TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 378

March 1990

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Iso. (% D)	Density (g/cc)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d4	CH_3COOD	99.5%	1.12	17	118	0.551 (32)
D-120	Acetic Acid-d4 + TMS		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%				

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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.	Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	HA-100, HA-100D, 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" X 17"	CFT-20, FT-80, FT-80A All Models use 16 1/2" or 16	Gridded-Two Color 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.

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Continued on page 59

January 26, 1990
(received 2/2/90)

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

Imaging and Diffusion

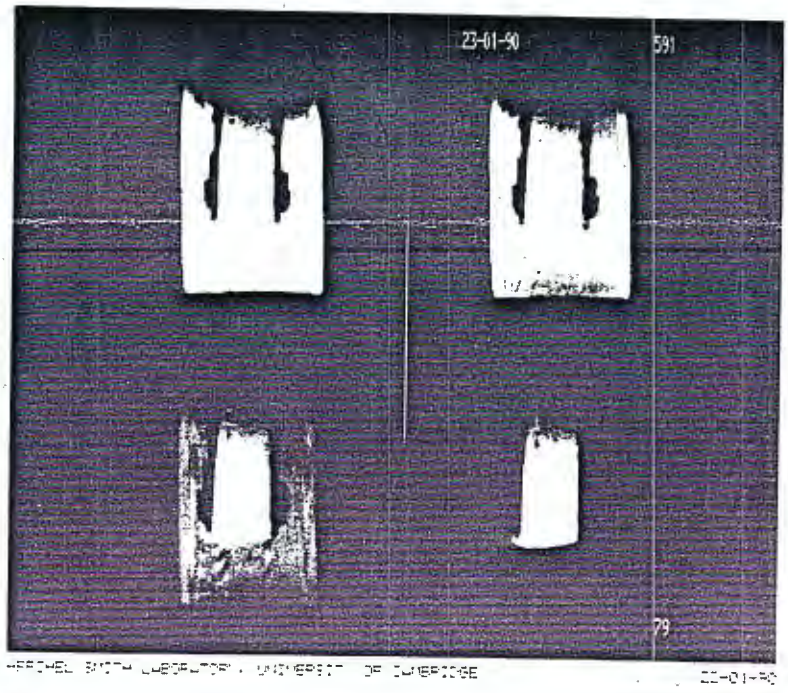
Dear Barry,

For the academic year 1989-90 I am on sabbatical enjoying the wonderful hospitality and instrumental facilities of Laurie Hall in Cambridge. Your reminder letter arrived very promptly even with a detour via Ottawa. Please credit this contribution to my NRC account even though the work is being done in Cambridge.

The attached figure shows four images of a 50 ml. beaker containing distilled water into which is inserted a glass tube closed with 'Saran' wrap and containing a 10 millimolar CuSO4 solution. As the delay between succesive scans is reduced from 4.0 sec. to 0.25 sec. the pure water signal saturates and disappears. The signal from the CuSO4 solution remains strong since T1 for this solution is short. We are using this system with various semi-permeable membranes to study a variety of diffusion processes.

Best wishes,

S. Brownstein



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NMR WITH A FUTURE

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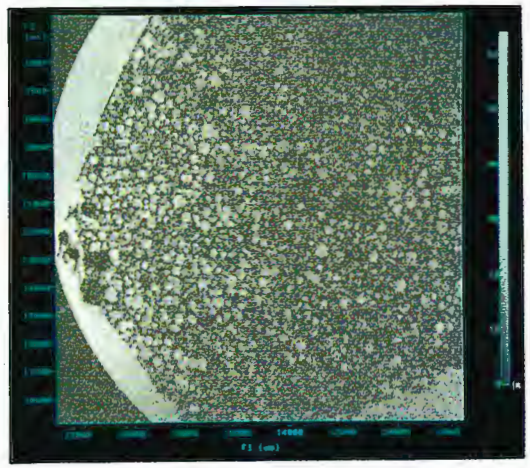
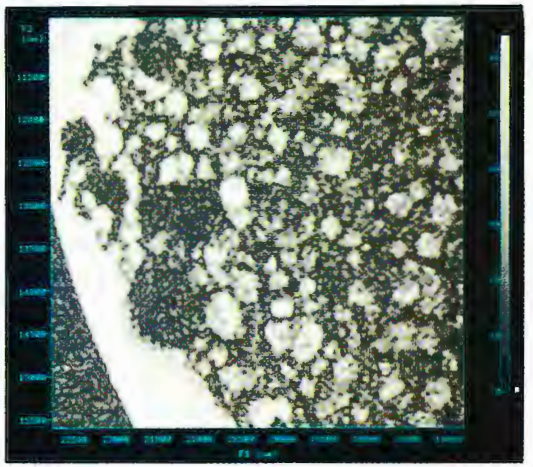
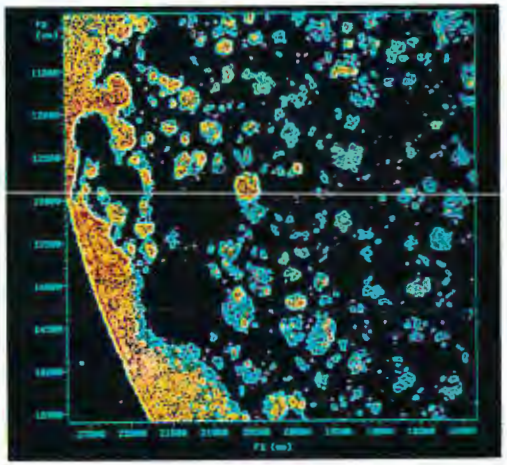
PVA 2

Run on a UNITY-300 Spectrometer, this PVA process-based sponge (courtesy of Prof. Dr. Gelan, Limburgs Universitair Centrum) demonstrates poor water absorption characteristics in practice. The sponge structure is cellular in nature and similar in dimensions to PVA 1 (see TAMU newsletter, December 1989).

The images below were obtained with a T_e of 0.1 sec. The images taken at a T_e 0.03 sec and a T_e of 0.1 sec show a marked contrast difference within the sponge, indicating limited water mobility.







