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FORTHCOMING NMR MEETINGS

- 1991 Joint Meeting FACSS/Pacific Conference, Anaheim, California, **October 6-11, 1991**; NMR/EPR Program Section Chairman: Prof. Cecil R. Dybowski, Chemistry Dept., Univ. of Delaware, Newark, DE 19716. Contact: FACSS, P.O. Box 278, Manhattan, KS 66502-0003.
- 13th Eastern Analytical Symposium, Somerset, NJ, **November 11-15, 1991**; NMR symposia November 12-14; See Newsletter 395, 43.
- Eighth Australian NMR Conference, Lorne, Victoria, Australia, **February 2-6, 1992**; See Newsletter 391, 38.
- 33rd ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, California, **March 29 - April 2, 1992**; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667
- Eleventh Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, Berlin, Germany, **August 8-14, 1992**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.
- High Resolution NMR Spectroscopy (a residential school), University of Sheffield, England, **April 1993[sic]**; Organizer: Dr. B. E. Mann (Sheffield); For information, contact Ms. L. Hart, The Royal Society of Chemistry, Burlington House, Piccadilly, London W1V 0BN, England; Tel.: 071-437-8656.

Additional listings of meetings, etc., are invited.

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Notice re 1991-92 Invoices

By the time this issue of the Newsletter reaches you, your 1991-92 subscription fee should have been received by me. Newsletter finances are such that we cannot afford to service subscriptions which are unpaid by the time the November issue is to be mailed. Please help by making it unnecessary to remove and then re-add your name. Thanks!

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Mailing Label Adornment: Is Your Dot Red?

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

Page Length Request Instruction

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All Newsletter Correspondence

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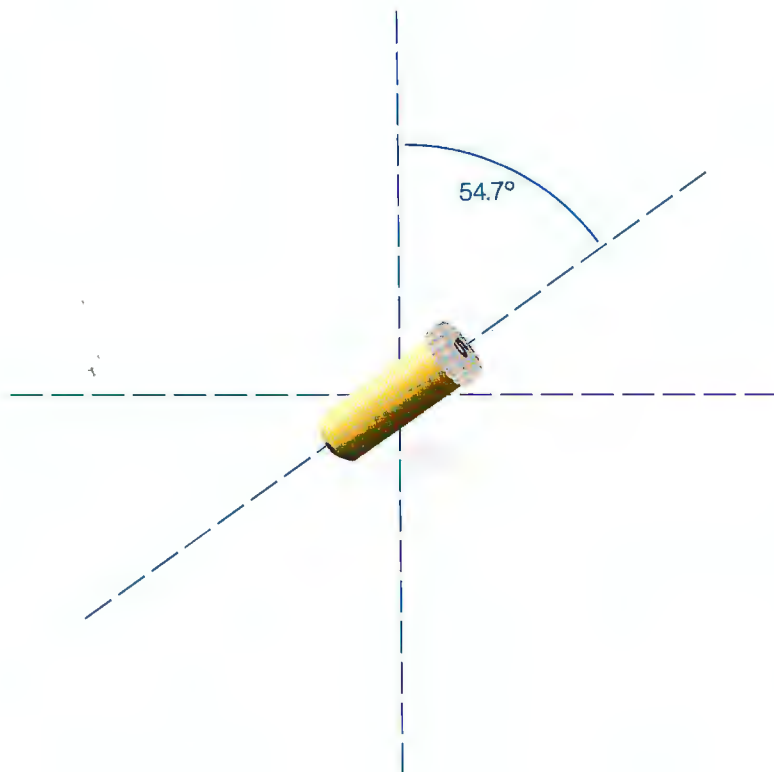
Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
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No. 399 (December) ----- 15 November 1991
No. 400 (January 1992) - 13 December 1991
No. 401 (February) ----- 24 January 1992

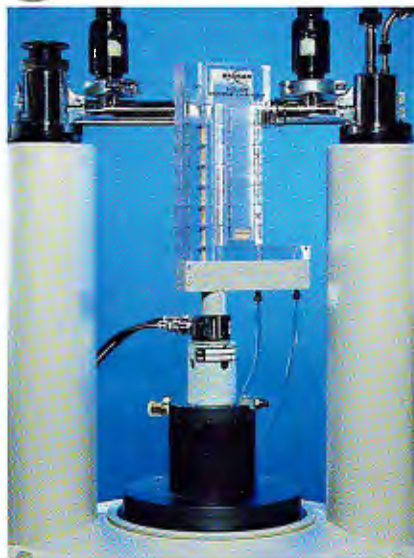
*** Please note that some of these dates are somewhat earlier in the month than has usually been the case !!**



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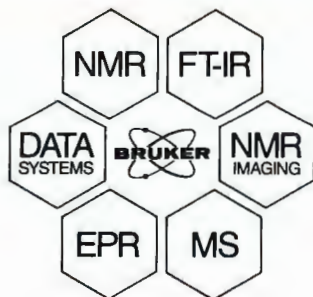
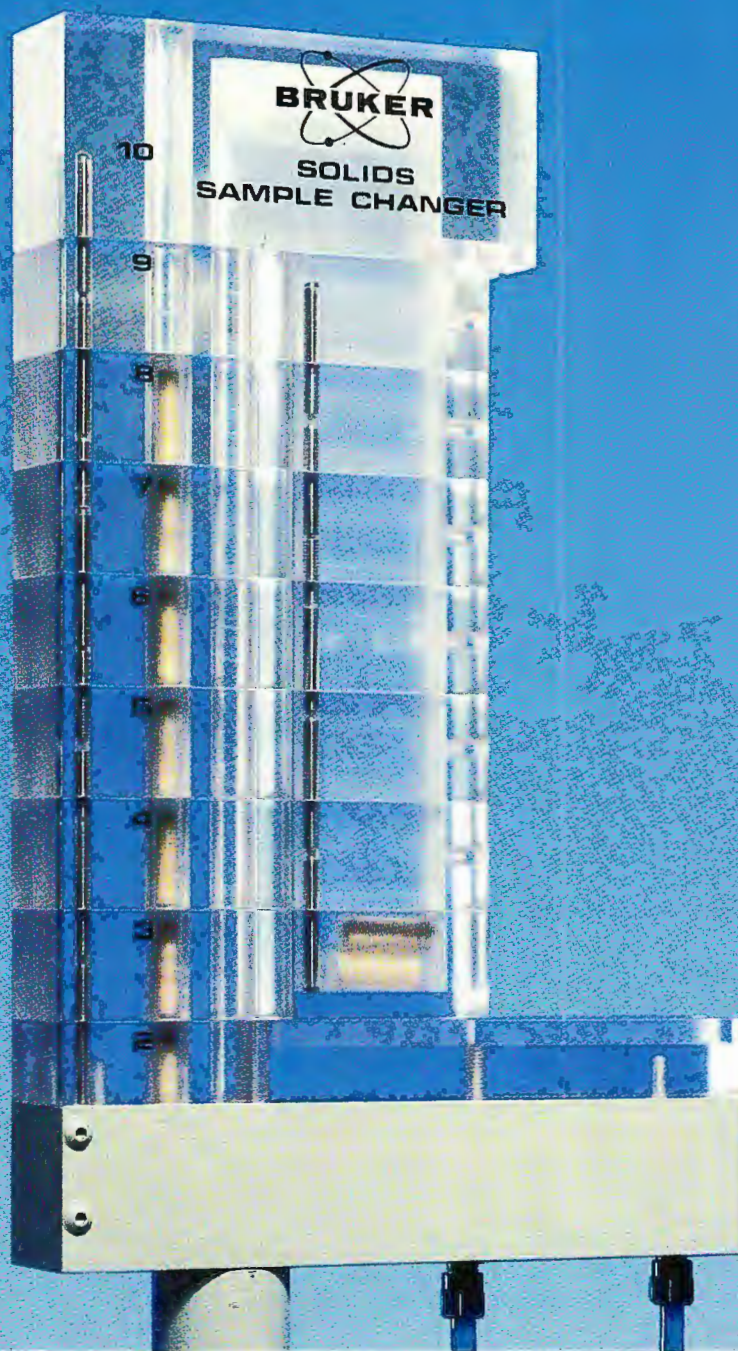


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Dr. Barry Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

(received 8/1/91)
August 10, 1991

Triple resonance and
new spectrometers

Dear Barry:

Much of our research nowadays concentrates on the study of larger proteins using triple resonance 3D and 4D NMR techniques. Much hardware was developed by our electronics expert, Rolf Tschudin, to make the old Bruker-AM consoles suitable for these experiments. Earlier this year we received a new AMX-600 spectrometer and we were rather skeptical about all the Bruker promises that we would not need any additional "home-built" hardware for our experiments with the new console. However, I must admit, even to date we have not installed any additional hardware and we have not been limited in the experiments we wanted to do (with the exception of a high power ^2H decoupling experiment which requires extensive fiddling with the ^2H lock).

Shortly after the AMX was installed, I tried to reproduce some results obtained previously on the old and heavily modified AM-600. The experiment in question is named HN(CO)CA and correlates amide protons with ^{15}N and with the $\text{C}\alpha$ shift of the preceding amino acid. The sequence and its use have been described in the Journal of Biomolecular NMR (the NEW journal) Vol.1, p.99-104 (for free copies of the first two issues write to the publisher, P.O. Box 214, 2300AE Leiden, Holland). The pulse scheme of the 3D experiment is illustrated in Figure 1. To speed up the testing I recorded the data in a 2D rather than a 3D manner, correlating the amide protons with the $\text{C}\alpha$ of the preceding residue, keeping the time t_1 fixed at 0 μs . Despite the large number of pulses involved in transferring magnetization from ^1H to $^{13}\text{C}\alpha$ and back, the experiment worked flawlessly and showed us how tremendously sensitive the experiment (or the AMX600) really is. A 2D spectrum was acquired in just over one hour for the protein calmodulin complexed with a 26-residue peptide (total mass of the complex ~20 kDa), using a 1.5 mM concentration. The 2D spectrum is shown in Figure 2.

Of course, we were very pleased that all these relatively complicated experiments can be done so easily. The turnside of the medal is that everybody else can do them too, making "the competition" a lot stiffer than it used to be.

Kindest regards,

Ad

Ad Bax

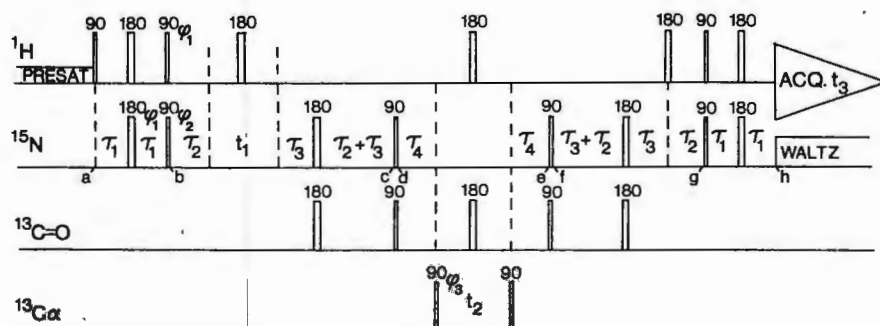


Fig.1. Pulse scheme of the HN(CO)CA experiment. For the 2D version illustrated here the evolution period t_1 was kept at zero. Full details can be found in J. Biomol. NMR 1, 99-104 (1991).

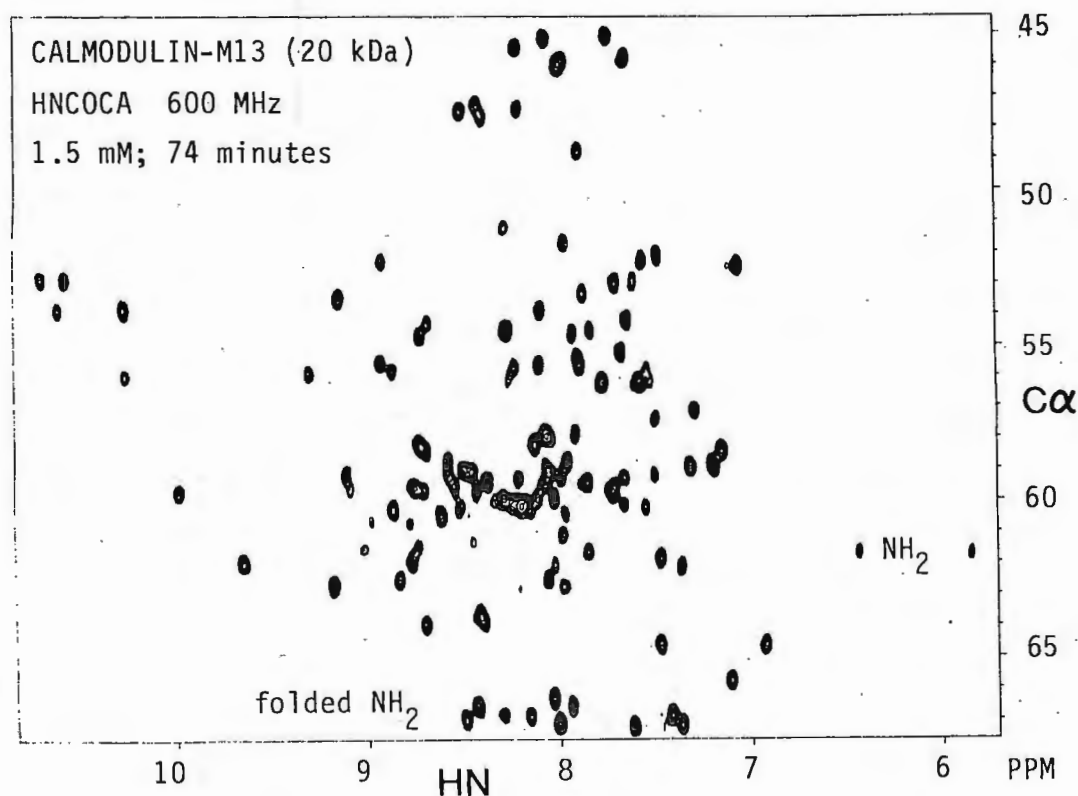


Fig.2. Region of the 2D H(N)(CO)CA spectrum displaying connectivity between amide protons and $C\alpha$ resonances of their preceding residues. The magnetization is relayed via the ^{15}N and $^{13}C=O$ resonances. Data were acquired with States-TPPI quadrature detection and consequently the side chain amides which correlate with $C\beta$ and $C\gamma$ carbons of Asn and Gln residues are folded and appear at the low field side of the spectrum instead of in the 35-40 ppm region, where they belong.

Sandia National Laboratories

Albuquerque, New Mexico 87185

August 14, 1991
(received 8/17/91)Dr. B. L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303

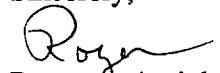
Quantitative Spectra of Silicate Materials

Dear Barry,

In the course of our ^{29}Si NMR studies of silicate materials we often need to record a quantitative spectrum, i.e. not only the relative but also the absolute intensities of each resonance must be quantitative. This means that we need to allow the magnetization to recover for several time constants before observation. In most instances, we estimate the spin-lattice relaxation time by varying the delay between $\pi/2$ observe pulses or by using a comb saturation sequence. Since the relaxation times are long and the signals are broad and therefore of low intensity, only a few points on the recovery curve are recorded. Figure 1 shows the recovery of a quartz sample, α -quartz powder from Alfa Products. The spectra were recorded at 4.7 T using direct polarization and magic angle spinning at 4 kHz. The solid line is a theoretical fit assuming exponential recovery of the magnetization. The $t=0$, $M=0$ was entered as a "theoretical" data point so that the curve fitting routine would draw the fit to short times. The exponential fit is reasonable, and leads one to believe that 90 % of the magnetization can be observed with a 100 s repetition time. This assumption would lead to a serious error.

