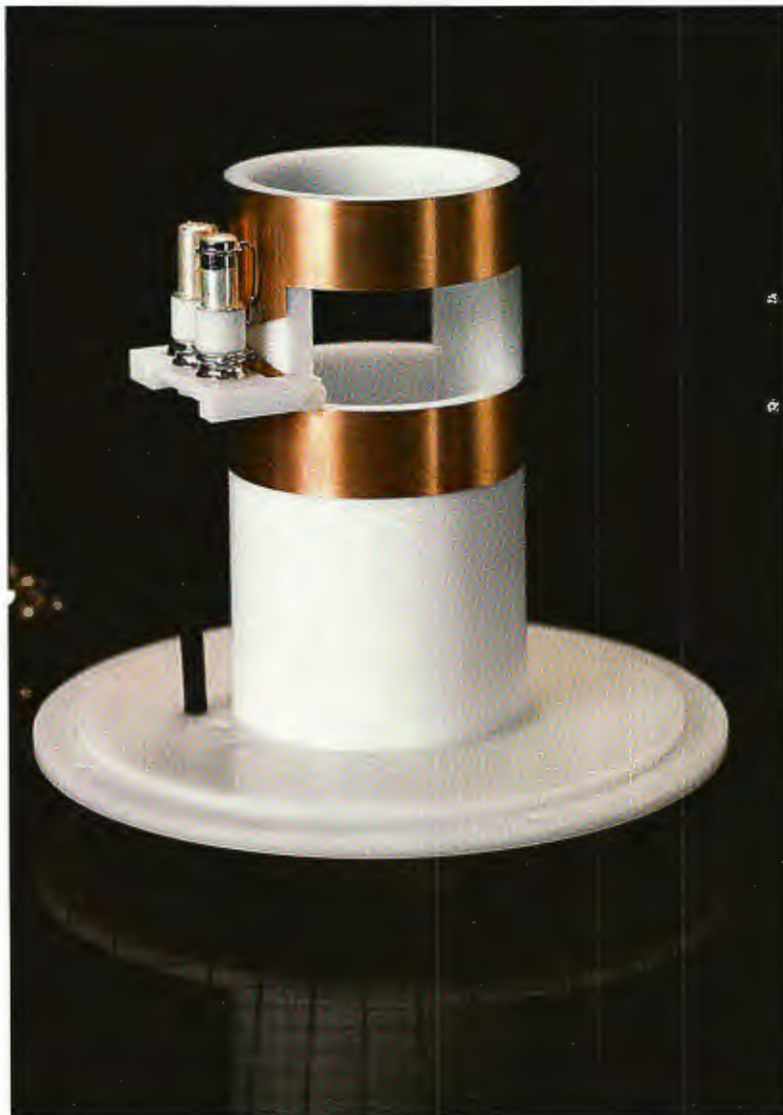
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November 8, 1989 (received 12/3/89)

Professor Barry Shapiro
966 Elsinore Ct.
Palo Alto, CA 94303

Dear Prof. Shapiro:

ZQC 2D NMR Spectra From 2D NOESY Data

Because Zero-Quantum-Coherences (ZQCs) are insensitive to magnetic field inhomogeneities,¹ several groups have been examining methods for obtaining ZQC correlation 2D NMR spectra.² I have been investigating the possibility of extracting ZQC 2D correlation spectra from phase-sensitive NOE 2D NMR (NOESY) data. Olejniczak and coworkers³ demonstrated that off-diagonal peaks in NOESY spectra are composed of ZQC and cross-relaxation components 90° out of phase with each other. I wondered if modifying the post-acquisition processing of the time-domain NOESY data could be used to obtain ZQC correlated 2D spectra.

