TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 366

March 1989

Continued on page 76

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotopic Purity (%D)	Density (g/cm³)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d4	CD_3COOD	99.5%	1.12	17	118	0.551 (32)
D-120	Acetic Acid-d4	CD_3COOD	99.5%				
D-13	Acetone-d_6	CD_3COCD_3	99.8%	0.87	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							543 (20)
D-21							0.611
D-122							
D-130							
D-28	Chloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

Need Deuterated Solvents?

WILMAD HAS YOUR SOLUTION!

Cost-conscious quality NMR solvents offered by Wilmad, such as $CDCl_3$, are frequently priced lower than more traditional sources. Included in this offering are the most common solvents, like Acetone-d_6, Benzene-d_6, D_2O, and DMSO-d_6, as well as some of the most unusual solvents for specialty applications, like 1,1,2,2-Tetrachloroethane-d_2, Octane-d_8, and Trifluroacetic Acid-d.

VARIAN

BOX/500 SHEETS

Cat. No.	Plot Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	HA-100, HA-100A, HA-100D	Gridded-Two Color
WCV-60 (A-60C)	11" X 26"	A-60, A-60A, A-60D	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100 (CW and FT)	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 (CW and FT)	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WC-11[illegible]	X 17"	All Models [illegible] 16 1/2" or 16	Blank

Use Plotter Paper and Pens?

WILMAD HAS YOUR CHART!

We provide the largest variety of paper and pens for NMR recorders or plotters available anywhere. Included in these listing are the newest spectrometers from Varian, Bruker, (and IBM), General Electric and JEOL, as well as the latest models, such as the Hewlett Packard 7475A, 7550A, and Thinkjet 2225A, Zeta 8 or 8A, and Western Graphtec 4730 plotters and printers.

Searching for the Unusual Requirement?
WILMAD HAS YOUR ANSWER!

The most comprehensive offering of "widgets, gadgets and specials" for NMR spectroscopy, including:

Spatula for 5mm NMR Tubes
Three types of Valve NMR Tubes
(including the new J. Young Valve Tube)
Solvent Jet NMR Tube Cleaners
pH Electrode for 5mm NMR Tubes
Taperlok ® NMR Tubes
A multitude of Coaxial Inserts
Alumina NMR Tube for Si-29 Studies
Ultra-thin wall NMR Tubes
Throwaway "THRIFT" and "ECONOMY" NMR Tubes

Serving the Spectroscopic Aftermarket

WILMAD GLASS COMPANY

Route 40 and Oak Road • Buena, NJ 08310 U.S.A.
609-697-3000 • TWX 510-687-8911
FAX 609-697-0536

TEXAS A&M NMR NEWSLETTER NO. 366, MARCH 1989 AUTHOR INDEX

TEXAS A&M NMR NEWSLETTER NO. 366, MARCH 1989 ADVERTISER INDEX

SPONSORS OF THE TAMU NMR NEWSLETTER

Abbott Laboratories
The British Petroleum Co., Ltd. (England)
Bruker Instruments, Inc.
Cryomagnet Systems, Inc.
The Dow Chemical Company
Eastman Kodak Company
E. I. du Pont de Nemours & Company
GE NMR Instruments
JEOL (U.S.A.) Inc., Analytical Instruments Division
The Lilly Research Laboratories, Eli Lilly & Company
Millipore Corporation, Waters Chromatography Division
The Monsanto Company
New Methods Research, Inc.
NMR Technologies Inc.
Norell, Inc.
The Procter & Gamble Company, Miami Valley Labs
Programmed Test Sources, Inc.
Shell Development Company
Siemens Medical Systems, Inc.
Spectroscopy Imaging Systems Corporation
Spectral Data Services, Inc.
Unilever Research
Union Carbide Corporation
Varian, Analytical Instrument Division

FORTHCOMING NMR MEETINGS

San Francisco Symposium - "In Vivo Magnetic Resonance Spectroscopy II", March 31 - April 2, 1989; San Francisco, California; See Newsletter 363, 17.

30th ENC (Experimental NMR Conference), April 2-6, 1989; Asilomar Conference Center, Pacific Grove, California; Conference Chair: A. N. Garroway; Contact Ms. Judith A. Watson, ENC Conference Center, 750 Audubon, East Lansing, MI 48823; (517) 332-3667. See Newsletter 362, 69.

11th Symposium - NMR: State of the Art and Future Directions, April 29, 1989; Dept. of Chemistry University of Rhode Island, Kingston, RI 02881; Contact: B. Sherman or R. Pothier, (401) 792-2203; See Newsletter 366, 55.

3rd Chianti Workshop on Magnetic Resonance Relaxation, May 28 - June 2, 1989; San Miniato (Pisa), Italy; Contact: L. Banci, Dipartimento di Chimica Bioinorganica, Universita degli Studi di Firenze, Via Gino Capponi 7, 50121 Firenze, Italy. See Newsletter 362, 68.

9th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, July 10-14, 1989; University of Warwick, Coventry, England; Contact: Dr. John F. Gibson; (01) 437-8656; See Newsletter 364, 72.

NMR Spectroscopy In Vivo (Clinical Applications), July 10-12, 1989; Lyon France; Contact Prof. M. Amiel - see Newsletter 364, 73.

10th ISMAR Conference, July 16-21, 1989; Morzine (Haute-Savoie), France. (Note the newly announced location.); Contact: P. Servoz-Gavin, Departement de Recherche Fondamentale, Centre d'Etudes Nucleaires de Grenoble, B.P. 85X, 38041 Grenoble Cedex, France.

The Society of Magnetic Resonance in Medicine - Eighth Annual Scientific Meeting and Exhibition, August 12-19, 1989; Amsterdam, The Netherlands; Contact: The S.M.R.M. Business Office, 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415)841-1899, FAX (415)841-2340.

Eastern Analytical Symposium, September 24 - 29, 1989; New York City; Contact: EAS, P. O. Box 633, Monchanin, DE 19710-0633; (302) 453-0785.

Additional listings of meetings, etc., are invited.

Continued on page 75

366-2

DEPARTMENT OF HEALTH & HUMAN SERVICES — Public Health Service

National Institutes of Health
Bethesda, Maryland 20892
Building : 10
Room : 7N315
(301) 496- 3341

Dr. Bernard L. Shapiro
TAMU NMR Newsletters
966 Elsinore Court
Palo Alto, CA 95303

January 18, 1989
(received 1/24/89)

Molecular Structures Using NMR and Restrained Dynamics

Dear Barry:

For some time now we have been determining the structures of peptides and small proteins in solution using the relevant 1D and 2D NMR techniques (such as HOHAHA and ROESY). During the course of these investigations we have wondered just how well these three dimensional structures are actually being determined. Do the NMR results actually lead to low energy conformations when one considers precision on the estimate of the internuclear distances?

In order to partially answer this question we have initiated two distinct lines of research. We have carried out extensive restrained (by experimentally determined distances) molecular dynamics simulations in the presence of solvent for times up to 100 psec. using many randomly generated starting structures. We find that the restraints are required to obtain acceptable low energy structures during the simulation period, and that it is necessary to explicitly include the surrounding solvent molecules.

We have also investigated the effect of digitization error on the precision in the estimate of cross peak volumes (in NOESY and ROESY) from which the restraints are derived. For typical situations in 2D NMR where a 1024 X 1024 data point matrix covers 3000-5000 Hz X 3000-5000 Hz (protons), errors can easily be as large as 100%. (Note that this error is only due to digitization and that instrument noise adds to the total error.) This translates to a 16 % error in the estimate of the internuclear distance and must be considered when estimating the restraint energies in the dynamics simulations. We are attempting to translate our results to the determination of an "R factor" in order to keep up with our crystallographer friends (if we have any left).

Please credit this contribution to Bob Highet's account.

Best regards,

James A. Ferretti

Jim

Kyou-hoon Han

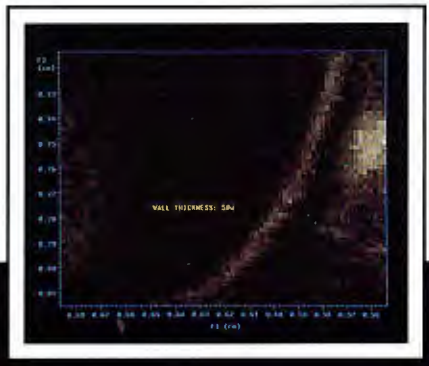
KHH

Now...Explore Higher Performance NMR Imaging!

A Siemens and Varian joint venture. You benefit from single-source access to systems combining Siemens expertise in clinical MR imaging with Varian's leadership in high-resolution NMR.

Only *Spectroscopy Imaging Systems* gives you both the resources of a large corporation and the innovative responsiveness of a dedicated company. You also receive the combined expertise of site planners, system engineers, technical support personnel, and application scientists.

For systems, service and support in imaging spectroscopy, call 415-659-2600 today. Or write to 1120 Auburn Street, Fremont, California 94538.



Spectroscopy Imaging Systems Corporation

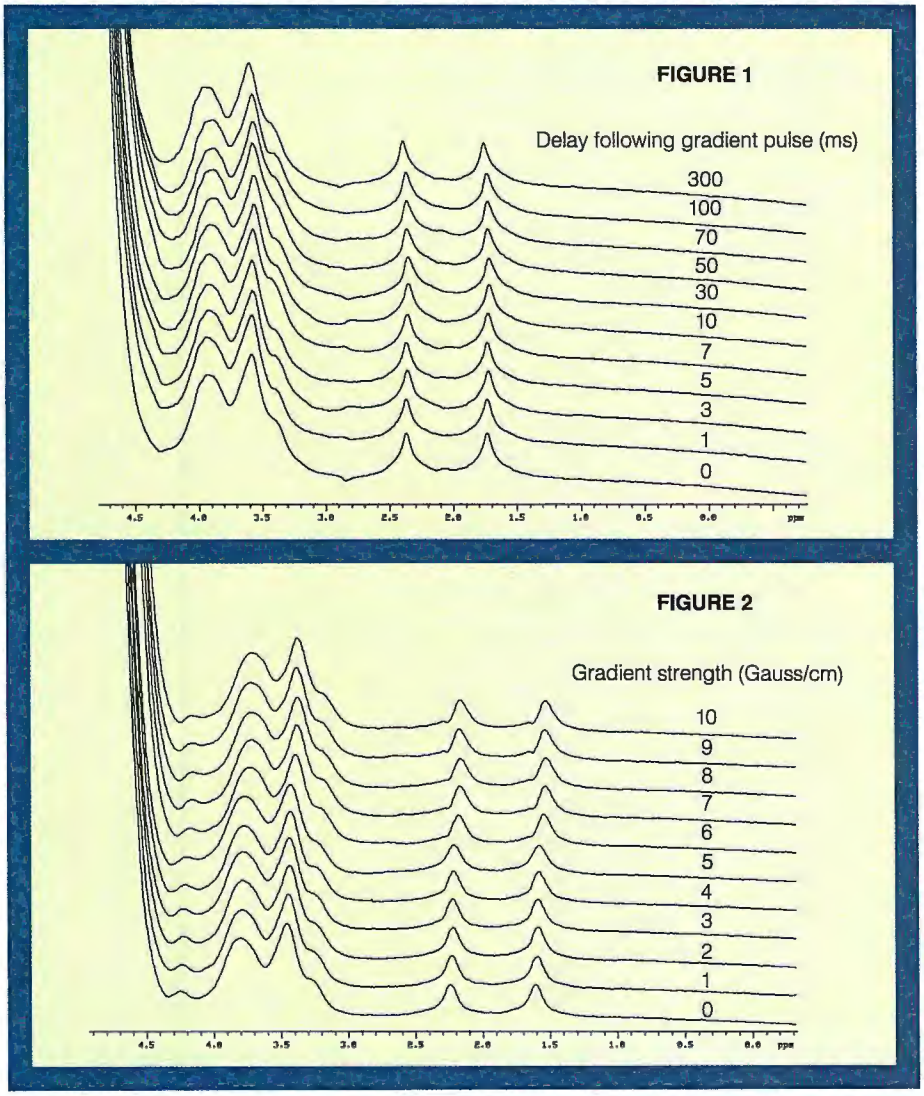
A Joint Venture of Varian and Siemens

(415) 659-2600

High Performance Auxiliary Gradient Coils

Dedicated RF coils, optimized for a specific application or a particular sample or both, have long been a tradition in NMR imaging as well as NMR spectroscopy. Similarly, dedicated gradient coils yield dramatic performance improvements in terms of rise and fall times, maximum gradient strength, duty cycle and eddy currents. Sophisticated eddy current compensation techniques reduce any residual spatial or temporal field variations due to eddy currents to a few hundredths of a percent, thus yielding distortion free spectra and images.

At Spectroscopy Imaging Systems we are able to obtain in-plane resolution of $20\mu \times 20\mu$ in an axial cut through an okra of 1.3 cm diameter. (See image shown on front side.) An enlargement of a portion of a seed capsule within the okra displays a wall thickness of 50μ (inset on front side). The gradient coil used to obtain this excellent performance has a diameter of 125 mm and was mounted in an Oxford Instruments 4.7 Tesla, 330 mm bore magnet without removing the standard set of gradient coils. The auxiliary gradient set is capable of generating greater than 10 Gauss/cm in all orientations using the same gradient power supply as the primary gradient coil.



The excellent control of eddy currents using the smaller gradient insert is demonstrated in Figures 1 and 2. Figure 1 shows a series of spectra of the high field region of a sample containing [2-^{13}C] acetate and [1-^{13}C] glucose in H_2O/D_2O. With the sample in the center of the gradient coil, a 1 second gradient pulse of 1 Gauss/cm was applied. The gradient pulse was followed by a variable delay, a 90° RF pulse and acquisition of the resultant free induction decay. All spectra are essentially undisturbed even at delay times below 10 ms.

Figure 2 shows a typical series of spectra of the same sample at an off-center location in the magnet. A gradient pulse of 5 ms duration with an amplitude range of 0 to 10 Gauss/cm was applied, and the FID recorded at a fixed delay time of 3 ms. The sample was positioned at 22.3 mm off the center, corresponding to local gradient field of 95 kHz. All spectra are virtually undisturbed, even at 10 Gauss/cm.

January 18, 1989
(received 1/28/89)

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

Dear Barry:

SUBJECT: Obtaining Symmetrical Deuterium Spectra of Solids

Symmetrical deuterium spectra of solids are sometimes difficult to achieve. Attempts to convert an unsymmetrical spectrum into a symmetrical spectrum through software phase correction often are futile. The source of the asymmetry generally is in spectrometer tuning, not in phasing.

The asymmetry problem reveals itself through the FID if the operator is unable to adjust the receiver phase to put the signal entirely into the real channel and there is only noise in the imaginary channel. The imaginary channel can be zeroed, of course, but this leads to spectral distortion if that channel does contain part of the FID.

To get symmetrical deuterium spectra we routinely use a tuneup procedure that was developed for proton multiple-pulse nmr. We, of course, adjust the four channels of the transmitter to have equal amplitude and differ in phase by exactly 90 degrees, but we also tune out any phase glitch that may exist. The specific method was developed by Burum, Linder, and Ernst (J. Magn. Reson., 1981, 43, 463). Other tuneup techniques in the literature may also be suitable, but we have not tried them. The test sample is a little deuterium oxide in a small spherical cell. Large test cells lead to problems with rf homogeneity. The tuning generally takes only 10 or 15 minutes during setup and then doesn't have to be repeated unless the spectrometer configuration is altered.

Very truly yours,

Mark

P. Mark Henrichs
Corporate Research Laboratories

PMH:

EASTMAN KODAK COMPANY
Bldg. 81/Fl. 2/RL, Rochester, NY 14650-2021
(716) 477-6229

BP Research International

Sunbury Research Centre, Chertsey Road, Sunbury-on-Thames, Middlesex TW16 7LN

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

Our ref: S/SYB/446/88

Telephone: 0932-76 2142

Date: 14th December 1988 (received 1/30/89)

Dear Professor Shapiro,

NMR AT HIGH PRESSURE

In recent years a number of high pressure nmr systems have been reported (1). At BP we have a number of applications for such equipment including the study of homogeneous catalysis and organic equilibria. However, we were reluctant to commit an entire spectrometer or probe for such use. The hpnmr system described by Roe (2) involving a sapphire tube with a titanium pressure valve seemed to be well suited to our needs and therefore we set about building a similar device.

Before actually commissioning the hpnmr system we had to take into account a number of safety aspects. Obviously, once the tube was pressurised the operator must be protected from it. Therefore we designed a brass carrier with a perspex window to allow the operator to observe the sample without any danger. This carrier slots on the spinner housing at the top of the magnet. It has a removable base plate so that it can easily be used with any of our spectrometers (to date we have used it on our JEOL GX400 and FX270 and Bruker MSL100 instruments).

Our second safety consideration was for our probes! We were unhappy about "dropping" the pressurised tube into the magnet on the compressed air supply, so we designed a device to lower the tube into the magnet which detaches itself automatically from the tube once it is in place in the probe.

Having achieved the required safety standards we tested out the tube. Although it reliably held pressure, we experienced problems with spinning it as it was very heavy. In addition, we noticed a very strong pull on the tube as it approached the centre of the magnetic field, even though the titanium contained negligible amounts of iron (eddy currents?). Therefore we totally redesigned the valve so that a substantial amount of it comprised PTFE rather than titianium. After a number of modifications we now have a system which holds pressure and spins reliably (it is also considerably lighter than the original system).

We now use our hpnmr setup routinely to study organic equilibria and homogeneously catalysed processes at temperatures and pressures approaching those used industrially. Figure 1 shows the ^{31}P spectra of a phosphine complex in a normal nmr tube and in the hpnmr tube. The resolution of the spectrum is not as good as that from the normal tube as the sample contained suspended solids. However, the results were more than adequate to study the fate of the complex during a catalytic cycle.

I hope this contribution will reinstate BP!

Yours sincerely

Dr J D Boyle
Spectroscopy Branch

References

(1) I Ando and G A Webb, Magn. Reson. in Chem. 24, 557 (1986).
(2) D C Roe, J. Magn. Reson. 63, 388 (1985).

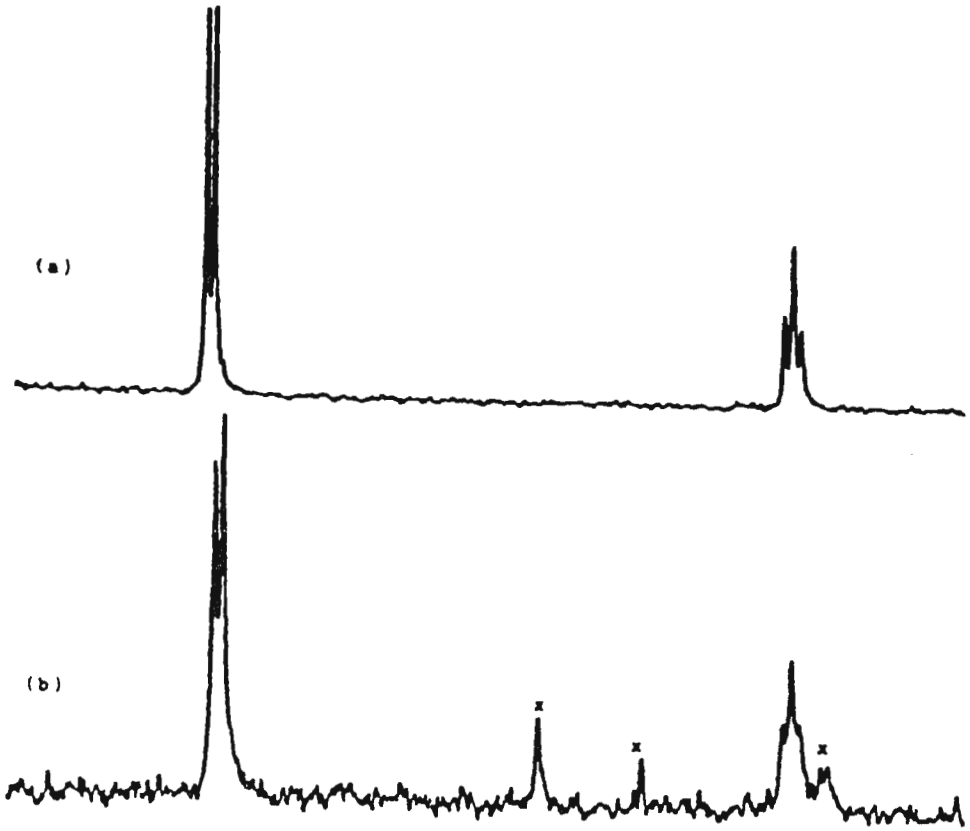


Figure 1 ^{31}P NMR spectrum of a transition metal phosphine complex (a) normal NMR tube (b) sapphire tube

x = impurity

UNIVERSITY OF CALIFORNIA, SANTA BARBARA

BERKELEY • DAVIS • IRVINE • LOS ANGELES • RIVERSIDE • SAN DIEGO • SAN FRANCISCO

SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY
Dr. Hellmut Eckert

SANTA BARBARA, CALIFORNIA 93106

2/3/89
(received 2/11/89)

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

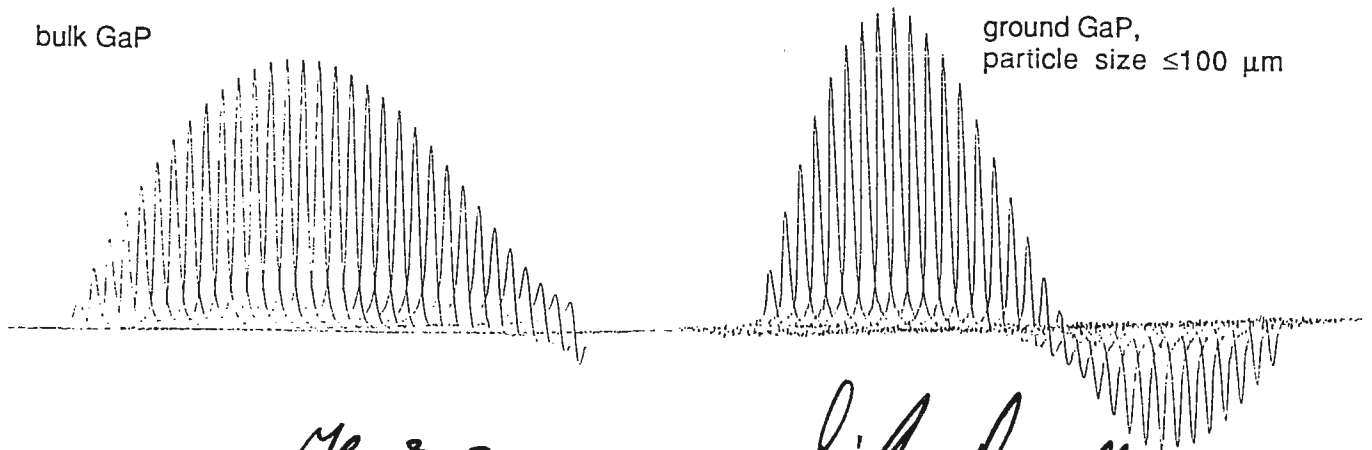
Measurement of Particle Sizes in GaP by ^{69}Ga Nutation NMR

Dear Prof. Shapiro:

In connection with our solid state NMR studies of colloidal semiconductors we have recently taken a look at particle size effects on solid state NMR parameters. Compared below are the ^{69}Ga nutation NMR spectra, obtained at 7.05 T and ω_1 = 33 kHz, of (a) bulk GaP and (b) the same material pulverized overnight in a mechanical alumina grinder in an N_2 atmosphere and passed through a 100 micron sieve. The figure shows normalized frequency domain spectra arranged in order of increasing length of the excitation pulse (from 0.5 to 16μs) in 0.5μs increments. The reduction of the 90° pulse length from 7.5 (bulk) to 4.5 μs (ground) is consistent with a high degree of excitation selectivity for the central 1/2->-1/2 transition of the spin-3/2 ^{69}Ga nuclei in the latter material. Although the introduction of first-order quadrupolar effects in cubic solids by grinding is already mentioned in "THE BOOK",[1] the detection by nutation NMR is more convenient than absolute signal intensity measurements, and leads to results that are easier quantifyable, especially when a distribution of quadrupole coupling constants is present. It should be possible to use the nutation effect for calibrating particle sizes in cubic powders containing nuclei with large quadrupole moments.

[1] A. Abragam, Principles of Nuclear Magnetism, Clarendon Press Oxford; paperback edition (1983), p. 246-249.

Please credit this letter to Tom Gerig's account.



Sincerely,

Hellmut Eckert

Jim MacDougall

One tough customer.

As a pioneer of NMR spectroscopy with over 30 years' experience, Roy Johnson knows NMR systems inside out. That's why he personally approves every one of our units before it's delivered.

And Roy is no easy sell.

As Senior Scientist, he pushes each system to its limits, checking test results and system operation thoroughly. So you'll get the high level of performance you expect—right from the start.

Roy's painstaking approach reflects GE's dedication to quality control. It's just one example of our commitment to your satisfaction.

That's why our systems meet your spectroscopy and imaging needs. With superior RF stability and a high performance digitizer to help provide dependable system performance. Plus data processing flexibility to help you keep pace with evolving sequence and analysis applications.

And you can count on GE service that includes equipment upgrades, software updates and advanced applications support.

At GE, we know NMR customers demand quality performance—from their equipment and their equipment manufacturer. And that's what we provide.

With help from people like Roy.

For more information or to arrange a demonstration, call us at (800) 543-5934.

Or call Roy at (415) 683-4408.

GE NMR Instruments

Put us to the test.

CSI 2T Applications

Shielded Gradients and Localized Spectroscopy

Eddy current effects are the leading cause of errors and lack of consistent results in gradient localization methods. It is not surprising, then, that actively shielded gradients, which have dramatically reduced eddy currents, represent a significant technology advance for all forms of B_o gradient volume localization and spectroscopic imaging methods. The fast rise time and high gradient strength characteristics of the coil used in these experiments are also important.

Even without pre-emphasis, shielded gradients recover fast enough to obtain spectroscopic information at 1 msec or less after a strong gradient has been turned off (Fig. 1).

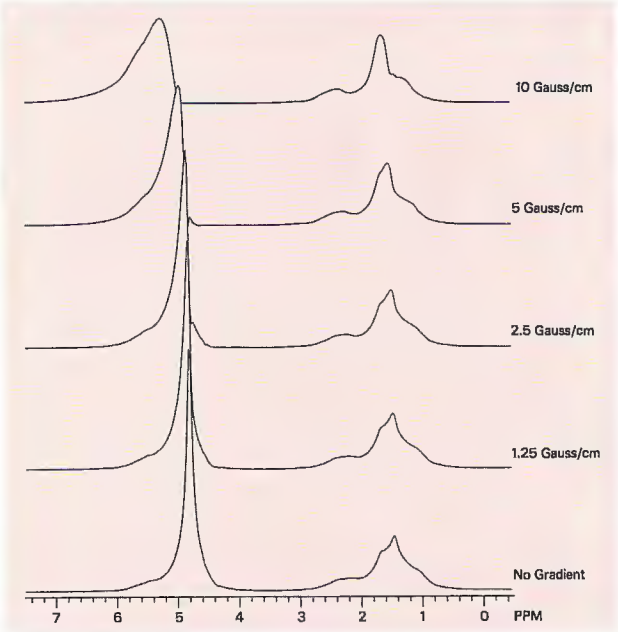


Fig. 1—Using an oil/water phantom, a 10 G/cm gradient will create a water frequency profile extending from 156 KHz to 280 KHz away from normal water resonance. Residual gradient effects of less than 0.01% (50 Hz at 10 G/cm) are observed in a spectrum acquired beginning 1 msec after a 20 msec gradient pulse.

As an example, a 4DFT spectroscopic imaging technique can resolve the four frequency domains that are associated with an NMR signal from an object: x-, y-, z-spatial coordinates and chemical shift δ. The above technique can be a practical alternative to single volume localized spectroscopy. This method allows phosphorous spectra to be obtained from well-defined regions as demonstrated in the following experiment, which was carried out on a GE CSI 2T system using high-strength, shielded gradient coils (Fig. 2). The phase-encode time is kept short (on the order of the dwell-time) to minimize phase-errors in the final spectra, as well as to avoid loss of signal due to T2 decay, which is significantly short in biological phosphates.

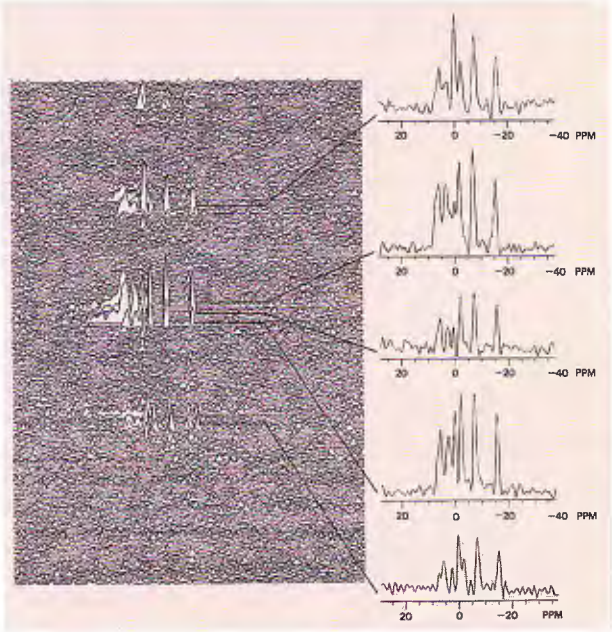


Fig. 2—Stacked plot showing 512 phosphorous spectra from 60 mm cubed region of a live rat. Each trace corresponds to 7.5 mm cubed region (voxel) from within the region of interest. The offset traces clearly show the achievable spectra and spatial resolution of the technique, as well as demonstrating localization of the liver phosphorous metabolites from that of overlying skeletal muscle. Total acquisition and processing time was two hours.