Figure 2 shows the recovery curve of the same quartz sample for much longer times. The curve is plotted on a log-log scale from which it is obvious that the magnetization follows power law behavior over 5 decades of time. We know from calibration of the spectrometer with samples having shorter relaxation times, that the magnetization is near equilibrium at the longest time recorded in Figure 2. Spectra recorded at 100 s would have accounted for only 2 % of the equilibrium magnetization. The relative intensities of multicomponent spectra could have suffered serious distortion. The power law behavior has been observed previously for intentionally doped sol-gel derived silicate samples (F. Devreux *et. al.*, Phys. Rev. Lett. **65**, 614 (1990)). Power law recovery was shown to be expected for recoveries dominated by randomly distributed paramagnetic impurities. Although the samples in this study were not intentionally doped, they may contain low levels of paramagnetic impurities. Devreux showed that the power law exponent is related to the fractal dimension of the material. We are using his treatment to study the spatial characteristics of multiphase silicates.

Sincerely,


Roger A. Assink

Mark B. Boslough

Randy T. Cygan

Please credit this contribution to P. Cahill. We hope that Figure 2 demonstrates why this contribution was late.

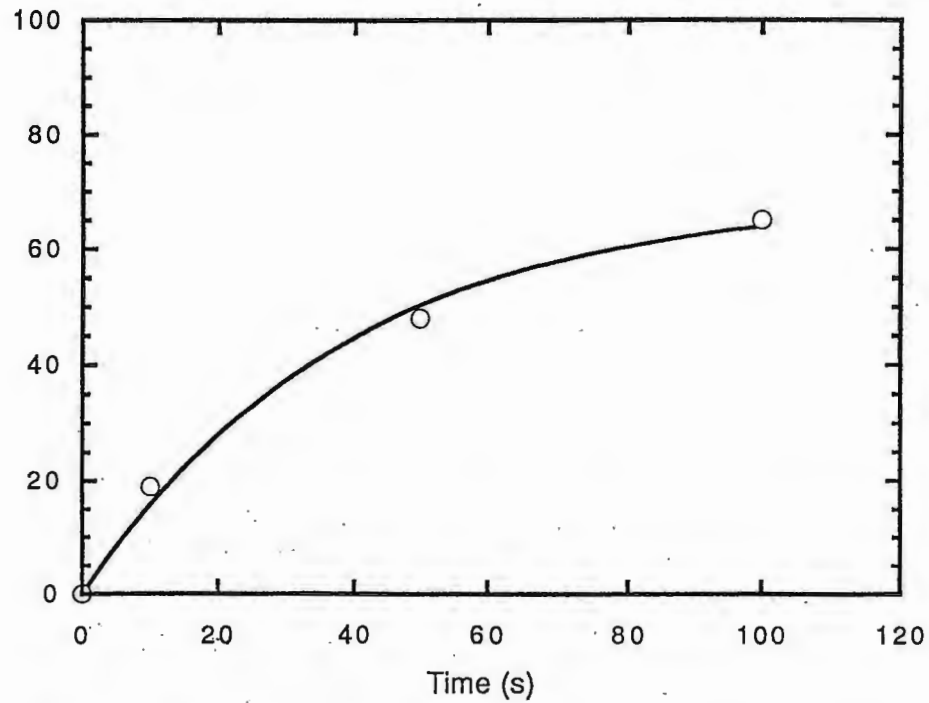


Figure 1. The magnetization recovery of a quartz sample for times up to 100 s. The solid line is a theoretical fit assuming exponential behavior.

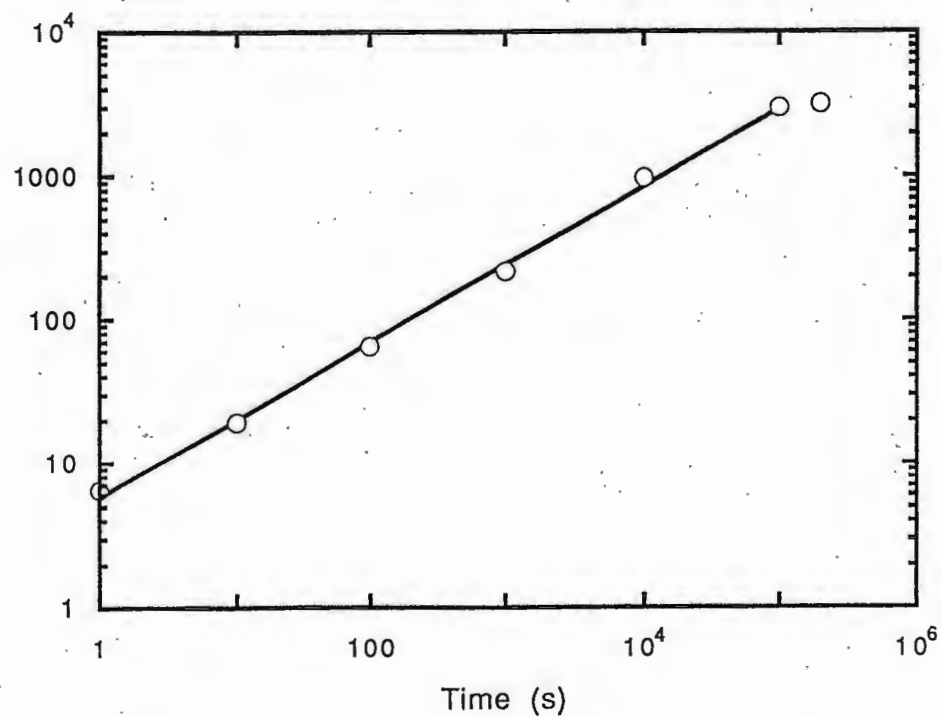


Figure 2. The magnetization recovery of a quartz sample on a log-log scale. The recovery follows power law behavior over 5 decades of time.

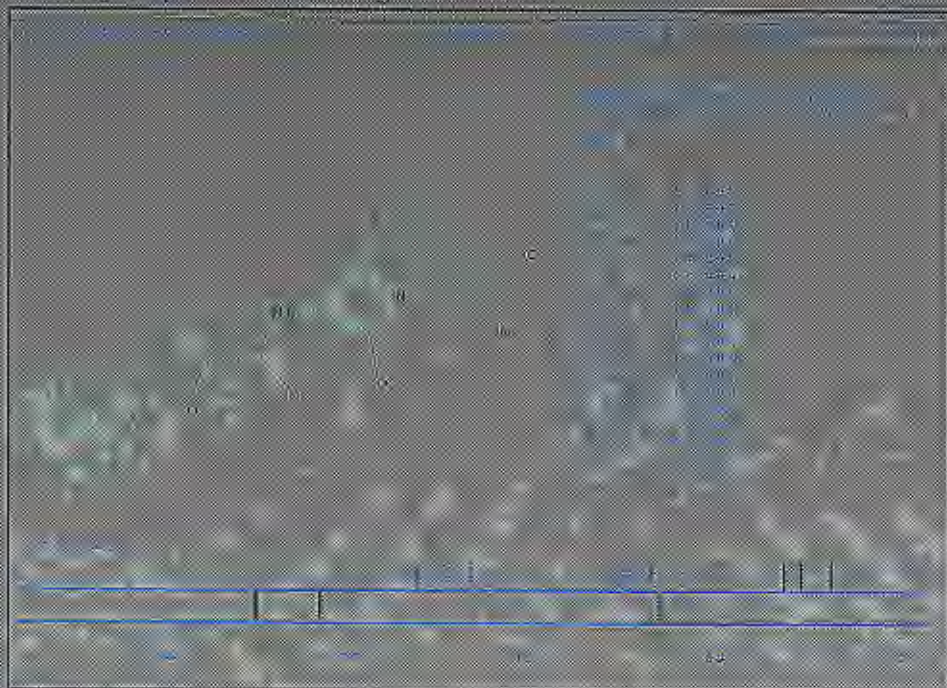
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July 14, 1991
(received 8/3/91)

Dr. Bernard L. Shapiro
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Measuring Diffusion Rates

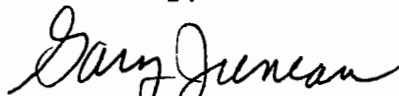
Dear Dr. Shapiro,

Staley has just marketed a new product, STELLAR, which is intended to replace fat in many food products. It has some interesting properties, one of which is its interaction with water. As a consequence, we became interested in the diffusion of water through the material. Lacking an imaging spectrometer or pulse field gradient equipment, the approach of Bansal and Dybowski¹ seemed appropriate. However, we used ^2H NMR rather than ^{129}Xe , and did a slightly different experiment.

We added about 2% D_2O to a sample of STELLAR and thoroughly mixed it. This preparation was placed at the bottom of a 10mm NMR tube. The height of the sample in the tube was about 5 cm. A layer of STELLAR, which didn't contain D_2O , of equal height was carefully added above it. The sample tube was then placed in the spectrometer so that only the bottom layer was monitored. The loss of ^2H signal over a period of time allowed us to estimate the diffusivity by using mathematics similar to that used by the above authors.

At the present time we have some preliminary diffusion coefficients. We are still optimizing and minimizing errors, though. We plan to complete our measurements in the near future.

Sincerely,


Gary Juneau

1. N. Bansal and C. Dybowski, *J. Magn. Reson.*, 89, 21-27 (1990)

Texas A&M University NMR Newsletter - Book Reviews

Book Review Editor:

William B. Smith, Texas Christian University, Fort Worth, TX 76129

"A Compilation of Chemical Shift Anisotropies"

by

T. Michael Duncan

The Farragut Press, P. O. Box 5102, Madison, WI 53705
1990; (ISBN 0-917903-01-3); paper \$19.95

For many years, NMR spectroscopists have been measuring not just the isotropic chemical shift, but all three principal elements of the chemical-shift tensor. These important quantities give greater definition of the local electronic structure than the isotropic shift. Indeed, some spectroscopists now use the values of specific elements as indicators of the existence of certain configurations or structures in solids.

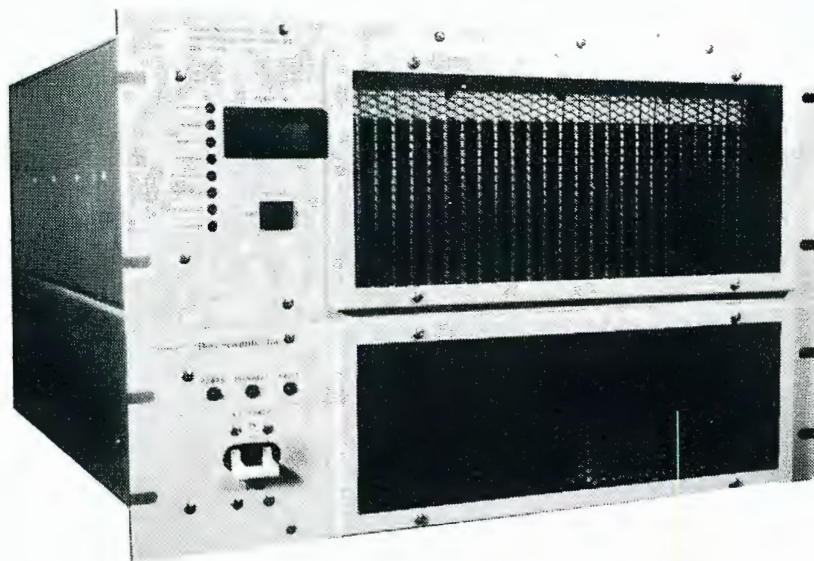
Mike Duncan has been interested in trends in chemical-shift tensor elements and their relation to the local chemical environment for a number of years. Like Charlotte Moore, he has amassed a compendium of spectroscopic data. It should be extremely useful for chemical identification and physical characterization. There are many tables (which Duncan calls chapters) of tensor elements. You can find information on ^1H , ^{13}C , ^{15}N , ^{19}F , ^{29}Si , ^{19}F , ^{31}P , ^{17}O , ^{23}Na , ^{35}Cl , ^{39}K , ^{51}V , ^{77}Se , ^{87}Rb , ^{111}Cd and ^{113}Cd , ^{119}Sn , ^{125}Te , ^{133}Cs , ^{183}W , ^{195}Pt , ^{199}Hg , ^{203}Tl and ^{205}Tl and ^{207}Pb . In his recent update, he also includes ^{129}Xe , ^{185}Re , and ^{187}Re . Each entry presents the name of a compound, a formula (indicating the particular position where necessary), the principal elements of the tensor, the isotropic shift, and a reference to where the data were obtained. (References are reasonably well done, although some "private communications" still crop up. In years to come, it will be difficult to track down the particular original data, if they are ever published.) Each table is divided by class of compounds and the "Contents" at the front of each chapter make it easy to get to the proper place quickly. At the beginning of each chapter is a schematic spectrum showing the positions of the isotropic shifts of representative materials. From this diagram, it is easy to determine the reference against which he defines shifts in the chapter. Where the original data were determined relative to another standard, he presents an explanation in a footnote of how the data were converted.

This is the kind of book everyone will ultimately use. It is also the kind of book whose author probably will not get as much credit for his scholarship as he deserves. Picking through all of the data in the literature, determining which direction in the spectrum is upfield (Yes, that's right!! It happens in solid-state NMR spectroscopy.), converting the data to a common basis, and organizing them by class must have been a Herculean effort which the casual reader will probably not appreciate. At the price, every NMR spectroscopist should have this important volume on his or her bookshelf. I would bet that Charlotte Moore would have had it on her shelf and used it regularly if she had been an NMR spectroscopist.

Cecil Dybowski
Department of Chemistry and Biochemistry
University of Delaware
Newark, Delaware 19716

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July 26, 1991
(received 7/29/91)

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

Efficient Calculation of NMR Powder Patterns

Dear Barry:

The ability to accurately simulate spectra is important for the design and interpretation of NMR experiments. For non-oriented solids, the calculation of spin-system responses in either the time-domain or frequency-domain requires summing the contributions from a collection of random-oriented single crystals. This powder averaging can be time-consuming and can limit the scope of such calculations. A key question that has been addressed by a number of groups is how to most effectively and efficiently calculate the powder average. Ideally, one would like to minimize the number of sampled points while maintaining the accuracy required to make meaningful comparisons with experimental spectra. We would like to report here our implementation of a simple, straightforward procedure that helps to satisfy these two seemingly conflicting requirements.

The method we use employs an algorithm that has been described in detail in a number of papers dealing with the general problem of evaluating multi-dimensional integrals.^{1,2} For convenience we shall refer to the algorithm as the C-W (Conroy-Wolfsberg) method. The first step in implementing the C-W method is to select the total number of points (N_p) to be used in computing the powder average. Tables in both referenced papers provide optimum values of N_p . Also tabulated for each value of N_p are incrementation step sizes for each of the integration angles. These step sizes are fixed, but not equal, for the different angles. In many integration schemes, the different angles in the powder average are incremented *sequentially*, with one angle being varied while the others are held constant. In the C-W method, the points at which the integral is evaluated are calculated, one from the next, by *simultaneous* incrementation of *all* of the integration angles. When incrementation would cause an angular value to exceed its upper limit, its value is 'folded back' (much like the path of a ball striking the edge of a billiard table) and its value is then decremented when selecting subsequent points.