Indeed, very good quality ZQC correlated 2D NMR spectra can be obtained by adjusting the phase parameters applied during phase calculations along f_1 and f_2 using the method of States et al.⁴ If 90° is subtracted from the zero-order phase term during f_2 -domain phase calculations and is added to the zero-order phase term during f_1 -domain phase calculations, the ZQC correlated spectrum shown in Figure 1 is obtained for cellobiose octaacetate dissolved in chloroform-d. A COSY spectrum of comparable quality to the spectrum in Figure 1, (along with resonance assignments) has been presented elsewhere.⁵ The data were collected and processed on a JEOL Model GX-400 NMR spectrometer by using two 1024 x 512 data matrices using the phase-sensitive NOESY pulse sequence, VPHNOE, with $\tau_m = 0$ msec. Using longer mixing times gives less intense ZQC correlations due to relaxation. Plotting of the spectra was accomplished with a modified version of Dennis Hare's FTNMR

program running on a VAX 8800. That the off-diagonal resonances observed in Figure 1 do arise from ZQC can be demonstrated by implementing the incremented mixing time technique of Macura et al.⁶ in the NOESY pulse sequence. This modification results in correlations due to ZQC being shifted along ω_1 in positive and negative directions. Figure 2 shows that the NOESY experiment using the incremented mixing time technique does in fact lead to displacement of the off-diagonal peaks along ω_1 as expected.

The advantages of this approach for obtaining ZQC correlated 2D NMR spectra are that the ZQC correlated spectrum is available without the need to develop a special pulse sequence, can be obtained as the result of a 2D NMR experiment already in wide use, is presented in a COSY-format, and provides information comparable to that obtained from a COSY experiment. In those cases where only limited sample is available and very long data collection times could be required to obtain both a COSY spectrum and an NOESY spectrum, only an NOESY data set needs to be collected, then processed either by the method of States et al. to give the NOESY spectrum or by the method described here to give the ZQC correlated spectrum, thus significantly reducing the time required for data collection. Connectivities usually extracted from the COSY spectrum can be extracted from the ZQC correlated spectrum while spacial proximities are analyzed from the NOESY spectrum if the NOESY spectrum was acquired at a suitable non-zero mixing time.

-
1. W. P. Aue, E. Bartholdi and R. R. Ernst, J. Chem. Phys. 64, 2229 (1976); A. A. Maudsley, A. Wokaun and R. R. Ernst, Chem. Phys. Lett. 55, 9 (1978)
 2. Cavanagh and J. Keeler, J. Magn. Reson. 77, 612 (1988); J. Jeener, B. H. Meier, P. Bachmann and R. R. Ernst, J. Chem. Phys. 71, 4546 (1976); L. D. Hall and T. J. Norwood, J. Magn. Reson. 76, 241 (1988)
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 4. D. J. States, R. A. Haberkorn and D. J. Ruben, J. Magn. Reson. 46, 286 (1982)
 5. C. M. Buchanan, J. A. Hyatt and D. W. Lowman, Carbohydrate Research 177, 228 (1988)
 6. S. Macura, K. Wuthrich and R. R. Ernst, J. Magn. Reson. 46, 269 (1982)

Sincerely yours,



Douglas W. Lowman
Senior Research Chemist
ECD Research Laboratories
P. O. Box 1972