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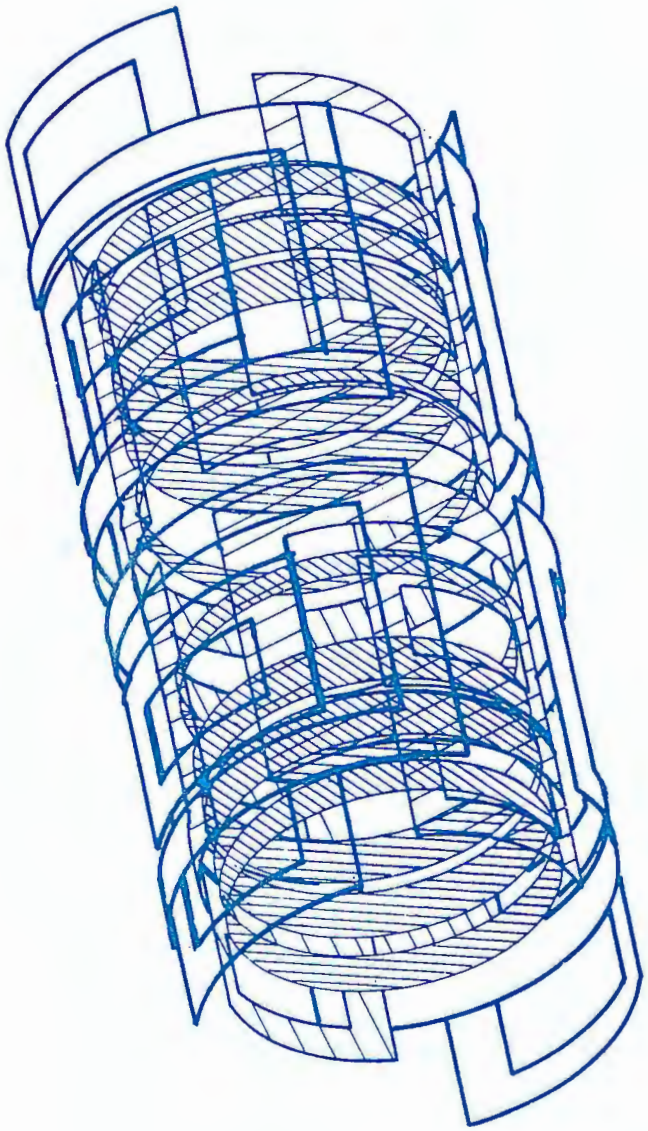

LOW TEMPERATURE

- MAS at 1 kHz below 8 K.
- High resolution below 90 K.

HIGH TEMPERATURE

- 7mm, 4 kHz MAS above 650° C.
- High resolution to 380° C.

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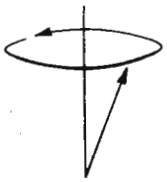
- **1987** First **CRAMPS** probe
- **1987** First **actively shielded** gradient probe
- **1987** First **5 mm 17 kHz** spinning
- **1987** First **1000 G/cm pulse** field gradient
- **1987** First **7 mm 9 kHz** spinning
- **1988** First **6 Kelvin** MAS
- **1988** First **650°C** MAS
- **1989** First **DAS** probe
- **1990** First **?**.



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January 27, 1990.
(received 2/6/90)

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

Correlation of ^{13}C methoxyl SCS values and aromatic ring P_{π}-bond orders.

Dear Professor Shapiro:

It is well documented that in aromatic compounds having two fused rings, the SCS values induced by a methoxy group at the two ortho positions have very different magnitudes. However the average of these two values is always close to the value found in anisole ($\sim$ -14.5 ppm). This behavior should be related to the electron distribution at the aromatic ring bearing the methoxy group.

With this in mind, we gathered the P_{π} values of naphthalene, quinoline, coumarin, indole and benzo[b]furan from the literature, while for flavone we used those of chromene. We also gathered the SCS values for 1- and 2-methoxynaphthalene, 8-methoxyquinoline, 6- and 7-methoxycoumarin, 5-, 6-, 7- and 8-methoxyflavone, 4-, 5-, 6- and 7-methoxyindole and 5- and 6-methoxybenzo[b]furan. In addition we remeasured the spectra of 5-, 6- and 7-methoxyquinoline and of 5- and 8-methoxycoumarin.

When the 40 pairs of values (P_{π} and ^{13}C SCS) of the 20 above mentioned compounds are treated by a least-squares procedure, the equation:

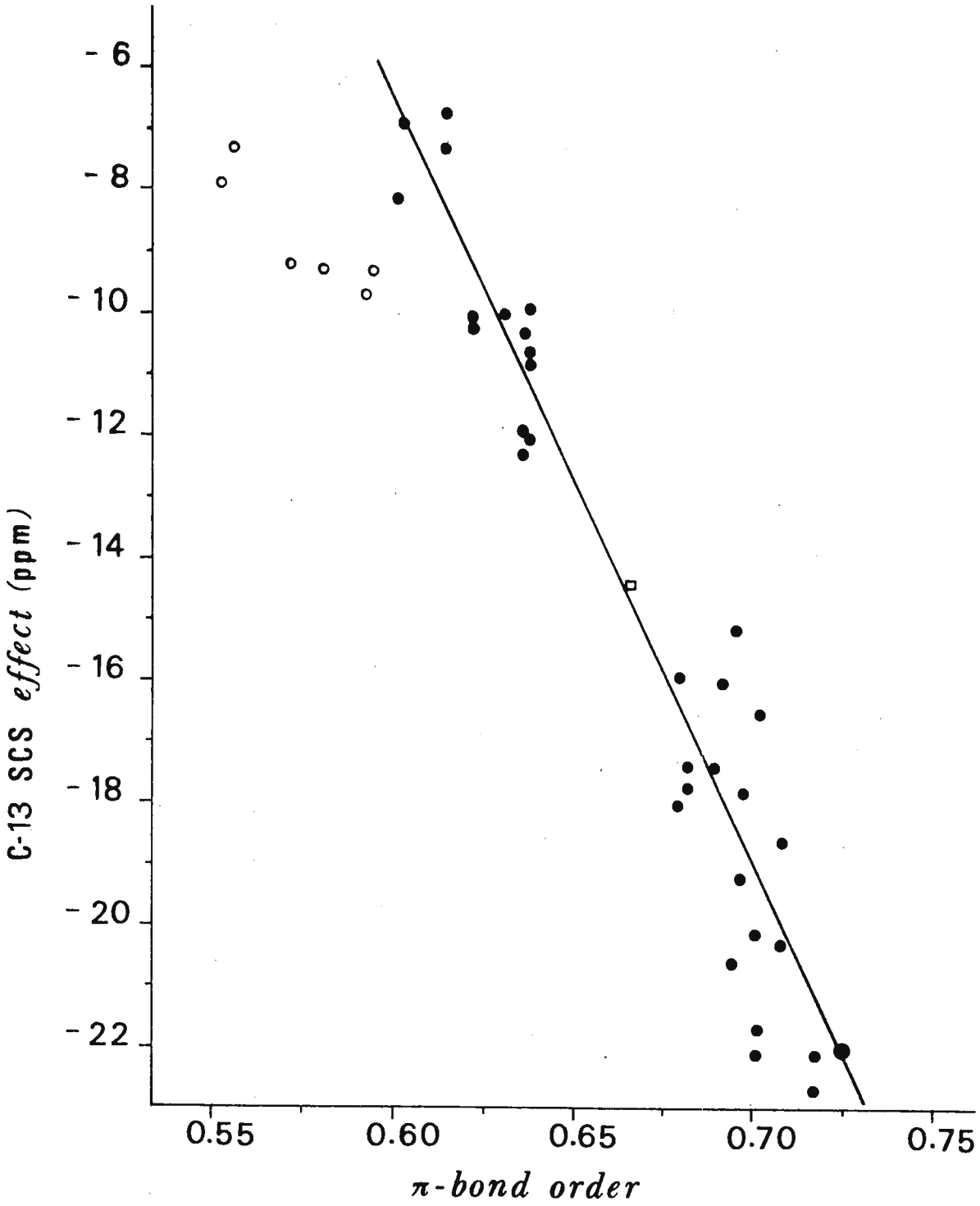
$$\Delta\delta_{ortho}\,(^{13}C) = -98.68\ P_{\pi} + 50.38$$