GE NMR Instruments

255 FOURIER AVENUE, FREMONT, CA 94539
(415) 683-4408, FAX (415) 490-6586

PRAUNHEIMER LANDSTRASSE 50, D-6 FRANKFURT 90
WEST GERMANY 4969 760 7431, TELEX 041 2002 GEG

7546 Printed in USA

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

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

National Institutes of Health
Bethesda, Maryland 20892
Building : 10
Room : B1D-123
(301) 496- 8140

In Vivo NMR Research Center
Feb. 6,1989

Dear Dr. Shapiro:

Additional information from In Vivo Localized Proton Spectra using short echo times.

It is well known that In Vivo proton spectroscopy offers advantages with respect to sensitivity and access to different metabolites. This has been demonstrated recently for relatively large volumes in humans (1,2) and for smaller volumes in cats (4) using stimulated echoes. However, in general, only long echo times (TE≥50 ms) have been applied because of the problems of water (and fat) suppression. The long echo time results in a) substantial T_2 losses for some compounds, b) unwanted signal modulation in multiplets due to homonuclear coupling and c) losses due to multiple quantum effects depending on the spin systems.

Now, we are convinced that water suppression is relatively straightforward, even for short echo times, as long as the linewidth of water is determined to a large extent by homogeneous broadening (pure T_2 effects). In vivo, this necessitates a very good localization technique with minimal susceptibility and resonance offset effects. Using the STEAM technique (3) and the shielded gradients (built by Dr. Pete Roemer of GE) on our 4.7T GE CSI instrument, we have shown that this is feasible (4). Of course, residual gradient effects should be really minimal.

With these conditions satisfied we use a combination of CHESS pulses (5) to suppress the water. Even with the inhomogeneous B_1 field of a surface coil it is possible to suppress water in this way by a factor of about 5000 or more as is shown in the spectrum of a nominal volume of $1cm^3$ in the cat brain using a TE of 14 ms (the residual water signal may be used for reference purposes). The full water suppression sequence will be published soon (6).

It is evident from the spectrum that a wealth of new resonances appear with these short echo times (we think that the spectrum contains no artifacts). Interestingly, at least one new peak at low field (about 8 ppm) comes up immediately after death of the cat. We are currently investigating the assignments. We hope that, with these improvements, we are again one step closer towards exploiting the full potential of localized proton spectroscopy.

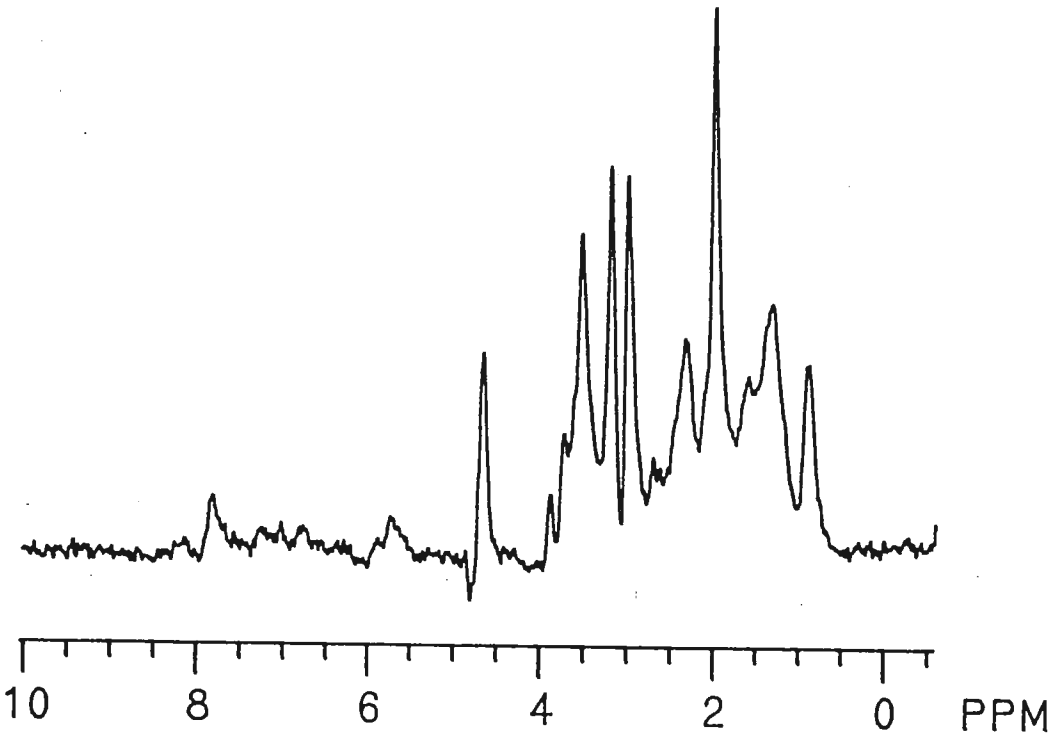
This work was performed in the NIH In Vivo NMR Research Center and will be submitted to Magnetic Resonance in Medicine.

[1] J. Frahm, M.L. Gyngell, H. Bruhn, K.-D Merboldt, W. Hanicke and R. Sauter, SMRM, 7th annual meeting, 1988, p. 613. [2] P.R. Luyten, A.J.H. Marien and J.A. den Hollander, SMRM, 7th annual meeting, 1988, p.327. [3] J. Frahm, K.-D Merboldt, W. Hanicke, J. Magn. Res. 72, 502 (1987). [4] P. van Zijl, C. Moonen, J. Alger, J. Cohen, S. Chesnick, submitted to Magn. Reson. Med.[5] A. Haase, J. Frahm, W. Hanicke and D. Matthaei, Phys. Med. Biol. 30, 341 (1985). [6] C. Moonen and P. Van Zijl, to be published.

Yours Sincerely,

Chrit Moonen, BEIB, DRS

Peter van Zijl, NCI



Proton spectrum of cat brain at 4.7 T. Nominal volume 1 cm^3; TE = 14 ms; TM = 50 ms. Total acquisition time 10 minutes (400 scans). Only exponential broadening and FT was applied for signal processing. The same surface coil (o.d. 6 cm) was used for transmit and receive. No surgery was performed on the cat. Some surface fat may have been detected in this particular example.

POSTDOCTORAL POSITION - PHYSIOLOGICAL NMR

A postdoctoral position is available immediately in the Biomedical NMR Center of the University of Arkansas for Medical Sciences. The ideal candidate should have a Ph.D. in a basic science and previous experience in physiological applications of NMR. The laboratory has a General Electric CSI-4.7T/33cm imaging/spectroscopy system, a GN-300WB high resolution NMR spectrometer and liberal access to a GE Signa clinical MRI system with spectroscopy. The laboratory has basic and clinical research projects in MR spectroscopy and imaging of ^{31}P, ^{1}H, ^{19}F, and ^{7}Li both in vivo and in vitro. Period is one year, renewable by mutual agreement. Salary is 23,000/yr. Interested individuals should send a resume and names of three references to:

Richard A. Komoroski Ph.D.
Director, NMR Laboratory
Slot # 582
University of Arkansas for Medical Sciences
4301 West Markham
Little Rock, AR 72205

UNIVERSITÉ DE LAUSANNE
INSTITUT DE CHIMIE ORGANIQUE
Rue de la Barre 2 — CH-1005 Lausanne
Téléphone (021) 44 42 50
Téléfax (021) 44 32 48

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

February 2nd 1989
(received 2/13/89)

GRAPH THEORY IN AUTOMATED ANALYSIS OF *Z*-COSY SPECTRA

Dear Dr Shapiro,

Over the last few years, we have attempted to improve algorithms for automated analysis of 2D spectra. We noticed some time ago that it might be useful to borrow some concepts from graph theory, but we hesitated to do so for fear of appearing too intellectual. Actually, we don't really feel a need for using any of the stunning theorems of graph theory (which saves us the trouble of trying to understand the proofs), but we like the semantics. Expressions such as 'nodes' and 'edges' sound so much better than the old-fashioned terminology of 'nuclei' and 'couplings'! Groups of magnetically equivalent nuclei are now called 'supernodes'. Who can resist the temptation of such a charming language? Actually, graphs provide an elegant description of the relationships between molecular structure and 2D NMR spectra.

Chemists routinely represent the structure of molecules by graphs (see Fig.1a). It is like Monsieur Jourdain who discovered to his amazement that he could speak in prose. To discuss ^{1}H NMR spectra, it turns out to be more suitable to use another type of graph that we call *global coupling network* (see Fig.1b). The nodes represent the chemical shifts of the protons, the edges stand for resolved scalar couplings between spins. Groups of magnetically equivalent spins, such as the three methyl protons symbolically represented by [3], can be fused into a supernode.

A global coupling network $G(\Omega, J)$ of nodes Ω and edges J can be subdivided into subgraphs $F^{kl}(\Omega', J')$ called *fragments* (Fig. 1c). A fragment consists of two active nodes Ω_k and Ω_l (chemical shifts of the active spins), their mutual active coupling J_{kl} and all passive nodes Ω_i that are connected to one or to both of the active nodes. A global coupling network can be subdivided into as many fragments as there are edges in the network. Graphically a fragment is drawn as in Fig.1c with the following conventions: active nodes are represented by ellipses, passive nodes by circles or by squares, depending on whether they are connected to only one or to both active nodes. Degenerate couplings are labelled with double cross-bars.

The cross-peaks in Fig.1d, extracted from an experimental *z*-filtered COSY spectrum, may be regarded as a 'mapping' of the fragments in Fig.1c onto the spectral domain. In fact, it is always possible to deduce the structure of a 2D spectrum from the molecular structure by following the path (a) → (b) → (c) → (d) in Fig.1. The aim of automated analysis is obviously to derive the molecular structure by following the inverse path (d) → (c) → (b) → (a). The analysis of individual cross-peaks is now sufficiently advanced so that one can get reasonable proposals for fragments. Assembling these into a global network turns out to be a more demanding task, bearing in mind that many of the fragments that are found may actually be misleading. In the language of graph theory, the problem amounts to deriving the supergraph G from the knowledge of the subgraphs F^{kl}. Although we have not found a completely fool-proof solution yet, we derive great pleasure from the fact that our difficulties can be described in noble terms.

Yours sincerely,

Peter Pfändler

Geoffrey Bodenhausen

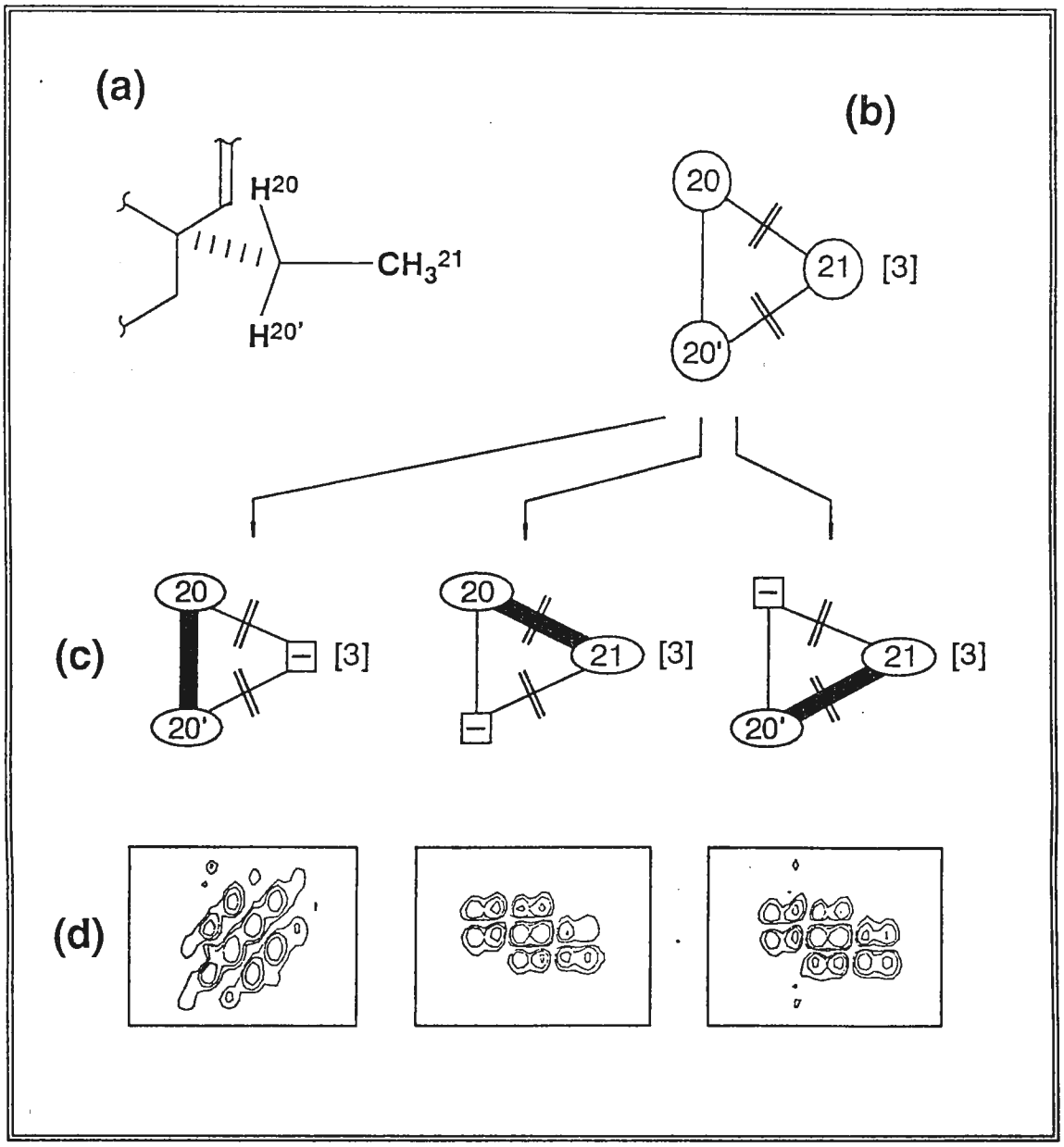


Fig.1 : (a) Graph describing part of the molecular structure of vinblastine. (b) Global coupling network of the protons H^{20}, $H^{20'}$ and the three H^{21} protons of the methyl group. (c) Fragments that can be extracted from the network. (d) Experimental cross-peak multiplets from a z-COSY spectrum of vinblastine.

Postdoctoral Position Available in Department of Chemistry & Biochemistry, University of Colorado, Boulder

A postdoctoral position is available immediately in my laboratory for studying structures of biologically active peptides or catalytic RNAs in solution by NMR spectroscopy. The laboratory is also involved in developing computational methods for generating solution structures of biopolymers from NMR data. Experience in 2D NMR of biopolymers, chemical or enzymatic synthesis of RNA or DNA oligonucleotides and/or computational chemistry desired. The appointment is for one year and is renewable upon mutual agreement.

The resources available include Varian VXR-500S and Varian VXR-300S spectrometers equipped with Sun computers as well as a Sun 4/260 computer for data processing and structure generation. Other resources include a PS 330 graphics system, use of an Alliant FX-8 minisupercomputer and access to supercomputer centers.

Interested applicants should send a resume and arrange to have two letters of recommendation sent to: Professor Arthur Pardi, Department of Chemistry & Biochemistry, University of Colorado, Boulder, Campus Box 215, Boulder, CO 80309. The University of Colorado has a strong institutional commitment to the principle of diversity. In that spirit, we are particularly interested in receiving applications from a broad spectrum of people, including women, members of ethnic minorities and disabled individuals.

SpecStation™ NETWORKING FROM NMRi

THE COMPUTER NETWORK THAT WILL GROW WITH YOU

DESIGN

- Turnkey networks designed utilizing your existing computers and spectrometers or adding new systems
- Currently running on Sun–3's and–4's, and VAXstation™ systems
- Currently porting software to Apollo, Ardent, Silicon Graphics, Stellar and Sun 386i
- SpecStation terminals starting at under $2,500 (high–speed Ethernet multi–window SpecStation graphics)
- PC SpecStation network graphics servers will be supported later in 1989.
- Supports serial graphics terminals for other users on network
- Share access to common devices including PostScript™ and color printer/plotters
- Transfer data from *all* modern NMR and many FT–IR instruments

SOFTWARE

- 1D and 2D NMR, FT–IR, IMAGE and statistical analysis software available
- Process and analyze data at any SpecStation

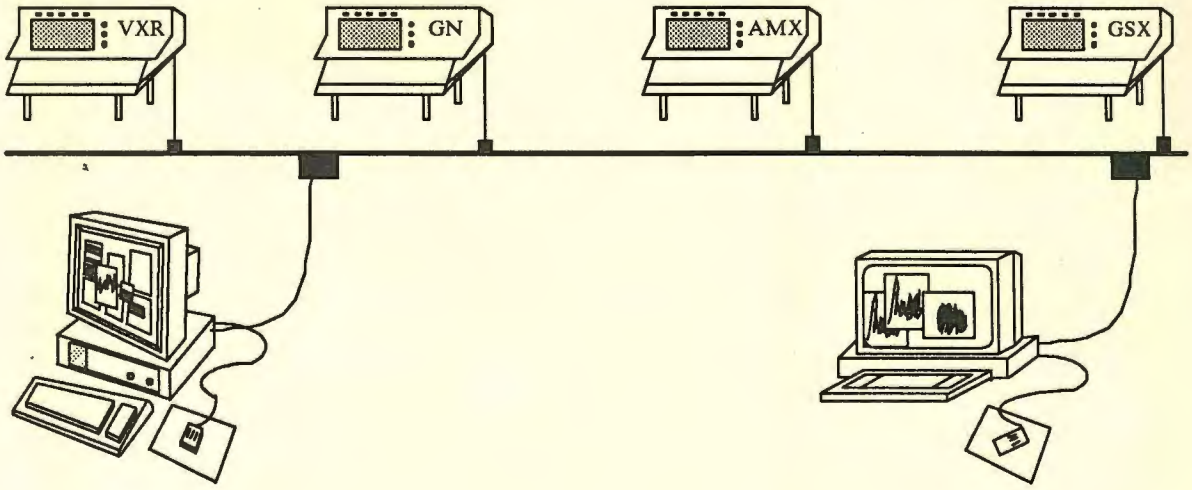
CUSTOMER SUPPORT

- On–site installation and training available
- Continuing technical support and software upgrade service available
- 800 phone and modem customer support
- Chemistry Ph.D. Customer Support team committed to solving your problems
- 48 SpecStations installed in 1988

New Methods Research, Inc., 719 East Genesee Street, Syracuse, New York 13210

THE NMR1/NMR2 SYSTEM INCLUDING NOW: NMR/MED™, NMR2/MODEL™ AND NMR2/IMAGE™

THE 100th SpecStation™ SYSTEM FROM NEW METHODS RESEARCH, INC. WILL BE DELIVERED THIS SPRING!



SpecStation™ COMPUTERS AND NETWORKS.

TURNKEY LABORATORY COMPUTER NETWORKING AND SOFTWARE FROM NMRi; OPTIMIZED FOR TODAY'S (*AND TOMORROW'S*) BEST WORKSTATION COMPUTERS.

FOR BRUKER•CHEMAGNETICS•GE NMR•JEOL•VARIAN INSTRUMENTS

New Methods Research, Inc., 719 E. Genesee Street, Syracuse, New York 13210
(315) 424-0329 FAX (315) 424-0356

THE NMR1/NMR2 SYSTEM INCLUDING NOW: NMR/MED™, NMR2/MODEL™ AND NMR2/IMAGE™

THE UNIVERSITY OF SYDNEY

DEPARTMENT OF BIOCHEMISTRY

SYDNEY N.S.W. 2006
AUSTRALIA

TELEPHONE: (02) 692-2222
TELEX: UNISYD 26169

23 January 1989
(received 1/30/89)

Prof. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
PALO ALTO CALIFORNIA 94303
USA

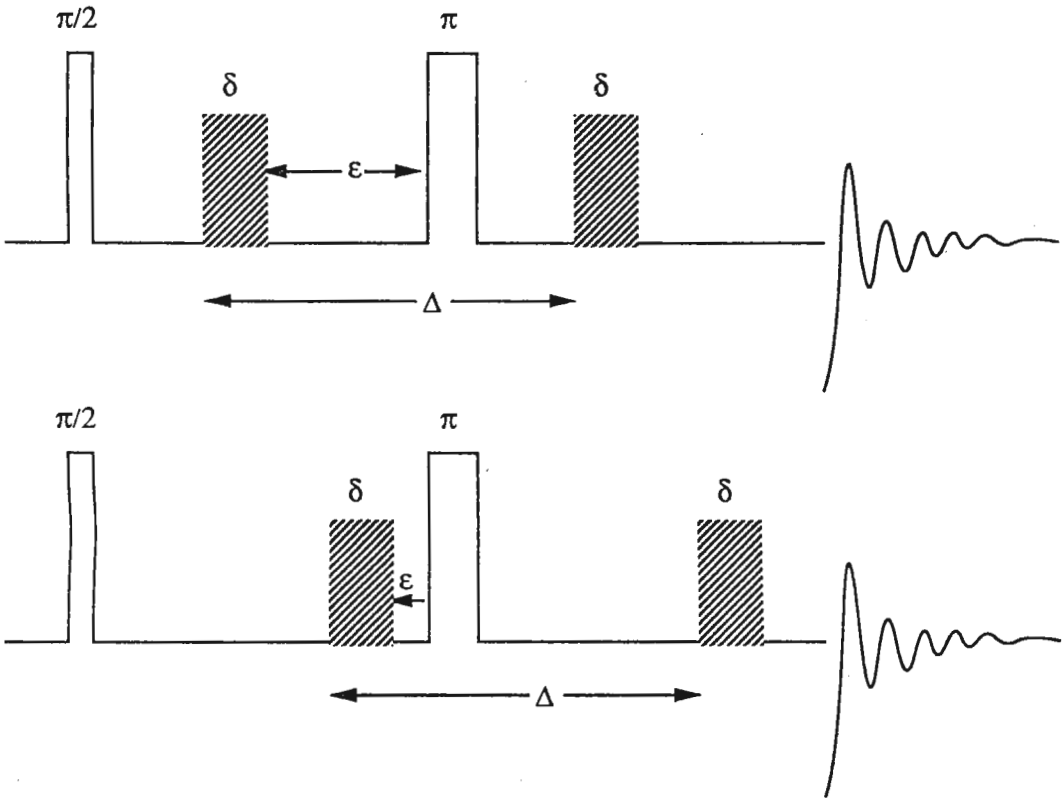
TITLE: High Resolution PFG Diffusion Measurements in Cells

Dear Barry,

We have previously measured the relaxation rate of [2,^{13}C]glycine to estimate the intracellular viscosity of the cytoplasm of human red cells [1,2]. A more direct approach to the problem is to use the pulsed field gradient (PFG) NMR experiment. We have recently met with success with this experiment after about 3 years of developing a system for our Varian XL/VXR 400 wide-bore (89 mm) spectrometer. The main developments which have been made are: (1) the anodized aluminium housing of the 30-105 MHz broad-band probe has been replaced by aluminium tube that extends only to the partition below the coil region; (2) we use a Maxwell pair (a pair of oppositely wound) solenoidal coils on a Perspex former which is attached to the outside of the glass-Dewar that supports the r.f. coils ; (3) the probe housing around the coils, and which supports the lower end of the spinner assembly, is made of Perspex; (4) an earth wire extends from the aluminium housing to a ring of aluminium which makes contact with the upper spinner tube; (5) the cables outside the magnet carrying the field gradient currents are all r.f.-shielded with copper braid which is earthed to the magnet; (6) the leads to the gradient coils in the probe are shielded by being in 4 mm Pyrotenax (copper coated 2-wire cable); (7) the fast-switching linear amplifier is home built and receives its logic pulses from the spectrometer via an optic coupler. Some of our first results obtained with the apparatus are about to appear in the refereed literature.

We have recently had cause to evaluate PFG diffusion apparatus from two leading manufacturers and in the course of those discussions it was necessary for us to define a "key" experiment which could be used to assess the quality of the apparatus. The experiment is the classical Stejskal and Tanner [3] one in which a linear field gradient pulse is applied in each of the two time intervals of a spin-echo r.f.-pulse sequence. For our test we suggest, for the common nuclei (^{1}H, ^{13}C, ^{31}P), a total echo time of 200 ms, a field gradient pulse of around 20 Gcm^{-1} of duration (δ) 5 ms and a time interval between the leading edges of the gradient pulses (Δ) of 100 ms. The time interval between the trailing side of the first gradient pulse and the start of the π r.f.-pulse is denoted ε in the figure below. In evaluating the PFG equipment we recommend holding δ and Δ constant and diminishing ε from ~50 ms to as small a value as possible. For a properly functioning apparatus the Fourier transform of the spin-echo FID is a spectrum whose phase parameters and amplitude are independent of ε. For our home-built apparatus this holds true down to ε values of 7 ms.

.../2



It is worth noting that our apparatus does not involve the use of pre-emphasis on the ~20Gcm^{-1} gradient pulses; this is probably the result of having a wide-bore magnet. We would like to hear of the experiences of any other research group with obtaining high resolution PFG spectra using probe-solute concentrations in the mM range.

Yours sincerely,

Philip W. Kuchel W.S. Price B.E. Chapman

PHILIP W. KUCHEL W.S. PRICE B.E. CHAPMAN

1. Z.H. Endre, E.B. Chapman and P.W. Kuchel, Biochem. J. 216, 655, 1983
2. Z.H. Endre and P.W. Kuchel, Biophys. Chem. 24, 337, 1986
3. E.O. Stejskal and J.E. Tanner, J. Chem. Phys. 42, 288, 1965

TEL AVIV UNIVERSITY
RAYMOND AND BEVERLY SACKLER
FACULTY OF EXACT SCIENCES
SCHOOL OF CHEMISTRY

אוניברסיטת תל-אביב
הפקולטה למדעים מדוייקים
ע"ש ריימונד ובברלי סאקלר
בית הספר לכימיה

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

Dear Dr. Shapiro:

January 18, 1989.
(received 1/26/89)

^{1}H - ^{31}P CORRELATED 2D NMR SPECTROSCOPY OF INTACT ORGANS.

The assignment of ^{31}P NMR signals in intact organs has been traditionally performed by the analysis of the spectra of tissues extracts. There are however cases where this approach does not lead to definite identification. In our previous ^{31}P NMR studies of taurocholate induced experimental pancreatitis a new transient signal appeared at -0.18 ppm (see Fig. 1). It was suggested that this compound can be a useful diagnostic and prognostic marker, and would aid in studying the basic pathophysiologic processes of the disease. Attempts for assignment of that peak by comparing the chemical shift to genuine compounds, and by using TLC and columns chromatographic methods of PCA extracts were unsuccessful. A ^{1}H-^{31}P correlated 2D NMR spectroscopy of the intact pancreas (Fig. 2a) gave the unequivocal assignment of the signal. In this technique, only those protons that are spin-spin coupled to ^{31}P nuclei are obtained. Thus the proton spectra are greatly simplified, the strong interfering signal of the water is eliminated, and it enables precise assignment of the phosphorous signals by correlating them to the proton chemical shifts of the groups to which they are coupled. The new phosphorous peak in the diseased pancreas correlated with two proton signals with chemical shifts of the choline αCH_2 and glycero CH_2 of lecithin. The compound was identified as taurocholate-lecithin complex. A 2D map of prepared taurocholate-lecithin solution (Fig. 2b) was identical. A signal with similar chemical shift was found in the PCA extracts of diseased pancreases. However, the 2D map of the extracts was completely different, and the signal must originated from a different chemical compound.

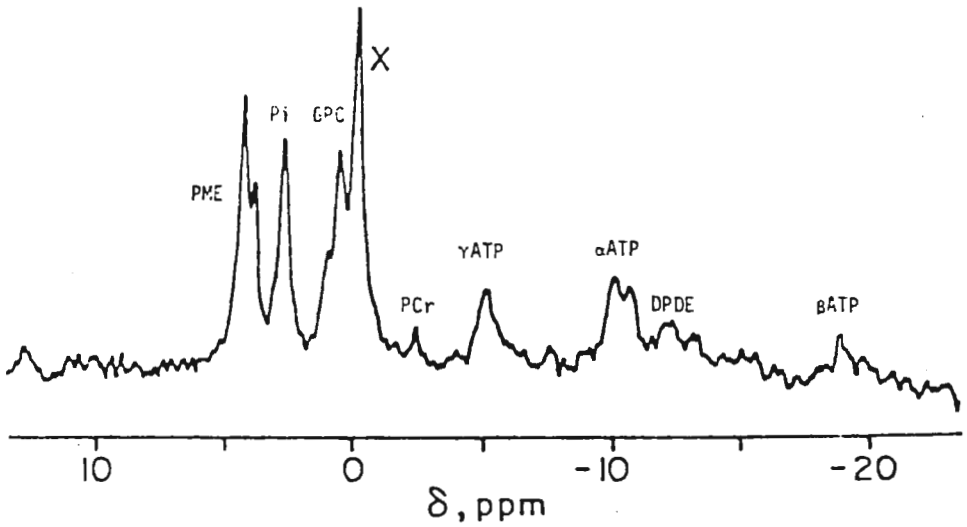
We recently performed ^{1}H-^{31}P correlated 2D NMR studies of experimental pancreatic carcinoma. The ^{31}P spectrum is dominated by the phosphomonoester peaks. 2D measurments demonstrated that this region is governed by phosphocholine (PC) and phosphoethanolamine (PEA), as well as other phosphorous compounds that overlapped PC and PEA in the ^{31}P spectrum but not in the ^{1}H dimension. Thus, this technique can provide a powerful tool for studying biochemical and metabolic processes in intact organs.