The success of the C-W method is demonstrated in the calculated powder spectra shown in Figures A - E. These powder spectra are Fourier transforms of the synchronously-sampled, time-domain ^{13}C signal for a randomly-oriented, dipolar-coupled ^{13}C - ^{15}N spin pair ($D_{\text{CN}} = 800$ Hz) rotating at the magic angle ($\nu_R = 2$ kHz) with two equally-spaced ^{15}N π pulses applied per rotor period. Figure A is the powder pattern

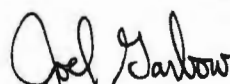
¹Conroy, H. J. *Chem. Phys.*, **47**, 5307 (1967).

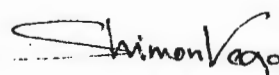
²Cheng, V. B.; Suzukawa, Jr., H. H.; Wolfsberg, M. J. *Chem. Phys.*, **59**, 3992 (1973).

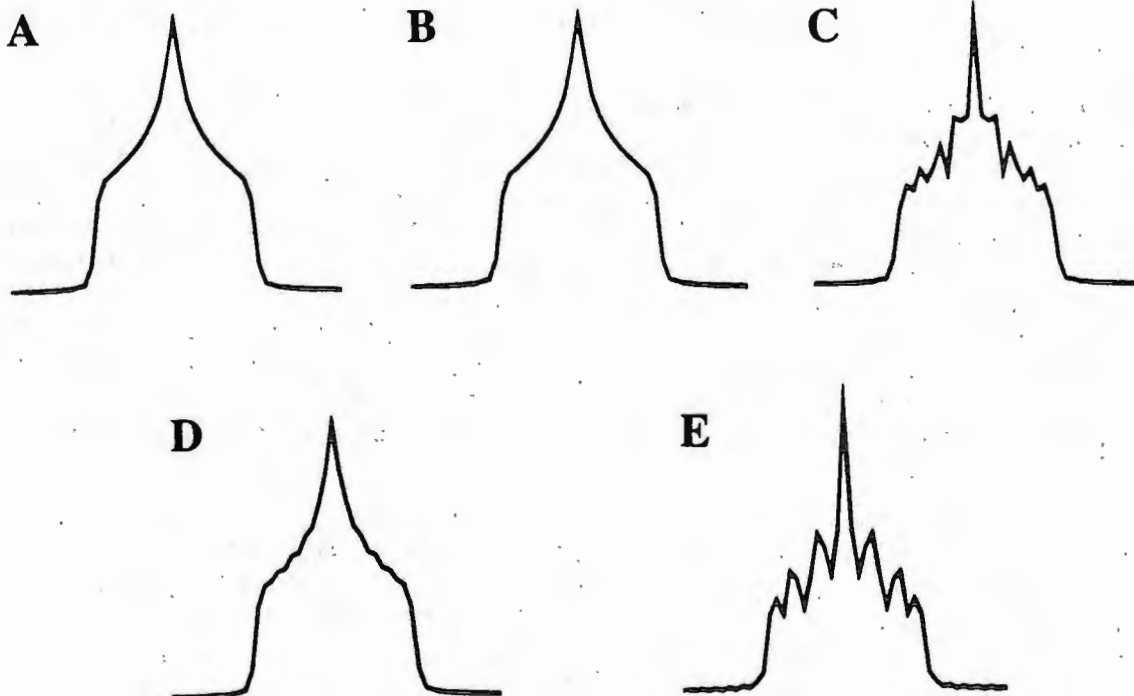
calculated for 4050 total points with sequential increments of 4.1° for first the polar angle, θ , and then the azimuthal angle, ϕ . Figure B is the corresponding spectrum calculated for 1154 points chosen according to the C-W method. The powder patterns in these two figures are indistinguishable from one another. Figure C, the calculated spectrum for 1250 points with sequential incrementation (7.5° increments in θ and ϕ), shows significant distortions that result from the undersampling of the powder distribution. When the number of integration points is further reduced, the errors become even more pronounced. Figure D shows the calculated powder spectrum for 538 different orientations selected via the C-W method. Although minor wiggles are now visible as a result of undersampling, the spectrum is still a reasonable representation of the true powder average. By contrast, the spectrum calculated for 578 points with a sequential incrementation for both angles of 11.25° , Figure E, is badly distorted.

These calculations demonstrate that the C-W method permits the number of points that must be evaluated to achieve accurate powder averaging to be significantly reduced. This in turn results in a significant reduction in computational time for powder-average calculations. This benefit is particularly valuable during computer-program development or for problems requiring the rapid survey of a large numbers of different parameters. We would be happy to provide further details of our implementation of the C-W method to anyone who might be interested. Please credit this contribution to the Monsanto account.

Best Regards,


Joel Garbow
Physical Sciences Center
Monsanto Corporate Research


Shimon Vega
Department of Chemistry
Washington University



Lawrence Livermore National Labs.
BOX 808 Livermore, CA 94550

July 25, 1991
(received 7/27/91)

Dear Barry:

BOUND WATER MEASUREMENT IN ROCKS FROM BORE HOLES IN NEVADA AS A FUNCTION OF DEPTH

I just received my "Reminder" notice on orange paper. It's too bad that it didn't come on white paper as we have just established a large recycling effort at Livermore and I would have been more than happy to contribute it, but they will only take white paper. One can only imagine how much paper a place this size can contribute to recycling even if it is limited to white.

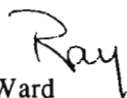
I have been trying to decide whether or not a subscription to the TAMU NMR Newsletter is worth my effort. Unfortunately because of the nature of our NMR group, I am the only one interested and available to contribute, and I sometimes find this difficult even if only once every eight to ten months. I was just about ready to write and say that I must cancel my subscription when I received the July 1991 edition of the Newsletter. The article by J. L. Ackerman describing how to modify a Bruker CPMAS Probe for ^{19}F detection caught my eye and I realized that if their method works the effort to continue to contribute to the Newsletter will be worth it both in money and time saved.

I have been working with members of our Earth Sciences Department determining bound water in rock samples retrieved from bore holes in Nevada. Although this is a classical NMR problem and the measurements not overly difficult with our Bruker MSL300, the results are turning out to be interesting to the Geologists and to me. The water content of the rocks varies between 0.5 and 10% and appears to be bound water because of the relatively large line widths- 10 to 30 KHz. Notably there is an absence of what the Geologists would call mobile water with a line width of ~2.5 KHz.

The accuracy of the proton concentrations by NMR are good to a few percent. We use benzoic acid as a proton standard. Reproducibility of the measurements on the same sample are likewise good to a few percent. The uniformity of the rock sample is probably the principal limitation in the reproducibility of the results. Without an exhaustive analysis of the problem, the uniformity of sample selection probably sets a limit our knowledge of the water content to 10-20%. One of the main interests was in the fraction of mobile to bound water. As the NMR line widths indicate essentially all the water is bound. Interestingly, in connection with the question of bound vs. mobile water in this rock system, D_2O exchange has been appreciably less effective in reducing the amount of bound proton water than has storing the rock sample in a dessicator over drierite. We are currently also exploring the question, "How relevant are the numbers we obtain in the laboratory to those actually in the native environment?". A number of experiments indicate that processing the rock has little effect on the bound water content.

Sincerely,

Raymond L. Ward



**Acorn NMR**

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FAX: (415) 683-6784

August 1, 1991

Murphy's Law Proof ???

Dear Barry,

We were reading a story in the paper the other day about a simple game show problem. The answer seemed obvious at first, but later thinking showed that the initial conclusion we jumped to was wrong. The situation reminded us of many we face in the laboratory each day. The game show scenario goes like this:

There are three doors to choose from. One has a great prize behind it and the other two have nothing. The contestant is asked to pick a door. After the choice is made, the game show host reveals one of the two remaining doors with nothing behind it and offers to let the contestant remain with the first choice or switch to the other remaining unrevealed door. Should the contestant remain with the first choice or switch to the other unrevealed door?

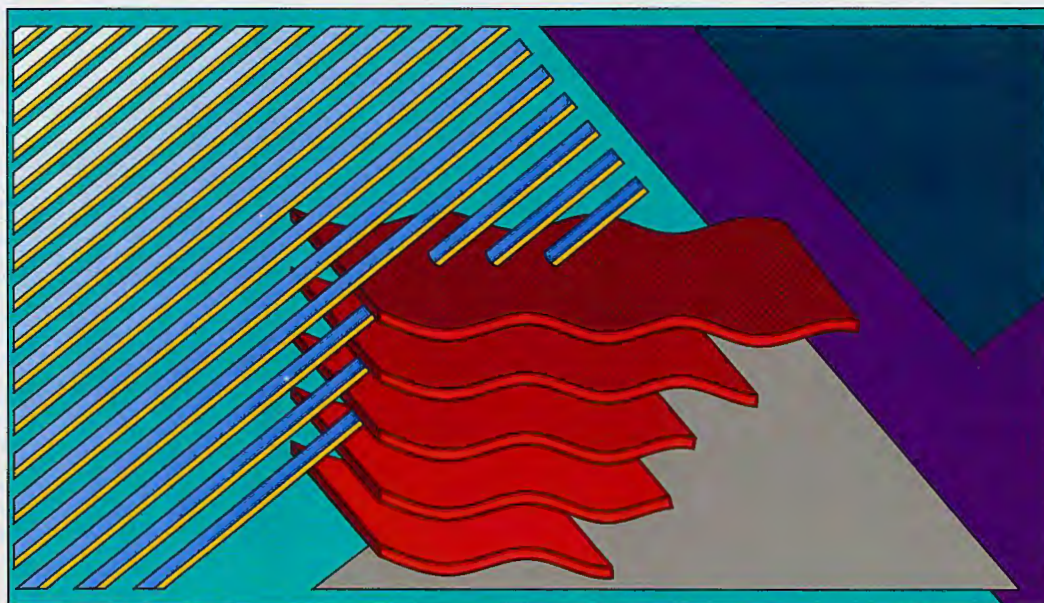
The conclusion we jumped to was that it didn't make any difference. We thought the odds were the same. They are not! When the first choice was made, the odds of a correct choice was $1/3$. The odds that the good prize was behind one of the other two doors was $2/3$. The all knowing game show host then reveals one of the two unpicked doors which has no prize behind it. This leaves two doors. The odds that the first choice was correct is still $1/3$. However; since the game show host knew which door to reveal from the two unpicked doors, the odds that the unpicked door is correct is $2/3$. The contestant then has the highest probability if he/she selects the other door.

We are sure that Murphy is just sitting around waiting to pull one over on us. We think of him as all knowing like the game show host. In diagnosing a broken NMR spectrometer or in doing a structural analysis, we are often faced with picking from three possible choices. As the decision process proceeds we sometimes make a choice as to which answer is correct. Then new evidence comes along to show that one of the other two possible choices cannot be correct. With the above example as evidence, we now approach the decisions differently; we switch choices. It seems to be working for us, maybe the readers should try it.

Gina Miner

Woody Conover

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August 6, 1991
(received 8/12/91)

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

RE: 3-D NMR Imaging of Coal

Dear Barry:

Recently, we have developed a new three-dimensional (3-D) nuclear magnetic resonance (NMR) imaging technique for spatially mapping proton distributions in whole coals and solvent-swollen coal samples. The technique is based on a 3-D back-projection protocol for data acquisition, and a reconstruction technique based on 3-D Radon transform inversion. In principle, the 3-D methodology provides higher spatial resolution of solid materials than is possible with conventional slice-selection protocols. The applicability of 3-D NMR imaging has been demonstrated by mapping the maceral phases in Utah Blind Canyon (APCS #6) coal and the distribution of mobile phases in Utah coal swollen with deuterated and protic pyridine.

In an earlier study, we had demonstrated the use of multi-pulse proton decoupling and back-projection reconstruction imaging methods to spatially resolve macroscopic resinite and vitrinite regions within a dried sample of Utah Blind Canyon coal.¹ Image contrast between the maceral phases was achieved on the basis of differences in proton density or spin-lattice relaxation (T_1). In the present study, we focus on the effects of solvent penetration to the swelling behavior of Utah coal.

The proton NMR spectrum of a specimen of Utah Blind Canyon coal swollen with "100%" deuterated pyridine displays three distinct proton resonances: there is a broad resonance (ca. 27 kHz) that corresponds to rigid protons in the sample, and there are two relatively narrow resonances that correspond to aromatic and aliphatic protons in mobile environments. Analysis of the broad and narrow components indicates that approximately 14% of the protons are in the mobile phase.

A 3-D surface-rendered NMR image of the Utah coal specimen swollen with deuterated pyridine is shown in Fig. 1. The image was recorded with a short recycle-delay time in order to suppress signal from the broad "solid" component having a longer T_1 . Thus, the image displays the proton distribution of mobile phases within the coal specimen preferentially. Surface rendering is performed by applying a user-adjusted threshold intensity to define a minimum intensity that is used to calculate the contiguous surface. The particular threshold employed was chosen to give the most accurate representation of the sample topology while suppressing low signal-to-noise structures near the sample surface. The image displayed is a good representation of the topology of the coal specimen; however, it should be realized that surface features seen in the image represent areas with more or less density of mobile protons. For example, the feature seen at the front left of the object (indicated by arrow) is a crack in the specimen resulting from swelling with pyridine. Other features on the surface may be due to regions where there is a higher density of solid phase.

An 80-micron thin section (slice) of the 3-D NMR reconstructed image taken as a horizontal plane near the center of the specimen is shown in Fig. 2. Areas of bright intensity represent highly mobile regions within the specimen. Low intensity regions indicate solid phases or voids within the coal. The crack is clearly visible at the lower right portion of the image (indicated by arrow).

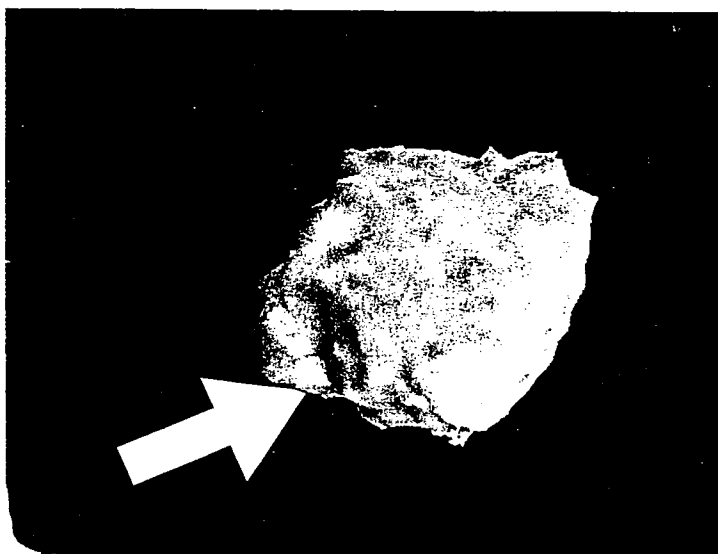


Figure 1. Surface reconstructed 3-D NMR image of Utah Blind Canyon (APCS #6) coal specimen swollen with deuterated pyridine.