is obtained (correlation coefficient: 0.929; root mean square error: 1.95 ppm). An inspection of the data shows that C-4a in 5-methoxyquinoline, 5-methoxycoumarin and 5-methoxyflavone, C-8a in 1-methoxynaphthalene, C-3a in 4-methoxyindole and C-7a in 7-methoxyindole deviate notably from the straight line. These six points belong to ring junction quaternary carbons and are represented by light dots in the graph.

When these six points are discarted, the least-squares treatment affords the equation:

$$\Delta\delta_{ortho}\,(^{13}C) = -126.85\ P_{\pi} + 69.66$$

(correlation coefficient: 0.961; root mean square error: 1.4 ppm), whose plot is represented in the graph. There, the heavy dot is the superposition of two points and the square, which was not used in the calculation, corresponds to anisole.



Sincerely Yours,

P. Joseph-Nathan C. García-Martínez M.S. Morales-Ríos

*csm

Preliminary Announcement

Industrial Applications of Magnetic Resonance Imaging

A One-Day Tutorial and Workshop Devoted to Nonmedical and Industrial Applications of Nuclear Magnetic Resonance Imaging

Massachusetts General Hospital
MGH NMR Center
Charlestown (Boston), Massachusetts

Date: Friday, April 20, 1990
(Week prior to the Boston American Chemical Society Meeting)

Nuclear Magnetic Resonance Imaging (NMRI or MRI) is a mature and well-proven noninvasive technique which has brought a revolution in medical diagnostic radiology. Quite recently, there has been a surge in the development of nonmedical applications of MRI covering a multitude of areas ranging from materials science to chemistry, physics, engineering, fluid flow and botany. Materials that have been studied include ceramics, polymers, elastomers, composites, geological materials, oil cores, wood, porous media, soils, biomaterials and medical prostheses.

The purpose of this tutorial and workshop is to acquaint industrial and academic scientists and engineers with the principles and practice of magnetic resonance imaging, especially as applied to potential problems in industrial R & D, quality control and nondestructive testing. The presentation will consist of lectures by several internationally known researchers, and will include a tour of the MGH NMR Center. This workshop should provide an excellent forum for the discussion of novel ideas for adapting MRI technology in many areas of interest to industrial scientists and engineers.

For more information, contact: Jerome L. Ackerman, Director of NMR Spectroscopy
MGH NMR Center
149 13th Street
Charlestown, MA 02129

Phone: 617-726-3083
FAX: 617-726-5819
Internet: jerry@mgh-rnmr.harvard.edu

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DEPARTMENT OF CHEMISTRY

12 January 1990
(received 2/3/90)

Barry L. Shapiro
TAMU Newsletter
960 Elsinore Court
Palo Alto, California
94303

Dear Barry:

This is a brief update on some of the recent activity here.

Assignment and Structural Analysis of a DNA Duplex Containing an Aldehydic Abasic Site: A DNA 11-mer containing a single abasic site has been assigned. The assignment of this sample was somewhat challenging since there are two anomeric forms of the abasic site present and thus the assignment was equivalent to having two nearly identical 11-mers in the same NMR tube. The DNA has a B-like conformation, like distortion of the phosphate backbone (unlike samples with analogs of the abasic site) and quite surprisingly the imino proton exchange rates for the DNA containing the abasic site are apparently slower than the equivalent DNA with a normal base pair. We are now moving on to other sequences and a more detailed analysis of the DNA conformation.

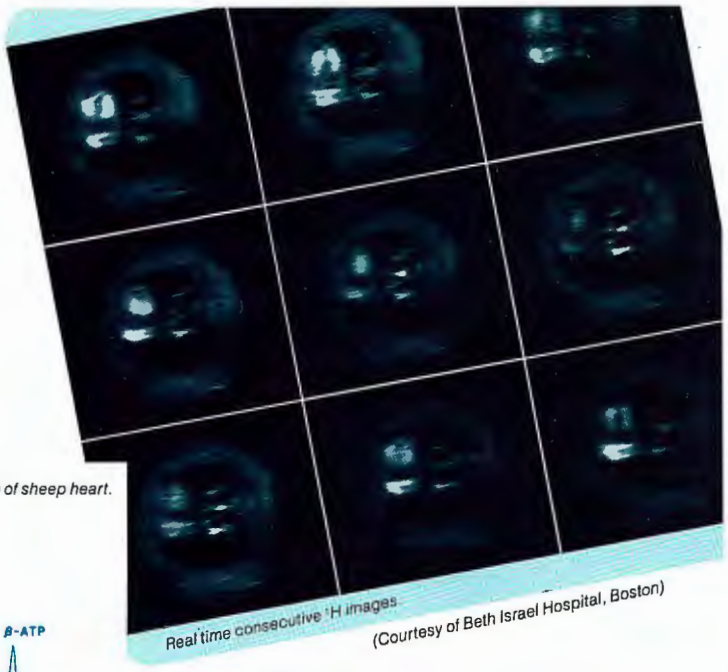
Effects of Single and Double Mutations and Deletions on the Structure and Activity of Staphylococcal Nuclease: An additional set of active site single and double mutants have been examined. It has been found that some double mutants have additive effects and some do not and the results so far can be rationalized in terms of known interactions between the proteins and substrates. A six residue deletion mutant, from the active site, of the protein has been found which is active and the conformation is now being characterized. We have also been fiddling around with cryogenic solvents for SNase but it is not clear that high quality NMR data can be obtained at -20 in 40% methanol.

NMR Relaxation Theory: There is an attempt to calculate NMR relaxation data directly from molecular dynamics trajectories more as a way to check the reliability of the trajectories than the NMR. There are some new wrinkles to relaxation in this, however.