Sincerely yours,

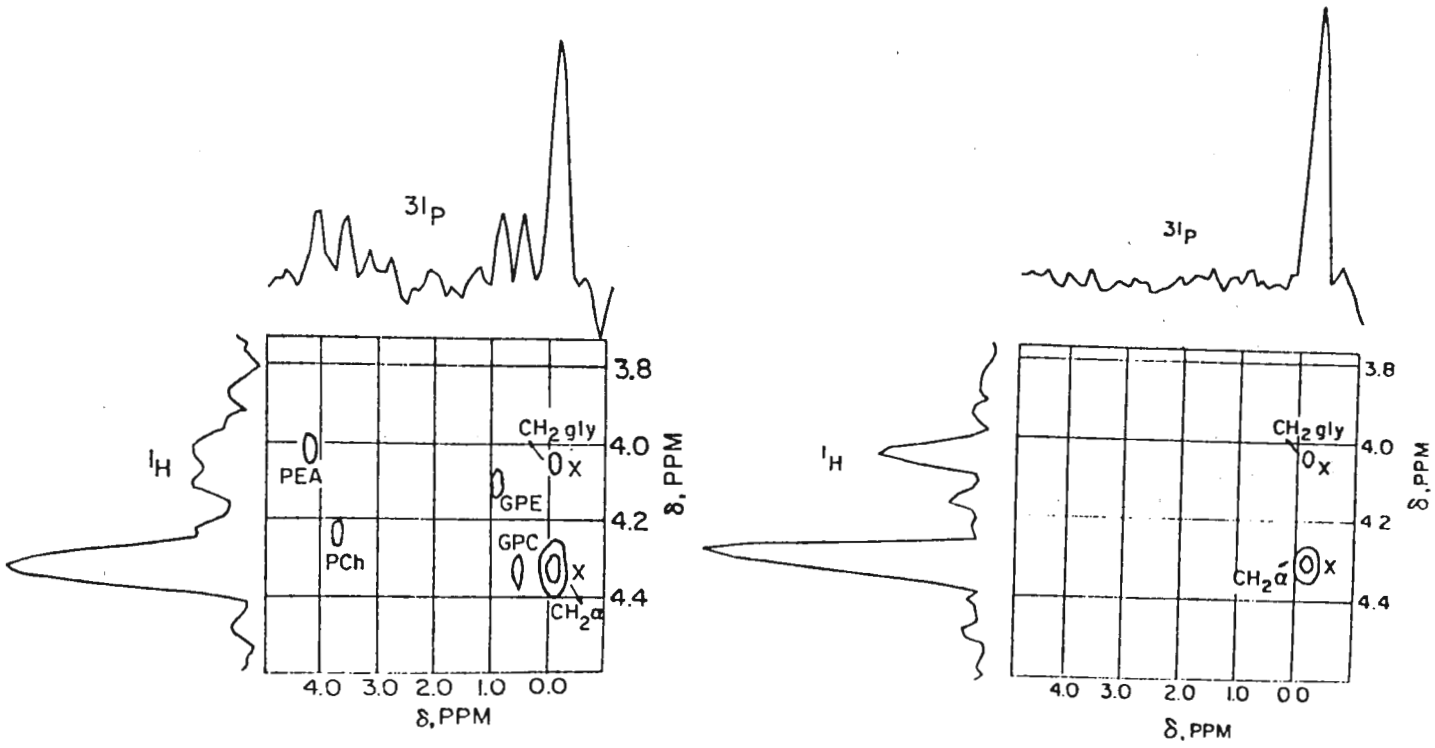
Tammar Kushnir Ofer Kaplan Nadir Askenasy Gil Navon

T. Kushnir Okaplan Ask Gil Navon

קרית האוניברסיטה, רמת אביב, תל אביב 69978. טל. 03-5450111. פקסימיליה 972-3-419513
RAMAT AVIV, 69978 TEL AVIV, ISRAEL, TELEX 342171 VERSY IL. CABLE VERSITY T. A. 5450111, FACSIMILE 972-3-419513



^{31}P NMR of a Pancreas from Rat
with Experimental Pancreatitis.



2D ^{1}H - ^{31}P NMR Correlation Spectra of:
a. Intact Pancreas from a Diseased Rat. b. Lecithin-Taurochlate Complex.

DOTY SCIENTIFIC:

EXPANDING THE BOUNDARIES OF NMR PROBE TECHNOLOGY

HIGH SPEED MAGIC ANGLE SPINNING

- 14kHz specified spinning (air drive) in high-performance, multinuclear, 5mm VT MAS probes.
- Speeds over 17kHz (air drive) under optimum conditions.

LOW TEMPERATURE

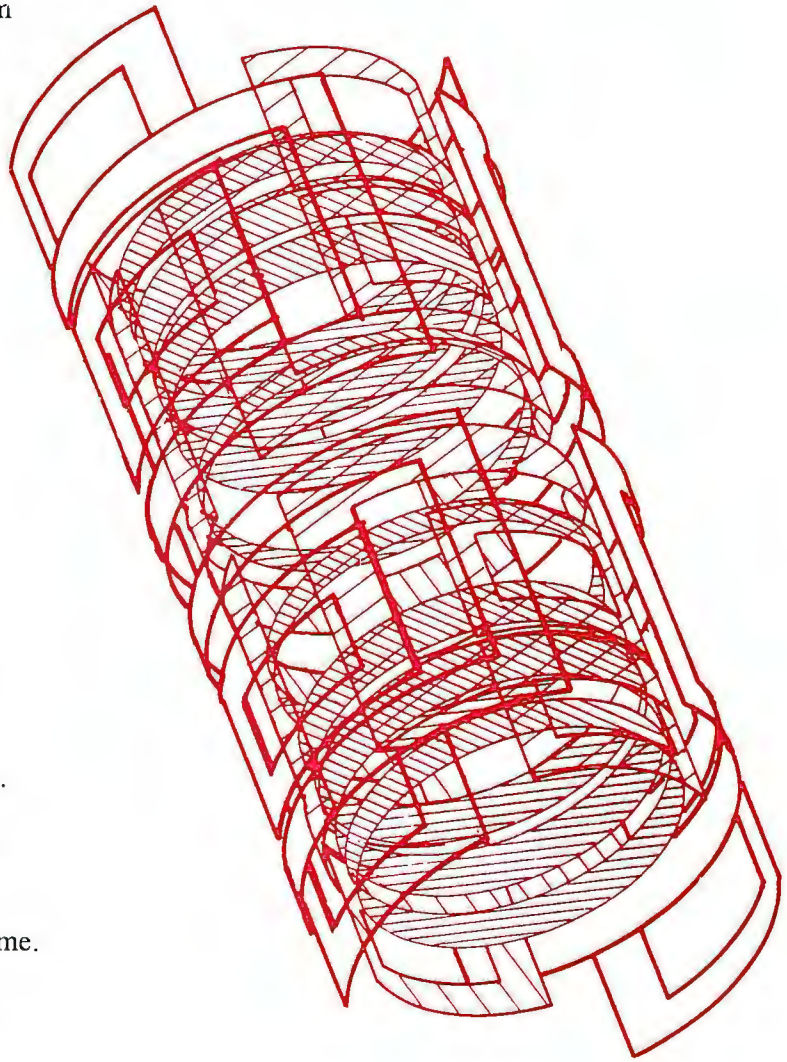
- Static, double-resonance below 3 K.
- MAS at 1kHz below 8 K.
- High resolution below 90 K.

HIGH TEMPERATURE

- Static, double-resonance up to 1200° C.
- 7mm, 4kHz MAS above 700° C.
- High resolution to 380° C.

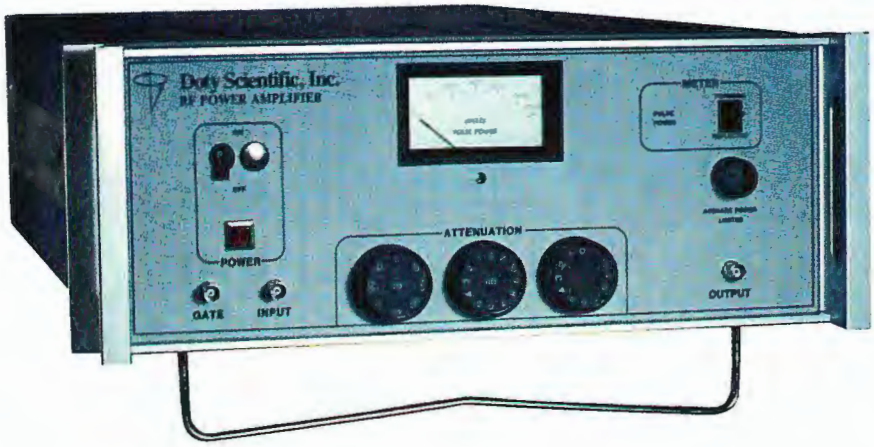
NMR MICROSCOPY

- X-Y-Z gradient coil systems for all supercon magnets.
- Pulse z-gradient fields up to 1000 G/cm (4mm).
- Fully shielded design minimizes eddy current and pre-emphasis problems.
- Gradient linearity ± 2% over full sample volume.
- High sensitivity double-resonance rf systems.



Doty Scientific, Inc.
600 Clemson Road
Columbia, S.C. 29223
(803) 788-6497

THE NMR AMPLIFIER



Amplifiers from Doty Scientific? Don't you make NMR probes?
Yes, we build NMR probes, all kinds of them. We are on the leading edge of NMR probe technology. Several years ago, however, we realized there were no amplifiers designed from the ground up for NMR. Everything was "adapted".
We knew how NMR amps should perform: Clean, precise pulse shapes. Pure power. Reliable and rugged. All solid state. Easy to use, no tuning. Just as importantly, we knew how to make them. So we did.
But, not overnight. Over years.
You'll like the results. We build the NMR amplifiers. The ones with the two year unconditional warranty.
Amplifiers from Doty Scientific? Yes.

SPECIFICATIONS

MODEL	POWER BANDPASS (MHz)	GAIN (dB)	PULSE POWER (W)	WEIGHT (lb.)	DOMESTIC PRICE
250A	7-140	53	250	52	$8,000
500A	8-130	56	500	57	9.800
1000A	8-125	58	1000	62	14,400

Rise Time: less than 50ns to 90%.
Fall Time: less than 50ns to -70dB.
80dB minimum signal blanking.

Total output noise:
Unblanked: 45dB above thermal noise.
Blanked: 25dB above thermal noise.

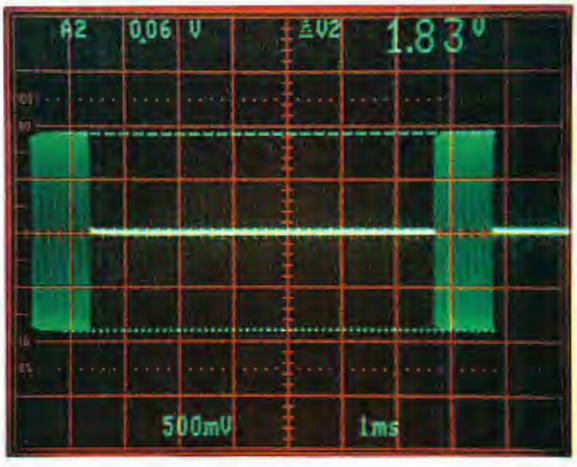
Flatness of response throughout power bandwidth:
± 2dB at half rated pulse power.
± 3dB at any power level.

THD is below 6% at any power level, any frequency.
Typical third order intercept is 60dBm.
Phase shift change (10% to 80% of rated power): 8° max.

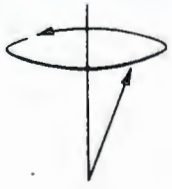
Half of rated power available to 170MHz. CW power is 20% of full pulse power.

Control panel includes three rotory decade attenuators, directional pulse power meter with 300ns response time, and adjustable average power limiter.

Model 1000A operates off of 220 VAC, 50 or 60 Hz.
Model 250A and 500A available in 120 VAC and 220 VAC, 50 or 60 Hz. Quiescent AC power demand is 8% of rated rf pulse power. Total efficiency at full power is 30%.



Typical 500 Watt pulse envelope.



Doty Scientific, Inc.
600 Clemson Road
Columbia, S.C. 29223
USA
(803) 788-6497
FAX: 803-736-5495

CENTRE FOR NUCLEAR MAGNETIC RESONANCE

Department of Chemistry
Tel: 523187
DR. OLIVER W. HOWARTH

University of Warwick
Coventry CV4 7AL

Department of Physics
Tel: 523523 Ext. 2403
DR. RAY DUPREE

Dr B L Shapiro
966 Elsinore Court
Palo Alto
California 94303
USA

18 January 1989
(received 2/1/89)

Dear Barry

OH GROUPS IN COAL TARS

One challenge we have recently faced is that of identifying and quantifying -OH groups (phenol, alcohols, carboxylic acids) in coal tars. These tars are, of course, very complex mixtures. Following Dereppe and Parboo[1], we derivatise the -OH groups, fairly quantitatively, using hexamethyldisilazane, and then take the ^{29}Si DEPT or INEPT spectrum of the O-$SiMe_3$ groups. The figure shows one example, from a high-rank bituminous coal.

Obviously we started by recording the shifts for the derivatives of single known compounds. These fall rather nicely into separated groups. Also, within a group, they correlate fairly linearly with the pK_a of the -OH group, unless there is an obvious ortho-interference. We are thus able to assign the resonances of unknown materials with some confidence. We have also shown that the peak areas correspond with known composition ratios in test samples.

The figure shows that many individual resonances can be resolved, even in the polyaromatic phenol region around 22 p.p.m.. The same remains largely true for source-fuels ranging from wood through to anthracite. We can thus trace the fate of individual compounds as coalification proceeds. Many of the changes are as expected. Wood tar is rich in aliphatic alcohols, but these have almost gone in lignites. Decarboxylation is complete by the sub-bituminous stage, and then the phenol balance swings somewhat towards polyaromatic phenols.

However, we have one unexpected general observation. In a few cases, such as with α- and β-naphthol, we can see a clear trend towards the thermodynamically favoured isomer as coalification proceeds. Another example is a trend from Ar(alkyl) to Ar(polymethyl). We are now assembling further evidence to support this detailed view of coalification as a multi-million year move towards thermodynamic equilibrium.

Yours sincerely

Oliver Howarth

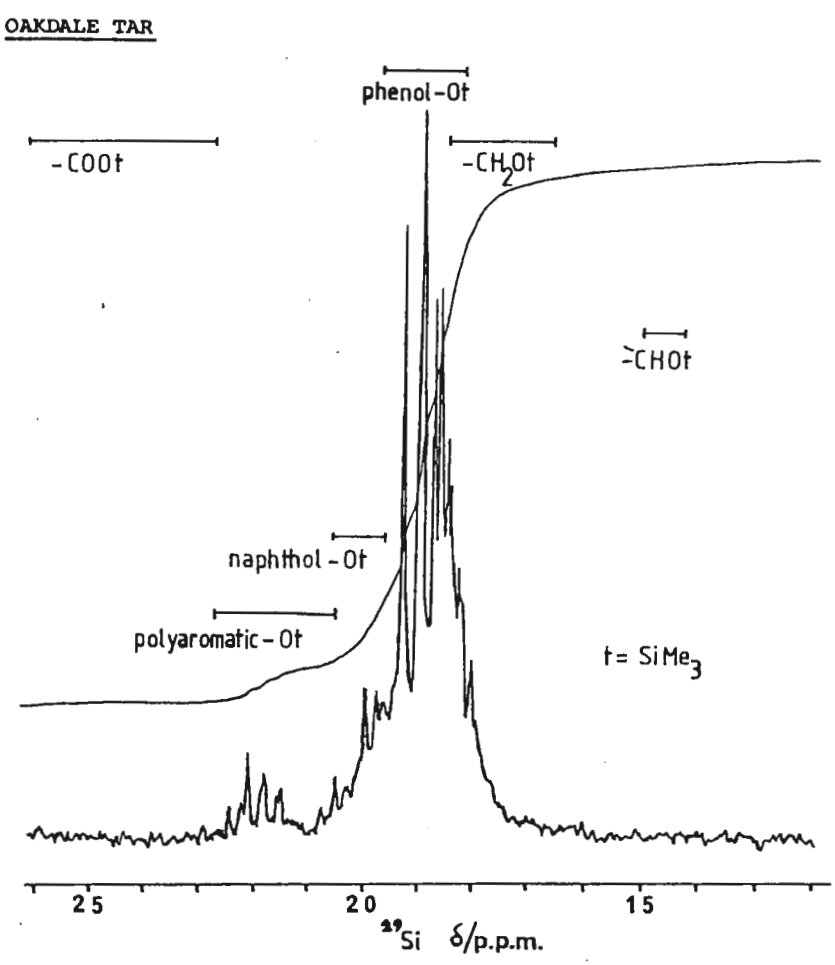
Giles Ratcliffe

Oliver Howarth

Giles Ratcliffe

---

[1] Dereppe, J.M. and Parboo, B. Anal. Chem. 58, 2641 (1986)



TECHNICAL SALES

Isotec Inc., a division of Matheson Gas Products, has an opening in its Labelled Compounds Group for a Technical Sales Representative.

This job provides an excellent opportunity for a bright, motivated person who is interested in promoting the advantages and uses of stable isotope labelled compounds to the research (chemical, physical, environmental, pharmaceutical, biomedical, etc.) community as well as interacting with the purchasing departments of the various institutions.

Qualifications:

- M.S. or Ph.D. in chemistry, preferably organic, with a background in the applications of NMR and mass spectrometry (Laboratory experience using stable isotopes will be a significant asset.)
- Ability to maintain current awareness of the various applications, present and future, of stable isotope labelled compounds
- Good command of both written and spoken English
- Willingness to spend considerable (>50%) time travelling

Please send your resume to Myra Gordon, ***Isotec Inc.*, 3858 Benner Road,**
Miamisburg, Ohio 45342.

UNIVERSITY OF LEICESTER
BIOLOGICAL NMR CENTRE

P.O. Box 138
Medical Sciences Building
University Road
LEICESTER LE1 9HN
Telephone:
Direct Line: (0533) 523054
Switchboard: (0533) 522522

Prof. G. C. K. Roberts
Dr. L. Y. Lian

(received 2/1/89)

24 January 1989

Dear Prof. Shapiro

Homonuclear Hartman-Hahn (HOHAHA or TOCSY) spectra have proved to be of considerable value in identifying spin systems in proton spectra of proteins. Recently Feng & Roder [1] reported that the HOHAHA spectrum of an exchanging system (a mixture of reduced and oxidised cytochromes) contains cross-peaks arising from the exchange process. This arises because both coherent and incoherent transfer of magnetization occur during the spin-lock period

We have observed the same effect in the commonly-studied situation of a ligand binding to a protein, in this case the inhibitor pyrimethamine (in slow exchange) to dihydrofolate reductase. As would be expected, rotation about the bond linking the phenyl and pyrimidine rings is highly restricted, and separate signals are observed for all four protons of the chlorophenyl ring of the bound inhibitor (2' 5.87; 3' 7.07; 5' 7.93; 6' 7.65 ppm, as determined by 2D exchange spectra). The cross-peaks between the two pairs of protons on either side of the ring are shown in the COSY spectrum (fig.2). In the HOHAHA spectrum (fig.1) these scalar coupled correlations are seen again (labelled J). In addition, however, cross-peaks are seen for both direct exchange (eg 2'bound ↔ 2'free; labelled D) and relayed exchange (eg 2'bound ↔ 2'free ↔ 3'free; labelled R). The relative intensities of these cross-peaks are a function of the spin-lock time, here 50 msec (this is too short to observe effects due to long range coupling across the ring).

An important feature of this experiment is that the concentration of the free inhibitor in the sample was very low - only about 1/50th that of the enzyme-inhibitor complex. This small amount of free ligand is nonetheless enough to have a considerable effect on both HOHAHA and NOESY spectra. in this case, where all the protons of the bound ligand had been assigned, interpretation was not too difficult, but we may not always be so lucky.

Please credit this to the account of G.C.K.Roberts.

Gordon C.K. Roberts

John R.P. Arnold.

Ref. [1] Y.Feng & H.Roder, J.Magn.Reson., 1988, 78, 597.

PYRIMETHAMINE:

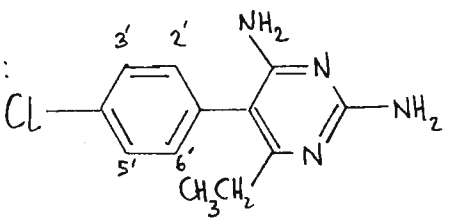


FIGURE 1: HOHAHA

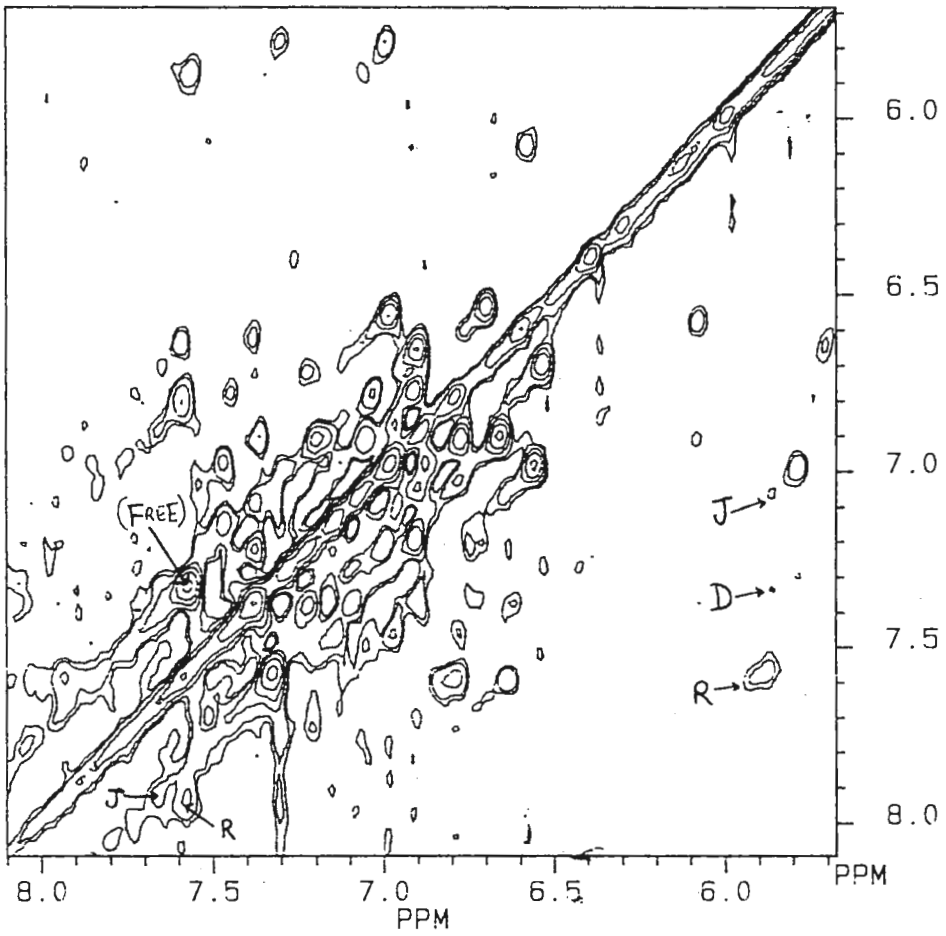
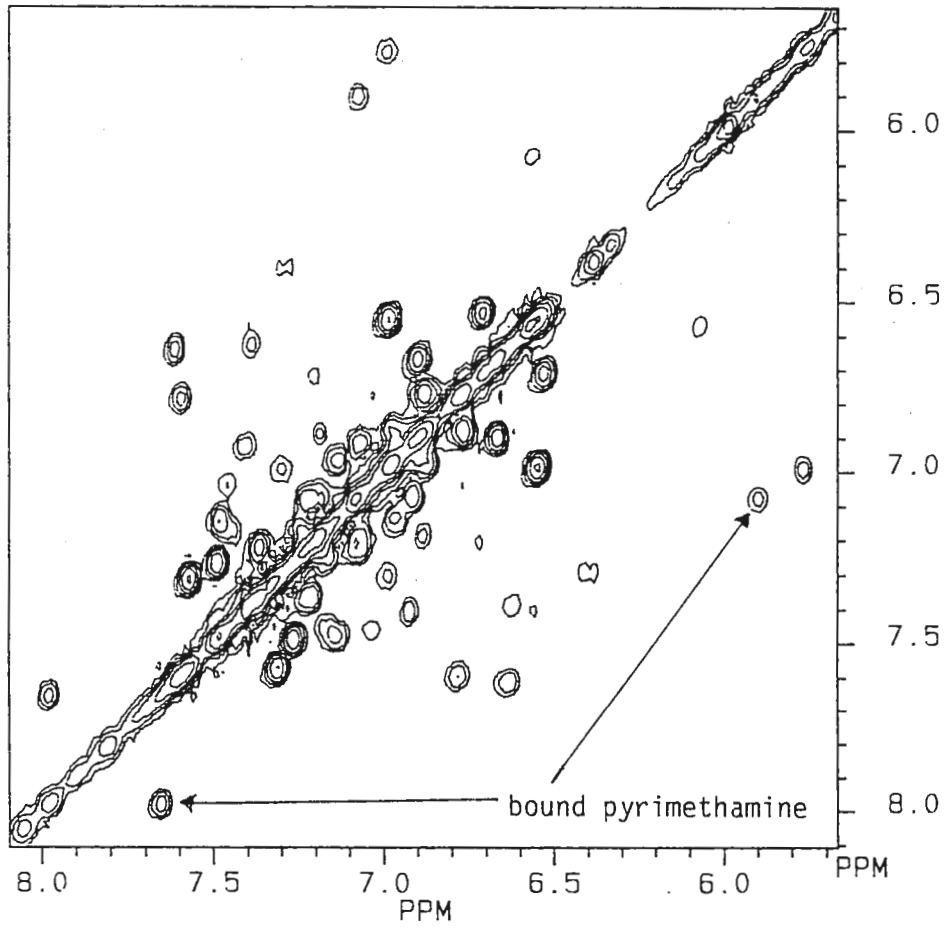


FIGURE 2: COSY



Z•Spec Probes

Offer

A complete line of high efficiency probes for all your NMR/MRI needs.

* *Surface coil*
* *High Resolution*
* *Imaging/Micro Imaging*
* *Solids*

Stop by our hospitality suite and talk to us about your probe needs during the 30th ENC at Asilomar April 2-6, 1989

For more information about Z•Spec Probe products contact Toby Zens, manager, Special Products Group.

NCC

NALORAC CRYOGENICS CORPORATION
837 Arnold Drive Suite 600, Martinez, CA 94553
Tel: (415) 229-3501 FAX (415) 229-1651

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service
National Institutes of Health

National Institute of Diabetes and
Digestive and Kidney Diseases
Bethesda, Maryland 20892

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

Predicting missing or erroneous data points by linear prediction or ... the revisited story of *CASSANDRA*

(title too long; shorter one provided for table of contents, by BLS)

(received 2/15/89)

Dear Barry,

In spite of its rather primitive nature, Fourier transformation is still widely used in NMR for obtaining frequency-domain spectra. However, there is some magic involved in this simple and fast algorithm, so that everybody just feign ignorance on its pitfalls or limitations... until inconvenient artefacts show up.

A naive spectroscopist may believe that the Cooley-Tukey algorithm (1) has been correctly implemented by the spectrometer manufacturer; but as any belief, this should be re-examined at least once. For example, are the first and last data points suitably scaled before FT (2), does the FT introduce a bias in the phase of the signal (a cosine-modulated signal should lead to a purely absorptive lineshape), does an inverse FT restore the time-domain signal at its original scaling?

As a result of the sampling theorem (3), the FT also plays strange tricks at the Nyquist frequency, especially if one is not aware of the type of FT (either *real* (4) or *complex*) employed. The striking observation, that NMR signals usually fold differently on the two sides of the Atlantic, is in no way related to the continental drift and remains beyond comprehension... until a correlation is made with the leading manufacturer on each side. Nobody runs single channel detection spectra anymore, and thus the term *quadrature detection* is generally useless if the sampling mode (simultaneous or sequential) is not specified. In fact, the folding pattern at the Nyquist frequency is only related to the sampling mode, which then implies the use of a given type of FT.