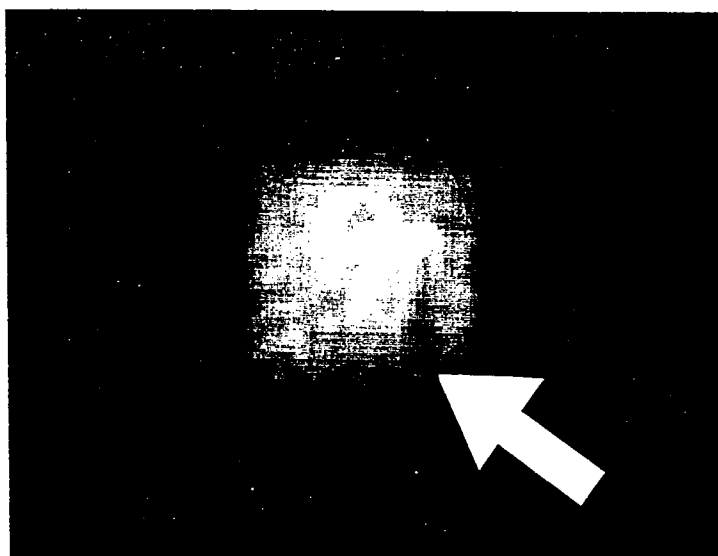


Figure 2. Internal 2-D thin section of 3-D NMR reconstructed image of Utah coal specimen as shown in Fig. 1.

REFERENCE

1. Dieckman, S.L.; Gopalsami, N; Botto, R.E. *Energy and Fuels* 1990, 4, 417.

D.C. French
D.C. French
CHM/ANL

S.L. Dieckman
S.L. Dieckman
MCT/ANL

Sincerely,
N. Gopalsami
N. Gopalsami
MCT/ANL

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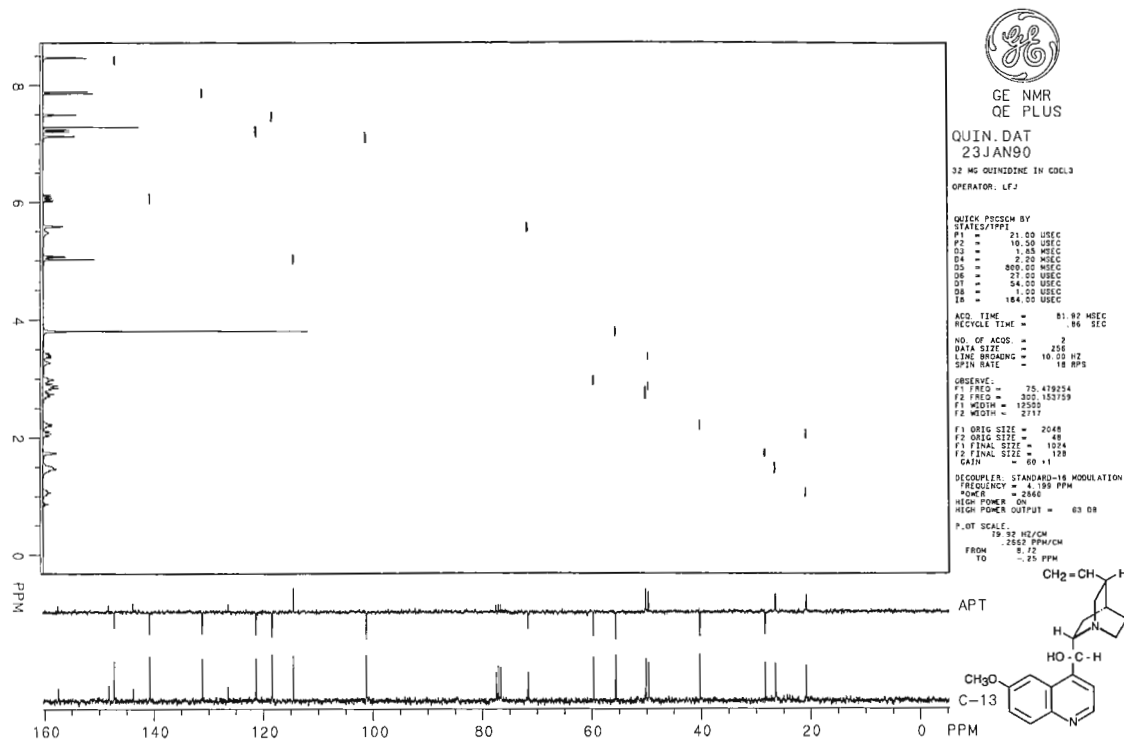
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Dr. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

August 12, 1991
(received 8/15/91)

Pi T₁ relaxation. A probe for paramagnetic oxygen localization in nitrogen fixing soybean nodules.

Dear Dr. Shapiro:

During the course of *in vivo* ³¹P studies of nitrogen fixing soybean nodules we observed a significant loss of the vacuolar Pi resonance magnetization when the tissue was perfused with N₂ relative to O₂. Approximately 80% of the magnetization was restored following 1.5 hr. of O₂ perfusion (data not shown). This is a novel finding never before observed in any of our or other previously reported ³¹P NMR plant tissue work (1). Figure 1 illustrates the loss of vacuolar Pi magnetization as a function of N₂ perfusion time. Upon dissecting the nodules into their component outer cortex and inner matrix we examined the ³¹P spectrum of each excised tissue and observed that the unusual loss of magnetization following N₂ perfusion was emanating from the vacuolar compartment Pi resonance (0.9 ppm, pH = 5.5) of the cortical tissue (outer layer of cells surrounding the nodule).

Earlier plant biochemical studies indicate that the cortical tissue of the nodule functions as an oxygen repository and barrier designed to maintain the anaerobic environment of the inner nodule matrix to protect the nitrogen reducing enzyme, nitrogenase (2). To clarify what mechanism is responsible for the changes in the observed Pi intensity we examined the T₁ relaxation behavior of Pi in the cortical cell tissue under aerobic and anaerobic conditions. Spin-lattice relaxation times for cytoplasmic and vacuolar Pi in intact nodules under N₂ and O₂ perfusion are given in Table 1. The significant increase in vacuolar Pi T₁ in going from an oxygen to nitrogen atmosphere appears to be responsible for the observed loss of resonance magnetization. As oxygen is sequestered in the vacuoles of the cortical cells significant shortening of the vacuolar Pi T₁ results. Isolated, excised cortical tissue shows the same relaxation behavior (Table 1). No such response is evident from the Pi signal representing the cytoplasmic Pi residing in the interior of the nodule (Table 1). Clearly, sequestered paramagnetic oxygen located in the cortical cell vacuoles can facilitate Pi relaxation *in vivo*. Thus Pi relaxation can function as a probe for localizing high concentrations of oxygen in compartmented tissues.

The same relaxation phenomena in soybean nodules is also evident using ¹H magnetic resonance microscopy. A detailed account of both the ¹H microscopy and ³¹P spectroscopy findings have been submitted to Plant Physiology.

1. Pfeffer, P.E. and Gerasimowicz, W.V. Introduction to high-resolution NMR spectroscopy and its application to *in vivo* studies of agricultural systems. In: Pfeffer, P.E. and Gerasimowicz, W.V. (eds.) NMR in Agriculture, CRC Press, Boca Ratan. 1988

2. Bergersen, F.J. Roots nodules of legumes: Structure and functions. John Wiley and Sons Ltd. 1982

Sincerely,

Phil Pfeffer

Dominique Rolin

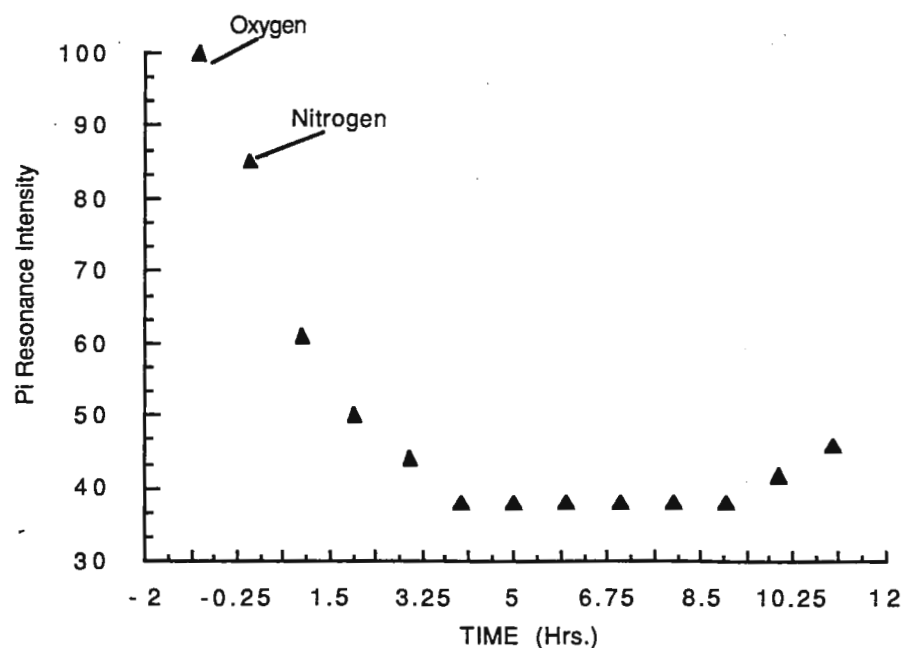


Figure 1. Area of cortical tissue vacuolar Pi resonance as a function of the duration of hypoxia treatment, (circulating N₂ saturated perfusate medium).

Table 1. T₁ and T₂ Relaxation Values for Pi *In Vitro* and *In Vivo* Under Aerobic and Anaerobic Conditions

Source of Pi	Perfusing Gas	T ₁ (s) ^a	T ₂ (s) ^a
25mM Pi Solution ^b	O ₂	6.66±0.05	0.141±0.004
	N ₂	6.64±0.07	0.115±0.002
Nodules(Vac. Pi) (Cyt. Pi)	O ₂	0.77±0.08	
	O ₂	0.95±0.19	
Nodules(Vac. Pi) (Cyt. Pi)	N ₂	3.98±0.69	
	N ₂	0.95±0.20	
Cortical Tissue(Vac. Pi)	O ₂	3.72±0.93 (59%)	0.41±0.02 (62%)
		0.20±0.08 (41%)	0.07±0.01 (38%)
Cortical Tissue(Vac. Pi)	N ₂	7.07±1.36 (86%)	0.37±0.01 (61%)
		0.39±0.07 (14%)	0.07±0.01 (39%)
Soybean Root Tissue (Stem Segments, Vac. Pi)	O ₂	6.30±0.93	1.53±0.08
Soybean Root Tissue (Stem Segments, Vac. Pi)	N ₂	5.80±0.65	

a) Average of three independent measurements±SD; b) Contained 100mM KCl, 10mM MES pH 6.0 no attempt was made to remove paramagnetic contaminants

Columbia University *in the city of New York*

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Friday, August 9, 1991 (received 8/12/91)

Dr. Barry Shapiro, Editor
TAMU NMR Newsletter
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Palo Alto, CA 94303

Big T_1 -Noise in Double Quantum Filtered COSY

Dear Barry:

The major problem in attempting to measure 2-D spectra as fast as possible is a lowering in the quality of the data due to the generation of repetition rate artifacts, sometimes called "Big T_1 -noise". If the data are acquired quickly, then the magnetization from one transient can carry over and interfere with the next. In theory, this interference between adjacent transients could be caused by either T_1 or T_2 , but in practice, T_1 is usually longer than T_2 , and thus T_1 is the more important problem. Despite this, artifacts generated by T_2 effects, while less important than those generated by T_1 , are often harder to remove. Either way, if we waited for complete relaxation between adjacent transients, the artifacts would go away. However, this is impractical. A routine 2-D spectrum would take ages. An attractive solution to this problem is to cancel the artifacts by phase cycling.

One of the most valuable techniques in the suppression of repetition rate artifacts is the two-step phase inversion process that we tend to think of as axial peak suppression. Nowhere is this better exemplified than in DQCOSY, where the four transient basic phase cycle suppresses axial peaks, since they are generated by single quantum magnetization which cannot pass through the double quantum filter, thus axial peak suppression is strictly speaking redundant. Nevertheless, the process of "axial peak suppression" offers a dramatic improvement in the quality of DQCOSY spectra, by cancelling repetition rate artifacts. Unfortunately, it also has the disadvantage of multiplying the minimum phase cycle by a factor of two.

In order to spend the minimum length of time measuring spectra we want to minimize the length of the phase cycle. Thus, we throw away half of the double quantum filter, leaving a two step cycle, but add artifact suppression, multiplying this by two, to end up with the four step phase cycle shown below.

First Pulse	3 0 1 2
Second Pulse	1 0 1 0
Third Pulse	0 0 0 0
Receiver	0 0 2 2

Loss of half of the double quantum filter produces data quite similar to PCOSY spectra (D. Marion and A. Bax, *J. Magn. Reson.* **80**, 528 1988), in that the diagonal peaks are in the positive absorption mode, and singlets are not suppressed. The starting point of the phase cycle has been adjusted so that the sequence ends with a step which causes the minimum disturbance of the z -magnetization (A. E. Derome and M. P. Williamson, *J. Magn. Reson.* **88**, 177 1990), thus eliminating the need for dummy transients between adjacent increments of the evolution time.

A more detailed account of this work is scheduled to appear in JMR next year.

Best Wishes



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Dr Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
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U. S. A.

5th August 1991
(received 8/12/91)

Deuterium NMR Studies of the *in vivo* Conversion of Vanillin to Capsaicin

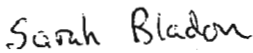
Dear Barry,

It has been postulated ¹, but not proven, that vanillin is a precursor in the formation of capsaicin in chilli peppers (*Capsicum frutescens* = *Capsicum annuum*). The earlier classical work of Bennett and Kirby² on capsaicin biogenesis did not consider this possibility and did not include experiments to test this hypothesis. In view of this, feeding experiments, using [5-²H]-vanillin, prepared by the method of Kirby and Ogunkoya³, were carried out.

A total of 5mg of the labelled vanillin (>99% atom D) was fed by injection into 5 developing fruits. After harvesting and extraction, the capsaicin fraction was isolated, and the proton and deuterium spectra were examined. In the proton spectrum, the doublet signal at $\delta=6.86$ had 95% of the intensities of the other two aromatic signals at $\delta=6.76$ and 6.81 , indicating that 5% of the capsaicin was labelled with deuterium. The deuterium spectrum in CHCl_3 showed the expected peak at $\delta=6.86$. By comparing the integral of this peak with that of the solvent peak, and assuming that the chloroform contained 15 ppm of CDCl_3 , it was calculated that 0.03% of the vanillin injected had been converted into capsaicin. From other experiments, it has become apparent that most of the labelled vanillin is incorporated into water-soluble glycosylated material.

While the experiment leaves no doubt that vanillin is converted into capsaicin under these (rather abnormal) conditions, it could be argued that the question of whether vanillin is on the biogenetic pathway, is still open. The experiment is, however, of interest as affording another example of the use of a relatively inexpensive technique, in a field dominated by the use of more expensive labelling methods.