Sincerely,

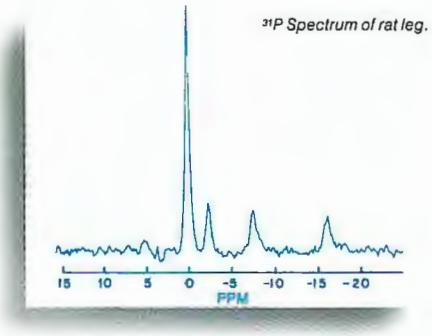
Philip H. Bolton.

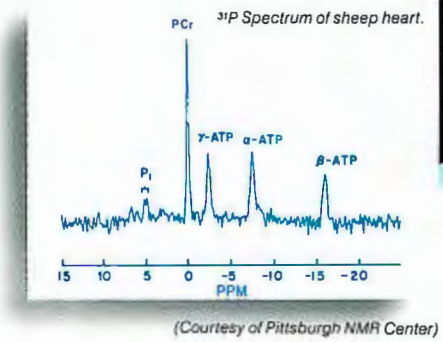
Real Answers.



Real time consecutive ^{1}H images

(Courtesy of Beth Israel Hospital, Boston)





(Courtesy of Pittsburgh NMR Center)

S/N performance.

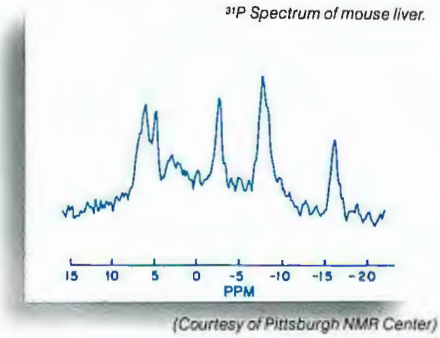
Unsurpassed performance in sensitivity. ^{31}P NMR spectrum obtained in a single acquisition at 81 MHz from muscle in rat leg. (1 msec adiabatic pulse) using a circular surface coil of diameter 28mm. The S/N on PCr is 60:1.

Large bore.

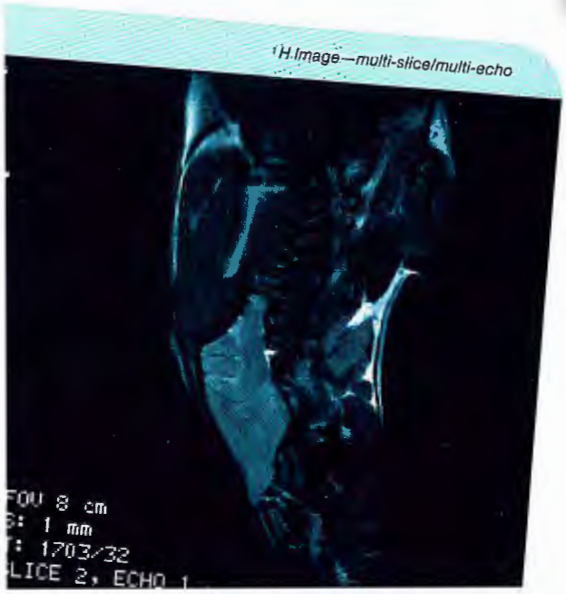
Wide access for large animal studies. ^{31}P NMR spectrum obtained at 81 MHz from heart muscle in a live sheep (64 x 90° pulses at 2 sec. intervals) using a circular surface coil of diameter 35mm.

Multi-slice/Multi-echo.

High quality image detail. An MSME image (256 x 256) taken of a rat head (TE = 33 msec, TR = 1700 msec). The slice thickness is 1mm and FOV is 8 cm. Details of the cerebellum, pharynx and the mystacial pads can be clearly discerned.



(Courtesy of Pittsburgh NMR Center)



In-vivo 7.0 tesla.

Horizontal-axis magnet systems at 7.0T. ^{31}P NMR spectrum obtained at 121 MHz from mouse liver (128 x 90° pulses at 5 sec. intervals) using a circular surface coil of diameter 10mm.

Real time.

A series of consecutive real time images can be obtained in a single heartbeat using a fast gradient echo technique with phase reversal. Each image can be obtained in only *58 msecs.* The in-vivo images shown above illustrate the diastole-systole-diastole cardiac cycle in a mouse.

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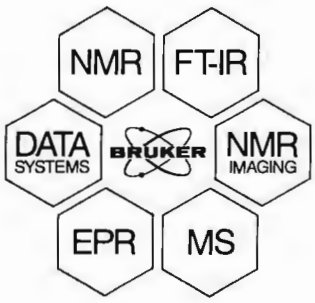

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Come Visit Us at the ENC

April 1-5, 1990.

Willows Living Room,
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See our software in action and "test drive" it for yourself.

Felix / PC

NMR Data Processing Software

FMR and Hare Research announce an inexpensive alternative for NMR data processing using IBM compatible PCs. This alternative is available as either a software package for 1D or Multi-D NMR data processing utilizing Hare Research's Felix/PC(tm) software or as turnkey data stations configured and ready to process NMR data. This software features a mouse oriented, menu based user interface. The menu software greatly increases the "user-friendliness" of the processing software for new and occasional users. The command line interface remains in place at all times for the experienced user.

Felix/PC software features:

- "Real-time" on screen phasing with mouse .
- Automatic baseline selection and corrections.
- Automatic peak-picking of 1D and 2D data.
- Plotting to HPGL or Postscript devices.
- Efficient Matrix oriented multi-D processing.
- Contour and "Image" 2D displays.

The NMR user can use her current PC and printers/plotters with Felix and be processing and plotting NMR data for $1000. Alternatively, she can purchase one of the available turnkey systems with the desired software and accessories and begin processing and plotting data immediately for as little as $6000.

ENC Special:

Between now and the end of April, file translation programs for Bruker Aspect and Nicolet/GE 1280 files will be included with each Felix/PC purchase at no additional cost.

PLOT

NMR Plotting Software

An NMR facility's efficiency and sample throughput can be substantially increased by transferring plotting operations to a separate data station, freeing spectrometers for data acquisition. FMR provides plotting software for a PC which performs the usual functions of FT, expansion, peak-picking, integration and plotting of 1D data from GE and Bruker spectrometers. Mouse-driven, pop-up menus and macros to perform command strings allow even the first-time user to manipulate data readily. PLOT imports GN, QE and Bruker files using Kermit or Xmodem at up to 19200 baud. Data can be plotted on HPGL plotters, PCL laser printers or dot matrix printers. This is an inexpensive means of optimizing the use of existing hardware.

At this time, preliminary versions of PLOT are available at the reduced prices. The finished PLOT 1.0 will have 80 MB of virtual memory (the ultimate limit is disk space) and will be priced at $895. Purchase a pre-release copy now and help us catch the bugs. For this effort you will receive a free update to version 1.0 when it is available, and save $395.