Baseline distortion is another devil of the FT pantheon. Without much regard to its physical origin, spectroscopists have performed cosmetic baseline corrections of one-dimensional spectra essentially in the frequency-domain. Techniques ranging from tedious adjustement on a flickering screen to highly sophisticated optimization routines have been used in the past for 1D NMR spectra; however, none of these are very suitable for 2D NMR. Optimization routines, whose efficiency is known to depend upon the starting point, often fail for a number of slices in a crowded 2D correlation map and nobody in our group is willing to spend weekends in a dark sub-basement correcting manually slices of high-resolution 2D spectra. Furthermore, mathematical models used for frequency-domain correction were obviously chosen for programming convenience (polynomial or spline functions) rather than based on the physical origin of the distortion.

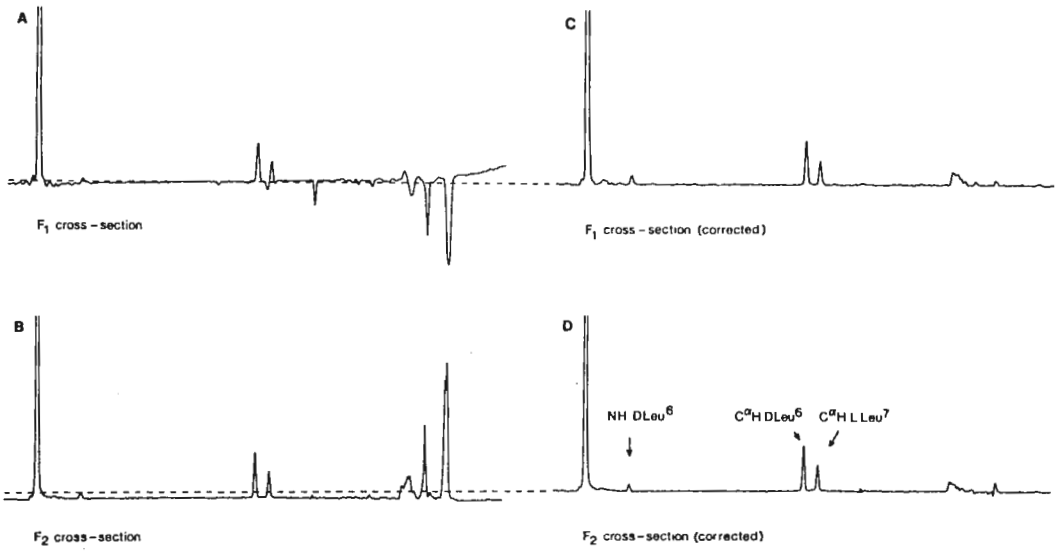
Two major causes of baseline distortion are the following; the sampling is not initiated at time t=0 because of the duration of the pulses (leading to a sizeable frequency-dependent phase correction) and a couple of points at the beginning of the FID are sampled incorrectly (due to the time response of the analog filter (5)). These distortions, encountered in the F_1 and F_2 dimensions of 2D spectra, can be eliminated *in the time-domain*, if the missing or distorted early points can be restored from the later "error-free" points.

In view of the etymological origin of its name, *linear prediction* is the suitable technique for extrapolating the future of a time series from its past and conversely. Using the fast algorithm developed by Burg, we were able to reconstruct the early data points of the FID and obtain almost perfect spectra using conventional FT. As compared to the regular use of LP for spectral estimation, our method (LP extrapolation + FT) is less demanding in computer time and is thus applicable for routine processing of 2D NMR spectra. Fig. 1 compares F_1 and F_2 slices for untreated and LP-corrected 2D data sets showing a dramatic improvement upon use of LP. By using the LP algorithm in conjunction with backward and forward FT, the calculation time needed for processing a typical 2D NOESY set is only about 15 min on a SUN-4. More details will be presented elsewhere (6).

Fig. 1 demonstrates that extrapolation using linear prediction is able to predict missing or corrupted data points in a reliable manner, leading to almost unbelievable spectra. Thus a suitable name for this method is *CASSANDRA* (7), a name that should be thought of as a mythological reference rather than as one more tortuous and mysterious acronym.

Dominique MARION

Ad BAX



------- Figure 1 -------

(1) J.W. Cooley & J.W. Tukey (1965) Maths Comput. 19, 297
(2) G. Otting, H. Widmer, G. Wagner & K. Wüthrich (1986) J. Magn. Reson. 66, 187
(3) R.R. Ernst, G. Bodenhausen & A. Wokaun (1987) *Principles of NMR in one and two dimensions* p. 104
(4) Additional baseline distortions may be encountered when using a real FT: see D. Marion & A. Bax (1988) J. Magn. Reson 79, 352
(5) D.I. Hoult, C.-N. Chen, H. Eden & M. Eden (1983) J. Magn. Reson. 51, 110
(6) D. Marion & A. Bax J. Magn. Reson. (in press - June 89)
(7) Homer *The Iliad* Book XIII (Date unknown) and Aeschylus *Agamemnon* (5th century B.C.)

Pharmaceuticals Division
CIBA-GEIGY Corporation
Summit, New Jersey 07901

CIBA–GEIGY

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

January 9, 1989
(received 1/26/89)

"NMR Spectroscopy and Recovery of a Surgically Modified Animal"

Dear Professor Shapiro:

In our recent in-vivo NMR studies, we have used a home-built surface coil probe to examine rat liver toxicity. This probe was designed for a Varian XL-400 vertical wide bore NMR spectrometer and tuned to ^{31}P at 161.9 MHz. In performing these experiments, it was necessary to surgically expose the liver in order to eliminate signals from muscle. A phosphorous NMR spectrum of muscle, obtained when the surface coil is placed directly over the skin of the rat's abdomen, is shown in Figure 1.

The surgical procedure, necessary to insure that the phosphate signals were predominately from the liver, required the removal of the abdominal musculature. The superficial skin flaps were then reflected and sutured laterally. The absence of the phosphocreatine peak in the ^{31}P spectrum (Figure 2) gave good indication that we were focussed on the liver. These experiments, however, required that the animal be sacrificed immediately following the NMR scan, making it impossible to follow the time course of a toxin in a single animal.

We now report a surgical procedure which not only allows ^{31}P NMR spectra to be obtained from the liver without interference from skin and muscle but also permits recovery and survival of the animal. The procedure involves the removal of the abdominal muscle overlying the liver and surgical implantation of a Mersilene mesh (Ethicon, Somerville, N.J.) to the remaining abdominal musculature. Following suturing of the skin flaps back to their previous anatomic position, the animals were allowed to recover from the Ketamine induced anesthesia. Three days post surgery, phosphorous spectra were taken through the skin of the underlying liver. The results (Figure 3) show an absence of the phosphocreatine peak indicating no observable contamination from skin or muscle. The animals survived for 6 weeks post surgery and were finally sacrificed. Muscle regeneration was observed after four weeks.

The implantation of the mesh and the successful recovery of the animals allows NMR studies of rat liver metabolism to be carried out over an extended time frame on a single animal. To the best of our knowledge, the implantation of the

Mersiline mesh for the purpose of NMR studies using a surface coil probe is the first of its kind and may prove useful for drug studies in which liver metabolism is monitored.

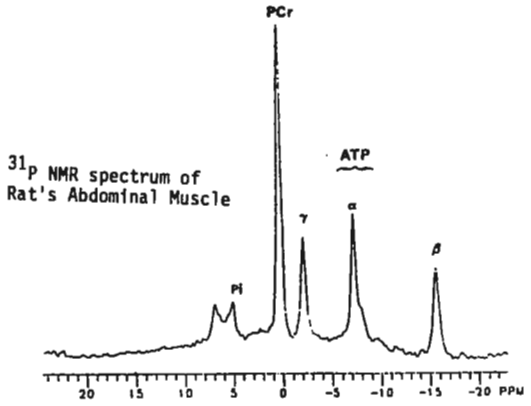
Sincerely,

Jeff Plutchok

Jeff Plutchok

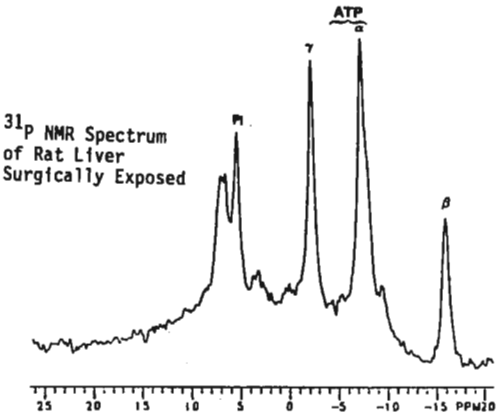
Nina C. Gonnella

Nina C. Gonnella



^{31}P NMR spectrum of Rat's Abdominal Muscle

Figure 1.



^{31}P NMR Spectrum of Rat Liver Surgically Exposed

Figure 2.

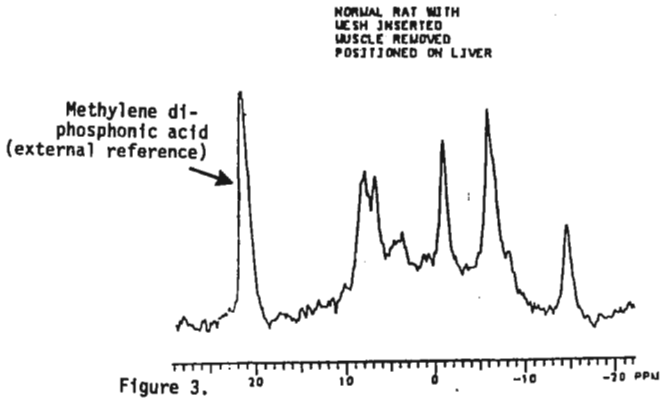
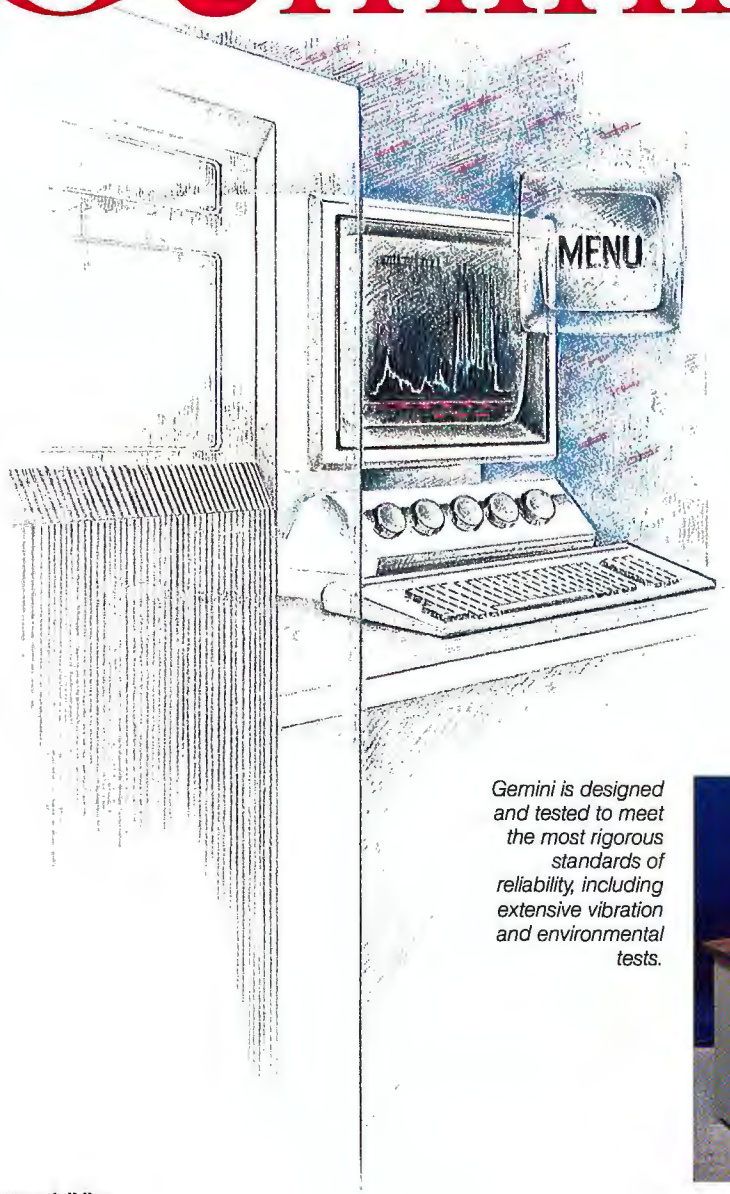


Figure 3.

How to acquire expert NMR data at the touch of a button

Gemini



Announcing Gemini: Never has any NMR spectrometer been this easy to learn and use

GuidePath™—our new advanced-menu user interface makes Gemini the world's easiest-to-use spectrometer. And gives you expert results!

Never has FT NMR been this reliable

Gemini's industry-standard design, the complete automation of all spectrometer functions, user-proven expert system software and extensive diagnostics all combine to produce the world's most reliable FT NMR results.

Call now

Get all the facts about Gemini—the new superconducting FT NMR from Varian. In the U.S., call 800-231-8134. In Canada, call 416-457-4130. In Europe, call Zug, Switzerland, at (042) 44-88-44; Darmstadt, Germany, at (06151) 7030. In Japan, call 3-204-1211.

Gemini is designed and tested to meet the most rigorous standards of reliability, including extensive vibration and environmental tests.

Varian is your full-line company for analytical instrumentation
UV-Visible-NIR
LIMS
Data Systems
Atomic Absorption
NMR
Liquid Chromatography
Gas Chromatography

MAG-4615A/801

NMR WITH A FUTURE

varian

How to bring true computer power to your NMR research

Introducing: the new VXR 5000 Series computers which integrate Varian's NMR processing and control software with computer workstations from one of the world's leading manufacturers—Sun Microsystems.*

Now, for the first time, you can add a broad range of true computer capabilities to your NMR research. These features make that possible:

- **Industry-standard computers and advanced version of the UNIX* operating system**—provide high reliability and peak performance
- **Advanced windowing system**—fast and extremely easy to use
- **Flexible data processing**—from routine 1-D spectra to automatic 2-D analysis
- **Image processing**—quickly processes large, multidimensional images
- **Multipurpose and multiuser**—expands laboratory capabilities and increases productivity

The VXR 5000 series computers, standard on all our VXR and imaging spectrometers, feature a choice of monochrome or color/grayscale displays and a variety of options for enhancing performance.

Call now for all the facts
In the United States, call 800-231-8134.
In Canada, call 416-457-4130.
In Europe, call Zug, Switzerland, at (042) 44 88 44; Darmstadt, Germany, at (06151) 70 30. In Japan, call (3) 204 12 11.

*Sun Microsystems is a trademark of Sun Microsystems, Inc. UNIX is a trademark of AT&T Bell Laboratories.

Varian is your full-line company for analytical instrumentation
UV-Visible-NIR
LIMS
Data Systems
Atomic Absorption
NMR
Liquid Chromatography
Gas Chromatography

MAG-4615A/801

NMR WITH A FUTURE

varian

DEPARTMENT OF HEALTH & HUMAN SERVICES
200 C St., S.W. (HFF-423)

Public Health Service

Food and Drug Administration
Washington DC 20204

Feb. 7, 1989
(received 2/11/89)

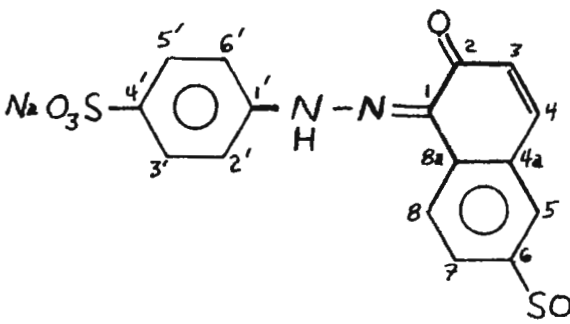
Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Ct.
Palo Alto, CA 94303

• Determination of Indirect C-H Connectivities by the FLOCK Sequence

Dear Barry,

The assignment of quaternary carbons in aromatic compounds is particularly difficult because "model" compounds cannot adequately mimic the conjugative effects which occur throughout these systems. Calculated chemical shifts, as can be seen below, are of little help in these efforts. The FLOCK variable evolution time sequence, developed by Bill Reynolds at the University of Toronto and scheduled to appear in the March issue of Magn. Reson. in Chem., enabled us to assign all of the carbons of Yellow No. 6 except 1 and 4a. The experiment would have permitted these resonances to be distinguished, but 2-bond C-H coupling constants are generally too small in aromatic systems to effect the necessary magnetization transfer. The structure of Yellow No. 6, carbon assignments, and calculated values, Gelbcke et al., Anal. Lett., 13, 975 (1980), are given below.

Position	Observed	n_H	Calculated
1	128.8*	0	133.4
2	178.4	0	149.7
3	126.2	1	120.5
4	142.6	1	134.2
4a	127.0*	0	128.3
5	126.4	1	125.9
6	140.6	0	137.5
7	126.1	1	122.8
8	122.1	1	127.8
8a	134.1	0	130.0
1'	142.8	0	155.9
2'/6'	116.7	1	123.6
3'/5'	127.3	1	126.8
4'	140.1	0	145.8



Sincerely,

John E. Bailey

E. P. Mazzola

UNIVERSITY OF CALIFORNIA, LOS ANGELES UCLA

BERKELEY · DAVIS · IRVINE · LOS ANGELES · RIVERSIDE · SAN DIEGO · SAN FRANCISCO 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

January 31, 1989

High-Accuracy Integrals; Comments on Integrals

Dear Barry:

We have recently become interested in measuring integrals to as high an accuracy as possible for a particular problem, but our experience may also be of general interest. As is known, the errors in such results depend on the signal-to-noise ratio, the spectrometer stability, the line width and shape, the peak separation, the dynamic range, the avoidance of saturation and unwanted NOE's, any base-line flattening procedure used, any line broadening or sharpening procedure used, the actual integration or peak fitting procedure used (including personal biases and the unwarranted rejection of any data), and (possibly) the phase of the moon! As the required accuracy becomes greater, so does the need to avoid systematic errors and subtle artifacts.

The system that we have been studying is a very simple one, namely the ^2H $\{^1H\}$ spectrum of cyclohexane-d_1 at -95 °C. There are just two rather well separated lines ($\Delta\nu$ = 36.2 Hz on a Bruker AM 500 MHz spectrometer; line width at half-height = 1.3 Hz in CS_2-5% CD_2Cl_2) of nearly the same intensities. The difference in the integrated intensities of the axial and equatorial deuteron signals gives the conformational preference (Winstein's A value) of deuterium in cyclohexane-*d1*, and this approach to obtaining that quantity was first reported a number of years ago (R. Aydin and H. Günther, *Angew. Chem., Int. Ed. Engl.* **1981**, *20*, 985-986) on a Bruker WH 400 MHz spectrometer (at -85 °C, these workers reported an equilibrium constant of $1/K_{ea} = 1.060 \pm 0.014$, i.e., $K_{ea} = 0.9434 \pm 0.0135$, favoring the equatorial position by 22 ± 5 cal/mol). However, later work in our laboratory using Saunders' isotopic perturbation method at room temperature on cyclohexane-*d10* isotopomers gave an A value of 6.3 ± 1.5 cal/mol (F. A. L. Anet and M. Kopelevich, *J. Am. Chem. Soc.* ***1986***, *108*, 1355-1356).

We have therefore tried to re-measure the A value of deuterium in cylohexane-*d1* by Günther's integration method. We have found that it is extremely easy to obtain spurious results, in large part because the lines are not extremely sharp and therefore overlap very slightly, and establishing symmetrical peaks and a flat base line is of critical importance. The alternating sampling of the real and imaginary part of the FID (which is used in Bruker spectrometers) introduces problems with obtaining a flat baseline unless a special non-standard procedure is used. This procedure, which we now describe, has been used by other workers, but does not seem to be widely known, and was brought to our attention by Dr. Mike Geckle, who also helped us to implement it. The frequency-independent and frequency-dependent phase corrections (PC0 and PC1) were set to 270 and 180° respectively. With the transmitter frequency set on resonance for a peak (CD_2Cl_2 in our case), a microprogram [1 ZE; 2 D1; 3 P1 PH1; 4 D5; 5 D5 PH0; 6 D6 ADC; 7 RCYC = 2 PH2; with PH1 = 0, 2, 1, 3 and PH2 = R0, R2, R1, R3] was used to phase that peak by adjusting the value of PH0 (360). The other peaks (i.e., those of cyclohexane-*d1*) were then phased by adjustments of the delay (D5) between the end of the pulse and the start of data acquisition and/or by slight variations in the filter band-width parameter (FW). The base line produced is very flat and requires little or no correction before peaks are integrated.

When the Challenge is Research,

the Choice is the Fully Capable, Cost-Effective, Accessible VIVOSPEC™ Magnetic Resonance Imaging Spectrometer.

New

Designed for MR Spectroscopy and Imaging by MR Researchers, the First Truly Accessible System that Really Puts Users First.

With the VIVOSPEC SPECTROMETER, you get a full range of spectroscopy and imaging features and functions, including—

- MULTINUCLEAR SPECTROSCOPY AND IMAGING CAPABILITY
- TRANSFORMED SPECTRA IN REAL TIME FOR SHIMMING
- HETERO- AND HOMONUCLEAR DECOUPLER OPTIONS
- OPTIONAL SELF SHIELDED GRADIENTS

. . . and much more.

Consider the Research Challenges Already Being Addressed:

- METABOLIC STUDIES
- MUSCULOSKELETAL STUDIES
- NEUROLOGICAL STUDIES
- ISCHEMIA STUDIES
- PHARMACEUTICAL STUDIES
- ANESTHESIA STUDIES
- IN-VITRO STUDIES

Discover the Savings

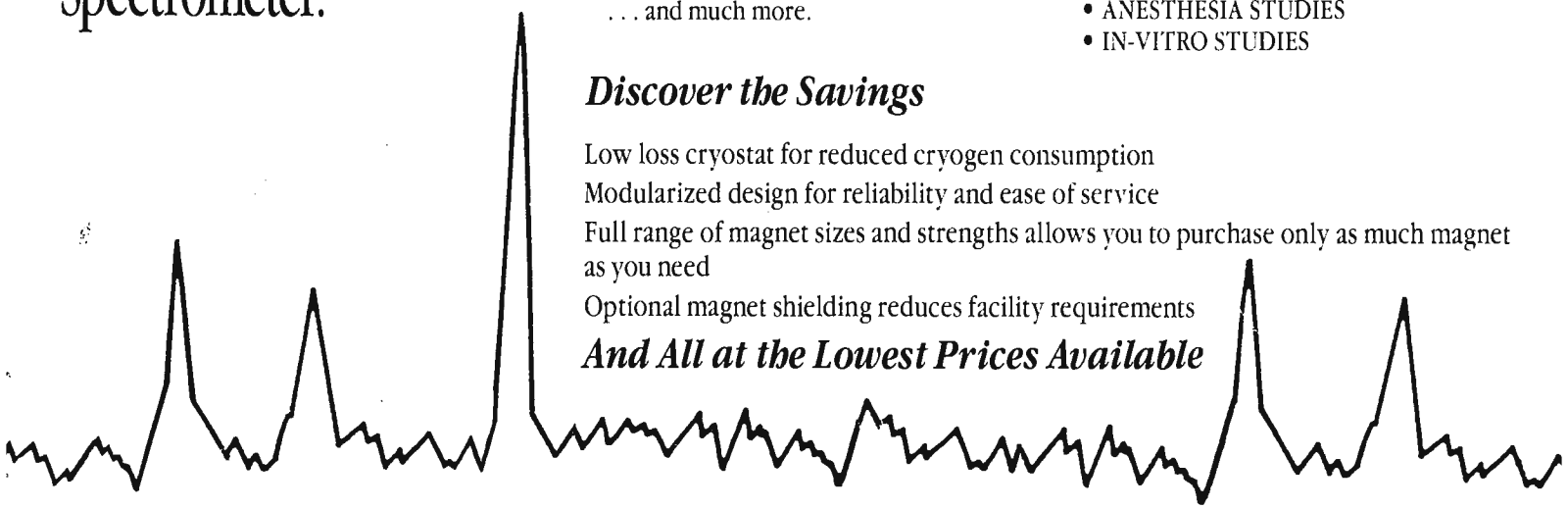
Low loss cryostat for reduced cryogen consumption

Modularized design for reliability and ease of service

Full range of magnet sizes and strengths allows you to purchase only as much magnet as you need

Optional magnet shielding reduces facility requirements

And All at the Lowest Prices Available



CALL NOW FOR MORE DETAILS ON THE VERSATILE, INNOVATIVE VIVOSPEC Magnetic Resonance Imaging Spectrometer . . . a product of Otsuka Electronics.

OTSUKA ELECTRONICS (U.S.A.) INC. 1 RAYMOND DRIVE • HAVERTOWN, PA 19083 USA • (215) 789-7474 • FAX (215) 789-8081

Our integration results, obtained as the average of three independent integrations (one done blindly, where the person knew nothing about the previous history of the problem) on 8 separate sets of high signal-to-noise FID data, give $K_{ea} = 0.977 \pm 0.002$, corresponding to an A value of 8.2 ± 0.9. The error in K_{ea} does not, of course, account for any possible remaining systematic errors, and we plan to test our integration result by integrations on *cis*- and *trans*-cylohexane-1,4-d_2.

We would like to hear through the Newsletter or personally of other attempts to measure integrals with an error of a small fraction of 1% (our definition of a *high-accuracy integral).* The question of integral accuracy in connection with a particular chemical problem has been investigated recently by Zvi Rappoport (see review in *Acc. Chem. Res.* **1988**, *21*, 442-449), although not at the level of accuracy needed for the cyclohexane problem. By the way, differences in intensities of the *same* peak under different conditions (as in difference spectroscopy) are much easier to obtain than the relative intensities of *different* peaks that more or less overlap.

The above work will appear in Tetrahedron Letters, and we hope to present it as a poster at the ENC in April.

Finally, a couple of comments on peak intensities and integrals in NMR (cf., Ray Freeman's treatment of intensities in his excellent *A Handbook of Nuclear Magnetic Resonance*):

(a) It is generally stated explicitly that quantitative NMR work requires that the area of each peak (or group of peaks) in a spectrum be *proportional to the number of nuclei* contributing to the peak(s). Whilst it is true that certain quantitative analyses, as in the work described above, demand this constraint, others do not. Quantitative work using ultraviolet, visible or infrared spectroscopy is routinely done, even though the above type of constraint is never met and each peak has its own extinction coefficient! Quantitative NMR analyses in many cases do not require that peak areas be proportional to the number of nuclei (thus, saturation or partial NOE's can be present), as for example in the analyses of mixtures of known compounds (especially in dilute solutions), where standard samples can be made for calibration purposes. The only requirement is that these saturation and NOE effects be reproducible between samples with the same spectrometer parameters. The distinction between these two types of integrals (**fixed-proportional** and **variable-proportional**) is not properly recognized in the NMR literature, and indeed, there is a lack of convenient names for these two methods. Discussions of quantitative aspects of NMR should not ignore the value of the latter type of integrals, as is generally done currently.

(b) The importance of a correct first point in the FID for obtaining accurate integrals is sometimes exaggerated. It is true, mathematically, that if the first point in the time domain is zero, then the total integral in the frequency domain is zero. However, integrals of sharp peaks over a narrow frequency range can still be reliably obtained! Also, Gaussian-Lorentzian line narrowing, which strongly affects the first part of the FID, does not necessarily invalidate peak integral information.