Yours sincerely,


Sarah Bladon


Peter Bladon

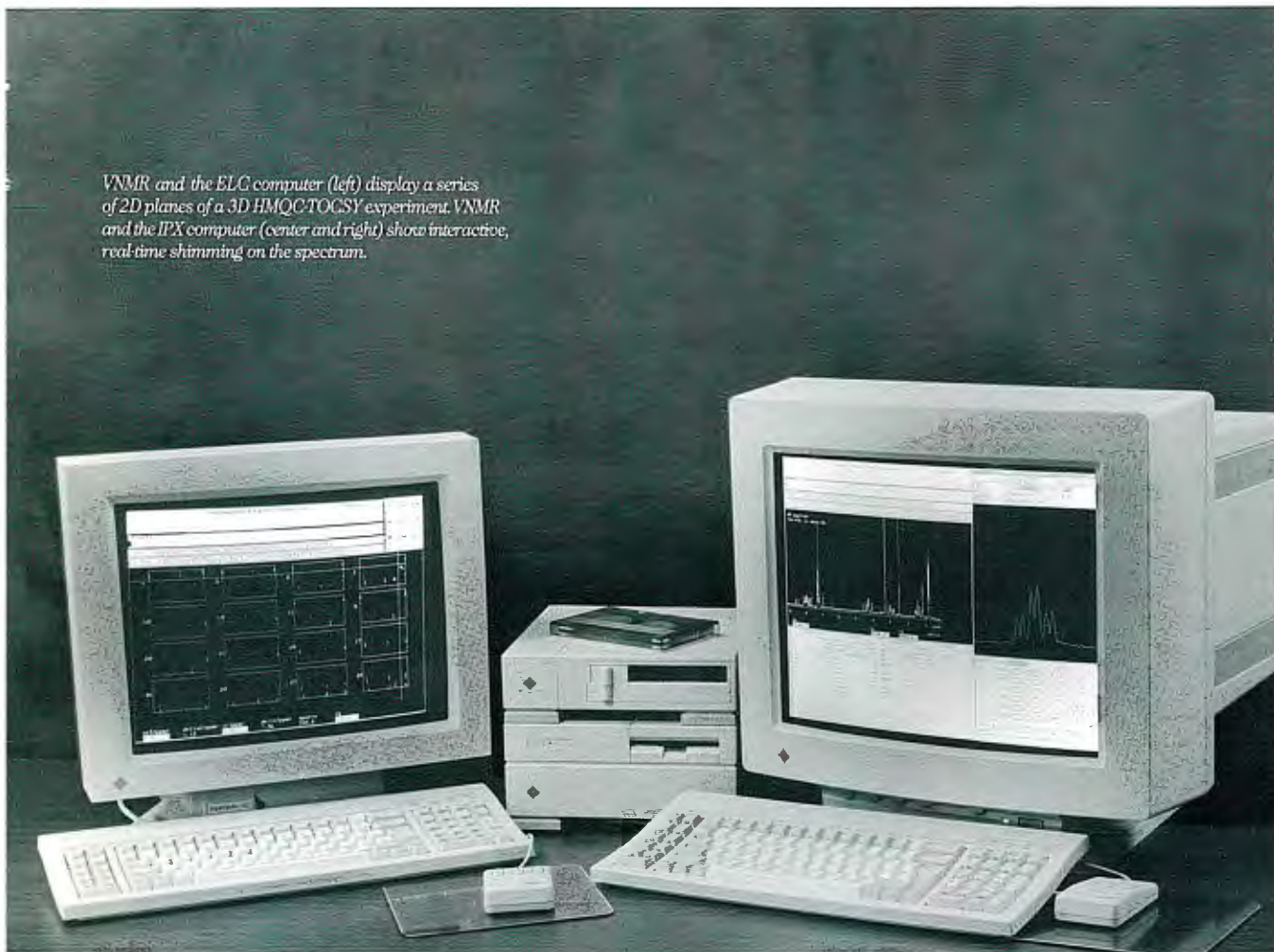
References

- 1 M. M. Yeoman, M. B. Miedzybrodzka, K. Lindsay, and W. R. McLauchlan, (1980). "Synthetic Potential of Cultured Cells", in "Plant Cell Cultures: Results and Perspectives", Elsevier-North Holland.
- 2 D. J. Bennett and G. W. Kirby, *J. Chem. Soc. (C)*, 1968,442.
- 3 G. W. Kirby and L. Ogunkoya, *J. Chem. Soc.*, 1965, 6914.

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Department of Chemistry

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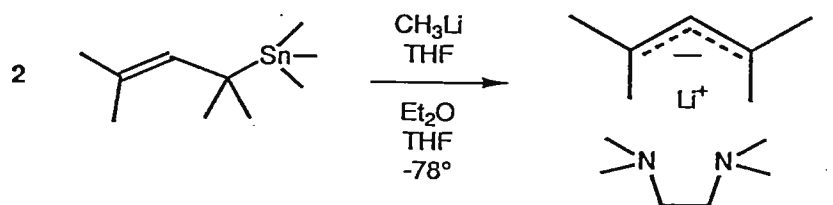
Answer Back Code: OSU CHEM UD

Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsimore Court
Palo Alto, CA 94303July 19, 1991
(received 8/2/91)

1,1,3,3-Tetramethylallyllithium

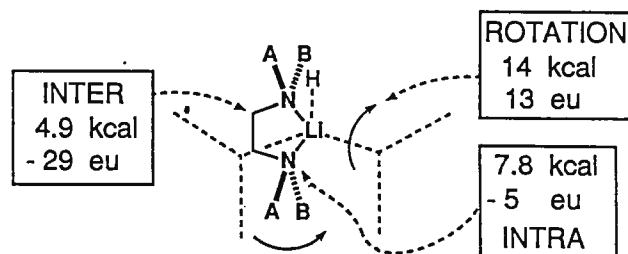
Dear Barry:

In response to the Dreaded Pink Note: In the course of some studies of the effect of alkyl substituents on the stability of π conjugated carbanions, we have prepared 1,1,3,3-tetramethylallyllithium, **1**, a somewhat elusive species which has defied synthesis for some time, see below. (Readers concerned about the origin of **2** should watch out for J. Org. Chem. ca. six months from now.)



Carbon-13 NMR of species **1** in diethyl ether at 180K includes equal doublets for the C-methyls of **1** as well as for the N-methyls of coordinated TMEDA (the diamine). These data support a proposed monomeric ion-pair in which coordinated Li^+ lies perpendicular to the allyl plane and TMEDA is unsymmetrically sited with respect to this plane.

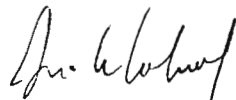
Above 180 K with increasing temperature both methyl doublets, just described undergo signal averaging to, ultimately, single sharp lines at their respective centers. That for C-CH_3 must be due to rotation about the π bonds of the substituted allyl anion. The rate of the process responsible for the N-methyl coalescence is independent of the concentration of **1**. It is most likely the result of rotation of coordinated Li^+ with respect to allyl counterion. A third slower process observed is the exchange of TMEDA between the complex and its free state in solution. NMR line shape analysis gives rise to the parameters listed around the diagram below. The π barrier is much like others we have measured. Note that the fastest process is rotation of complexed Li^+ .



Dynamic effects seen in NMR data for three other coordinated lithium compounds were also ascribed to slow rotation of coordinated lithium with respect to counterion. Three species are 1,3-bis(trimethylsilyl)-allyllithium¹, 1-trimethylsilylallyllithium² and 1-bis(2-ethoxyethyl) aminomethyl-dimethylsilylallyllithium³. All three exhibited ΔH^\ddagger values of 7-8 kcal for motion of coordinated Li^+ with respect to counterion. That appears to be typical for dynamics of motions of ions within ion-pairs.

I trust this revives our subscription. Best regards.

Yours sincerely,



Jose A. Cabral
Research Associate



Gideon Fraenkel
Professor of Chemistry

JAC/GF:jlj

1. Fraenkel, G.; Chow, A.; Winchester, W.R.; *J. Am. Chem. Soc.* **1990**, *112*, 1282.
2. Fraenkel, G.; Chow, A.; Winchester, W.R. *J. Am. Chem. Soc.* **1990**, *112*, 2582.
3. Fraenkel, G.; Cabral, J.A.; Winchester, W.R. To be published.



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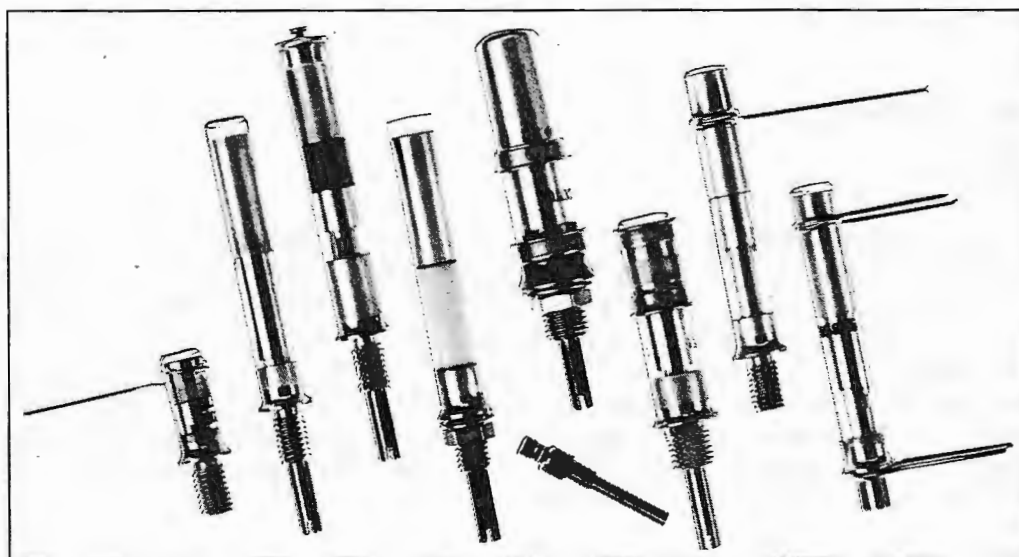
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Using your knowledge and at least 2 years' experience with NMR spectroscopy, you will design and implement software for the analysis and display of NMR data. Working in the Research and Development group, in Palo Alto, CA, you will be actively involved in the latest NMR innovations from the fields of liquids, solids and microimaging. MS or PhD in Chemistry, Physics, or related scientific discipline with "C" programming experience is required.

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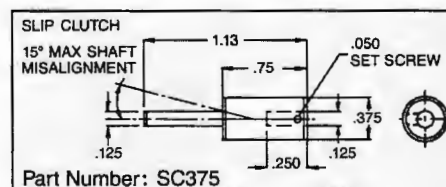
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18. Juli 1991

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 Heinrichstraße 28
 Tel. (0316) 380

Unser Zeichen:

Prof. B. L. Shapiro
 TAMU NMR NEWSLETTER
 966 Elsinore Court
 Palo Alto, CA 94303

"Felix" an open program!

Dear Prof. Shapiro :

Like many other spectroscopists we installed "Felix" distributed by D. Hare on a sun spar station interfaced via Ethernet to our Bruker AM 360 spectrometer. The program is mostly used in manipulating, processing and displaying nmr-data. However, as D.Hare delivers the file formats of "Felix" the nmr-data files can be easily accessed by the users "home-made" programs for the purpose of further numerical analyses of different kinds. To encourage colleagues to take this opportunity we would like to present two subroutines which might provide an interface to "Felix".

```
subroutine retelix(datfil)
common c, isize, irc, sw, sf
character datfil*10
complex c(1024)
real r(2048)
integer ip(2048)
equivalence (c,r), (c,ip)
open (21, file=datfil, form='unformatted', status='old')
read(21) i, (r(j), j=1, abs(2*i))
if (i.gt.0) then
  isize=i
  irc=1
  close (21)
  return
endif
isize=float(ip(1))
irc=ip(2)
sw=r(17)
sf=r(18)
read(21) i, (r(j), j=1, 2*i)
close(21)
return
end
```

```
subroutine wrfelix(outfil)
common c, isize, irc, sw, sf
character outfil*10
complex c(1024)
real r(2048)
equivalence (c,r)
real p(32)
integer ip(32)
equivalence (p,ip)
open(22, file=outfil, status='unknown')
close(22, status='delete')
open(22, file=outfil, status='new', form='unformatted')
np=16
do i=1, np
  ip(i)=0
  p(i+16)=0.
end do
ip(1)=isize
ip(2)=1
ip(3)=0
p(17)=sw
p(18)=sf
write(22) -np, (p(i), i=1, 2*np)
if (ip(2).eq.1) then
  write(22) ip(1), (c(i), i=1, ip(1))
else
  write(22) ip(1), (r(i), i=1, ip(1))
endif
return
end
```

Yours sincerely

H. Sterk
 H. Sterk

G. Kontaxis
 G. Kontaxis

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

July 30, 1991
(received 8/10/91)

"Making Plots in the FELIX program with an HP Draft Plotter (Draft Master RX HPGL/2)"

Dear Professor Shapiro:

During the past half year we have been actively engaged in establishing a peptide/protein NMR facility. Our acquisitions included a Bruker AMX-500 NMR spectrometer connected via TCPIP to a Silicon Graphics 4D/35GT workstation with an HP-Draft plotter. We currently have three software packages residing on our SG 4D/35GT which include FELIX, DSPACE and MADNMR. All of our data processing and analysis is conducted on the SG workstation using the FELIX program. We have been very pleased with the quality of the processed data, however we did experience a problem with plotting on the draft plotter using the FELIX program. In order to make a plot on the draft plotter one had to exit from the FELIX program and modify the plot file. For example, we found that plots of the size (15 X 14 in.) could be generated by replacing the first line of the plot file with the following expression:


PS; IP; SC-500, 20000, 0, 14000;

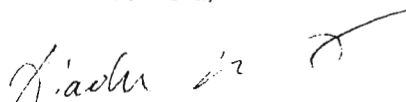
and generating a plot outside of the FELIX program.

This problem was subsequently brought to the attention of Dennis Hare. A new version of FELIX (1.1) was generated which enables plotting from inside the FELIX program, however the program automatically sets the size of the plot to the entire width of the page. To control the size of the plots on the draft plotter, it is still necessary to exit from FELIX and follow the procedure described above. This problem only exists with our draft plotter but not with our HP-7550B plotter.

If anyone having the same Draft Master RX HPGL/2 plotter has found an alternate means of generating custom sized plots from inside the FELIX program, we would appreciate receiving your input.

Sincerely,


Nina C. Gonnella, Ph.D.


Xiaolu Zhang, Ph.D.



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

Dr. David E. Axelson
Group Leader
Magnetic Resonance Imaging Centre
Petroleum Recovery Institute
3512-33rd Street N.W.
Calgary, Alberta
Canada T2L 2A6

August 10, 1991
(received 8/19/91)

Materials MR Imaging Centre

Dear Barry,

This is just a brief letter to describe a new materials MR imaging centre that will be in operation later this year, and to indicate a change of address for me personally.

We have recently purchased a Bruker Biospec/3 24/30 2.35T instrument with a number of multinuclear probeheads for doing everything down to microimaging at high gradient field strengths. A consortium of companies with primary interests in petrophysical applications has also been assembled. While a major part of the instrument time will therefore be dedicated to oil cores and related experiments using high temperature, high pressure and flow - related experiments, we also have a considerable interest in applications to a variety of other fields / industries including: forestry / pulp & paper, agriculture, polymers / composites / advanced materials and emulsions, to name but a few.