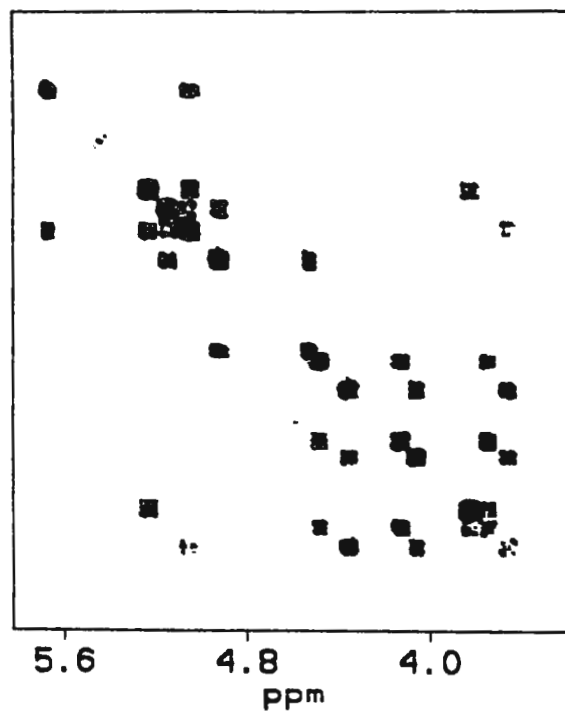


Figure 1. ZQC-Correlated 2D NMR Spectrum

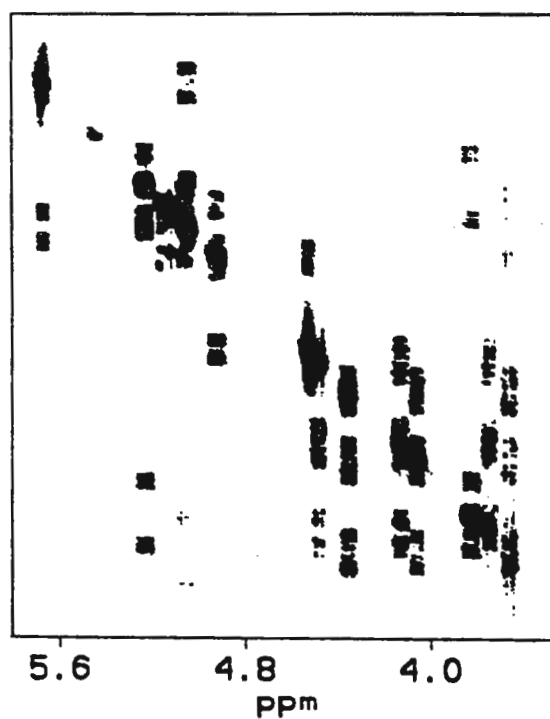


Figure 2. NOESY 2D NMR Spectrum using the Incremented-Mixing Time Technique and Processed in the Manner Described Above

Laboratorium für Physikalische Chemie

Dr. O.W. Sørensen

0430

Universitätstrasse 22

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Telefonzentrale 01/256 22 11

Postadresse:

ETH-Zentrum
8092 ZürichProfessor B.L. Shapiro
966 Elsinore Court
Palo Alto, California 94303
USA

Zürich, November 23, 1989

Dear Professor Shapiro,

The number of pulse sequences in NMR is so large that most people probably have stopped counting them years ago. In this jungle of experiments it is not only necessary to have a quality measure for the pulse sequences but also to know what the theoretical limits of their performance are. In this way one can (ignoring instrumental artifacts) stop searching for improved experiments when the theoretical maximum has been achieved.

A theorem answering exactly this question is now available [1]. Given an arbitrary initial state σ_i and a desired final state A , that should be approached by a unitary transformation X like

$$\sigma_f = X \sigma_i X^{-1} = aA + bB, \quad \text{Tr}\{A^\dagger B\} = 0,$$

the maximum coefficient of A , a^{\max} , is determined by the following expression:

$$a^{\max} = \frac{\text{Tr} \{ \sigma_i^{D_1} A^{D_1} \}}{\text{Tr} \{ A^\dagger A \}}$$

where $\sigma_i^{D_1}$ and A^{D_1} are the respective diagonal forms of the matrices σ_i and A in which the eigenvalues have been ordered in decreasing (or increasing) size. They are obtained by the diagonalizing transformations

$$\sigma_i^D = U \sigma_i U^{-1}, \quad A^D = V A V^{-1},$$

and the ordering transformations

$$\sigma_i^{D_1} = U' \sigma_i^D (U')^{-1}, \quad A^{D_1} = V' A^D (V')^{-1}$$

As a fringe benefit the above theorem also provides the propagator leading to the maximum possible transfer efficiency, namely

$$X = V^{-1} (V')^{-1} U' U.$$

From this propagator it is, however, not always obvious how to design a corresponding pulse sequence. Further work along these lines is planned.

Please credit this letter to Prof. R.R. Ernst's account.