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

Sincerely yours

Frank

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

Altona and Karplus type calculations on a MacIntosh

M. J. Minch and C. Ward, Departments of Chemistry and Mathematics
University of the Pacific, Stockton, CA 95211 (received 2/17/89)

Vicinal proton coupling constants are a function not only of the time averaged orientation of each substituent with respect to the coupled proton pair, but also the electronegativity of all substituents bound to the H-C-C-H fragment. A number of useful general rules and semi-empirical Karplus-type equations, correcting for substituent electronegativity, have been developed from J-values for rigid ring couplings. This includes the useful equation of Altona Haasnoot, and DeLeeuw (Tetrahedron, *36* (1980) 2783-2792) which is a six-term expression based on MO theory with parameters that are functions of both the electronegativity of the first (α) and second (β) atoms of each substituent, as well as the torsion angle and orientation, with the whole expression parametrized by statistical correlation to 315 experimental coupling constants, covering a wide range of rigid compounds and substituents.

$$^3J = P_1\cos^2\phi + P_2\cos\phi + \Sigma\Delta\chi_i \{P_3 + P_4\cos^2(\zeta_i\cdot\phi + P_5 \mid \Delta\chi_i\mid)\} \qquad (1)$$

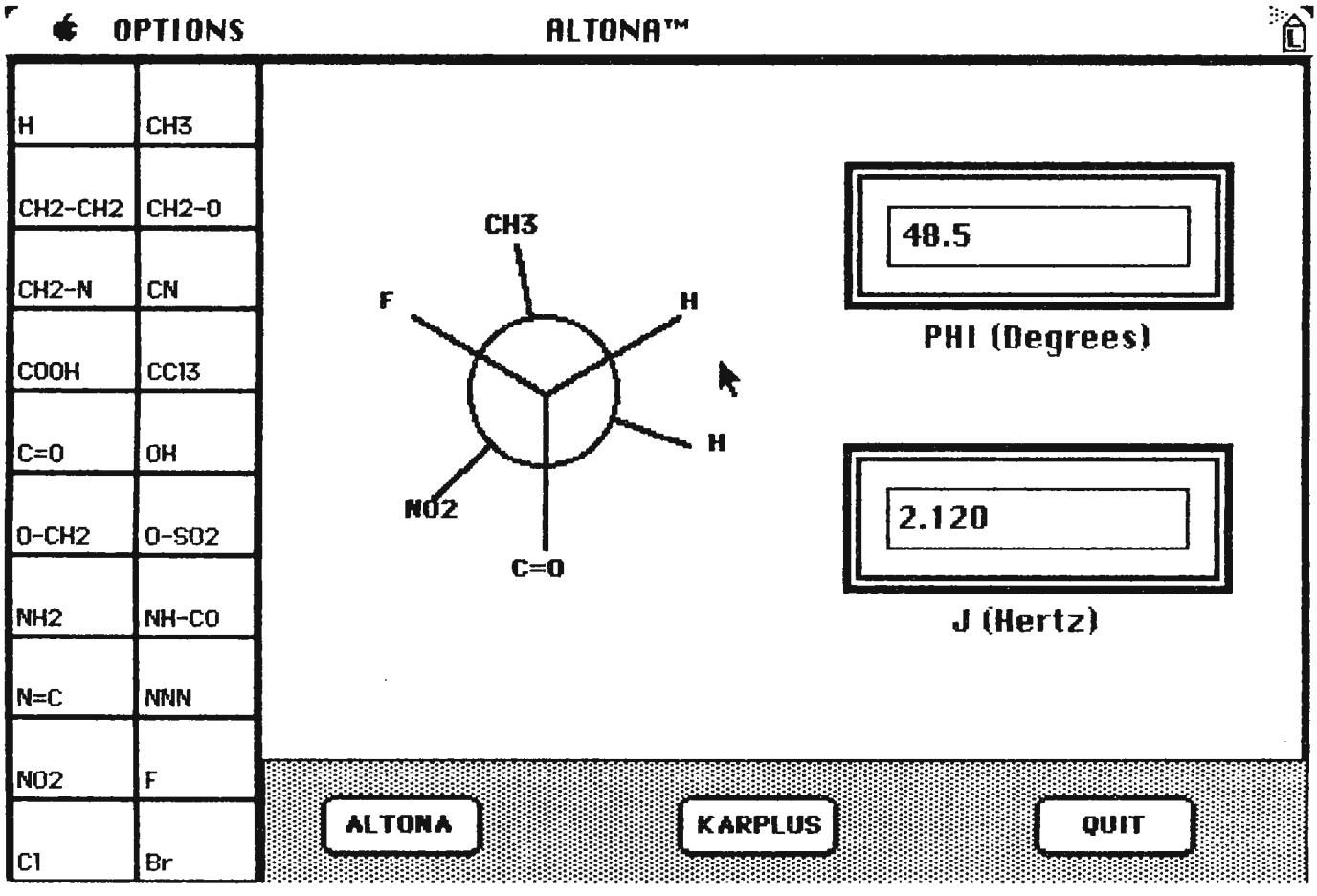
For substituents containing β-atoms

$$\Delta\chi_i = \Delta\chi^{\alpha\text{-atom}} - P_6\,\Sigma\Delta\chi^{\beta_i\text{-atoms}} \quad \text{where } \Delta\chi_i = \chi_{\text{atom } i} - \chi_H$$

ζ_i is ±1 depending on the substituet orientation , and $\Delta\chi_i$ is calculated from the Huggins electronegativity.

We have recently developed an easy-to-use program, ALTONA based on Equation (1) and employing interactive graphics written for the MacIntosh™ computer. The program calculates torsion angles from observed J-values or the reverse, J-values from known torsion angles, using four different versions of the Karplus equation or the Altona equation. The calculated geometry is revealed as a Newman projection down the C-C bond. The user selects the substituents about this C-C bond from a menu and types in a dihedral angle to calculate coupling constants. Plots of J vs. angle are also provided.

This program is available to interested spectroscopists with MacIntosh computers for $10.00. Please do not send disks! Make checks out to Coburn Ward and allow three-to-four weeks for delivery of one copy of the program on a 800 K 3.5 inch disk with instructions for its use. Call for additional details (209) 946-2442.



Colorado State University

Department of Chemistry
Fort Collins, Colorado 80523
(303) 491-6480

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

January 26, 1989
(received 2/1/89)

Solid State NMR Results on the AM-600; Postdoctoral Position Available.

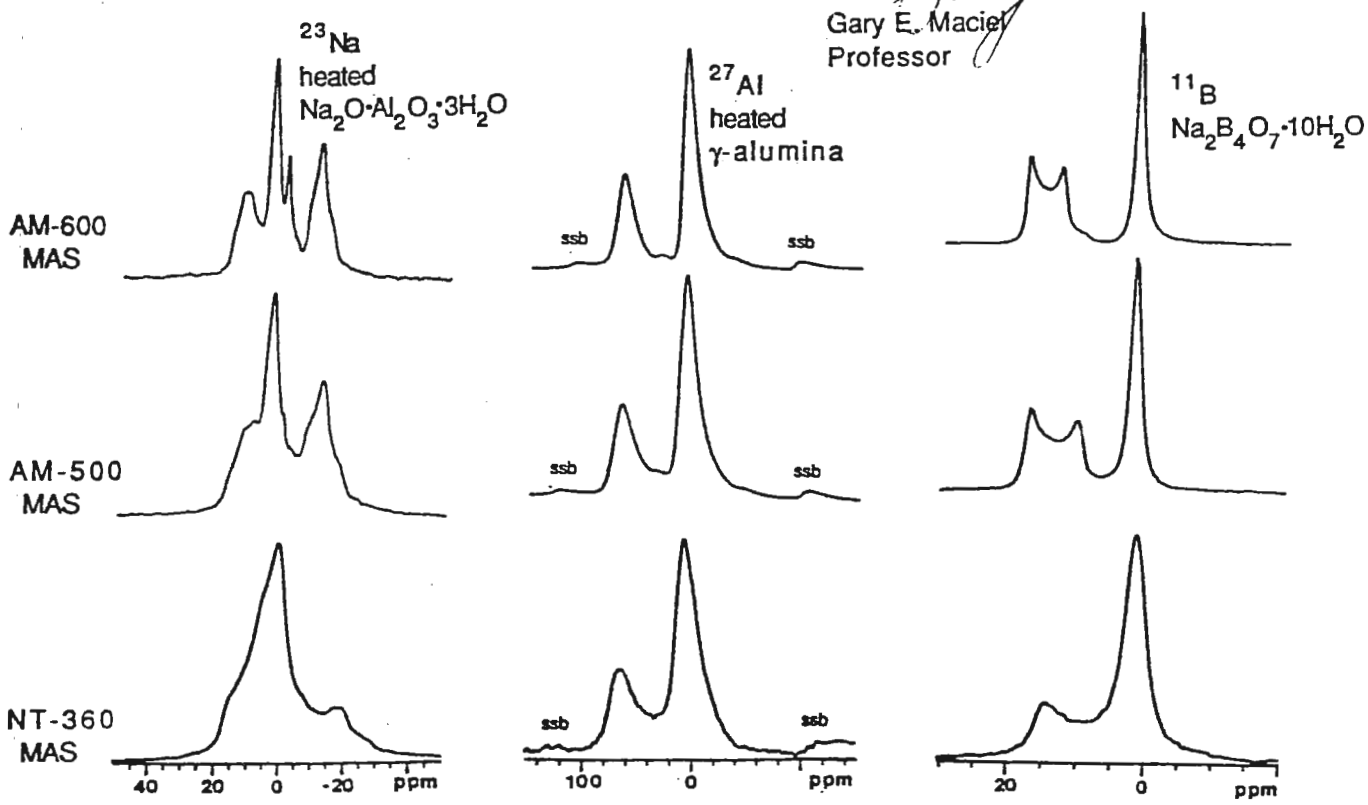
Dear Barry:

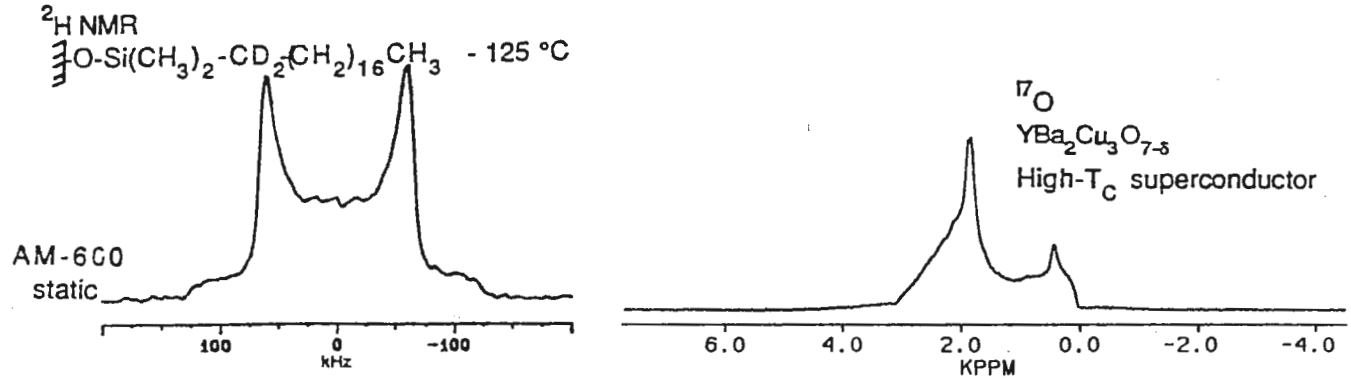
Performance of the AM-600 spectrometer, installed in the C.S.U. Regional NMR Center (303-491-6455) several months ago, has been outstanding. We have expended a considerable effort examining the field dependence of -1/2↔1/2 spectra of quadrupolar nuclides under conditions of high-speed MAS. Thanks to a high-speed digitizer built by Bruce Hawkins, we have also obtained excellent wide-line performance. The spectra below demonstrate the kinds of results we have been obtaining.

Beginning about July, 1989, a postdoctoral position will become available in the Center. Applicants interested in details should contact me.

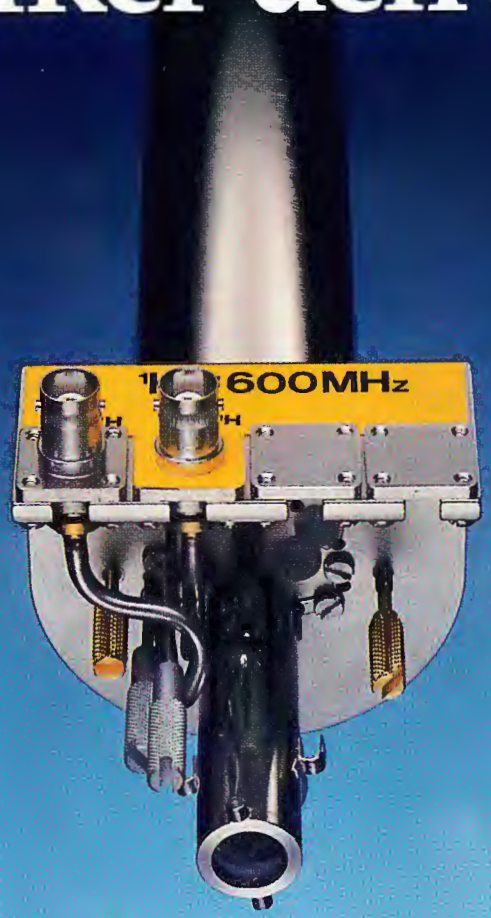
Sincerely,

Gary E. Maciel
Professor





Bruker delivers.



Again.

Since its foundation in 1960, Bruker has continued to make major contributions to the field of commercial analytical instrumentation, especially NMR:
1963: World's first pulsed NMR spectrometer.
1967: World's first truly multinuclear high resolution NMR spectrometer.
1969: Introduction of Fourier transform techniques for NMR.

Since then Bruker has entered the fields of FT-IR, NMR Imaging (MRI), and in-vivo spectroscopy.

We introduced a fiberoptics link for high-speed data transfer and ultra-fast array processors for the new generation of NMR data processing systems.

Then in 1986 we introduced the first microscopy accessory for NMR.

And now Bruker announces the 600 MHz high resolution NMR spectrometer.

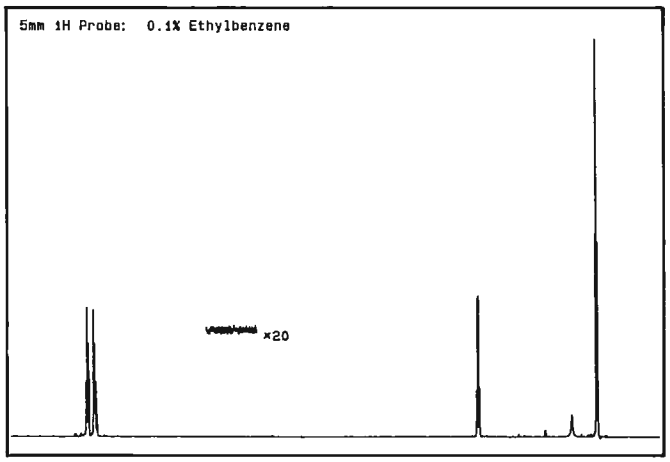
In short, no other manufacturer has advanced NMR as significantly as Bruker. And no one delivers as complete a line of systems and accessories for research and routine NMR, and as comprehensive a support package:
From hardware to software, from technical service to applications support. Bruker delivers. To find out more, drop us a line or use the reader service card.

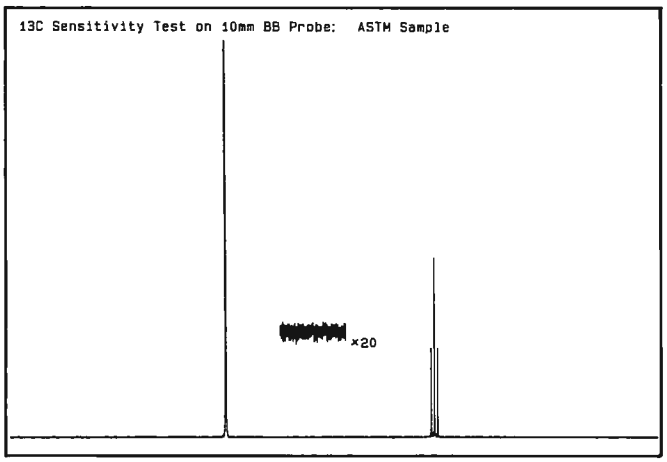
Bruker Instruments, Inc.
Manning Park, Billerica, MA 01821

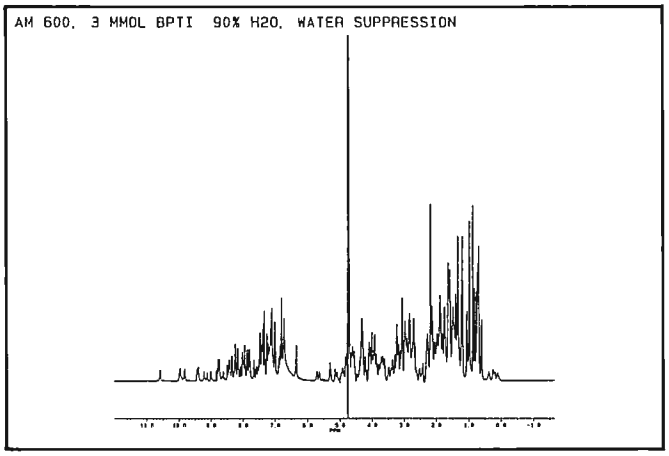
In Europe: Bruker Analytische Messtechnik GmbH, Silberstreifen, D-7512 Rheinstetten 4, W. Germany

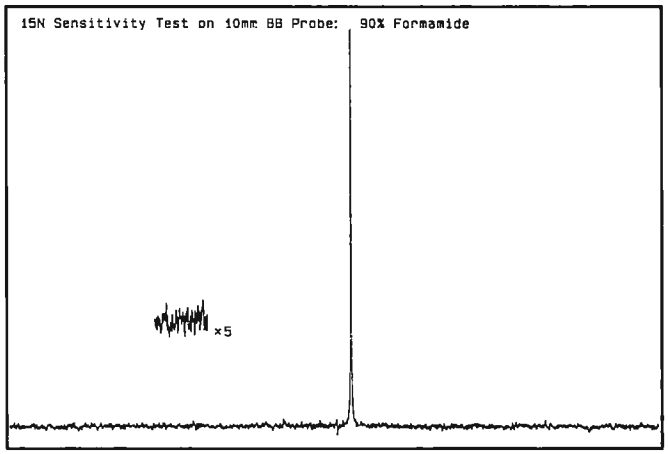
BRUKER *NMR Systems designed to solve problems.*

AM 600 NMR Spectra









Current AM 600 specifications (subject to change):

Probehead	Nucleus	Test	Value
^{1}H selective (5mm)	^{1}H	lineshape	10/20
		S/N	600:1
Broadband (5mm)	^{13}C	S/N (ASTM)	220:1
		S/N (10% EB)	180:1
		lineshape	6/15
	^{15}N	S/N (FORM)	25:1
Broadband (10mm)	^{13}C	S/N (ASTM)	650:1
		S/N (10% EB)	375:1
		lineshape	6/15
	^{15}N	S/N (FORM)	80:1

Resolution (all probes) 0.25 Hz

Magnet drift: ca. <40 Hz/hr

EB = ethylbenzene with ^{1}H decoupling
ASTM = 60% C_6D_6 in dioxane
FORM = Formamide (^{1}H decoupling without NOE)
LINESHAPE: ^{1}H = $CHC1_3$ linewidth at height of ^{13}C satellite/at 20% this level
^{13}C = C_6H_6 linewidth at 0.55%/0.11% of peak height

NMR FT-IR DATA SYSTEMS BRUKER NMR IMAGING EPR MS

Australia: BRUKER (Australia) Pty. LTD., Earlwood, New South Wales, Tel. 02-5589747
Belgium: BRUKER SPECTROSPIN S.A./N.V., Brussels, Tel. (02) 648 53 99
Canada: BRUKER SPECTROSPIN LTD., Milton, Ontario, Tel. (416) 876-4641
England: BRUKER SPECTROSPIN LTD., Coventry, Tel. (0203) 463770
France: SADIS BRUKER SPECTROSPIN SA, Wissembourg, Tel. (088) 94 98 77
India: BRUKER INDIA SCIENTIFIC Pvt. LTD., Andheri (West), Bombay, Tel. 22 62 72 32
Italy: BRUKER SPECTROSPIN SRL, Milano, Tel. (02) 23 50 09, 2 36 40 69
Japan: BRUKER JAPAN CO. LTD., Ibaraki, Tel. 0298-52-1234
Netherlands: BRUKER SPECTROSPIN NV, Wormer, Tel. (75) 28 52 51
Scandinavia: BRUKER SPECTROSPIN AB, Åkersberga, Sweden, Tel. (07 64) 6 80 60
Spain: BRUKER ESPANOLA S.A., Madrid, Tel. 341-259-20-71
Switzerland: SPECTROSPIN AG, Fällanden, Tel. 1-82 59 111
W. Germany: BRUKER ANALYTISCHE MESSTECHNIK GMBH, Rheinstetten, Tel. 0721-5161-0
BRUKER ANALYTISCHE MESSTECHNIK GMBH, Karlsruhe, Tel. 0721-5967-0
BRUKER-FRANZEN ANALYTIK GMBH, Bremen, Tel. 0421-8700-80
USA: BRUKER INSTRUMENTS, INC., Billerica, MA 01821, (617) 667-9580
Regional Offices in Chicago/IL, Wilmington/DE, Houston/TX, San Jose/CA

The University of Iowa

Iowa City, Iowa 52242

The University of Iowa Hospitals and Clinics
Department of Radiology

319/356-2188
If no answer, 356-1616

1847

February 13, 1989
(received 2/17/89)

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

Dear Dr. Shapiro,

In clinical settings the inherent insensitivity of NMR mandates the need for optimal acquisition methods to permit decreased patient examination time. Multi-dimensional imaging and spectroscopy are most often performed with pulses sequences in which the raw data are obtained on a regular rectangular sampling grid. This pattern is inefficient for the isotropically band-limited objects commonly studied. Data points and time can be eliminated if efficient sampling patterns are used instead. I am evaluating the potential of these optimally-efficient methods in collaboration with Bill Thoma and our clinicians.

For isotropically band-limited functions in 2 dimensions, a hexagonal sampling grid is most efficient; in 3 dimensions, a body-centered cubic (BCC) grid is most efficient (see Figure 1). These optimal sampling patterns are advantageous because they require 13.4% and 29.3% fewer data points, for 2 and 3 dimensions respectively, than rectangular sampling methods. This gain can be used to either reduce the data acquisition time while maintaining image resolution or to improve the image resolution while maintaining the imaging time. For echo-planar imaging optimal sampling would lower the data rate requirements. A disadvantage of these efficient sampling techniques is that they use less data; hence, the signal-to-noise ratio of the results will be lower. For some 2-dimensional NMR spectroscopy applications this technique may not be useful because complete coverage of a rectangular frequency domain is required.

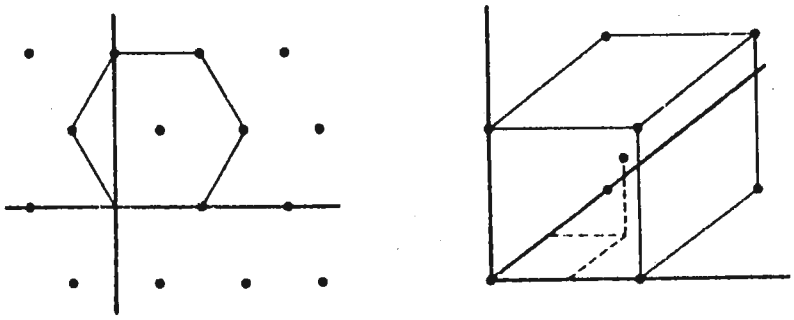
A modified DFT was derived which uses non-rectangular input points. However, the output points from this new transform are calculated on a rectangular grid because this is the pattern used by commercial hardware and software for image display and analysis. Fast algorithms for each of the efficiently-sampled multi-dimensional cases have been developed.(1,2) Without these algorithms, lengthy reconstruction times would render the efficient sampling schemes impractical.

Imaging in 3-dimensions may be implemented with a frequency-encoding gradient and 2 phase-encoding (PE) gradients. Hexagonal sampling in the 2 PE dimensions is accomplished by proper choice of the PE gradient stepping pattern. Thirteen percent fewer PE steps are required. In 4-dimensional chemical shift imaging optimal sampling in the 3 spatial dimensions with a 29% efficiency gain is achieved by choosing the stepping pattern of the 3 PE gradients appropriately.

The method assumes a band-limited source. This is equivalent to imaging a finite sized object. If the actual object is larger than the assumed size, the DFT produces aliasing. The pattern of this aliasing is given by the reciprocal lattice vectors of the sampling pattern. For rectangular sampling the aliasing occurs along the coordinate axes, but for hexagonal and BCC sampling the aliasing occurs primarily along the diagonals. This is an advantage because, in most imaging situations, the body tends to be elongated along an axis. Thus, optimal sampling may reduce aliasing problems.

References: (1) J.C. Ehrhardt, Soc. Mag. Res. Med., Book of Abstracts, 1087, 1988. (2) R.M. Mersereau and T.C. Speake, IEEE Trans. ASSP, 29,1011, 1981

Figure 1. On the left is a hexagonal grid of sampling points. On the right is a body-centered cubic grid of sampling points.



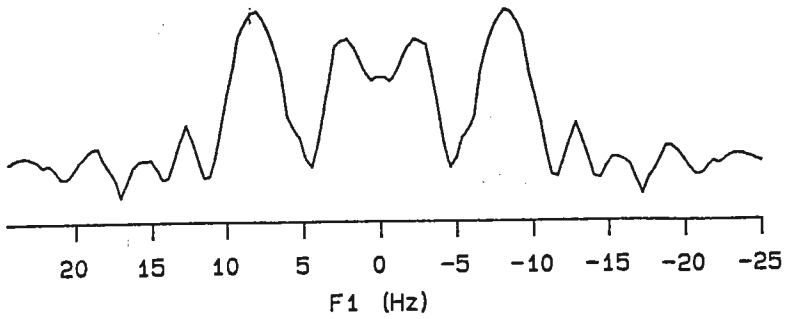
Sincerely,

Jim Ehrhardt

James C. Ehrhardt, PhD
Professor

- Continued from p. 45

The spectrum is a slice showing the J_{HH} of the H_1' proton of the labeled U of the DNA duplex. The spectrum shown is for the minor form of the DNA duplex. Both forms are in the C2'-endo family and a complete report on comparing the two conformational forms is in progress.



Wesleyan UNIVERSITY

HALL-ATWATER LABORATORIES
MIDDLETOWN, CONNECTICUT 06457
TEL.: (203) 347-9411

DEPARTMENT OF CHEMISTRY

February 15, 1989
(received 2/21/89)

B.L. Shapiro
Tamu NMR Newsletter
966 Elsinore Court
Palo Alto, CA 99303

Proton Detection Spectroscopy of DNA

Dear Barry:

Recently we obtained ^{13}C NMR data on a sample of duplex DNA of the sequence d(CCGUGCC) paired with d(GGCACGG) in which the U was labeled in the 1' and 3' positions with ^{13}C. The data showed that both the 1' and 3' sites were present in at least two environments. The ^{13}C NMR results showed that both sites exhibited two signals with the ratio between the intensities of the two forms changing with temperature and the signals from the two forms coalescing when the duplex was melted (J. Am. Chem. Soc. 109, 7217). The temperature dependent behavior was found to be completely reversible.

To obtain more information about this conformational equilibrium we have begun to perform a series of proton detection experiments on the labeled DNA. The proton signals of the 1' and 3' protons also exhibit two sets of signals each. One of the more informative experiments has been the use of ^{13}C filtered proton-proton J spectroscopy which allows, in principle, rather high resolution determination of the proton-proton couplings which can be related to DNA conformation. We have been using the pulse sequence

^{1}H: 90-tau- t1/2-180-t1/2-tau-acquisition

^{13}C: 90-90- -decoupling

to obtain data such as that shown in the figure for the two 1' sites. These and other data are now being analysed to determine the conformations of the two forms of this DNA duplex and how the two conformations differ.

Sincerely,

Philip H. Bolton
Associate Professor

- Continued on p. 44

ARGONNE NATIONAL LABORATORY

9700 South Cass Avenue, Argonne, Illinois 60439

ANL BITNET: BOTTO@ANLCHM
TELEX: 9102583285
PHONE: (312) 972-3524
FAX: (312) 972-4470

February 7, 1989
(received 2/15/89)

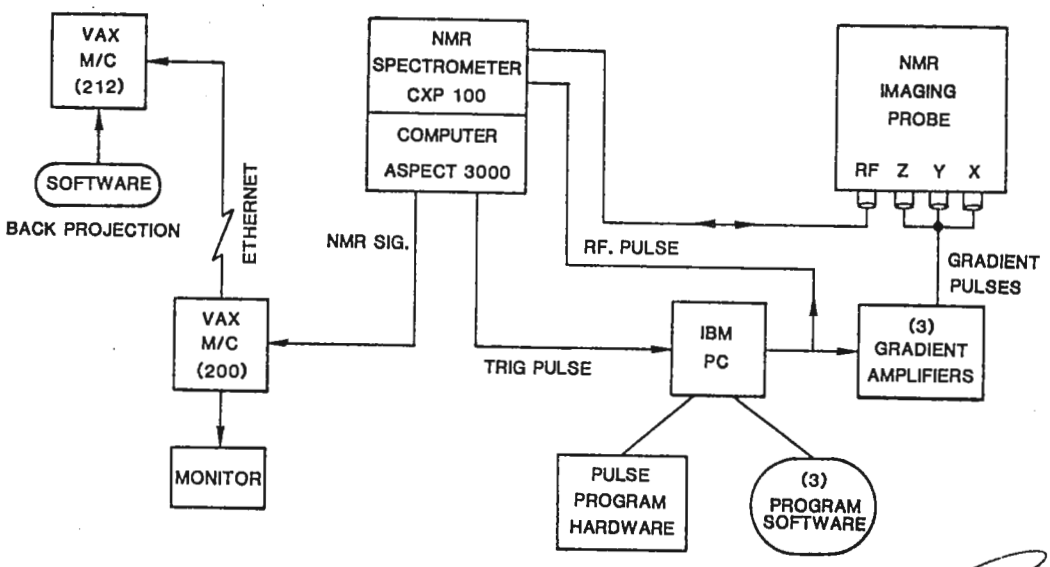
Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elisnore Court
Palo Alto, CA 94303

RE: Imaging of Solids on CXP-100 Spectrometer

Dear Barry:

We have become increasingly interested in the NMR imaging of short T_2 polymeric materials as applied to aspects of materials research. Our present application involves the determination of the distribution of multicomponent polymeric additives in advanced green ceramic components [Development of Nuclear Magnetic Resonance Imaging Technology for Advanced Ceramics, Argonne National Laborjatory Report ANL-87-53 (November, 1988).] Because of the short proton T_2s (typically less than 0.5 msec) of these materials, large magnetic field gradients are required to obtain the desired spatial resolution in the image. Additionally, the length of the T_2 also makes filtered back projection imaging techniques the method of choice over more widely used recalled echo techniques. To facilitate these experiments, we have designed and have begun to test a home-built imaging accessory for our Bruker CXP-100 spectrometer.