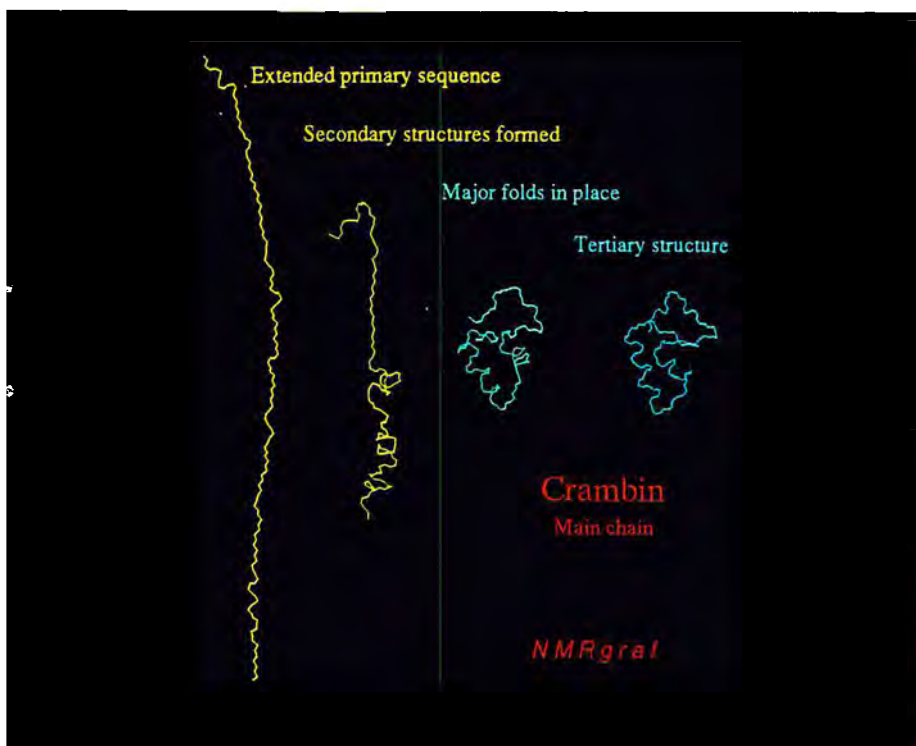
Interested parties should contact me at the PRI address given above for details.

Sincerely,
Dave
Dr. D.E.Axelson

Petroleum Recovery Institute
3512 - 33 Street N.W., Calgary, Alberta, Canada
T2L 2A6

Telephone: (403) 282 - 1211 Fax: (403) 289 - 1988

NMR Structure Prediction Software



Four snapshots of the main chain of crambin as it is folded from its primary sequence into its proper tertiary structure by NMRgraf. This trajectory utilizes NOE annealing that incorporates both experimental NMR data and molecular modeling techniques to predict complex molecular structures.

Structure Determination With NMR Spectroscopy

NMR spectroscopy is gaining wide acceptance as an essential technique for the determination of the 3-D structure of molecules and macromolecules.¹ It is especially useful with molecules in solutions or membrane bound complexes (such as drug receptor sites) to which X-ray diffraction cannot be applied due to the difficulty in obtaining crystals.

The interpretation of NMR data to predict 3-D molecular structures has been derived from two complementary techniques, molecular dynamics simulation and distance geometry. Molecular dynamics simulation attempts to find low energy structures by searching conformational space, while applying experimental constraints as an energy pseudopotential. Distance geometry obeys all known experimental or covalent distances, but ignores electrostatics and non-bond interactions. While each technique has made a significant contribution to the 3-D interpretation of NMR data, neither incorporates all the experimental and empirical information that is available to determine candidate structures.

Combining restrained molecular dynamics simulation and distance geometry takes advantage of both the experimental NOE distance constraints and interatomic energetics within the molecule so that candidate structures reliably conform to experimental data and exhibit low energies.² Improved data analysis tools and the advent of 3-D and 4-D NMR analysis techniques are now providing excellent structure information on both small and large

proteins.³ Because of the ability to determine structure for molecules that can't be solved using other methods, NMR will become an increasingly important tool for determining molecular structure.

Application of Molecular Dynamics to Experimental NMR data

NMRgraf is a fully-integrated program that uses Nuclear Overhauser Effect (NOE) and J-coupling data obtained from NMR experiments as a starting point for molecular mechanics and molecular dynamics simulation in order to predict, verify, and visualize the 3-D structure of molecules, both large and small.

NMRgraf uses pre-tested simulation and structure refinement strategies to incorporate the chemical properties embodied in inter-atomic force fields (such as bond lengths, angles, dihedrals, electrostatics, and van der Waals interactions) with experimental inter-proton distance and angle data obtained from NMR spectroscopy. The resulting 3-D structure both satisfies the NOE restraints and is consistent with the chemical properties embodied in force fields.

No Previous Modeling Experience Required

NMRgraf has been developed specifically for use by NMR spectroscopists. It requires no special training or experience in modeling techniques. NMRgraf has a windowed, icon-driven, user-interface, and on-screen, interactive menus to help novice users achieve rapid, accurate data entry and analysis. Advanced users are equally well accommodated with a macro language and full functionality that is accessible from the command line.

Back Calculation & Interactive Model Validation

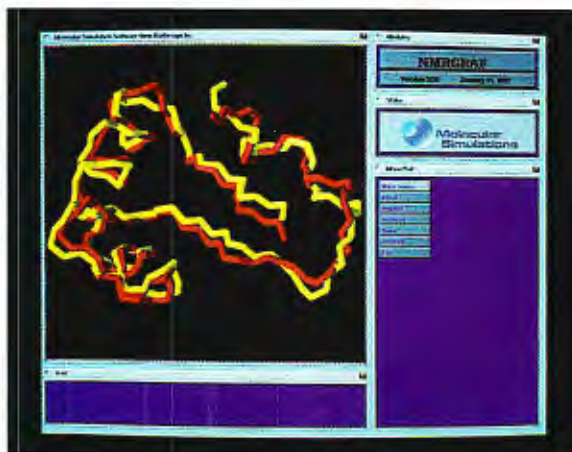
The most tedious and time consuming aspect of NMR research is resonance assignment. NMRgraf has been integrated with NMR data analysis software so that it allows spectroscopists to run both programs simultaneously and interactively. This ability to work with both programs in a windowed environment reduces the "guess work" involved in peak assignment.

For example, a spectroscopist may point to any peak in the NOE spectrum in one window, and NMRgraf will draw lines in a second window between atom pairs of the predicted 3-D structure that could be assigned to that peak, based on intensity information.

1. Wüthrich, K., *Science* 243, 45 (1989)
2. van Gunsteren, W.F., Boelens, R., Kaptein, R., Scheek, R.M., and Zuiderweg, E.R.P., "Molecular Dynamics and Protein Structure" (J. Hermans, Ed.), p.92. Polycrystal Book Service, Western Springs, IL, (1985)
3. Pool, R., *Science*, 249, 364-365, (1990)

After predicting a 3-D structure, NMRgraf back-calculates the NMR spectrum by calculating a relaxation matrix incorporating all spin diffusion contributions resulting from proton-proton interactions.⁴ NMRgraf calculates peak intensities for display and comparison with the experimental spectrum. The scientist can also point to any back-calculated peak and NMRgraf will draw a line between the atom pairs that correspond to that peak in the 3-D structure.

Comparison of the predicted structure of crambin (main chain atoms only), with the native structure obtained from X-ray crystallography. The NOE data set of approximately 600 interatomic distances was generated from the experimental structure. The RMS difference for the main chain is 0.9 Angstroms.



This interactive link between NMR analysis software and NMRgraf helps the scientist rapidly identify and reassign any incorrectly assigned peaks. Changes in the assignment of experimental peaks are reflected immediately in the 3-D predicted structure and also in the back-calculated NOEs.

Embed Technique To Determine Starting Structures

NMRgraf employs the metrization and embed techniques to automatically generate multiple starting structures. Embed generates multiple structures by assigning known NOE distances and then randomly assigning unknown distances.⁵ By generating a variety of structures that conform to the known NOE

data, embed confines the molecular dynamics simulation to the maximum relevant conformational space and eliminates in advance any structures which are inconsistent with the NOE's.

Initial structures also can be read-in from an outside data bank (such as Brookhaven Protein Databank or Cambridge Crystallographic Database), or in-house company database. They also can be built from sequence information using NMRgraf's specialized, menu-driven builders and libraries of fragments covering L- and D-amino acids, RNA/DNA, lipids, carbohydrates, solvents, and various organic fragments.

NOE and J-coupling data also can be read directly from NMR analysis programs, including Hare Research's FELIX and New Methods Research, Inc.'s NMR2™ and NMR2/Model™.

Interface to BIOGRAF™ & POLYGRAF™

NMRgraf is completely integrated with BIOGRAF molecular simulation software for biological systems and with POLYGRAF molecular simulation software for polymeric and materials systems for more extensive modeling studies.

Supports Industry Standards and Platforms

NMRgraf supports industry standard X-windows and OSF MOTIF, and operates on a variety of 3D workstations, including Silicon Graphics IRIS™, IBM RS6000™, and Stardent TITAN™ series.

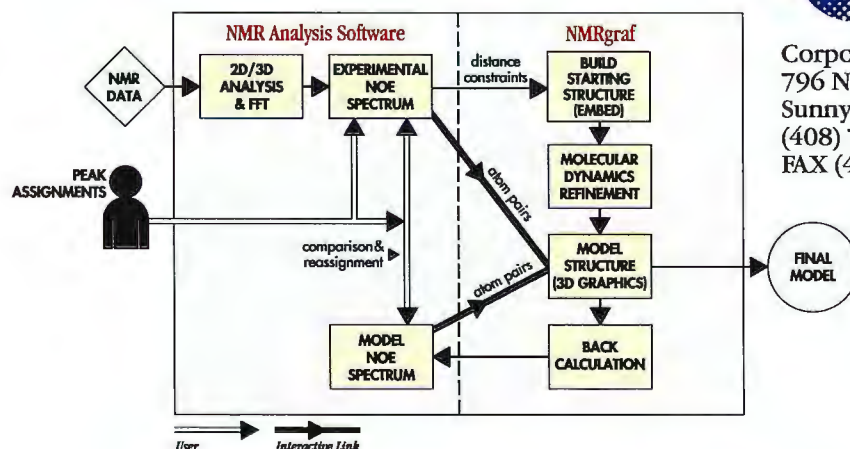
Free 30-day Trials Available

NMRgraf is available to qualified scientists for a thirty-day free trial. Scientists interested in evaluating NMRgraf are urged to contact their Molecular Simulations sales representative.



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NMRgraf's direct interface to NMR data analysis allows the user to operate both programs simultaneously and interactively in a windowed environment. This interactive link between NMR analysis software and NMRgraf helps the scientist rapidly identify and reassign any incorrectly assigned peaks.

4. Kaptein, R., Zuiderweg, E.R.P., Scheck, R.M., Boelens, R., and van Gunsteren, W.F., *J. Mol. Biol.* 182, 179, (1985)

5. Crippen, G.M., *J. Comp. Phys.* 24, (1977)

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August 7, 1991
(received 8/10/91)

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

RE: Addition of a Check Valve to Bruker AC/AM Spectrometer with
Sample Changer

We have recently installed a Bruker AC-300 equipped with a BACS-60 sample changer, BVT-1000 variable temperature controller and 10 mm carbon probe. This system is to be kept at 105°C to study the compositions of polyolefin resins using C-13 NMR. The high temperature operation requires the use of 10 mm ceramic spinners. These ceramic spinners are considerably heavier than the plastic spinners normally used at room temperature. The combined use of the heavier ceramic spinners and the sample changer created an unexpected problem.

This problem developed during the first weekend of high temperature operation using the sample changer and automatic software. We discovered that one sample tube was broken, spilling its contents inside our new 10 mm probe. After cooling the probe and carefully removing all residual traces of solvent and polymer, we pondered the possible causes of the disaster. We quickly realized that the sample tube was broken because it descended too quickly when lowered into the probe under computer control. This happened despite the presence of a buffer tank designed by Bruker to specifically slow the descent of the sample tube by providing a cushion of air when the sample lift air is shut off.

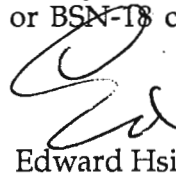
After considerable discussion with Bruker engineers and an application chemist, we were able to verify that all air connections were correctly installed. We were then told to look for a leak which allowed the buffer tank

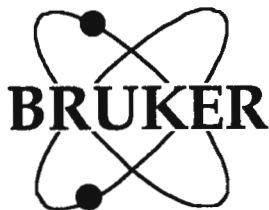


air to dissipate prematurely leading to an excessively rapid descent. We isolated the "leak" to the sample changer side by plugging the air line from the sample changer and using the BSN-18 to lift and drop the sample. The buffer tank system worked flawlessly under these conditions. Careful examination of the sample changer air lift valve confirmed that the valve functioned properly. It opened and closed under computer control. Our problem was caused by the sample changer air lift valve leaking air in the reverse direction than it is designed to operate, i.e., the air lift valve shut off the lift air but allowed the air from the buffer tank to leak. This small leak was enough to cause the observed problem when a ceramic spinner is used.

The solution to this is simple. We installed a one inch air check valve between the sample changer air lift line and the buffer tank. The large size of the valve is determined by the high air flow rate requirement. The check valve prevents the buffer tank air from leaking through the air lift valve. The buffer tank now works flawlessly in lowering the samples with ceramic spinners under both sample changer control or BSN-18 control.