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Probe Coil Leads

Instrumentation Note 17

One very critical part of a probe is the probe coil itself. Great care is taken to design probe coils for the best RF homogeneity and highest Q as well as many other important parameters. In this design process the leads between the active probe coil and the probe's tuning and matching capacitors are often overlooked. These leads do however play a very important role in the overall probe performance.

The first area of consideration is the length of the probe coil's leads. The longer the leads, the farther removed the capacitors and other probe components can be from the active area of interest. This is good because these components have a magnetic susceptibility which affects probe resolution, lineshape and shimability. It is bad because the leads add inductance to the total probe coil inductance AND are not surrounded by sample. In effect this condition reduces the probe coil's filling factor. The maximum sensitivity would occur when all of the sample coil inductance was filled with sample. In probe design, the probe sensitivity can be improved when the ratio of active coil inductance to coil lead inductance is increased. This is one reason why multiple turn coils give better results than single turn coils. This holds true whether the multiple turns are in parallel or series.

One way to minimize the effect of the probe coil's leads is to get as much of the tuning capacitance near the active part of the coil as possible. Since the circulating current in a probe is proportional to the Q and impedance level of the coil, having most of the capacitance at the bottom of the coil and before the leads would reduce the ability of the leads to pick up signal. It also however, reduces the tuning range from the variable capacitor at the end of the leads. This range is not only needed for tuning the probe to other nuclei but for samples of different ionic strength with the same nucleus.

Where the leads are located relative to the sample is also very important. If the probe coil's leads are run up the sample insert, then they will be near the sample in areas removed from the main active region of the probe coil. These removed areas have at least two major problems. The first is that they do not have good B_1 homogeneity. Possible consequences of poor B_1 homogeneity are poor inversion and poor water saturation. The second major problem area is that the sample near the leads experiences a different magnetic field value, resulting in poor spectral lineshape (hump). This phenomenon gives rise to a strange set of conditions with unexpected results. If the magnetic field were perfect in all areas where there is sample and coil inductance, then the lineshape would be perfect. If the magnetic field is perfect in the active area of the coil but of an extremely different value (*i.e.*, would give rise to an NMR signal outside the spectral window) in the area of the leads the lineshape would be okay. This means that often a very good magnet or a very poor magnet can give good lineshape results, but an in between magnet would give poor results.

To get around the "lead pickup" problem, the coil's leads either must not pick up sample signal or must be shielded from the sample signal. This can be done in many ways:

- Geometrically position the leads to minimize differential signal pickup by each lead. This works because if both leads see the same signal there is no induced voltage across the coil's output and therefore no signal from the leads.
- Shield the leads with a band of copper or another material (plating) in the area of the leads.
- Arrange a virtual ground area of the coil to shield the leads.
- Get as much of the coil's tune capacitance as close to the active coil as possible. This lowers the current in the leads and thereby the lead pickup.

One way to test your current ^1H probe's lead response is to take a very small drop of water in the bottom of a 5mm tube and place it in the center of the probe coil. Run a spectrum using a very wide sweep width, process and integrate the spectrum. Set the integral to a value such as 100. Now repeat the process changing the sample depth in 2 mm increments using the same normalization and processing constants. Move in both directions until the signal goes to zero. Be certain to go far enough since the signal often goes to zero and returns as the sample is moved from the coil to the lead area. Use these data to create a plot of integrated intensity versus sample depth. This plot has a wealth of information. It shows the proper sample depth, the length of the coil, and any lead pickup taking place. Sometimes lead pickup can be minimized by pulling the bottom of the sample up above the area of lead pickup. This makes shimming more difficult due to end effects of the sample but should reduce lead pickup.

Once again we can see that probe design comes down to a series of details and compromises between conflicting requirements.

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Prof. Dr. Horst Kessler

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01/23/1990

(received 1/29/90)

Dr. B. L. Shapiro
Editor/Publisher
TAMU NMR Newsletter

966 Elsinore Court
Palo Alto, California 94303
U S A

Move to the TU München, Proton-Detected DEPT Spectroscopy

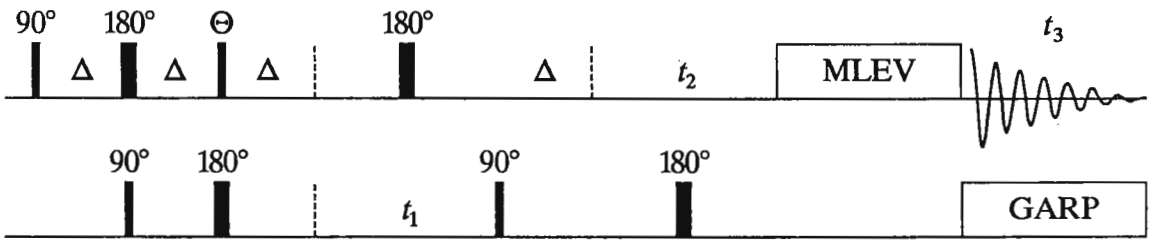
Dear Barry,

After a long period of silence I will again contribute a letter on the credit of Prof. F.H. Köhlers submission.

We recently moved from Frankfurt to the Technische Universität München. Now our installation of Bruker instruments AMX 600, AMX 500 and AC 250 connected to two X 32 data stations is complete. We seem, for the first time in my scientific life, to have enough measuring time for our group. The data stations are connected via ETHERNET to a Silicon Graphics Power Station 4D - 240.
Continuing our work on the structure determination of peptides we are enthusiastic and active.

Recently we achieved, in our opinion, the first total assignment of all non-carbonyl carbons of a small protein: tendamistat (74 amino acids, Biopolymer in print) and are working with some new techniques in one, two, and three dimensions.

Among these, the implementation of DEPT to proton-detected heteronuclear shift correlation seem to offer special advantages for editing (discrimination of CH, CH_2, CH_3) and multiplicity selection, e. g. a spectrum which contains only CH_2 groups needs a small spectral range in the carbon dimension and thus allows for higher resolution in a 3D DEPT TOCSY spectrum, pulse sequence:



Such an example (carbon in natural abundance) is shown in the spectrum of the cyclic hexapeptide cyclo(-D-Ala-Phe-Trp-Lys(Z)-Val-Phe-).