A block diagram of the imaging system is presented in the figure below. The probe has been designed to provide 54 g/cm and a 28 mm working sample diameter. An IBM PC based pulse programmer will provide RF pulse shaping and magnetic field gradient control. Experimental data will be acquired on the Bruker spectrometer and will then be ported from the Aspect 3000 to Argonne's VAX 8700 via ethernet for image reconstruction and processing.



Sincerely,

S. L. Dieckman S. Gopalsami P. S. Wong W. A. Ellingson R. E. Botto

REB/jmb

Fremont Magnetic Resonance
47647 Hoyt Street
Fremont CA 94539
(415)656-2398
(415)623-0851 FAX

If You Want To:

- Use your PC to Annotate and Plot Spectra on Lasers, DotMatrix Printers and HPGL Plotters.
- Incorporate Spectra into Desktop Publishing Programs.
- Routinely Determine NMR System Noise Figures.
- Add an X Nucleus Decoupler to your NMR System.
- Upgrade Existing NMR Probes.
- Obtain New Customized Probes and Coils.
- Utilize an NMR RF Module and Probe Repair Service.

Come See us at the ENC

April 2-6, 1989.

Willows Living Room,
Second Floor.

Or Call or FAX us anytime for details.

FMR is dedicated to upgrading installed NMR instruments. Our goal is to provide materials and services which allow your existing NMR equipment to perform *your* experiments at levels equal to or exceeding that of new NMR spectrometers.

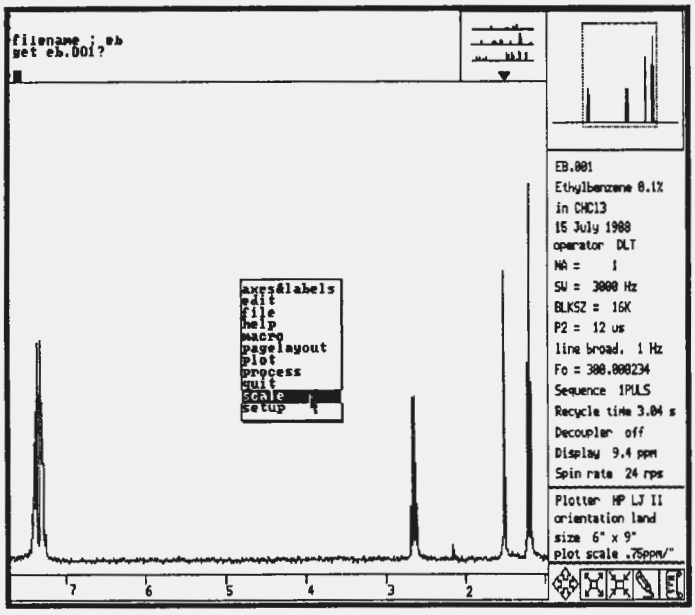
We offer an RF module and probe repair services. Modules and probes can be expressed to our facilities for repair by our trained technicians at reasonable rates. Repaired items can be returned air express for minimum down-time. Call us for time and price estimates. Detailed quotes can be provided after inspection and before repair if desired.

Due to our internal corporate structure and low overhead, we can provide responsive services at very competitive prices.

Fremont Magnetic Resonance
47647 Hoyt Street
Fremont CA 94539
(415)656-2398
(415)623-0851 FAX

PC based Plotting

Plotting takes a lot of time, even if your system is capable of multi-tasking and plotting in the background. And when its plotted all you have is a hard copy of the spectrum. With FMR's PC based plotting software package you can:



Screen Image

- Plot off-line in the convience of your office or home.
- Plot spectra using PC based laser printers, dot-matrix printers or HPGL plotters.
- Annotate the spectra with labels, notes, figures, arrows and etc..
- Incorporate the annotated spectra as figures in articles, memos and letters using popular desktop publishing software packages.
- Perform minor spectral editing to a data file for publication quality results.
- Archive the data using inexpensive PC based disks and tape drives.
- Have a convienent digital library of your spectra in your office for immediate reference and comparision.

PLOT is a software tool requiring an IBM PC or compatible, and supports common graphics devices, mice, plotters and printers. The transformed data file is transferred to the PC by RS-232, Kermit, Ethernet, or by emulating the spectrometer's plotter. PLOT is WYSIWYG, which means you can preview your plot on the computer screen as it will appear on paper. Recently accessed spectra are kept in a stack similar to an HP calculator for easy manipulating and viewing.

PLOT will work with CGA, EGA, VGA, Hercules, AT&T, or Tandy graphics systems. It will employ a 8087 coprocessor if it finds one. It can output your data to an HP-compatible laser printer, an HPGL plotter, A Zeta plotter, an Epson-compatible dot matrix printer, to a file in any of the above formats, or to a file for input into Aldus PageMaker or Xerox Ventura Publisher.

Online HELP is available for each command, icon, and screen area. The user may view the help text on topic "xxx" by typing "help xxx", or get help on screen items by pressing the F1 key and then placing the cusor over the questionable item.

AT&T Bell Laboratories

600 Mountain Avenue
Murray Hill, NJ 07974-2070
201 582-3000

February 16, 1989
(received 2/20/89)

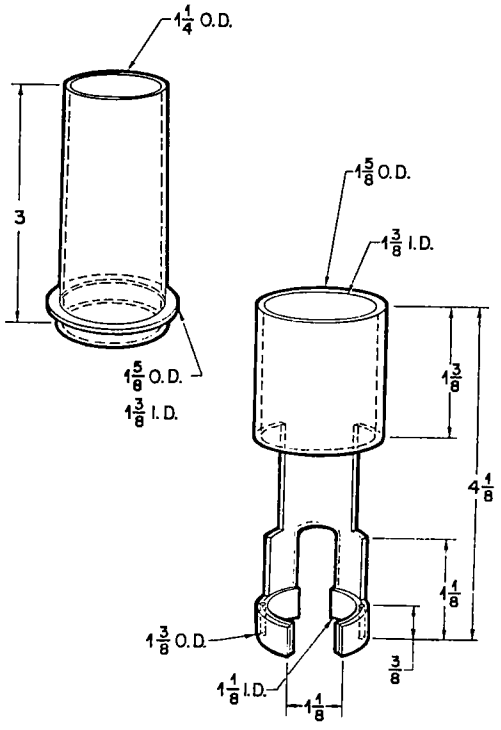
Dr. Barry Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Rodent Holder and Non-invasive Modification of a Bruker NMR Microscope Probe

Dear Barry:

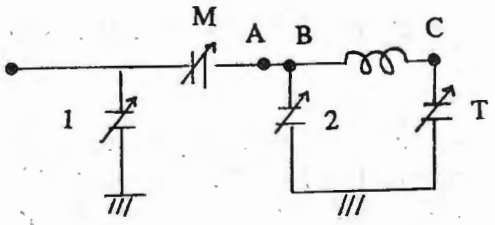
As you know, two of the most important requirements for microimaging on animals are (1) a highly reliable system for positioning and restraining the animal, and (2) excellent signal-to-noise. We have developed two modifications for the Bruker NMR Microscope probe that are invaluable for obtaining high resolution, high sensitivity surface coil images of rodents.

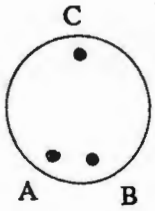
We show here a rodent holder that can comfortably accommodate an 80 gram rat. The dimensions are in inches. The significant aspects of this design are (i) it attaches firmly to the probe body, using the same screws and holes that were originally used to hold the volume imaging coil in place; (ii) it provides reliable and reproducible positioning; (iii) the tapered top allows easy insertion into the bore of the magnet; and (iv) breathing artifacts are minimized because the rodent can be gently taped into place.

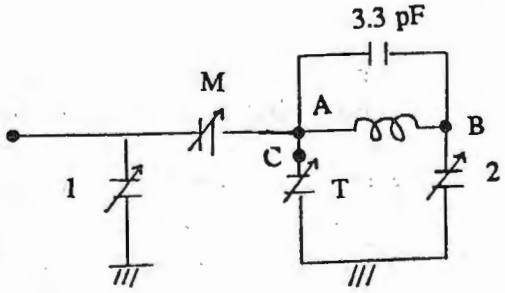


The holder is made of clear acrylic tubing, available from Acme Plastics of Patterson, New Jersey. Chloroform or Craftics #33 clear thickened cement can be used to glue the parts together. The top is tapered and milled out on the inside.

The other modification concerns a plug-compatible surface coil. The original circuit for the 20 mm volume imaging coil (left) can be modified "non-invasively" by removing the volume imaging coil from its plug-in socket (center, top-down view of socket). A teflon coated copper surface coil is fabricated (1 turn, 1 cm diameter) that has a 3.3 pF capacitor soldered across leads A and B. The coil, with legs A and C connected together, is placed in the circuit (right). The capacitor connecting A and B removes the "legs" from the circuit and greatly reduces the inductance of the coil, making the coil "loop" the only source of radio emission and detection. In these diagrams, 1, 2, M (match), and T (tune) are the original capacitors in the circuit for the original 20 mm coil. The inductor in the left diagram is the volume imaging coil; the inductor in the right diagram is the surface imaging coil.







Helen K. Tubbs Michael D. Cockman

Helen. K. Tubbs **Seiji Ogawa** **Michael D. Cockman**

Ron Behling Tso-Ming Lee Paul Glynn L. W. Jelinski

Ronald. W. Behling **Tso-Ming Lee** **Paul Glynn** **Lynn W. Jelinski**

McMASTER UNIVERSITY
Department of Chemistry
1280 Main Street West, Hamilton, Ontario L8S 4M1
Telephone: (416) 525-9140
FAXMAIL (416) 528-5030

February 2nd, 1989
(received 2/11/89)

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

DEUTERATED SOLVENT FOR HIGH TEMPERATURE POLYMER NMR

Dear Barry:

Carbon-13 NMR is commonly used to determine branching in polymers particularly in polyethylene and polypropylene. These spectra are usually obtained as 15% solutions in 1,2,4 trichlorobenzene at 120-140C. Long term resolution stability becomes difficult at this temperature and hence it was decided to deuterate the solvent. The FID shown in figure 1 was the result of 16 hours accumulation and the resulting spectrum in figure 2 indicates the resolution that can be obtained.

A limited amount of this solvent is available; contact NHW for further information and costs.

B.G. Sayer G. Timmins N.H. Werstiuk

Please credit this contribution to the account of A.D. Bain.

Fig.1. FID of polypropylene in 1,2,4 trichlorobenzene after 16 hours

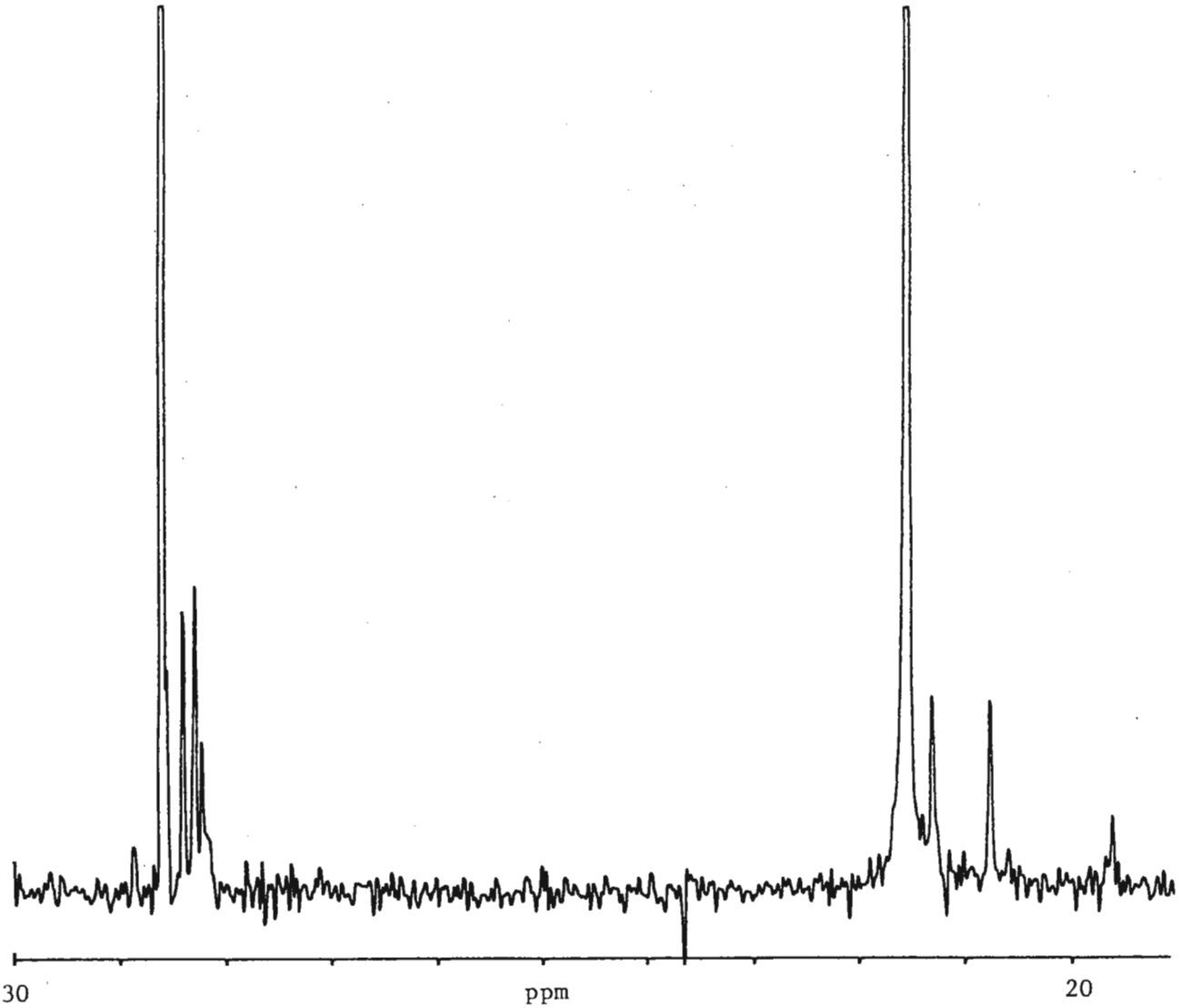


Fig.2. Part of carbon-13 NMR spectrum of polypropylene

Myra

and

ISOTEC INC.
A Matheson, USA Company

invite

you to join us

in our suite

VIEWPOINT EAST

Asilomar Conference Center

at the

30th ENC

Monday, Tuesday and Wednesday evenings

9:30 p.m.———?

D, ^{13}C, ^{15}N, ^{17}O, ^{18}O

Are you aware that ***ISOTEC INC.*** is a reliable, affordable source of compounds labelled with the above stable isotopes?

Our product line ranges from the very simple — $^{1}H_2O$ (deuterium-depleted), D_2, D_2O, ^{13}CO, $^{13}CO_2$, $^{13}CH_3I$, $K^{13}CN$, $KC^{15}N$, $K^{13}C^{15}N$, $^{15}NH_4Cl$, $H^{15}NO_3$, $H_2{}^{17}O$, $H_2{}^{18}O$, $^{18}O_2$, etc. — to the more complex — Benzaldehyde-α-^{13}C,d, Glycine-2-^{13}C,^{15}N, D-Glucose-1-^{13}C, D-Glucose-$^{13}C_6$, Styrene-α,β-$^{13}C_2$, etc. We also have the major NMR solvents in stock—very pure and very highly enriched, all checked by ^{1}H NMR at 400 MHz.

We now have hundreds of compounds available from stock and are making more each day. We are also able to carry out many custom syntheses; please let us quote on your requirements.

ISOTEC INC. is also a primary supplier of ^{3}He and the other noble gas stable isotopes, including ^{21}Ne, ^{83}Kr, ^{129}Xe and ^{131}Xe.

Numerous other stable isotopes, enriched and depleted, are available as well as most enriched metal stable isotopes including NMR-active ones such as ^{29}Si, ^{57}Fe, ^{65}Cu, ^{77}Se, ^{87}Rb, ^{113}Cd, ^{125}Te, ^{157}Gd, ^{199}Hg, etc.

ISOTEC INC. provides very high quality stable isotope labelled compounds at very, very affordable prices. Please call *Myra Gordon* or *Maureen Kalloo* to place your orders or to receive additional information.

ISOTEC INC.

A Matheson® USA Company

3858 Benner Road • Miamisburg, Ohio 45342 U.S.A.

(513) 859-1808 • (800) 448-9760 • Telex: 288278 • Fax: (513) 859-4878

January 30, 1989
(received 2/13/89)

Dear Dr. Shapiro,

The graduate students of the University of Rhode Island's Chemistry Dept. present their 11th Symposium entitled, "NMR STATE OF THE ART AND FUTURE DIRECTIONS" on April 29, 1989.

Dr. Joseph J.H. Ackerman (Washington Univ.) will speak on the topic of, Invivo NMR Spectroscopy, Dr. Ad Bax (NIH) will discuss recent developments in, Two- and Three- Dimensional NMR. Dr. Gary E. Maciel (Colorado State Univ.) will talk about "High Resolution ^{1}H NMR of Solids". Dr. Alexander Pines (UC Berkeley and National Academy of Sciences) entitles his lecture "There's No Free Induction" and Dr. Jacob C.A. Schaefer (Washington Univ.) will discuss the detection of weak $^{13}C-^{15}N$ dipolar coupling by "Magic-Angle Spinning NMR".

We extend an invitation to all graduate students working in the field of NMR to present their research at our Poster Session during the symposium.

Registration is requested and the charge is $18.00 per person (nonstudent). For more information please contact Brad Sherman or Robyn Pothier, (401) 792-2203 Dept. of Chemistry, University of Rhode Island, Kingston, RI 02881.

The Graduate Student Symposium Committee would greatly appreciate your listing of our symposium in the March or April issue.

Thankyou for your time,

Robyn Pothier

Robyn Pothier co-chairperson

DOW CHEMICAL U.S.A.

TEXAS OPERATIONS
FREEPORT, TEXAS 77541

February 10, 1989
(received 2/17/89)

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

Dear Dr. Shapiro,

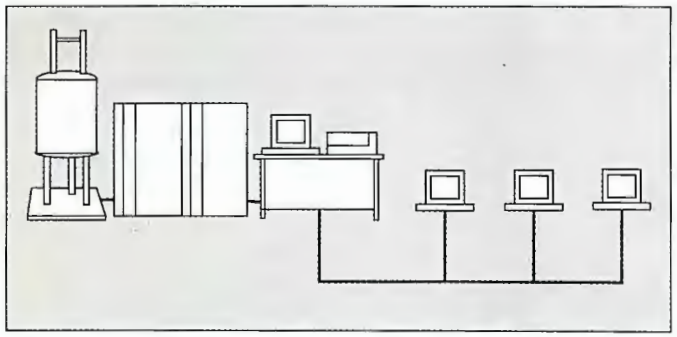
Although automatic sample changers are available for most commercial NMR spectrometers, not all manufacturers allow for the changing of samples which are at temperatures other than ambient. The need for maintaining samples at an elevated temperature prior to introduction into the superconducting magnet is particularly important when, as in our laboratory, numerous polyolefin copolymers must be analyzed. These samples need to be kept at 130 degrees C both before and during analysis by carbon-13 NMR. We describe here a system which utilizes a Zymark robot to automatically load, run and unload up to nine samples on a JEOL GX-400 NMR. Our goal was to attach the robot to the NMR in such a manner that samples could be picked up from a hot block, placed in the NMR and then retrieved after the run was complete. Also, we did not wish to become involved in modification of the basic vendor supplied NMR programs or hardware.

The layout of our system is shown in Figure 1. In operation, NMR tubes which are already fitted with spinners are placed in an insulated metal block which is heated by a standard laboratory hot plate (Thermolyne type 2200) and equipped with an over-temperature cutoff. The basic sequence of movements by the robot is triggered by the ejection of a sample. The detection of an eject signal by the power and event controller of the robot uses the only spectrometer hardware modification necessary for the system. On the JEOL GX-400 this signal was easily obtained from the spin and sense amplifier (connector SRIN). Upon detection of an eject signal the robot retrieves the sample, confirms the sample's presence in the hand, and places it in the receiving rack. The new sample is removed from the hot block, confirmed and placed on the air cushion at the top of the superconducting magnet. During this process the macro programs of the spectrometer are in a pause mode. The robot then types a continue command on the keyboard accessible to it. Control of the instrument is restored to the spectrometers, which then proceeds to lower the sample, set up the parameters, perform necessary "touch up" shimming and begin the data acquisition. A Tektronix 4107 terminal is the normal second terminal on the JEOL system and this keyboard is used by the robot to pass control back to the spectrometer. When the robot is used, the spectrometer is thus run from the Tektronix rather than the "main" spectrometer terminal. The keyboard must be used with the autorepeat feature off and symbols such as @ must be redefined so that only a single keystroke is needed. Throughout the system, standard vendor supplied programs (such as autoshim routines) were incorporated without modification into the macro programs.

Here Is Your Brand New $10,000, 500 MHz FT-NMR Spectrometer.

Our GSX Series FT-NMR spectrometer was built for the laboratory which has a large family of NMR users, but the budget for only one NMR spectrometer.

When you invest in one GSX, you can get up to three others just by adding data stations, each of which costs approximately $10,000.



The GSX FT-NMR spectrometer can be shared by up to four users simultaneously. While one does an experiment, others can be analyzing data, preparing manuscripts, or generating slides for the next scientific meeting.

The GSX is an advanced technology NMR instrument equipped with a multi-user, multi-tasking DEC 'microPDP-11 based' data system.

It offers the highest available sensitivity and resolution with automatic shimming, and such advanced technology features as automatic probe matching and menu-driven control.

Best of all, for the large laboratory, a single GSX can be configured to accommodate multiple users simultaneously. And that means more productivity and less aggravation for everyone.

While one user is running an experiment, a second can be analyzing data from a previous run, a third working on a manuscript, and a fourth preparing slides for the next scientific meeting.

No one even needs to be in the NMR lab. Experiments can be queued up on the autosampler and run from remote locations — another lab, another office, even another building.

Come see our multi-user GSX FT-NMR spectrometer in operation.

For a demonstration or literature, call (508)535-5900. Or write JEOL U.S.A. Inc., 11 Dearborn Road, Peabody, MA 01960.

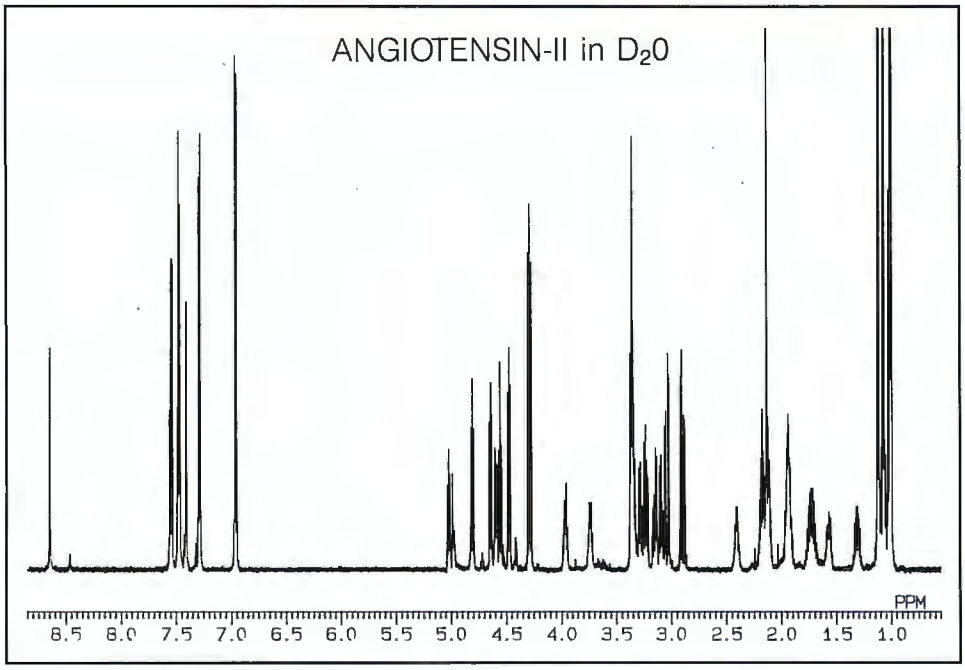
GSX FT-NMR spectrometer

JEOL

Serving Advanced Technology

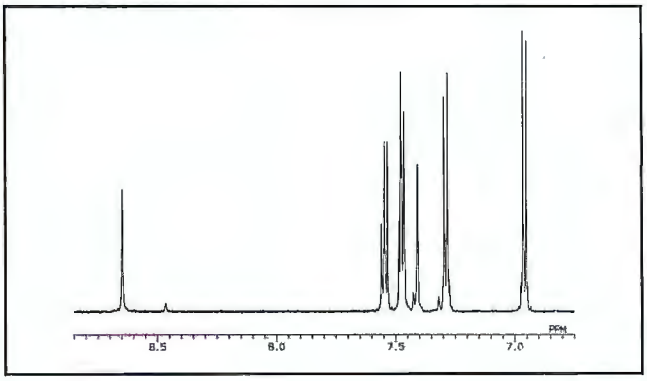
microPDP-11 is a trademark of Digital Equipment Corporation

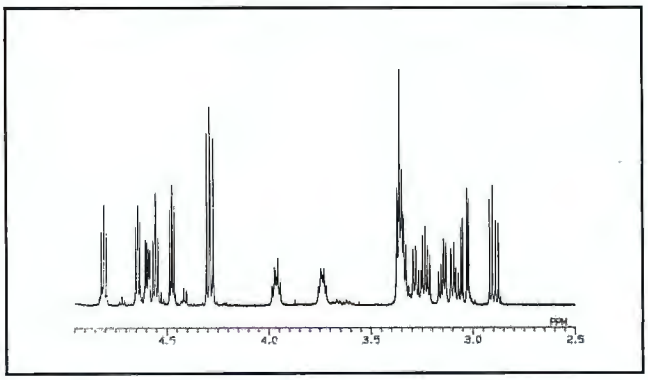
GSX-600

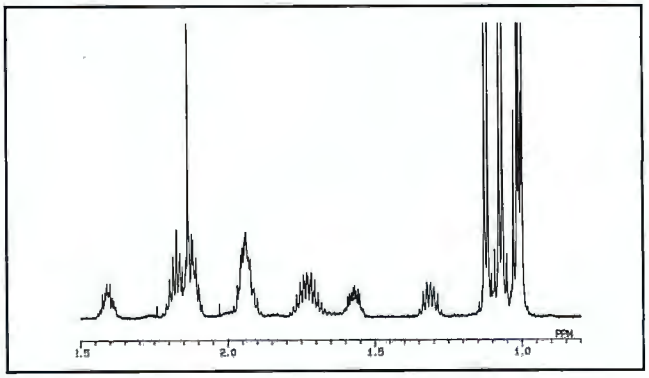


JEOL in its continuing commitment to the scientific community announces an operational 600 MHz NMR spectrometer at its Akashima applications laboratory. The Oxford Instruments 14.1 Tesla superconducting solenoid with JEOL's well proven matrix shim system, benchmark Rf technology, and excellence in probe design combine to provide the highest dispersion in 600 MHz performance. If your research and applications in macro molecules or low gamma nuclei requires 600 MHz performance, you owe it to yourself to call JEOL.

The above data is of Angiotensin-II in D_2O and was processed under normal one dimensional conditions. If you are presently evaluating 600 MHZ NMR spectrometers, we would be pleased to arrange for you to see the GSX-600.







Please Contact:

JEOL

Serving Advanced Technology

11 Dearborn Road, Peabody, MA 01960
(508) 535-5900

The design of the hand is of importance, since the retrieval of an ejected sample involves the grasping of a moving target as the sample bobs on the air cushion that ejects it. This bobbing motion is greatly reduced by the plastic "cover" shown in Figure 2. The hand is lowered over the sample with the jaws at maximum separation; at the appropriate height the top of the NMR tube bobs against the plastic cover stabilizing the tube's motion. The jaws then close to grasp the sample. Confirming the presence of the sample in the hand is done by having the robot place the sample so as to interrupt the light source to a photodiode. This simple device consists of a photodiode and a light bulb mounted six inches apart and 6.5 inches high. Both the bulb and photodiode are under the control of the Zymark power and event controller.

In order to minimize the effects of the system on magnet homogeneity, the steel skirt of the robot was replaced with one made of aluminum. Also, a steel plate at the base of the Tektronix keyboard was removed and replaced with plexiglas. The table for the system consists of a plywood base with aluminum legs.

Although some steel portions of the Zymark robot could not be easily replaced, we have not experienced any significant degradation of magnet homogeneity. Small changes in the field were easily shimmed out. The homogeneity effects are minimized by locating the robot and associated hardware at the maximum possible distance (both horizontally and vertically) from the magnet. Also, the robot arm is programmed to return to a position which causes the least effect on the field and to remain there during the run. Our robot spectroscopist system is used quite frequently, and has proven to be reliable and convenient.