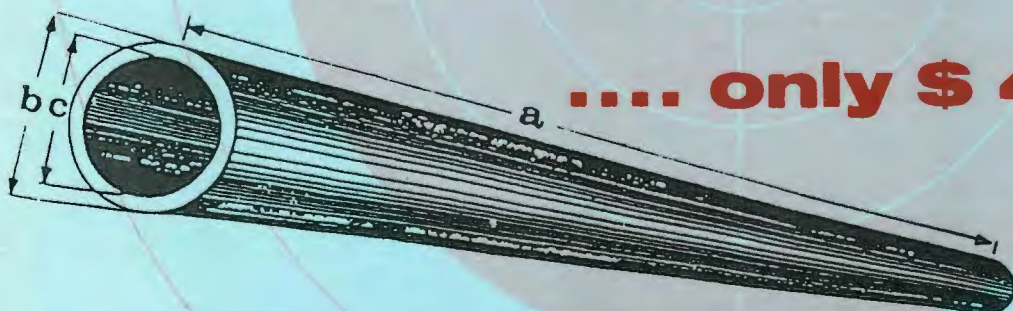
Yours sincerely,


O.W. Sørensen

[1] O.W. Sørensen, Prog. NMR Spectrosc. and J. Magn. Reson., in press.

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

(received 12/16/89)

Title: *In Vivo* ^{31}P NMR and Paramagnetic Contamination.

Dear Dr. Shapiro,

On occasion in the course of our *in vivo* ^{31}P NMR studies of plant root tissues we have noted that spectral lines including our reference peak (HMPA) have been considerably broader, making the S/N less than tolerable (Fig. 1A). We investigated our instrumental parameters and found none at fault. In addition, we checked the source of the plant tissue, the NMR tubes, perfusate and glass wool spacer in the NMR tube but all checked out OK. However, one parameter, the CaCl_2 used to prepare the growth medium (0.1mM) for the 3 day old seedlings was found to be impure. That is, the CaCl_2 used contained low levels of Cr(11.0ppm), Fe(1.0ppm) and Mn(1.0ppm). In general, these levels would not be considered high enough to cause paramagnetic broadening in a spectrum, however, after 3 days of rapid growth of immature root tissue, a significant concentration of metals can build up within the cells. Signal loss seems especially severe in the case of the vacuolar P_i signal (Fig. 1A) at 0.89 ppm since the vacuole is a principal metal ion sequestering compartment [1]. Following 23h of continued perfusion with metal ion free buffer we see that much of the Cr, Fe and Mn can be washed out of the tissue (Fig. 1B) to restore the spectrum to a state in which it is only slightly broader than the spectrum of the control which contains no measurable paramagnetic ions (Fig. 1C).

Obviously, when performing *in vivo* experiments, care should be taken to check your growth medium carefully since a little paramagnetic goes a long way.

Tom Boswell Dominique Rolin Julian Schmidt Mike Kurantz Phil Pfeffer*

Tom Boswell Dominique Rolin Julian Schmidt Mike Kurantz Phil Pfeffer

*Please credit this contribution to my account.

- [1] Kime, M.J., Ratcliffe, R.G. and Loughman, B.C. (1982) J. Exp. Bot. 33 656-669; Pfeffer, P.E., Tu, S.-I., Gerasimowicz, W.V. and Cavanaugh, J.R. (1986) Plant Physiol. 80 77-84.