Jack Perry


Edward Hsi



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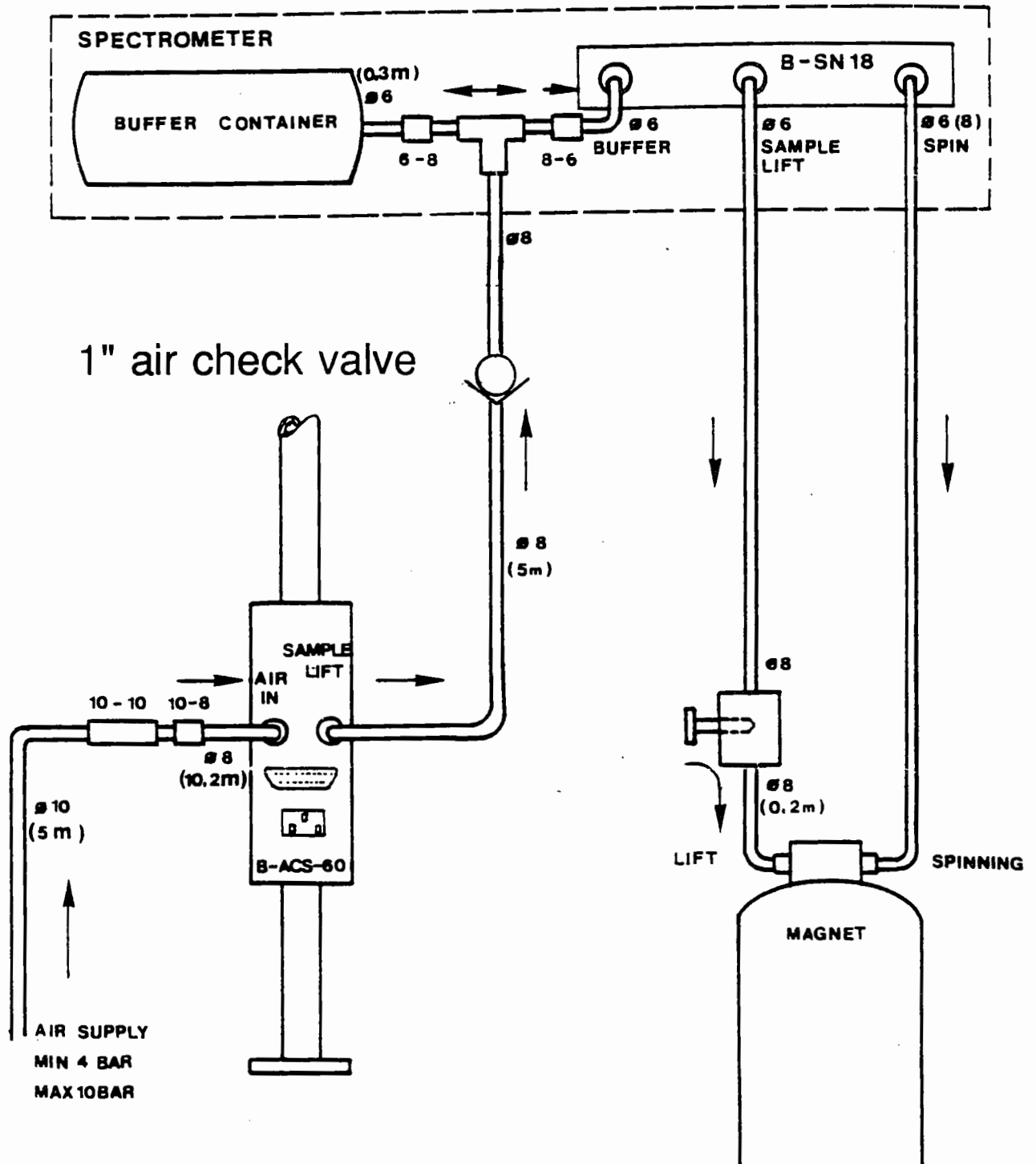
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Figure 1. Diagram of BACS-60 Connection To BSN-18 With Check Valve Installed



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Technology Department

August 2, 1991

Professor Bernard L. Shapiro
Texas A&M NMR Newsletter
966 Elsinor Ct.
Palo Alto, CA 94303

Improving the performance of "older" spectrometers by using narrow-band preamplifiers

Dear Barry,

Since it is time to send in another contribution, we felt that it would be useful to say something about a few of the modifications made to our vintage NT-300. For those that are not aware of the improvements that can be made by utilizing narrow-band preamplifiers, it may be worthwhile mentioning. We have been using Janel preamplifiers (Janel Labs, Corvallis, OR) for both ^1H and ^{13}C to eliminate the unwanted signals going into the broadband preamplifier. The Janel preamplifiers that we are using have a 1.5 db noise figure and a gain of 16 db. We have also installed back-to-back diodes (IN4148) to protect the preamplifier input and allow the transcoupler to function properly. The diodes are placed across the input to the Janel preamplifier which is installed before the input to the broadband preamplifier. The Janel preamplifiers require +15 volts, which may be taken from the broadband preamplifier.

The advantages gained will naturally depend on the type of spectrometer and whether the modification is even necessary. However, improvements should be more noticeable on older spectrometers. Overall, we have found that this type of modification is a good, relatively inexpensive way (about \$40.00 each) of improving the performance of a spectrometer.

Best regards,

A handwritten signature in dark ink, appearing to read "Ken", positioned above the printed name "Kenneth D. Hope".

Kenneth D. Hope

Larry D. Sims¹

¹University of Houston, Department of Chemistry, Houston, Texas 77204-5641

The NMR Data

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Software

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tecThe logo for tecmag, featuring the word 'tec' in a bold, sans-serif font, followed by 'mag' in a similar font. The 'a' in 'mag' is stylized with a dot, and the 'g' has a long, horizontal tail that ends in an arrow pointing to the right.

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July 22, 1991
Strasbourg, le (received 7/29/91)
Prof. B; Shapiro
966 Elsinore Court
Palo Alto

Phase transition and rotor breaking in MAS experiments

Dear Barry

We have recently broken a MAS spinner; this happens sometime! but in our case, the accident seems strange and after analysis we find that a phase transition may be at the origin of such events.

We have solid state MAS NMR experiments in progress on tellurium compounds. This nucleus has often, as all heavy nuclei, large chemical shift anisotropies and high speed rotation is needed when several chemical shifts are present. When we buy our MSL 300, during the tests, we were able to spin the adamantane sample up to 21 kHz with our 4 mm Bruker probe. We then have decided to spin a tellurium sample at 20 kHz. The experiment goes on without problems about three hours and looking from time to time to the spectrum we were very satisfied, when the spinner suddenly crash. As we were a bit surprise by that event, we look to the spectrum and we discover on the left side of the main peak a very small line which does not exist before. We then have thought to a possible phase change.

The pressure at the surface of the spinner may be obtained by a simple calculation which gives: $P = 4\pi^2 n^2 R^2 \rho / 3$ where n is the number of rotations per second, R the internal radius of the spinner and ρ the mass per unit of volume. Applied to our situation, $n = 2 \cdot 10^4$ Hz, $R = 1.5$ mm and $\rho = 5.6$ g.cm⁻³. This gives a pressure of $66.3 \cdot 10^6$ Pascals or 650 atmospheres! Between zero at the center and this pressure, conditions are met for some phase change in many circumstances. When the transition occurs, the small change of the cristal shapes is sufficient for a small imbalance of the rotor.

The literature saids that this compound has two possible phases at room temperature. A usual one with $\rho = 5.6$ g.cm⁻³ and a second one of higher density with $\rho = 5.9$ g.cm⁻³. The second has been obtained by the effect of the pressure.

This result has also be found recently by S. Hayashi and K. Hayamizu [Bull. Chem. Soc. Japan **64**, 1386 (1991)] who have observed conformational changes according to the spinning rate on a silicon rubber.

If we apply the previous formula to a usual situation: $n = 5$ kHz, $\rho = 1$ g.cm⁻³ (a usual value for organic compounds) and $R = 1.5$ mm, one obtains $P = 7.25$ atmospheres which is a more comfortable situation.

Conclusion: Pay attention to phase transitions not only to avoid rotor breaking but also in structural problems.

Yours sincerely

P. GRANGER

POST-DOCTORAL POSITIONS

As part of a new initiative in structural biology several new NMR laboratories with interests in protein structure and function are being established in Toronto, Ontario, Canada. Research interests of the new labs include protein structure, folding and dynamics (J.F.-K. & L.E.K), design and optimization of multidimensional NMR experiments (L.E.K), protein-DNA and protein-protein interactions (C.H.A. & M.I.), in addition to the established program of J.P.C. (polysaccharide and glycoprotein structure and dynamics). There will be the opportunity for collaboration among these laboratories as well as with other molecular modelling and crystallographic groups in the city. Currently available instrumentation includes 500 MHz (4) and 600 MHz (1) spectrometers (4 of these are brand-new with multichannel, multinuclear capabilities). An additional 500 and 600 will be acquired within the next two years. Candidates should have experience in one or more of the following areas: multidimensional NMR, structure determination of macromolecules, isolation and purification of proteins, molecular biology, and/or scientific computing. To apply for positions, interested candidates should send their CV to the appropriate investigators listed below. Openings are available starting from Dec. 1991 onward.

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Stanford University
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Dr. Julie Forman-Kay
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Biochemistry Research Division
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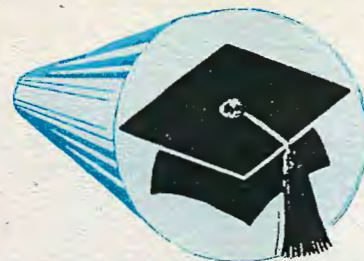
Dr. Lewis E. Kay
Depts. of Medical Genetics and Chemistry
Medical Sciences Bldg.
University of Toronto
Toronto, Ont. M5S 1A8
FAX (416) 978-4194

Address until Jan. 1992
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PW 5 0
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AQ 3 277
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NS 72
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D2 7925 000
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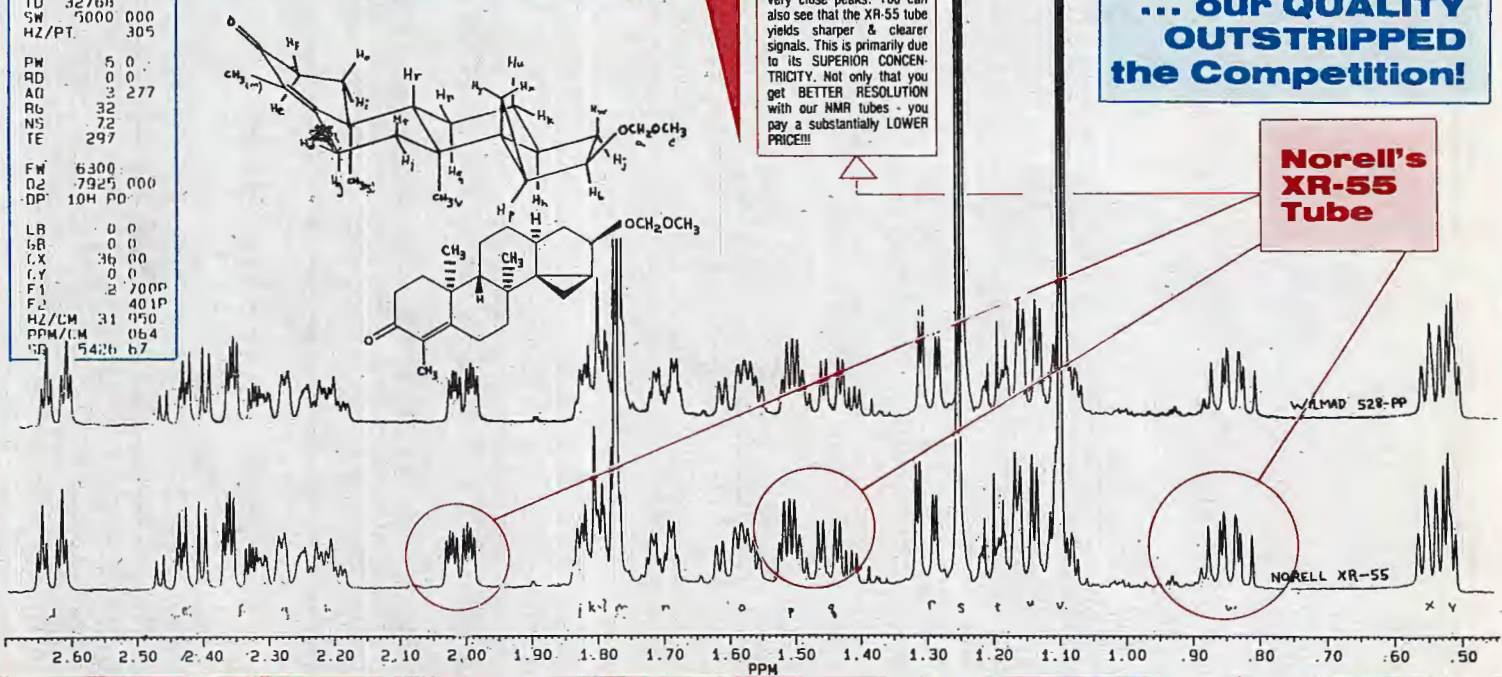
Comparison and evaluation carried out at Harvard University by Scott Virgil on a Bruker AM-500 NMR unit, on November 30, 1988.

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Spectroscopy Centre

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

Kosygina-4
117977, Moscow
USSR

25 July 1991
(received 8/12/91)

WHAT IS THE NMR USERS CHOICE ?

Dear Dr. Shapiro,

We'd like to present for your kind consideration our brief comparison of various NMR manufactures by the popularity of their equipment among the users.

We suppose that the "popularity" of a certain company correlates with the number of researches performed on its NMR instruments. In this respect we analyzed a wide range of publications in such traditional fields of NMR as chemistry, biochemistry and molecular biology, and development of NMR techniques. These publications involved more than 1000 articles from "Magnetic Resonance in Chemistry", "Biochemistry" and "Journal of Magnetic Resonance" for 1989,90,91.

The collected data were transformed into the diagrams shown in figures 1-4. They demonstrate that Bruker NMR instruments are most frequently used in the recent NMR researches.

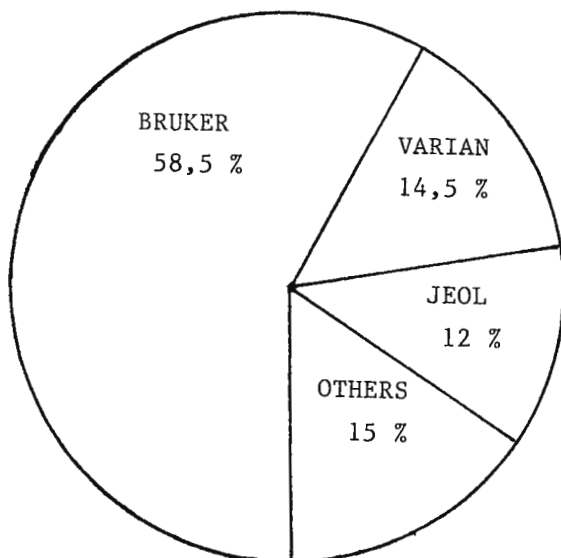
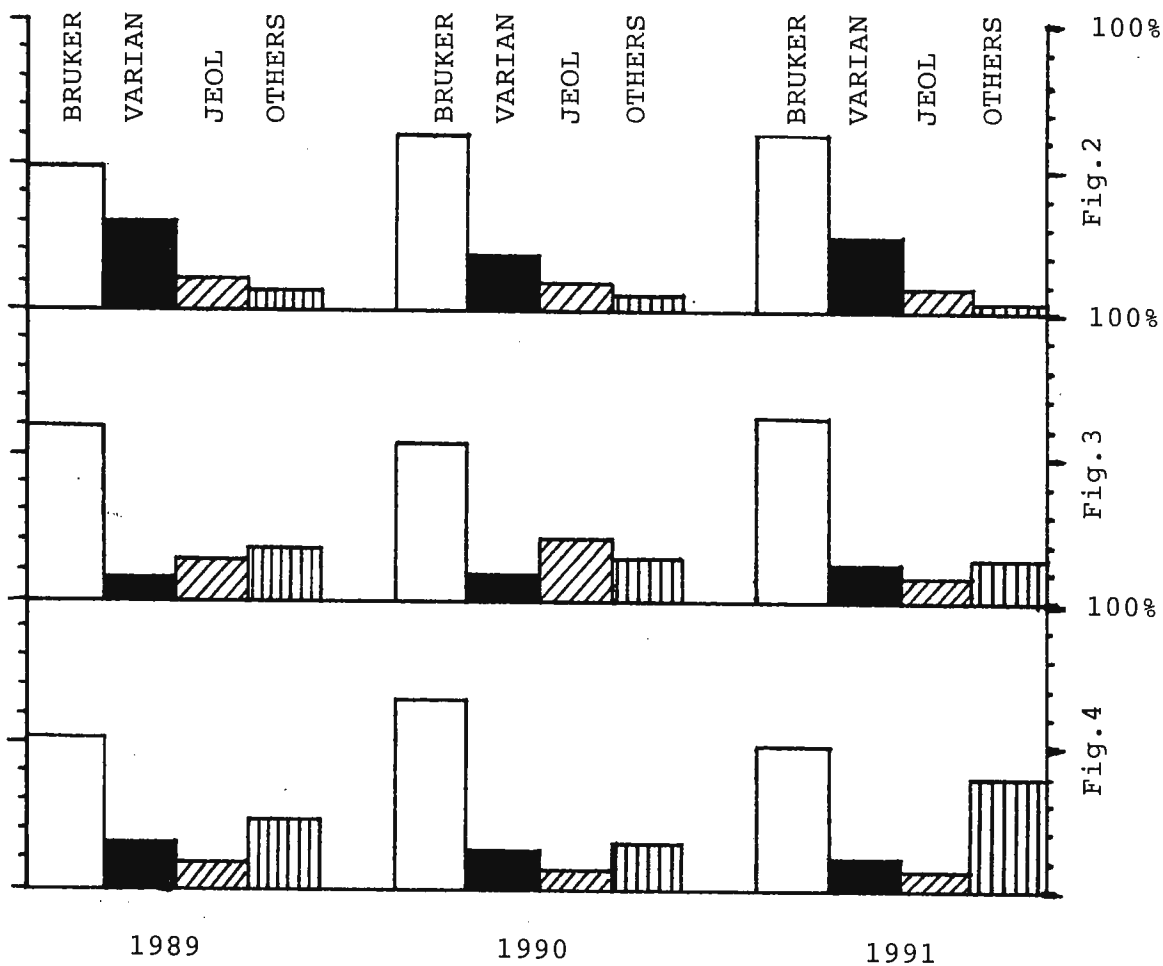


Fig.1.
Generalized data for
1989, 90 and 91 years.