M. D. Meadows R. P. VanderWal G. V. Bettoney

Figure 1

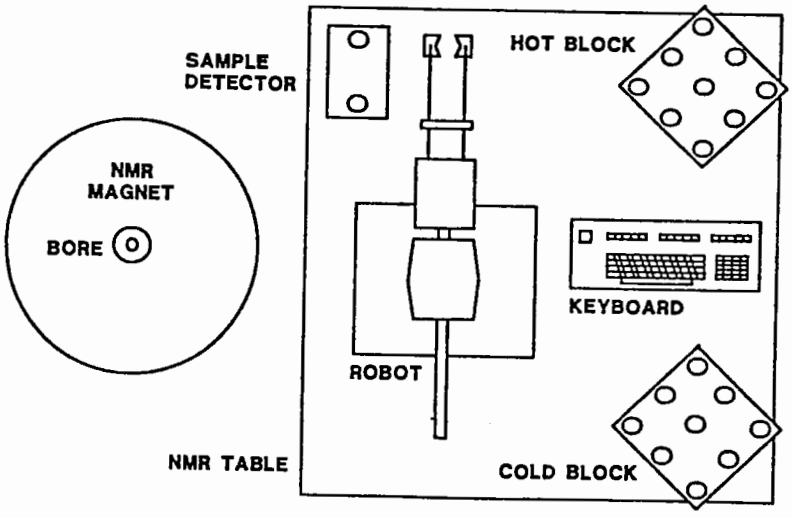
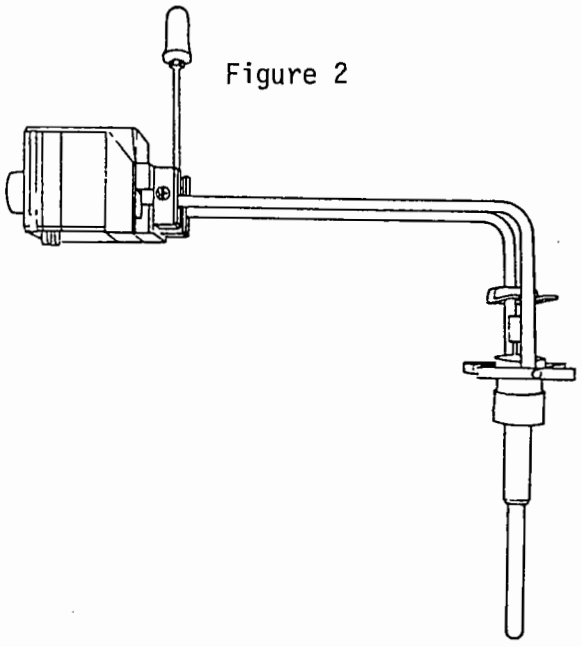


Figure 2



UNIVERSITY OF CALIFORNIA, SANTA CRUZ

BERKELEY • DAVIS • IRVINE • LOS ANGELES • RIVERSIDE • SAN DIEGO • SAN FRANCISCO SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY SANTA CRUZ, CALIFORNIA 95064

February 15, 1989
(received 2/17/89)

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

FREQUENCY RANGE SENSITIVITY ON GE PROBES

Dear Barry:

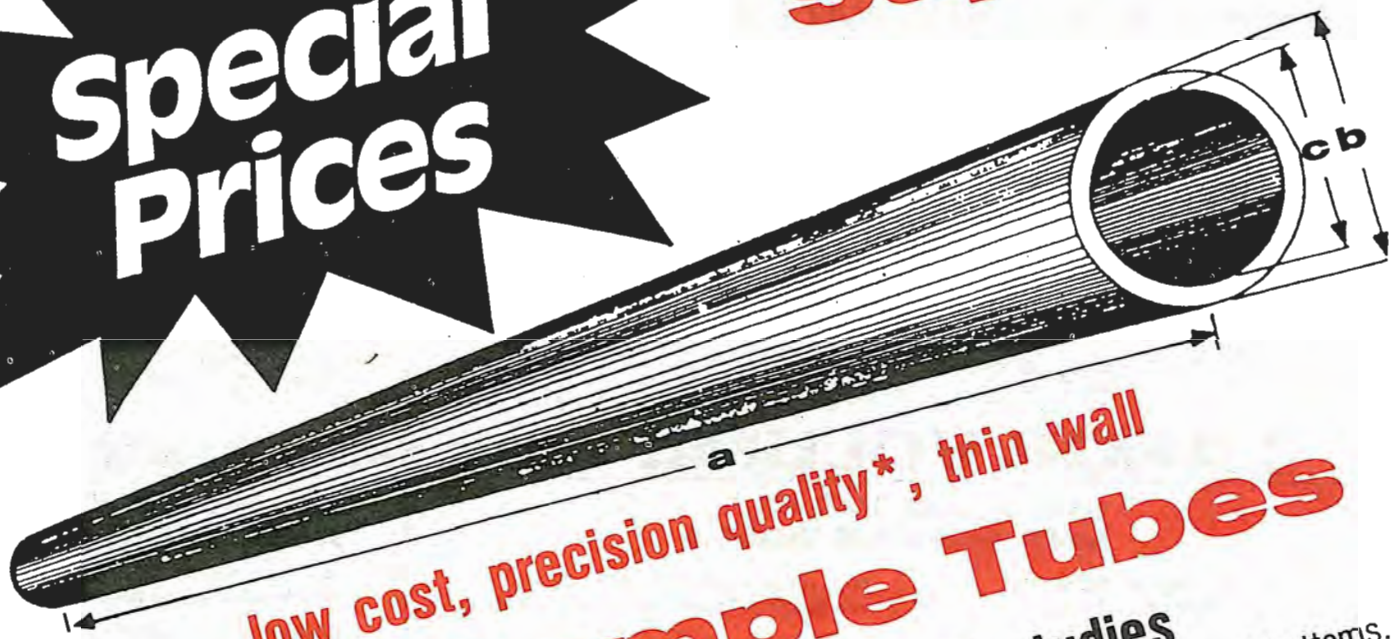
The high dielectric constant associated with samples of high ionic strength in aqueous solutions can alter the resonant characteristics of a probe coil beyond the capabilities of the tuning capacitors for a given frequency. We encountered this problem recently (1.2 grams of KCL in D_2O) on our dedicated GE (GN-300) Widebore 12mm Potassium Probe (14.MHz).

Applying the concept that in a resonant circuit, the order of circuit elements in series is irrelevant [Alderman & Grant, JMR 36, 447-451 (1979)] we have been successful in increasing the frequency range of our Potassium probe as well as "Broadbanding" a dedicated 5mm C/H probe to tune from 70 MHz (^{59}CO) through 121 MHz (^{31}P) with no observable decrease in signal to noise and line shape. At 5mm the Proton 90° pulse at approximately 75 watts (60db) remained 13 usec; 90° pulse for ^{13}C is 12 usec (ASTM) and for Ammonium Phosphate in D2O the ^{31}P 90° pulse is 10 usec after the modification.

Although the circuitry is well known to all, [Murphy-Boesch, Biomedical Magnetic Resonance (1984)] we used the same basic LC configuration on both probes. Figure 1. is the Carbon-13 circuit modification and Figure 2. the Potassium-39 modification. All the variable capacitors are tunable to 12 pf. The fixed porcelain capacitor was empirically selected. The "shorting" stick (Figure 1) is disengaged for frequencies between 79 MHz (^{23}Na) and 121 MHz.

Special Prices

Brand New Super-5™

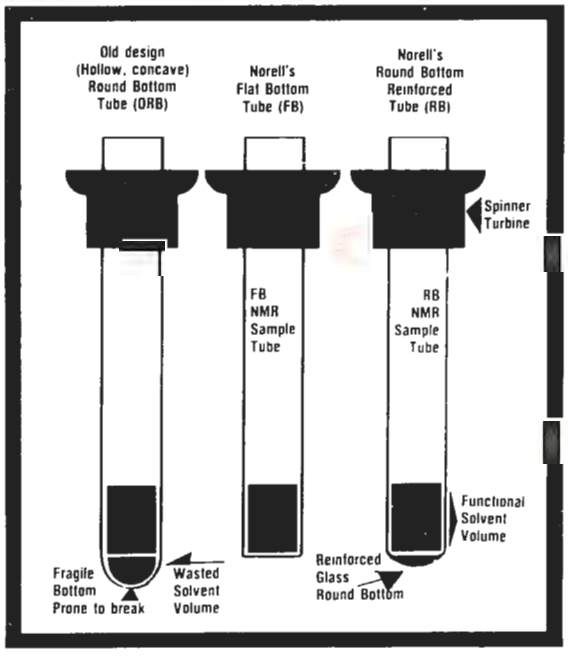


low cost, precision quality*, thin wall

NMR Sample Tubes

for routine, all purpose NMR studies

Super-5 NMR tubes are supplied with either flat or concentric-reinforced round bottoms. When ordering, specify either FB (for flat bottom), or RB (for concentric round bottom).



SPECIFICATIONS:

Super-5™

Tube length: 178mm (7 in.)
o.d. 4.97mm ± 0.04mm
i.d. 4.20mm ± 0.04mm
camber: ± 0.04mm

Price:

25 tubes ($1.50 ea.) $37.50
50 tubes ($1.40 ea.) $70.00
100 tubes ($1.30 ea.) $130.00

*Controlled in production by high accuracy laser beam.

Please Note: More recently, we developed a specially reinforced and concentric flat bottom tube on the inside with a round bottom on the outside. Not only that you save on solvent with this tube, it is less prone to break when tapped on a hard surface. **When a round bottom tube is ordered, we expedite this new, solvent saving, reinforced RB tube.** If you insist on the older, hollow and concave design (used primarily by competition) - simply add letter ORB to our catalog number - there is no extra charge.

NORELL, INC., 314 Arbor Ave., Landisville, NJ 08326
Call toll-free 1-800-222-0036, in NJ 609-697-0020

NMR Sample Tubes

1988-1989 PRICE LIST

5mm o.d. *THIN WALL* NMR SAMPLE TUBES

No. 508-UP ULTRA PRECISION for ultra high resolution NMR

Our top quality tube!

Standard tube lenght:178mm (7 inches);
o.d. 4.97 ± 0.013mm (0.0005 in.);
i.d. 4.20 ± 0.025mm (0.001 in.);
camber ± 0.013mm (0.0005 in.).
For additional length,
add $0.24 per cm ($0.60/in.).

1-99 tubes...$4.50 ea.
100-299.....$4.25 ea
300 + ...$4.00 ea.

No. 507-HP HIGH PRECISION for high resolution NMR

Standard tube length: 178mm (7 inches);
o.d. 4.97 ± 0.013mm (0.0005 in.);
i.d. 4.20 ± 0.025mm (0.001 in.);
camber ± 0.025mm (0.001 in.).
For additional length,
add $0.21 per cm ($0.50/in.).

1-99 tubes...$3.00 ea.
100-299.....$2.95 ea.
300 +.....$2.90 ea.

No. 506-P PRECISION for medium and high resln. NMR

Standard tube length: 178mm (7 inches);
o.d. 4.97 ± 0.025mm (0.001 in.);
i.d. 4.20 ± 0.025mm (0.001 in.);
camber ± 0.025mm (0.001 in.).
For additional length,
add $0.16 per cm ($0.40/in.).

1-99 tubes...$2.50 ea.
100-299.....$2.45 ea.
300 +.....$2.40 ea.

No. XR-55 PRECISION FOR medium and high resln. NMR

Standard tube length: 178mm (7 inches);
o.d. 4.97 ± 0.025mm (0.001 in.)
i.d. 4.20 ± 0.025mm (0.001 in.);
camber ± 0.03mm (0.0015 in.);
For additional length,
add $0.08 per cm ($0.20/in).

1-99 tubes...$2.20 ea.
100-299.....$2.00 ea.
300 +.....$1.90 ea.

No. 505-P PRECISION for medium and high resln. NMR

Standard tube length: 178mm (7 inches);
o.d. 4.97 ± 0.03mm (0.001 in.);
i.d. 4.20 ± 0.03mm (0.001 in.);
camber ± 0.04mm (0.002 in.);
For additional length,
add $0.08 per cm ($0.20/in).

1-99 tubes...$1.80 ea.
100-299.....$1.75 ea.
300 +.....$1.70 ea.

No. 502 THROWAWAY TYPE

Standard tube length: 178mm (7 inches)
o.d. 4.97 ± 0.05mm (0.002 in);
i.d. 4.20 0.05mm (0.002 in);
camber ± 0.07mm (0.003 in);
For additional length,
add $0.08 per cm ($0.20/in).

1-99 tubes...$0.80 ea.
100-299.....$0.75 ea.
300 +.....$0.70 ea.

LARGE VOLUME NMR SAMLE TUBES

10mm o.d. *THIN WALL* NMR SAMPLE TUBES

No. 1008-UP ULTRA PRECISION for ultra high resolution NMR

Standard length: 178mm (7 inches);
o.d. 10.00 ± 0.013mm (0.0005 in.);
i.d. 8.76 ± 0.025mm; (0.001 in.)
camber ± 0.007mm (0.0003 in.).
For additional lenght,
add $0.20 per cm ($0.50/in.).

1-5 tubes....$14.50 ea.
6-25.....$13.75 ea.
26 +.....$12.95 ea.

No. 1005-P PRECISION for medium and high resln. NMR

Standard length: 178mm (7 inches);
o.d. 10.00 ± 0.013mm (0.0005 in.);
i.d. 8.76 ± 0.025mm (0.001 in.);
camber ± .013mm (0.0005 in.).
For additional length,
add $0.20 per cm ($0.50/in.).

1-5 tubes....$9.50 ea.
6-25.....$9.25 ea.
26 +.....$9.00 ea.

20mm o.d. *THIN WALL* NMR SAMPLE TUBES

No. 2005-P PRECISION for medium and high resln. NMR

Standard length: 178mm (7 inches);
o.d. 20.00 ± 0.01mm;
i.d. 17.70 ± 0.02mm;
camber ± 0.03mm.
For additional lenght,
add $1.00 per cm ($2.50/in.).

1-5 tubes....$38.00 ea.
6 +.....$37.25 ea.

NOTE: Other tube sizes available, please inquire.

PTFE VORTEX PLUGS
All sizes - specify tube size...$40/lot of 5
Rods for all Vortex Plugs.....$10 each.

PTFE Machined Tube Caps
5mm ... $25/25 caps; $45/50 caps
10mm ... $28/25 caps; $50/50 caps

For HIGH PRECISION and ULTRAPRECISION work, we recommend our No. 507-HP and No. 508-UP NMR Sample Tubes.

Don't waste your money!

Why waste money on so called "Thrifty-Royal-Emperor" tubes when you get the same or better results with Norell's low cost No. 502 ($0.70 each in lots of 300) and precision XR-55 ($2.00 each in lots of 100). Choose either flat bottom (standard) or reinforced-concentric round bottom (by adding RB to our catalog number) .... available only from Norell.

Don't let them fool you!

... with claims of **infinitely better bargains.** Such claims serve no purpose **other than to mislead you,** and you end out a looser by paying double or tripple the price!

You save More with us!

Norell has the lowest prices in the industry.

To place your order, call toll-free
1-800-222-0036; in NJ call 609-697-0020

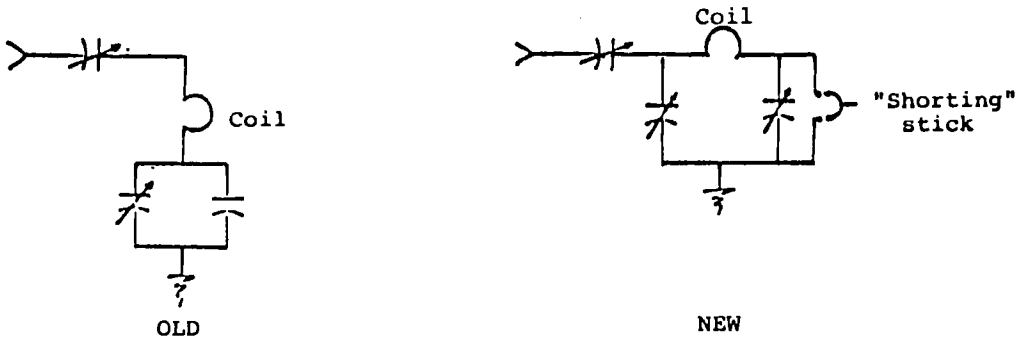


Figure 1.
Carbon-13 Probe Coil

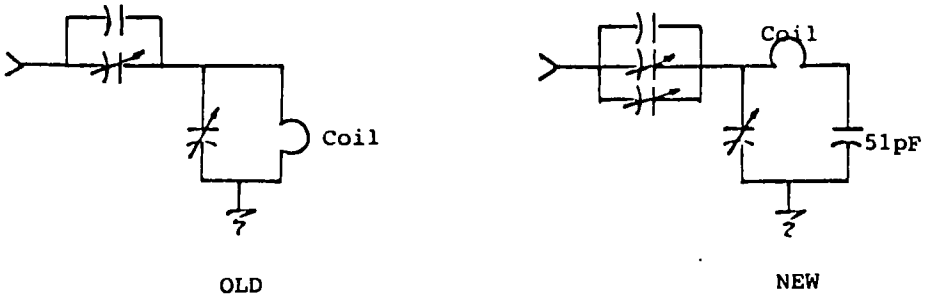


Figure 2.
Potassium-39 Probe Coil

Sincerely,

Jim Loo

Wilton Materials Research Centre

ICI Advanced Materials
ICI Films
ICI Chemicals & Polymers Ltd

PO Box No 90 Wilton Middlesbrough
Cleveland TS6 8JE England

Telephone (0642) 454144 (Switchboard)
Telex 587461 WWC G
Cables ICI Wilton Middlesbrough
Facsimile (0642) 432 444

Professor B L Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto
California CA 94303
USA

Your ref.	Our ref	Direct line	Tel Ext	Date
	S1PJ201M\LE		6413	23 January 1989 (received 1/28/89)

Dear Barry

VARIABLE TEMPERATURE IMAGING

We have been using a Bruker MSL-200 spectrometer, equipped with mini-imaging accessories, to investigate applications of MRI in materials science. Many of our samples have T_2S that are too short to allow signals to be generated by standard biomedical Fourier techniques. Our modified probe and improved gradient performance now enables echo times of the order of 2 ms to be used. Even so, T_2S from rigid solids, cross-linked polymers and strongly bound absorbed solvents may still be too short for observation. T_2S may be extended by operating at elevated temperatures by increasing the degree of molecular or chain-segment motion present in solids. In particular, polymers observed above the Tg can give liquid-like signals.

We have constructed a variable-temperature imaging probe consisting of a horizontally mounted solenoid, wound from 3 mm copper ribbon (3 cm x 1.5 cm) surrounded by a standard Bruker wide-line probe dewar and fitted with a thermocouple, supply gas dewar and heater. The probe can operate at temperatures between -120°C and +100°C, though, as yet, only high temperature operation has been investigated thoroughly. The low-Q r.f. circuit also allows multiple-pulse (MREV-8, etc) line-narrowing sequences to be performed, enabling solid materials to be imaged directly. The coil has good r.f. homogeneity over a volume approximately 1.2 cm by 2.0 cm, enabling small-scale materials to be imaged. With gradients up to 12 G/cm, resolution of ca 100 microns can be routinely achieved on typical samples.

Polyflon Company

Non-magnetic Capacitors for NMR Applications

An array of Polyflon non-magnetic capacitors, many of which are used in NMR/MRI applications.

Polyflon's rugged trimmer and variable capacitors meet rigid NMR/MRI specifications.

Polyflon's variable, trimmer and fixed capacitors meet critical non-magnetic, high voltage, high power, high Q NMR specifications.

A test report was recently submitted to Polyflon by a University deeply involved in NMR research. Polyflon trimmers were subjected to various tests in order to gather essential data (Q, DF, L, ESR, EPR) for use in the design of NMR RF equipment. Tests were conducted at 10, 100, 200, 400, 500, and 600 MHz. These trimmers were first exposed to a 4.7 Tesla magnetic field for detection of the presence of any magnetic particles in the trimmers. These tests conclusively showed there were no magnetic particles present.

Maximum dissipation displayed by the trimmers was 0.000125 with a minimum less than 0.0001, resulting in Q measurements from 8,000 to more than 10,000, at 600 MHz. (NOTE: Customers, who have tested Polyflon trimmers in their own laboratories, have measured Q's greater than 20,000 at 500 MHz.) The inductance of the trimmers was less than one nHy and maximum ESR at 160 milliohms with minimum EPR at 100 megohms.

Polyflon trimmers and variable capacitors provide linear tuning with no reversal in capacitance during capacitor adjustment. They are used in NMR pulsed applications with operating voltages up to 15 Kv peak and duty cycles ranging up to 10 %. Standard range of variable capacitor designs are available with a minimum capacitance of less than 1 pF to a maximum capacitance of 125 pF.

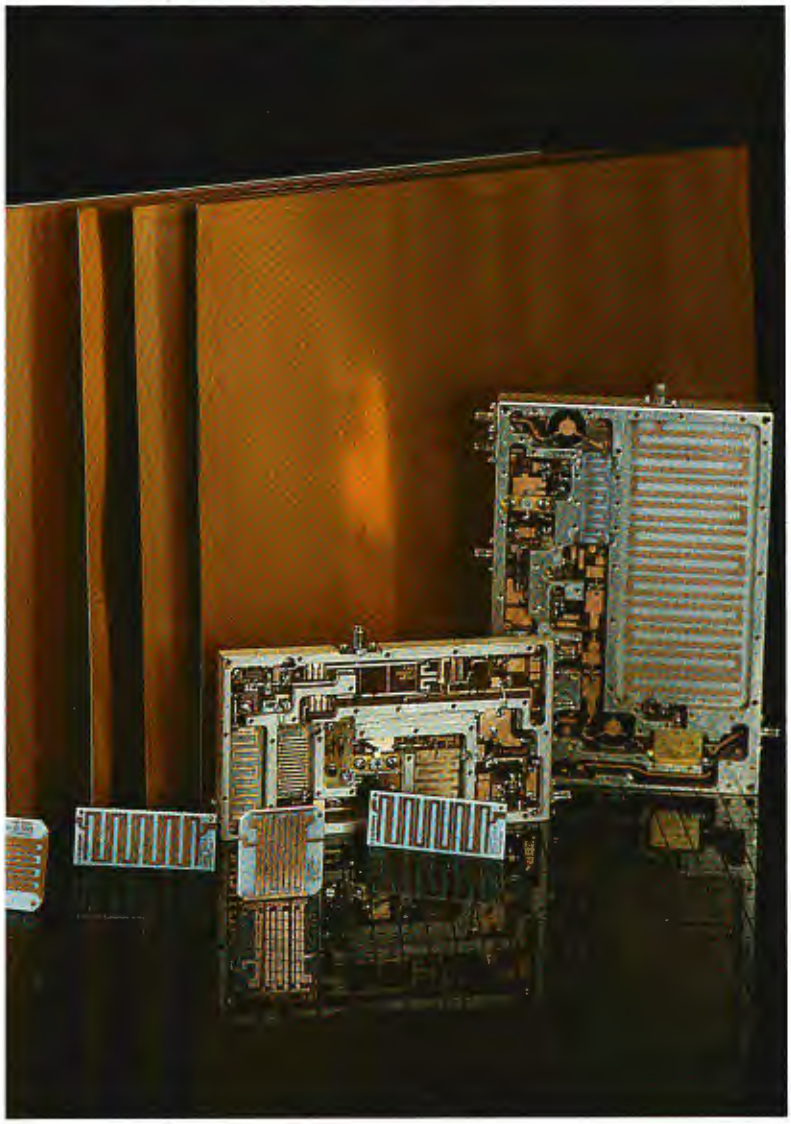
Fixed capacitors, used for 6 Kv peak pulsed applications, are available with capacitance values from 50 pF to 250 pF. For 10 Kv peak voltage applications, fixed capacitors are available with capacitance values ranging from 25 pF to 150 pF.

Polyflon also designs and manufactures variable, trimmer and fixed capacitors to customer specifications.

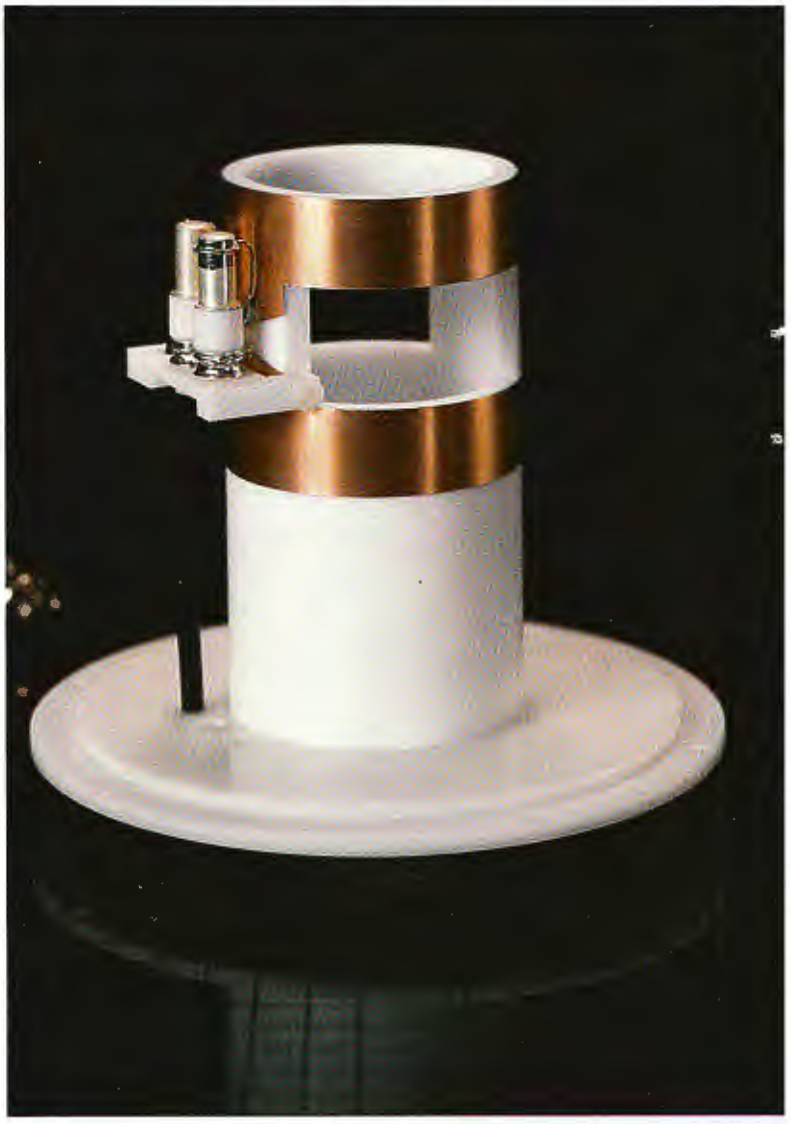
Polyflon Capacitor Advantages:

- **Non-magnetic**
- **Low dissipation factor (low loss)**
- **Extremely High Q**
- **Low ESR**
- **High EPR**
- **Linear tuning (no capacitance reversal)**
- **High voltage capability**
- **Rugged design**
- **Long operating life**

Material Technology for NMR Product Applications



Pure PTFE dielectric is used in all Polyflon CuFlon® microwave substrates, assuring low loss.



80 MHz copper plated PTFE saddle coil assembly.

Polyflon uses only pure PTFE dielectric in its CuFlon® microwave substrates which provides one of the best low loss dielectrics in the industry.

Since nothing is added to the PTFE, the dielectric constant of 2.1 remains consistent over an extremely wide range of frequencies.

The unique, proprietary electroplating technology for PTFE, developed by Polyflon, permits design and fabrication of superior RF components.

An intimate bond is produced between the metal surfaces and the PTFE, with no other element or media between them. The dissipation factor and Q is then that of the PTFE substrate itself.

Very low loss components are produced with this technology that can be used with excellent results at very high microwave frequencies.

Polyflon's CuFlon substrate materials, found in many NMR/MRI applications, are used in various coil designs such as surface, solenoid and saddle configurations. CuFlon is also used as a substrate in pulsed RF amplifiers, wide-band RF transformers, and chip capacitors for tuning and matching elements.

Polyflon's expertise in the PTFE plating technology can and does provide customers with rugged and reliable products for critical NMR/MRI applications.

Call or write for information.

CRANE®

POLYFLON

Polyflon Company • 35 River Street, New Rochelle, NY 10801
Tel: (914)636-7222 • FAX 914-633-3664

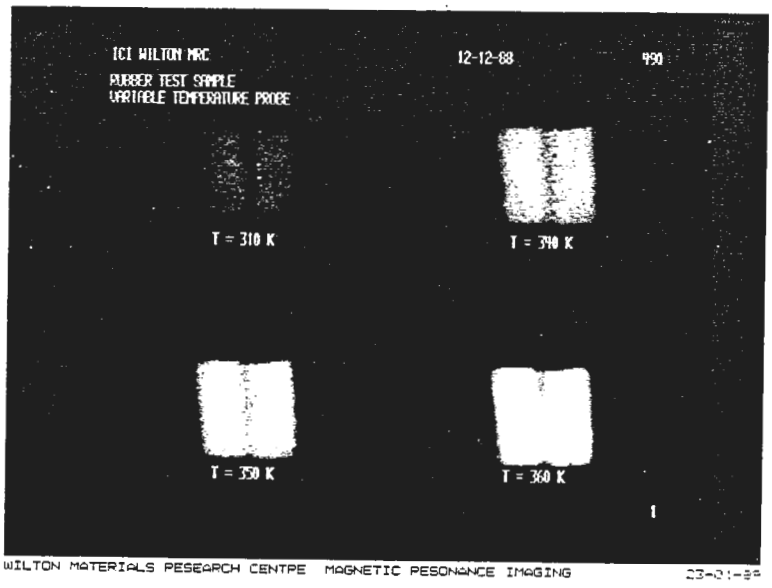
The figure shows images generated from a sample comprised of two blocks of a crosslinked rubber separated by a sheet of paper, obtained at 310, 340, 350 and 360K. The 128 x 128 images were obtained with single-transient acquisition, requiring only 64 seconds to complete. A hard 90 - hard 180 sequence was used, with an echo time of 2.2 ms. At room temperature, no signal is observed without extensive signal averaging. The benefits from high temperature imaging of such samples are apparent.