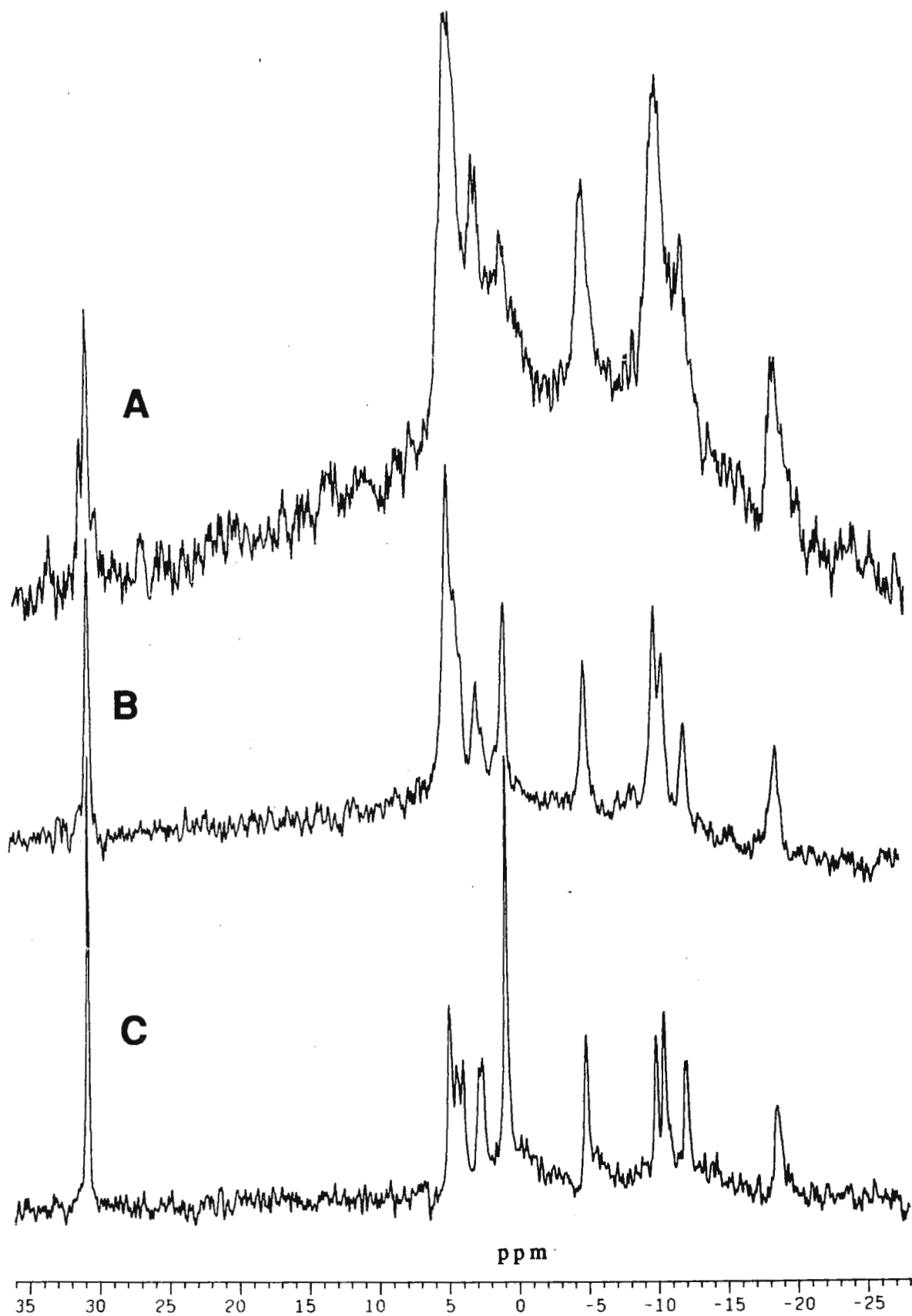


Fig. 1 (A) 161.7MHz ^{31}P spectrum (27 minute accumulation, 10,000 transients) of 700 (3-5mm) maize root tips from seedlings grown 3 days on impure 0.1mM CaCl_2 ; (B) Same as (A) following 23h of perfusion; (C) same as (A) except uncontaminated CaCl_2 was used.

TAMU NMR Newsletter

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B. L. Shapiro
2 January 1990

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* * * * *

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* * * * *

CSI 2T Applications

Shielded Gradients: Theory and Design

NMR imaging and localized spectroscopy depend on the use of pulsed magnetic field gradients. As these techniques have grown more complex, it has become apparent that eddy currents created in the magnet cryostat and other structures by pulsed gradients have become the chief limitation to many sophisticated applications.

Figure 1a illustrates the design problem for unshielded gradients. Figure 1b illustrates the shielded gradient arrangement. Figures 2 and 3 show the contours of constant flux for an unshielded and shielded Z gradient coil, respectively. This demonstrates that, for the shielded gradients, most of the flux has been kept away from the magnet bore.