The histograms for "Magnetic Resonance in Chemistry" (Fig. 2), "Biochemistry" (Fig. 3) and "Journal of Magnetic Resonance" (Fig. 4).

Yours sincerely,

M.Yu. Pokrovskaya

D.Yu. Artemov

ANNOUNCING

A BREAKTHROUGH IN GRADIENT SPECTROSCOPY:

PHASE - SENSITIVE GRASP

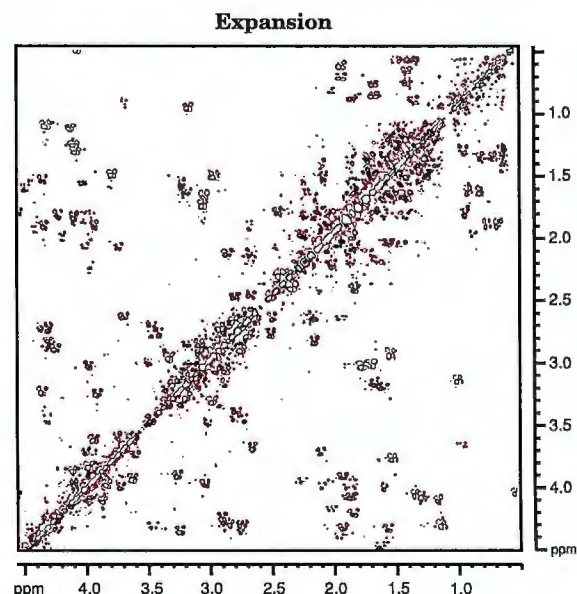
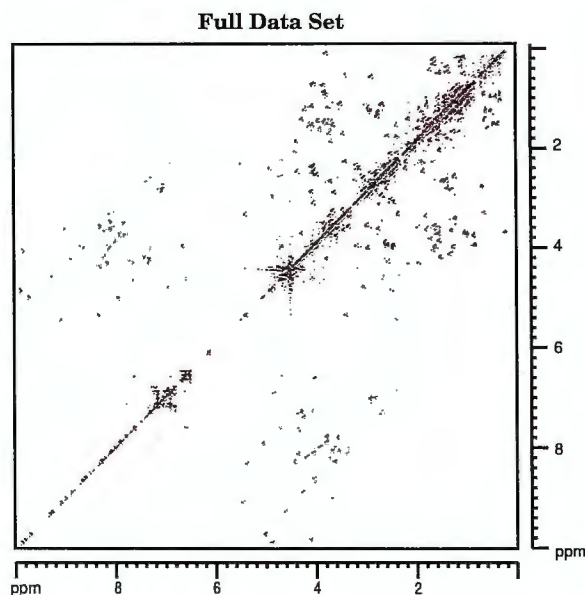
Phase-sensitive gradient DQF COSY of 3mMBPTI in 90% H₂O/10% D₂O on an AMX 600 using a GRASP II accessory including a 5mm triple-resonance probe [¹H {¹³C, ¹⁵N}] with integrated Z-gradient. 60G/cm, no phase-cycling, no baseline correction, symmetrized; (D. Moskau, Spectrospin AG)

Gradient Spectroscopy (GRASP) has tremendous potential for accelerating the acquisition of long 2D/3D NMR spectroscopy experiments, by eliminating the need for lengthy phase-cycling for quadrature detection in ω_1 . Other GRASP benefits are its superb water-suppression capability, and reduced T₁-noise.

However, until now GRASP was not practically applicable to NMR spectroscopy of larger molecules, because only magnitude-mode spectra could be obtained (except in artificial low-bandwidth cases of e.g. 1 ppm ranges at 300 MHz). Recently, important advances were reported (1) towards obtaining phase-sensitive GRASP-data at high field over a broad frequency range on an AMX 600 equipped with a GRASP II accessory.

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(1) A. L. Davis, E. D. Laue, J. Keeler, D. Moskau, J. Lohmann, *Journal of Magnetic Resonance*, in press.



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Department of Physics

August 10, 1991
(received 8/12/91)

Dr. Bernard Shapiro
966 Elsinore Court
Palo Alto, CA 94303

Dear Dr. Shapiro:

We are studying physisorbed layers of HD, H₂, and D₂ on unusually uniform MgO substrate. A published preparation¹ yields MgO powder of ~ 8 m²/gram surface area and high surface uniformity (100 faces), as indicated by the sharpness of adsorption isotherms. Our MgO was prepared by our collaborator Dr. John Larese of Brookhaven National Laboratory and has ~ 28 m²/g.


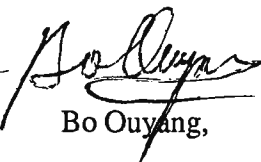
Here we present deuterium NMR results of HD at 4.2 K. Figure 1 shows the spectra for $x = 0.9, 1.0$, and 1.13 ($x = 1.0$ is approximately a filled layer). As x approaches and then exceeds one, a sharp central feature appears from the molecules in the second layer. Evidently the broad spectral component is from the tightly bound first layer. The ratios of intensities (areas) are in accord with this assignment. For $x = 1.50$ (not shown), the sharp feature is very prominent (off scale).


In Figure 2, the 4.2 K spectrum of 0.5 layers HD plus 1.0 layers H₂ is displayed. This system was annealed at 40 K and then cooled. Because no central sharp line appears, we conclude that none of the HD is in the second layer. We attribute this isotope effect to quantum zero-point motion: the binding energy to the surface is the attractive potential minus the $\hbar\omega/2$ zero-point energy. We note that as little as 1% HD in the second layer would be evident in Fig. 2.

Figure 3 shows the time domain *multiple-echoes* for a mixed system of 1.1 layers of ortho-D₂ (even J, nuclear spin 0 and 2) plus 0.4 layer of HD, at 4.2 K. The data begin after the second of two rf pulses. The occurrence of three echoes in HD and ortho-D₂ has been explained; we refer the reader to that treatment². The three echoes in Fig. 3 have times appropriate for ortho-D₂; HD echoes would occur at the times marked by the arrows. The long, smooth decay is from second layer molecules, HD and ortho-D₂. The sharp echoes come from the first layer. Thus, the first layer has only D₂ molecules, not a random mixture. Again, this is the result expected for large zero-point quantum effects: the more massive molecules are preferentially nearest the MgO.

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2. M. P. Volz, P. Santos-Filho, M. S. Conradi, P. A. Fedders, R. E. Norberg, W. Turner and W. Paul, Phys. Rev. Lett. **63**, 2582 (1989).

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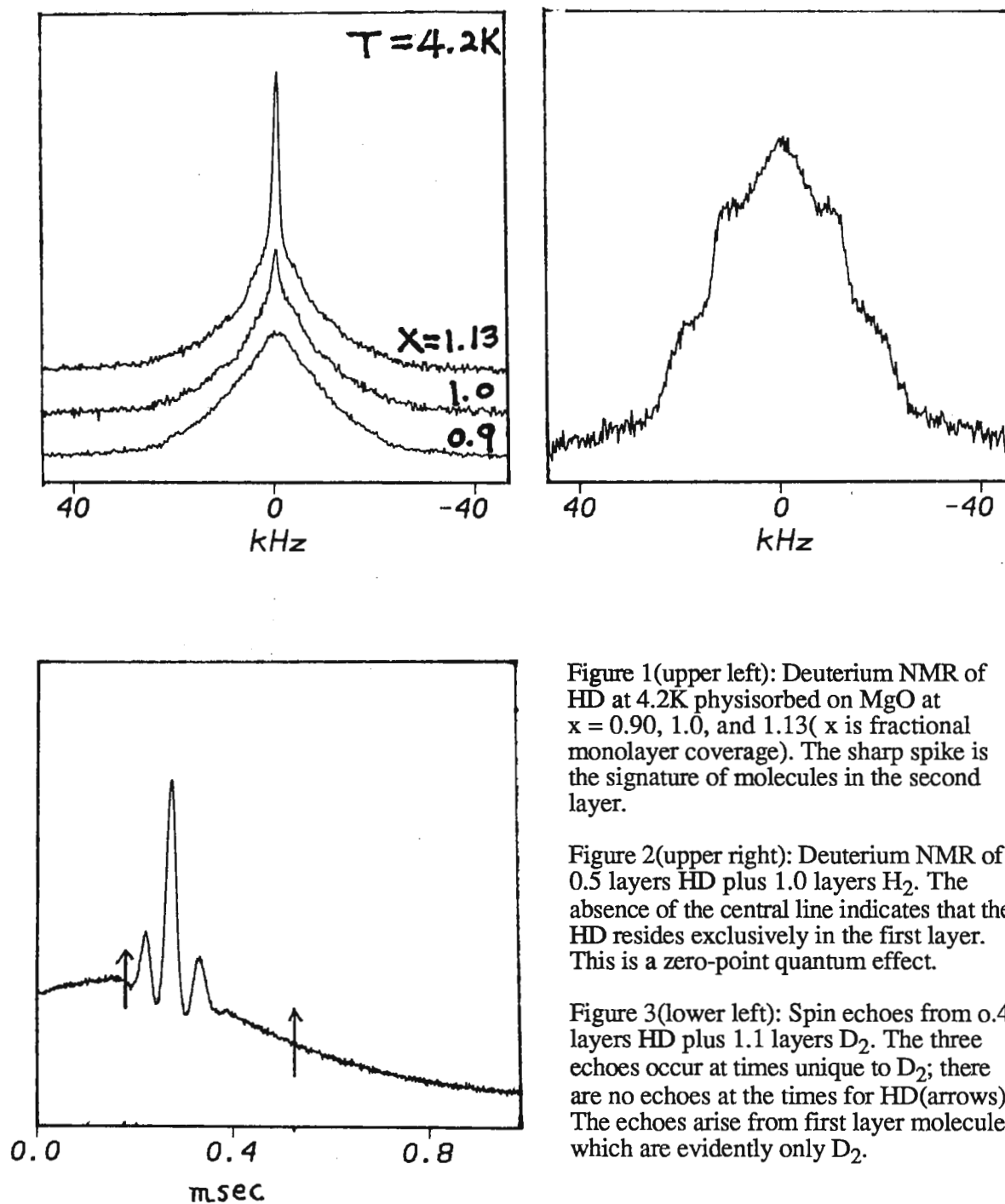


Figure 1(upper left): Deuterium NMR of HD at 4.2K physisorbed on MgO at $x = 0.90, 1.0$, and 1.13 (x is fractional monolayer coverage). The sharp spike is the signature of molecules in the second layer.

Figure 2(upper right): Deuterium NMR of 0.5 layers HD plus 1.0 layers H_2 . The absence of the central line indicates that the HD resides exclusively in the first layer. This is a zero-point quantum effect.

Figure 3(lower left): Spin echoes from 0.40 layers HD plus 1.1 layers D_2 . The three echoes occur at times unique to D_2 ; there are no echoes at the times for HD (arrows). The echoes arise from first layer molecules, which are evidently only D_2 .

Gradient Enhanced Spectroscopy SWAT

GE introduces the use of Switched Acquisition Time (SWAT) gradients to achieve pure phase 2D spectra with quadrature detection in both the acquisition (ω_2) and evolution (ω_1) dimensions without any phase cycling and without an additional set of t_1 data.

One example of a pure phase gradient enhanced COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene- d_6 is shown in Fig. 1. SWAT gradients and a single acquisition per block were used. Data was collected on an Omega 300WB with Microstar actively-shielded gradients. A 5mm inverse probe was built for use within the gradient coils.

Digital resolution of 1.2 Hz in ω_1 and 2.4 Hz in ω_2 was achieved by collection of a 512 x 512 matrix with t_1 evolution time of 840ms and a t_2 acquisition time of 420ms. A single acquisition per t_1 evolution data block and an average recycle time of 1.84s resulted in a 15 minute total collection period.

Since the SWAT gradient method encodes the necessary information in a single t_2 acquisition time, it avoids the collection of additional data blocks required by traditional pure phase methods. This time efficiency is especially important for collection of large multidimensional data sets.

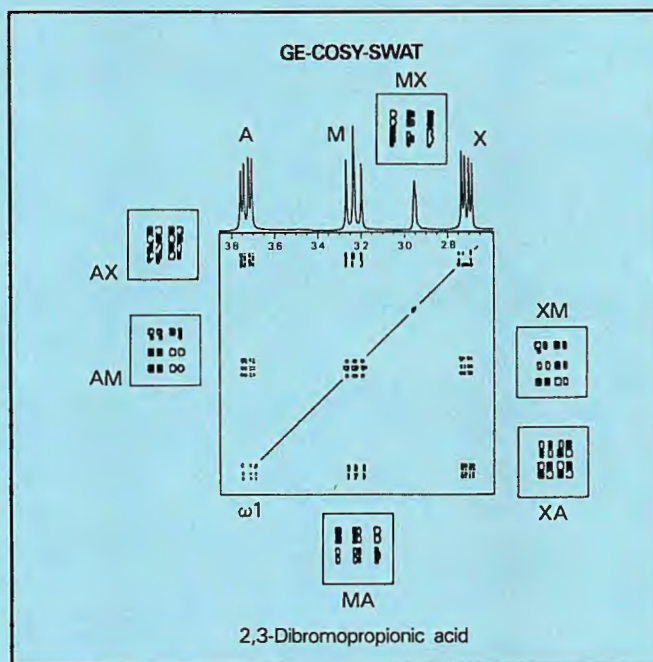


Fig. 1

Contour plot of a 300 MHz pure-phase COSY spectrum of a solution of 2,3-dibromopropionic acid in benzene- d_6 acquired with only a single acquisition per t_1 evolution time increment using the GE-COSY-SWAT method. Cross peaks are shown in expanded insets with positive peaks as darkened contours and negative peaks as open contours. A one dimensional spectrum is plotted across the top of the 2D spectrum.



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