Can you please credit this contribution to A Bunn's account.

Yours sincerely

Pete

PETE JACKSON



I can't abide empty space, and there is nothing on hand which is the right size for this 'hole', so herewith a *tout* for a wee book related only indirectly to the pursuit of salvation through NMR spectroscopy. The sub-title notwithstanding, it will, I am sure, be found both entertaining and useful by those who toil outside of Academe as well as those within.

"**Microcosmographia Academica** - *Being a guide for the young academic politician*" - by **F. M. Cornford.**

Bowes & Bowes (Publishers) Ltd., 9 Bow Street, Covent Garden, London WC2E 7AL, England. ISBN 0 370 00145 1. First published in 1908, and reprinted numerous times, including in a very inexpensive paper-back edition in 1983 (and perhaps even more recently). The price at the Bowes & Bowes shop in Cambridge in 1983 was £1.95.

B.L.S.

ES-3740

DUPONT
REG U.S. PAT. & TM. OFF.
ESTABLISHED 1802
E. I. DU PONT DE NEMOURS & COMPANY
INCORPORATED
P.O. BOX 80356
WILMINGTON, DELAWARE 19880-0356

CENTRAL RESEARCH & DEVELOPMENT DEPARTMENT
EXPERIMENTAL STATION

February 13, 1989

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

Copper Spin Counting in High T_c Superconductors

Dear Barry:

Our experience with solid-state NMR applications in zeolites has made us aware of the great importance of spin counting in inhomogeneous and nonstoichiometric systems. Thus, when we began Cu NQR and NMR measurements on the high-T_c superconductor $YBa_2Cu_3O_x$ ($6<x<7$) a little over a year ago, we first wanted to find out whether we were seeing 1%, 10% or 100% of all the coppers in the sample and whether relative signal intensities were quantitatively reliable. This letter summarizes some of what we did to answer these questions.

The Cu NQR spectra consist of 1 to 5 MHz wide peaks that cover the frequency range from 20 to 31 MHz. They must be obtained point-by-point with a spin echo method. All measurements are done with a CXP300 at room temperature. We use pulses of 4 and 8 μs, giving rise to echoes that represents 0.3 MHz-wide slices out of the broad spectral distribution. Since the FT of the echoes have a universal shape that is entirely determined by the pulse amplitude and widths, measuring the echo amplitude by visual curve fitting to a standard signal is simple and straightforward. Doing this in frequency space has the added advantage that spurious signals from external transmissions (of which Wilmington has plenty around 30 MHz) are usually well distinguishable from the spin echo signal.

The real difficulty is the calibration of signal intensities over a wide frequency range. If we start with the simplifying assumption that at the top of the echo all the resonant spins point along the "x direction", then the detected signal amplitude, S, at resonance frequency, ν, is related to the number of spins, N, through the relation (Abragam, page 72) $S=N\gamma I(I+1)\nu^2 f(\nu)$, where f(ν) stands for the combined response function of the rf coil, probe, transmission line, amplifier, and detector. In this form, the equation allows comparison between signals of nuclei with any γ and at any resonance frequency. Since f(ν) is the only uncertain factor in the equation, a full characterization of its behavior completes the quantitative evaluation of our detection method. In the hope that f(ν) could be made frequency independent, we constructed a probe insert that is tunable from 18 to 40 MHz. However, above and below 30 MHz we have to use different power-amplifier inserts and preamplifier matching boxes, while for each frequency point in the spectrum we must tune the probe, the power amplifier, and preamplifier. The following quantitative signal comparisons were made to test f(ν).

1. NMR in 7.05 T of an aqueous solution of NH_4Cl at the three resonance frequencies of ^{35}Cl, ^{37}Cl, and ^{14}N. At each frequency the FID amplitude was measured following an optimized 90° pulse. The numbers of spins were proportional to the natural abundances. The normalized results were:

Hare Research, Inc.
14810 216th NE
Woodinville WA 98072
Tel: (206) 789-7559
FAX: (206) 788-2552

Barry—

Subject: Automated NOE Identification

To determine the three-dimensional structure of a biopolymer, one must first perform the tedious task of assigning the resonances in the spectrum. Following that a second time-consuming and potentially tricky process is the identification of the hundreds to thousands of NOEs in a NOESY spectrum. This step entails not only identifying as many NOE cross peaks as possible, but also determining a distance estimate (or distance range estimate) from the NOE buildup rate. This information is stored for use in calculating the molecule's structure.

During this long and tedious process it is quite possible that one misidentifies an NOE given human nature, or perhaps by forgetting the possibility that the peak could arise from a proton that was not assigned (few large molecules are completely assigned). A second error that has happened to several researchers is identifying the NOE correctly, but writing it (or the distance range) incorrectly in the input file for the structure determination program.

These problems are avoided and the process greatly facilitated if an automated NOE identification routine is used. This routine involves comparing an assignment list to a NOESY cross peak list—a simple process, though now performed much faster without the chance of transcriptional errors. Distance ranges are automatically determined (either strong/medium/weak ranges, or determined from a scaled distance estimate) and the output can be read directly by the structure calculation program (in this case DSPACE).

This is only possible, of course, when the identity of the NOE is unambiguous. When several possible identities exist for a given NOE cross peak it is useful to utilize available distance information such as an average structure obtained from several structures where the standard deviation in atomic position is also calculated. This information is used to check interatomic distances for all possible parent pairs for ambiguous NOE cross peaks. For example, a peak in a protein spectrum occurs at 8.54 ppm, 2.89 ppm); there are three amide protons assigned at 8.54 ppm and two protons assigned at 2.89 ppm, giving rise to six different pairs of parents. A check of the average structure however shows only one possible pair within 4Å (including a standard deviation or two). This peak is now identified!

But maybe not, if there is an unassigned amide proton within 4Å of either proton at 2.89 ppm. Thus feature of an automated NOE identification program should be to check for the presense of unassigned protons. In this case it is useful to write the "identified" NOE and distance range, but comment that line out. What happens now is up to the user. Maybe he or she will use this information to get some more resonance assignments, an easier process with the tools provided by intelligent automation programs.

Paul —Paul L. Weber

If you liked that idea—

VISIT OUR BOOTH AT THE ENC!

Software Demos on Personal IRIS, SUN, and PC.

Plus a sneak preview of our new NMR program, FELIX:

- Realtime phasing, expansion, and difference spectra.
- 1D Processing supported on the PC (>1 soon).
- Flexible and friendly integration and peak picking.
- Process data matrices in up to 6 dimensions.
- Linear least squares cross peak fitting.
- Semiautomatic connectivity analysis.
- Built-in NOESY backcalculation from structure data.
- Automatic NOESY cross peak identification (maybe).

We will also be glad to show you our software for NMR-based structure determination (DSPACE).

Play with our computers, have a beer, tell us what you like and don't like about our software, have another beer, or just hang out and talk science with Paul and Dennis.

See you in April!

Hare Research, Inc.
Committed to providing tools
to keep you on the forefront of science.

Nucleus	Frequency	$S/(N\gamma I(I+1)\nu^2)$
^{35}Cl	29.4 MHz	0.96
^{37}Cl	24.5 MHz	1.04
^{14}N	21.7 MHz	0.34

2. ^{35}Cl NQR in zero field of weighed samples of K_2PtCl_6 and K_2IrCl_6. These Cl compounds were identified after an extensive search in Ref. [1] and an intensive consultation with du Pont colleagues concerning the availability, safety, and purity of prospective samples. The signal amplitudes were determined from extrapolation of echo intensities to $\tau=0$ in a two-pulse sequence. Normalized results:

Sample	Frequency	T_2	$S/(N\nu^2)$
K_2PtCl_6	25.81 MHz	1.3 ms (Gaussian)	1.13
K_2IrCl_6	20.73 MHz	150 μs (expon'l)	0.88

3. NQR in zero field of ^{63}Cu and ^{65}Cu in Cu_2O. Normalized results:

Nucleus	Frequency	N	γ	$S/(N\gamma^2)$
^{63}Cu	26.0 MHz	69.1%	11.28 MHz/T	0.93
^{65}Cu	24.0 MHz	30.9%	12.09 MHz/T	1.07

4. All measurements of Cu NQR intensities at 30 MHz were routinely double checked with tuning inserts for >30MHz and for <30MHz. The data always agreed within 5%.

The agreement is not perfect, but with the exception of the puzzling ^{14}N result, we found that all intensity measurements indicate that $f(\nu)$ is a constant, within ±15%.

The remaining question is that of uniform excitation by the echo sequence. There are theoretical worries related to the orientation depencence of NQR pulse flip angles and the resulting small differences that arise in the powder summations for different asymmetry parameters. On the other hand, we found empirically that the rf power could be varied over quite a wide range without producing an appreciable change in NQR echo amplitudes. Moreover, rf power adjustment for maximum echo intensities at different Cu NQR frequencies consistently ended up at the same transmitter-gain setting. This gave us confidence that there are no serious problems with the excitation aspect of the NQR spin counting. More or less encouraged by this evaluation, we finally dared to draw some cautious quantitative conclusions from our NQR results of '123'.

Please credit this to Dr. Don Bly's account.

Sincerely,

Alex

Alexander J. Vega

[1] G. K. Semin, T. A. Babushkina, and G. G. Yakobson, "Nuclear Quadrupole Resonance in Chemistry", transl. from Russian by A. Barouch, Keter, Jerusalem, 1975.

For Sale

A Varian XL-200 with a V-77 Data System Purchased new in 1983
Includes Console, Hard Disk Drive, and Dewar
5mm $^{13}C/^{1}H$ switchable probe and 10 mm $^{13}C/H$ probe
Make us an offer we cannot refuse
Michael J. Minch
Department of Chemistry UOP
Stockton CA 95211
(209)946-2442

CALIFORNIA INSTITUTE OF TECHNOLOGY

Manager of NMR Facilities, California Institute of Technology. The California Institute of Technology seeks an individual, preferably at the Ph.D. level, to oversee an NMR facility consisting of a range of NMR spectrometers. The ideal candidate will have a broad-based knowledge of NMR hardware and maintenance, the design and implementation of modern pulse sequences and related techniques, as well as versatility with associated computer software. Please send resume and salary history to Dr. John Sibert, Division of Chemistry and Chemical Engineering, 164-30, California Institute of Technology, Pasadena, CA 91125. Applications from minority and women candidates are encouraged; the California Institute of Technology is an equal opportunity employer.

TAMU NMR Newsletter

Policies and Practical Considerations

(Revised February 1989)

The TAMU NMR Newsletter (formerly the IIT NMR Newsletter, and originally the Mellon Institute NMR Newsletter) continues with the same name, under the aegis of Texas A&M University, although the undersigned Editor/Publisher now resides in California. The Newsletter, now in its thirty-first year of consecutive monthly publication, continues under the same general policies as in the past. All communication with the Newsletter must be directed to the address overleaf.

1. **Policy**:

The **TAMU NMR Newsletter** is a means for the rapid exchange of information among active workers in the field of NMR spectroscopy, as defined broadly, including imaging. As such, the Newsletter will serve its purpose best if the participants impart whatever they feel will be of interest to their colleagues, and inquire about whatever matters concern them.

Since the subscriber/participant clearly is the best judge of what he or she considers interesting, our first statement of policy is "We print anything." (This usually is followed by the mental reservation, "that won't land us in jail.") Virtually no editorial functions are performed, although on rare occasions there is the need to classify a contribution as 'not for credit'. I trust that the reasons for this policy are obvious.

The **TAMU NMR Newsletter** is not, and will not become, a journal. We merely reproduce and disseminate exactly what is sent in. Foreign participants should not feel obliged to render their contributions in English.

2. **Public Quotation and Referencing:**

Public quotation of Newsletter contents in print or in a formal talk at a meeting, etc., is expressly forbidden (except as follows), and reference to the TAMU NMR Newsletter by name in the scientific literature is never permissible. *In order to quote results or use material from the Newsletter, it is necessary, in each individual case, to obtain the prior permission of the author in question and then to refer to the material quoted as a "Private Communication".* If your copy of the Newsletter is shared with other readers, it is your obligation as the actual recipient of the Newsletter to see that these other readers of your copy are acquainted with, and abide by, these statements of policy.

3. **Participation is the prime requisite for receiving the TAMU NMR Newsletter:**
In order to receive the Newsletter, you must make at least occasional technical contributions to its contents.

We feel that we have to be quite rigorous in this regard, and the following schedule is in effect: Eight months after your last technical contribution you will receive a "Reminder" notice. If no technical contribution is then forthcoming, ten months after your previous contribution you will receive an "Ultimatum" notice, and then the next issue will be your last, absent a technical contribution. Subscription fees are not refunded in such cases. If you are dropped from the mailing list, you can be reinstated by submitting a contribution, and you will receive back issues (as available) and forthcoming issues at the rate of nine per contribution.

Frequent contributions are encouraged, but no "advance credit" can be obtained for them. In cases of joint authorship, either contributor, but not both, may be credited. Please indicate to whose account credit should be given. Please note that meeting announcements, as well as "Position Available," "Equipment Wanted" (or "For Sale"), etc., notices are very welcome, but only on a not-for-credit basis, *i.e.,* such items do not substitute for a *bona fide* technical contribution. Similar considerations must occasionally be applied to a few (quasi-)technical items.

4. **Finances**:

The Newsletter is wholly self-supporting, and depends for its funds on advertising, donations, and individual subscriptions.

The **Subscription** fee is currently US$120.00 per year, with a 50% academic or personal discount. Subscriptions are available only for the twelve monthly issues which begin with the October issue and run through that of the following September. However, a subscription can be initiated at any time, and the issues back to the previous October will be provided as long as copies remain available.

Companies and other organizations are also invited to consider joining the list of **Sponsors** of the Newsletter. Sponsors' names appear in each month's Newsletter, and copies of the Newsletter are provided to all Sponsors. The continuation of

Continued

this non-commerical Newsletter depends significantly on the interest and generosity of our Sponsors, most of whom have been loyal supporters of this publication for many years. We will be happy to provide further details to anyone interested.

Another major, indeed most essential, source of funds for the Newsletter is **Advertising**. We earnestly encourage present and potential participants of the Newsletter to seek advertising from their companies. Our rates are very modest - please inquire for details.

5. **Practical Considerations**:

a) All technical contributions to the TAMU NMR Newsletter will always be included in the next issue if received before the published deadline dates.

b) Please provide short titles of all topics of your contributions, so as to ensure accuracy in the table of contents.

c) Contributions should be on the *minimum* (NOTE!!) number of 8.5 x 11" (21 x 27.5 cm) pages, printed on one side only. Contributions may not exceed three pages without prior approval. Each page must have margins of at least 0.5 - 0.75" (1.3 - 2.0 cm) on all sides. Please observe these limits. Black ink for typing, drawings, etc., is essential. All drawings, figures, etc., should be mounted in place on the 8.5 x 11" pages. We are not equipped to handle pieces of paper larger than 8.5 x 11" (21 x 27.5 cm).

Foreign subscribers are reminded that regardless of the standard paper length you use, *all material* - letterhead, text, figures, addresses printed at the page bottom, *everything - must not exceed 10" (ca. 25.3 cm) from top to bottom.*

Significant savings of Newsletter pages and total space can be made by exercising close control over the formatting and type sizes of the contributions. **Please consider the following:**

i) For those with computers, try using a smaller type font. The body of this page is printed in 10 point type, which I believe is adequate for most purposes. Even 12 point is acceptable, I suppose. Those who are computerized can also employ non-integral spacing of lines so that sub- and superscripts don't collide with lines below and above.

ii) **PLEASE** avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard'!* This page has margins on both sides of 0.6" (*ca.* 1.55 cm), which is very adequate. Margins of the same size at the top and bottom are sufficient also, but don't worry if there is more space at the end of your document, for I can often use such spaces for notices, etc.

Also, please avoid large amounts of unused space at the top of letters. Give thought to the sizes of figures, drawings, etc., and please mount these so as to use the minimum space on the page.

iii) 'Position Available', 'Equipment Wanted', and Similar Notices. These are always welcome, without charge, but not for subscription credit, of course. Such notices will appear, however, *only* if received with these necessarily rigid constraints: a) Single spaced; b) both side margins 0.6 - 0.7" (1.5 - 1.7 cm.)- NOT WIDER; c) the minimum total height, please, but definitely no more than 4.5" (11.5 cm.) This will let me place such notices wherever a bit of space occurs.

iv) **AVOID DOUBLE SPACING LIKE THE BLACK PLAGUE ! ! !** This is extremely wasteful of space. Even sans computer, small type and 1.5-line (if needed) spacing can be had with a little effort.

6. **Suggestions**: They are always welcome.

B. L. Shapiro

Address for all correspondence:

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

Telephone: (415) 493-5971.

16 February 1989

TAMU NMR Newsletter

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. 368 (May)——————21 April 1989
No. 369 (June)——————19 May 1989
No. 370 (July)——————16 June 1989
No. 371 (August)——————21 July 1989

To those who have been so helpful in conserving space by the careful formatting of their technical contributions, using small type fonts, avoiding the sin of double-spacing lines, etc., many thanks. To those who have yet to help me save pages (*i.e.*, expense), please join the ranks of the righteous.

Again I ask that 'Position Available', 'Equipment Wanted', and similar notices be constructed so as to use the ***minimum vertical space***. Avoiding large margins helps greatly: 1/2 - 3/4" on each side is quite sufficient. DO NOT DOUBLE SPACE THESE NOTICES!! - in future I'll send badly done notices back for re-working. I am most happy to have such notices appear in the Newsletter, without charge, for Subcribers, Sponsors, Advertisers, or indeed anyone else, but your cooperation in utilizing space efficiently will truly be appreciated.

It is heartening to note the increase in the number of **Advertising** pages in recent issues of the Newsletter. Some of this increase is connected with the imminent occurence of the ENC, but a more general development of the advertising component is clear. This is of obvious benefit in keeping the Subscription fee down to a tolerable level. Please assist in this process by making sure that our loyal Advertisers know that you have seen their ads in the Newsletter and appreciate their support. Despite the increased amount of advertising, still more is needed, as are additional **Sponsors**. Please do what you can to help . . . .

Let me again acknowledge with thanks the clever and artistic efforts of **Wendy Goldberg** and **Uwe Oehler** for their stodge-reducing contributions to the Newsletter pages. My inventory of these NMR-related cartoons is getting very low, but I still hope that others will also step forward to provide some pictorial rays of sunshine and mirth.

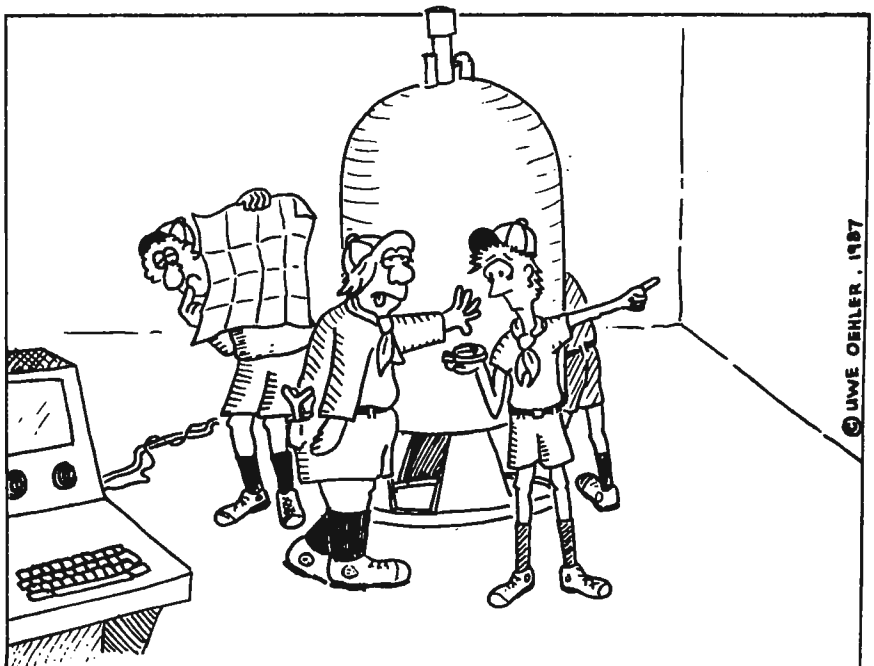
B.L.S.

TEXAS A&M UNIVERSITY
NMR NEWSLETTER

No. 366
March 1989

Table of Contents, cont'd.

* * * * *



ONE MORNING, HAROLD ARRIVES AT WORK TO FIND FOUR LOST CUB SCOUTS FROM REMEDIAL ORIENTEERING.

CSI 2T Applications

Analytical techniques: localized spectroscopy

Nuclear magnetic resonance (NMR) imaging has proven to be a valuable analytical technique. But the possibility of combining imaging with NMR spectroscopy has always been a desired goal. Recently developed localization techniques now make it possible to collect NMR spectra from a region of interest determined by an NMR image.[1-5]

Seven vials, each containing a different material, were imaged (Fig. 1). Using the stimulated echo technique,[5] 5 mm cubes (125 ul) were selected, and proton NMR spectra were collected. Although this method may limit the type of RF pulse sequence used, many experiments can be performed within a specific volume, including solvent suppression (Spectrum d) and relaxation measurements (Spectrum a). Such techniques may have applications for nondestructive testing and analysis of materials.

References:

[1]Bottomley, PA; Foster, TB; Darrow, RD: *J. Magn. Reson.*, 1984, 59:338.

[2]Aue, WP; Mueller, S; Cross, TA; Sellig, J: *J. Magn. Reson.*, 1984, 56:350.

[3]Ordidge, RJ; Connelly, A; Lohman, JAB. *J. Magn. Reson.*, 1986, 66:283.

[4]Luyten, PR; Marien, AJH; Sijtsma, B; den Hollander, JA. *J. Magn. Reson.*, 1986, 67:148.

[5]Frahm, J; Merboldt, KD; Hanicke, W. Works in Progress, 5th annual meeting of the Society of Magnetic Resonance in Medicine. Montreal, August, 1986, p. 158.

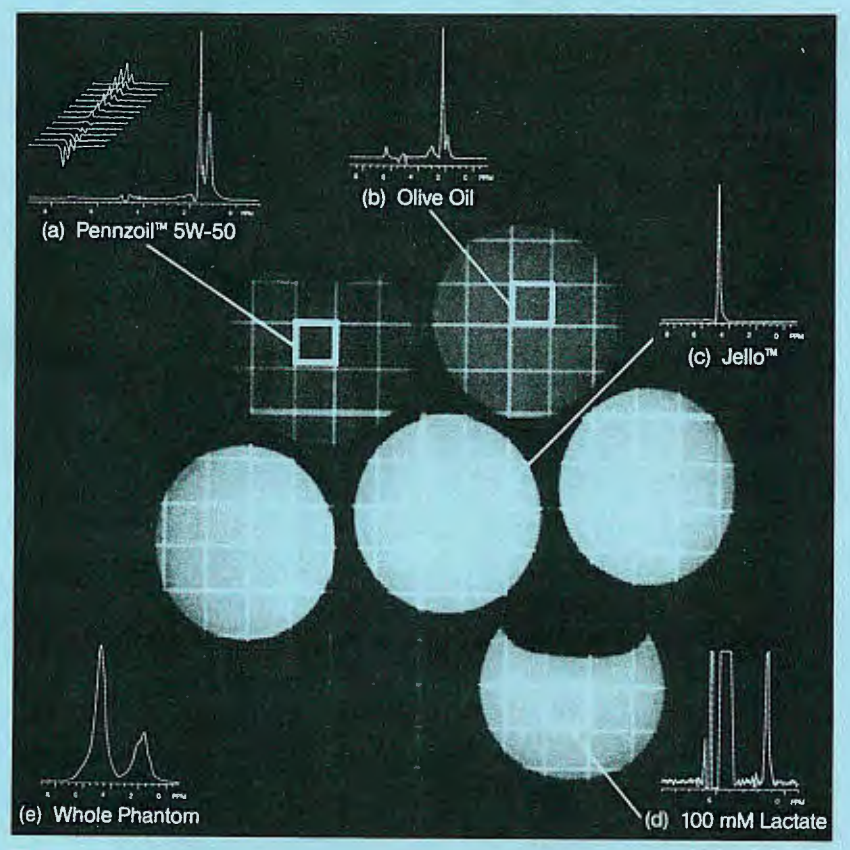


Fig. 1

GE NMR Instruments

255 FOURIER AVENUE, FREMONT, CA 94539
(415) 683-4408, FAX (415) 490-6586

PRAUNHEIMER LANDSTRASSE 50, D-6 FRANKFURT 90
WEST GERMANY 4969 760 7431, TELEX 041 2002 GEG

7131A Printed in U.S.A.

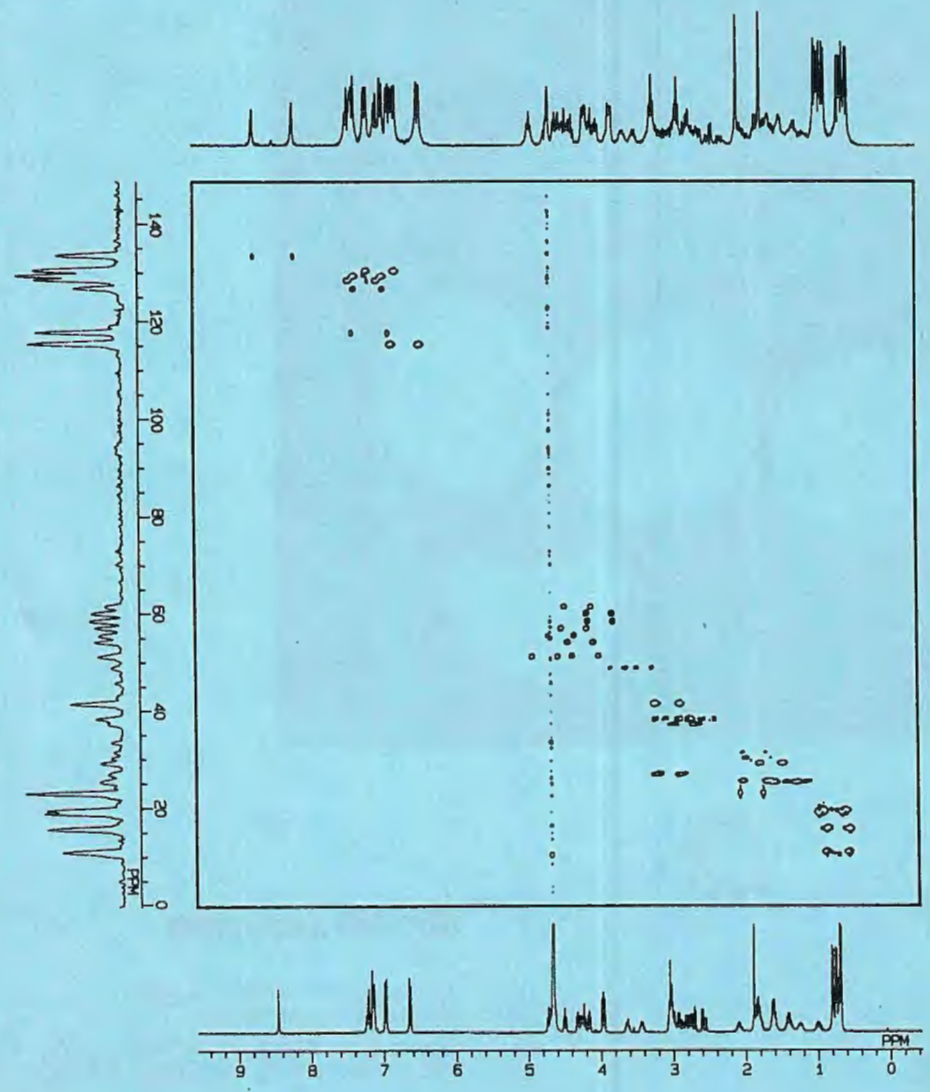
JEOL GSX-FT NMR Systems

Subject: Rf STABILITY

All of the automation, elegant experiments, and high speed computer processing will do nothing for an NMR experiment if the spectrometer is not stable. The Rf section of the spectrometer must be reproducible and clean of spurious signals over periods of days for some experiments.

One of the most demanding experiments for spectrometer stability is the reverse detection (H [C]) experiment without C decoupling. Between the relatively sharp lines and the low magnitude of the satellites, this experiment graphically demonstrates the stability of the spectrometer. As the data below shows, a standard JEOL GSX Spectrometer has the Rf stability to do these experiments.

ANGIOTENSIN-II, 3mg.in.D20



*Sample courtesy of Dr. Jeffrey Hoch (Rowland Institute For Science, Inc.)

For further information call:

JEOL
Serving Advanced Technology
11 Dearborn Road, Peabody, MA 01960
(617) 535-5900