The dramatic reduction of eddy currents which can be made over the conventional, unshielded gradients is shown in Figures 4a, b. These graphs show frequency as a function of time following the application of a long, constant amplitude gradient pulse which is suddenly cut off. Soon after cut off, a 90° pulse is applied and the complex FID recorded. The instantaneous frequency is then obtained from the FID and normalized by dividing by the frequency offset at the sample during the gradient pulse.

Figure 4a shows a typical decay of extra magnetic fields in a CSI 2T instrument caused by eddy currents in the conventional, unshielded gradient set with compression.

Figure 4b shows the decay of the uncompensated shielded Z gradient and Figure 4c shows the Z gradient decay with compensation. Note that the time scale for 4b and 4c is five times shorter than that for the unshielded gradients.

Fig. 1a.

CONVENTIONAL GRADIENT COIL

- Currents are constrained to flow on a single cylinder.
- There is only one degree of freedom.



Fig. 1b.

SHIELDED GRADIENT COILS

- Currents on two cylinders.
- Two degrees of freedom.

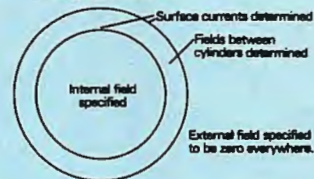


Fig. 1a—Design problem for unshielded gradients. The field inside the winding is specified to be a linear gradient and the current pattern on the cylinder is determined. **Fig. 1b**—Design arrangement for shielded gradients. The field inside the inner cylinder is specified to be a linear gradient and the field beyond the outer cylinder is specified to be close to zero. The current patterns on both inner and outer cylinders are then determined.

Fig. 2.

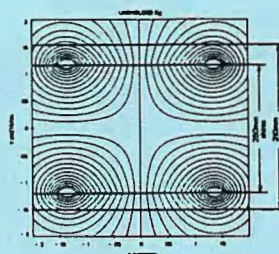
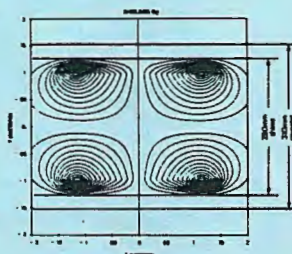


Fig. 3.



Lines of constant flux for Z-gradient. **Fig. 2**—Unshielded gradient. Note that flux lines extend well beyond the cryostat bore. **Fig. 3**—Shielded gradient. Flux lines are kept within the outer gradient cylinder.

Fig. 4a.

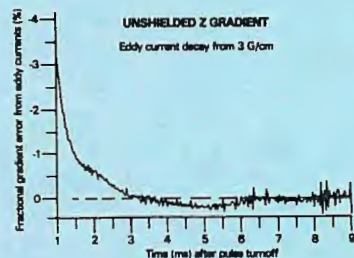


Fig. 4b.

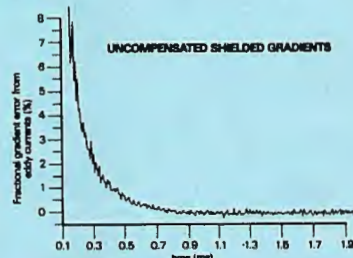


Fig. 4c.

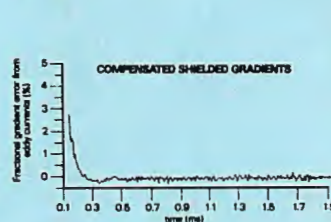


Figure 4b

Decay of field following application of square gradient pulse. **Fig. 4a**—Unshielded gradients. **Fig. 4b**—Shielded S150 gradient with no waveform compensation. **Fig. 4c**—Shielded S150 gradient with waveform compensation.



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**JC Hoch, S Hengyi, M Kjaer, S Ludvigsen, and FM Poulsen, "Symmetry Recognition Applied to Two-Dimensional NMR Data", Carlsberg Res., Commun., Vol. 52, p.111, (1987).