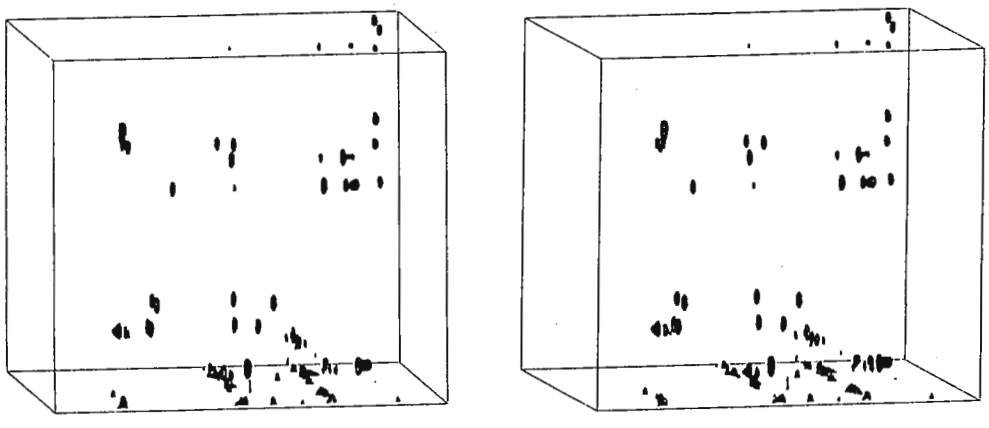
The trick of this technique is to use the heteronuclear multiple quantum term after the Θ - pulse at protons. This term, which is not used in conventional DEPT spectroscopy evolves in t_1 as usual in "inverse" techniques. The relevant transfer functions after the Θ - pulse are

CH	: $S_x \sin\Theta$	$+ 2 I_x S_y \cos\Theta$
CH_2	: $S_x \sin\Theta \cos\Theta$	$+ 2 (I_{1x} + I_{2x}) S_y \cos 2\Theta$
CH_3	: $S_x \sin\Theta \cos^2\Theta$	$+ 2 (I_{1x} + I_{2x} + I_{3x}) S_y \cos\Theta (1 - 3\sin^2\Theta)$

Selection of CH_2 groups is possible by $\Theta = 90°$ (whereas a 180° pulse give the sign discrimination between CH/CH_3 and CH_2).

Yours sincerely

(H. Kessler) (P. Schmieder)



3D-DEPT-TOCSY

exclusive excitation of CH_2-groups
net aquisition time: 10 hrs

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Phone: (314) 694-1000

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

January 2, 1990
(received 1/22/90)

C Points the Way to VXR Phase Table Flexibility

Dear Barry:

We have recently been investigating and comparing a number of pulse sequences on our SUN-based Varian VXR-500 spectrometer that differ only by the radio frequency (rf) phase of one or more of the elements in the pulse sequence. We have found the use of the real-time acquisition processor (AP) phase tables (VNMR Software Version 2.2a) to be an enormous improvement over the older modulo arithmetic calculation of the AP phase variables. (Thanks, Varian!)

However, after examining a few similar pulse sequences, each using three or four different AP phase tables, we ran into minor file management and nomenclature problems. In order to examine the effects of different phase cycles on an experiment, one has to either change the existing phase table(s) or create an essentially identical pulse sequence that loads a different phase table(s). The first case hinders direct comparisons between separate phase cycles by requiring that the AP phase table be physically altered, while the second case requires both recompilation of the pulse sequence and the existence of nearly identical files and tables. Neither of these two approaches permit the phase cycle to be arrayed (like other parameters) so that direct comparisons between different phase cycles can be made within the same experiment.

To overcome these difficulties and minimize the number of files and tables that were being created, we took advantage of features of the C programming language by defining an array of pointers which refers to the run-time phase vectors (*e.g.*, t1 to t60) contained in the AP phase table. We then created a keyboard enterable VNMR acquisition parameter (*e.g.*, pulse_phase) to serve as the array index. By employing this modification, different phase cycles for a particular pulse sequence element can be selected from one AP phase table by simply changing the assignment of a single acquisition parameter. To investigate a new phase cycle, one needs only add a new phase vector to the existing phase table and set the acquisition parameter accordingly. This approach not only provides more flexibility with easier control over individual phases, but also allows the phase of individual pulse sequence elements to be arrayed within the same experiment. Of course, the number of phase vectors and, hence, the number of available pointers, are subject to the same restrictions and limitations as the AP phase tables. The variable phase pointers can be implemented by including lines, similar to those given below, in appropriate places in the pulse sequence. Their use is illustrated by the following examples.

```
/* VARIABLE DECLARATION */
     codeint *pointers[61];              /* Declare pointer array */
     int pulse_phase;                    /* Example of pulse phase variable
                                            declaration */

/* INITIALIZE PHASE VARIABLE POINTERS TO AP PHASE TABLE VECTORS */
     pointers[1] = &t1;
     pointers[2] = &t2;
       .
       .
       .
     pointers[m] = &tm;                  /* m is limited by AP table
                                            restrictions, m ≤ 60 */

/* LOAD VARIABLES */
     loadtable("phasetable");
     pulse_phase = (int)getval("pulse_phase");

/* BEGIN PULSE SEQUENCE */
     pulse(pw,*pointers[pulse_phase]);   /* Example of a rf pulse that uses
                                            phase variable pointer to assign
                                            the phase of the pulse */
```

Examples

Given a pulse sequence that includes the above statements and the following AP phase table, the phase of the rf pulse is determined by phase vector t1 (*e.g.*, x y -x -y) when the acquisition parameter pulse_phase = 1 and phase vector t2 (*e.g.*, x -x y -y) when pulse_phase = 2. If the pulse_phase parameter were set to an array (*i.e.*, pulse_phase = 2,3), then arrayed data are collected where the first array element uses phase vector t2 and the second array element uses phase vector t3 (*e.g.*, x x x x y y y y -x -x -x -x -y -y -y -y) for the phase of the rf pulse.

phasetable:

```
t1 = 0 1 2 3                          /* x  y -x -y */
t2 = 0 2 1 3                          /* x -x  y -y */
t3 = (0)4 (1)4 (2)4 (3)4              /* x  x  x  x  y  y  y  y
                                         -x -x -x -x -y -y -y -y */
```

An alternative to using the phase variable, of course, would be to create a variable that names the AP phase table. This would also allow phases to be arrayed. However, this approach requires separate tables for the same experiment, which becomes annoying, especially if only one phase in the pulse sequence is being altered at a time.

Sincerely,

John Kotyk Norman G. Hoffman Bill

John J. Kotyk Norman G. Hoffman William C. Hutton

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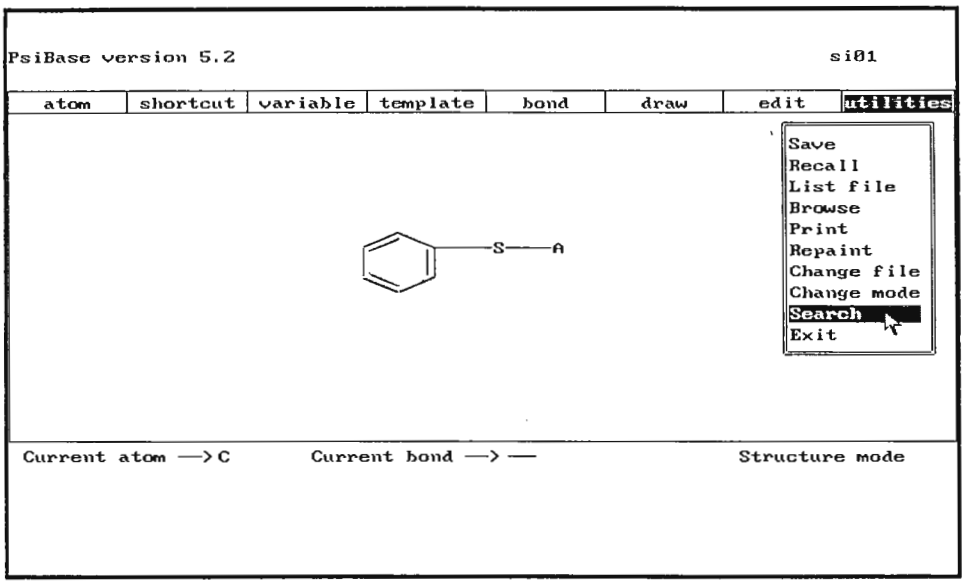
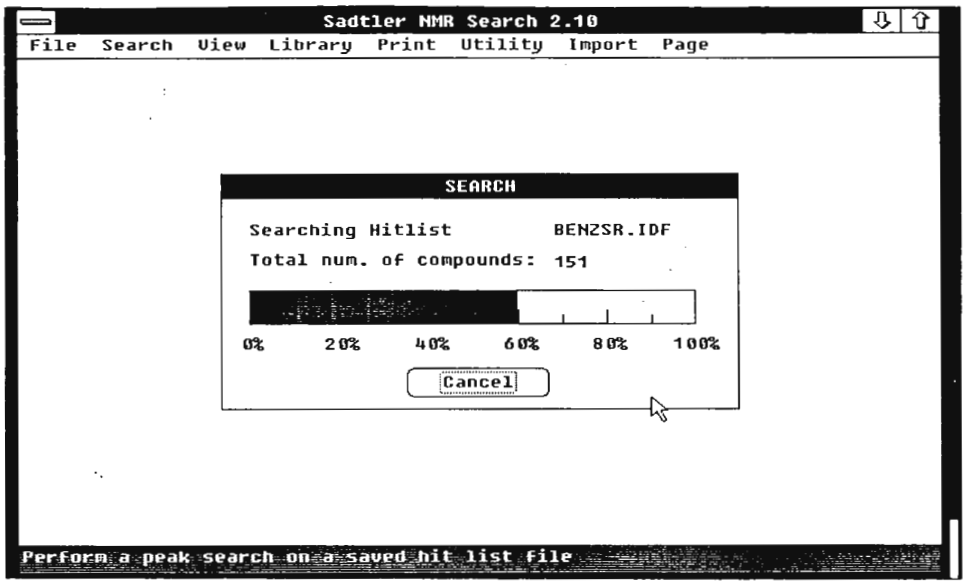
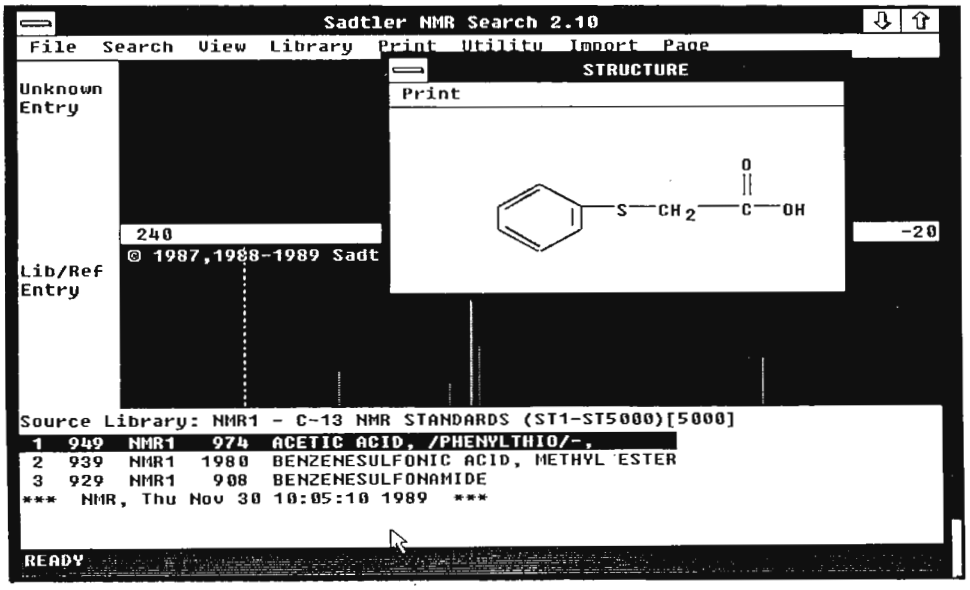


Step 1: Search for all thiophenol derivatives.



Step 2: Search for those derivatives that match your spectrum.



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ST. GEORGE CAMPUS
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STATEN ISLAND, NEW YORK 10301

January 11, 1990
(received 1/29/90)

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

Dear Barry:

We wish to claim the dubious honor of observing the world's longest ^{1}H-^{13}C contact-time dependence from CPMAS spectra (Figure 1), obtained in this case for gel-state multilamellar vesicles of 50% (w/w) dipalmitoylphosphatidylcholine in D_2O. (Our Doty probe even survived, but z-axis storage techniques were needed to avoid baseline distortions from overload of our IBM[sic] WP-200 preamplifier.)

The oscillatory variation in ^{13}C signal intensities, which is most pronounced for the relatively mobile headgroup methyls in this and other phospholipids, is reminiscent of the behavior reported some time ago in J-cross polarization experiments for liquids. Even though reorientation on the MHz timescale is not efficient enough to yield high-resolution ^{1}H MAS NMR spectra (Figure 2), the gel-state multilayer organization appears to permit sufficient motion at mid-kHz frequencies to interfere with polarization transfer from ^{1}H to ^{13}C.

We leave it to your readers to decide whether we are really doing solid-state or solution-state spectroscopy... and we would be interested in communicating with other investigators who might provide some insight into similar anomalies.

Very truly yours,

Helen N. Halladay
Postdoctoral Research Associate

Ruth E. Stark
Associate Professor of Chemistry

Please credit this contribution to the account of the City University of New York.

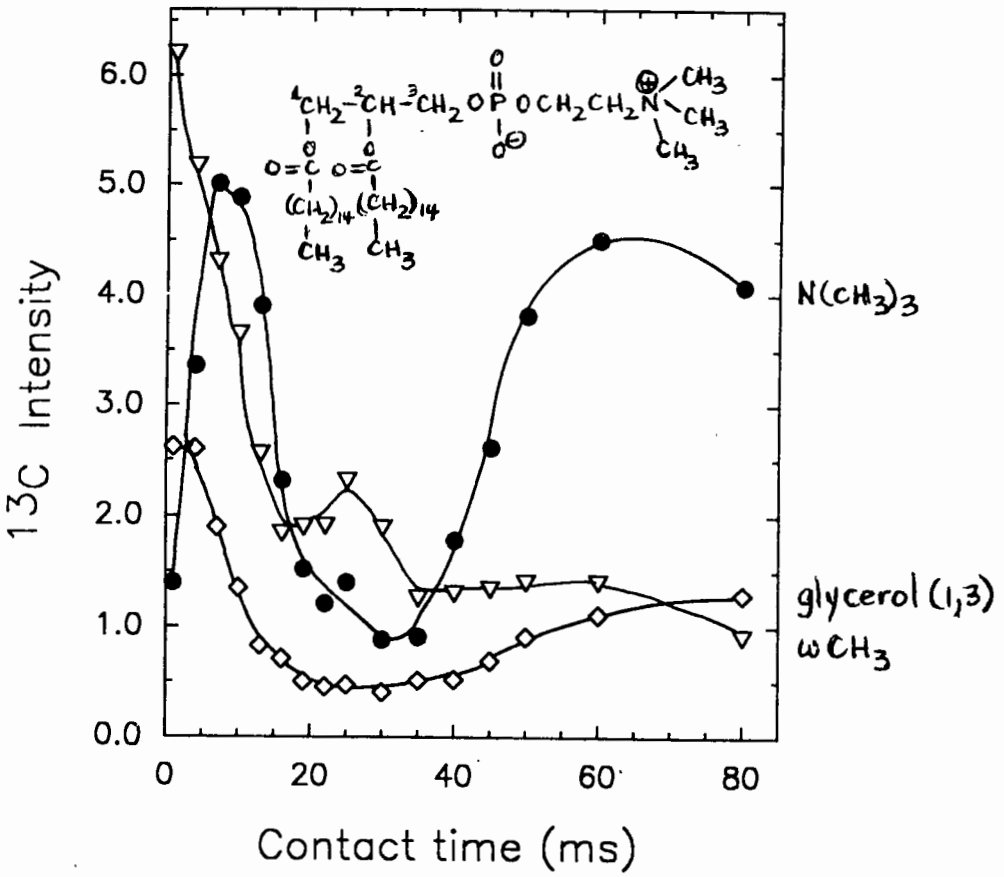

Figure 1. Variation in CPMAS ^{13}C NMR signal intensities for gel-state DPPC in D_2O (reduced temperature -0.048). For contact times exceeding 2 ms, recycle delays of 5 s were used to avoid sample heating. Similar variations in signal intensity were observed for C(18):C(10)PC.

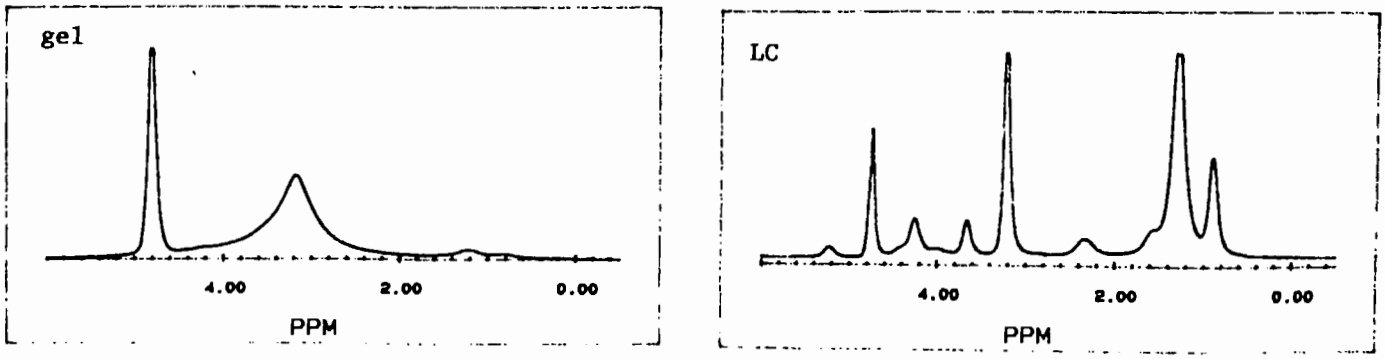

Figure 2. 1.8 kHz MAS ^{1}H NMR spectra of C(18):C(10)PC at 280 K (gel) and 292 K (liquid crystal).

Shell Development Company
A Division of Shell Oil Company

Westhollow Research Center
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Houston, TX 77251-1380

November 10, 1989
(received 1/25/90)

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

Dear Dr. Shapiro:

The use of magic angle spinning (MAS) in solid state NMR has become so pervasive that it will be meaningless for me to extoll its virtues. A great many ways are in vogue as to how best to set the angle which range from the esoteric but elegant method of Bodenhausen, Caravatti, Deli, Ernst and Sauter [1] involving lasers to the relatively simpler but very effective technique of using the spinning side bands of ^{79}Br in potassium bromide [2]. In most cases that use such spinning side bands to set the angle, the ease of setting is mostly determined by the nature of the nucleus that needs to be studied. Thus, KBr is quite popular with researchers studying carbon at 4.7 or 9.4 Tesla while KI is used when the nucleus of interest is silicon. However, for studies involving aluminum which quite often requires the use of 9.4 Tesla or higher fields, use of KBr poses some problems in the sense that the preamplifiers, receivers and the transmitters of the observe channel have to be retuned after setting the magic angle with ^{79}Br (100.21 MHz at 9.4 Tesla) as its Larmor frequency differs considerably from that of ^{27}Al at that same field (104.23 MHz at 9.4 Tesla). We have observed that the use of aluminum metaphosphate (Alfa Products) to set the magic angle circumvents the above problem quite easily and has the added advantage of being a good chemical shift reference. Shown below are the free induction decay and the FT spectrum of aluminum metaphosphate after one scan. The sample was spun in a 9 mm DELRIN rotor at approximately 3 KHz and a tip angle of 30^0 was used. Our experiments indicate that the number of echoes/side bands and their intensity observed for this aluminum salt are extremely sensitive to the magic angle and the angle can be set as accurately as was possible with KBr.

The apparent isotropic chemical shift with reference to aqueous aluminum chloride solution is -22 ppm and is quite unique when compared to other aluminum phosphates most of which occur in the +30 to +40 ppm region. The extremely intense signal makes this salt particularly useful when the demands of high speed spinning impose the use of relatively small volume rotors. The line width at half height is about 250 Hz which is considerably narrower than that observed in most zeolites.

Please credit this contribution to the account of Dr. Larry Sterna.

Sincerely,

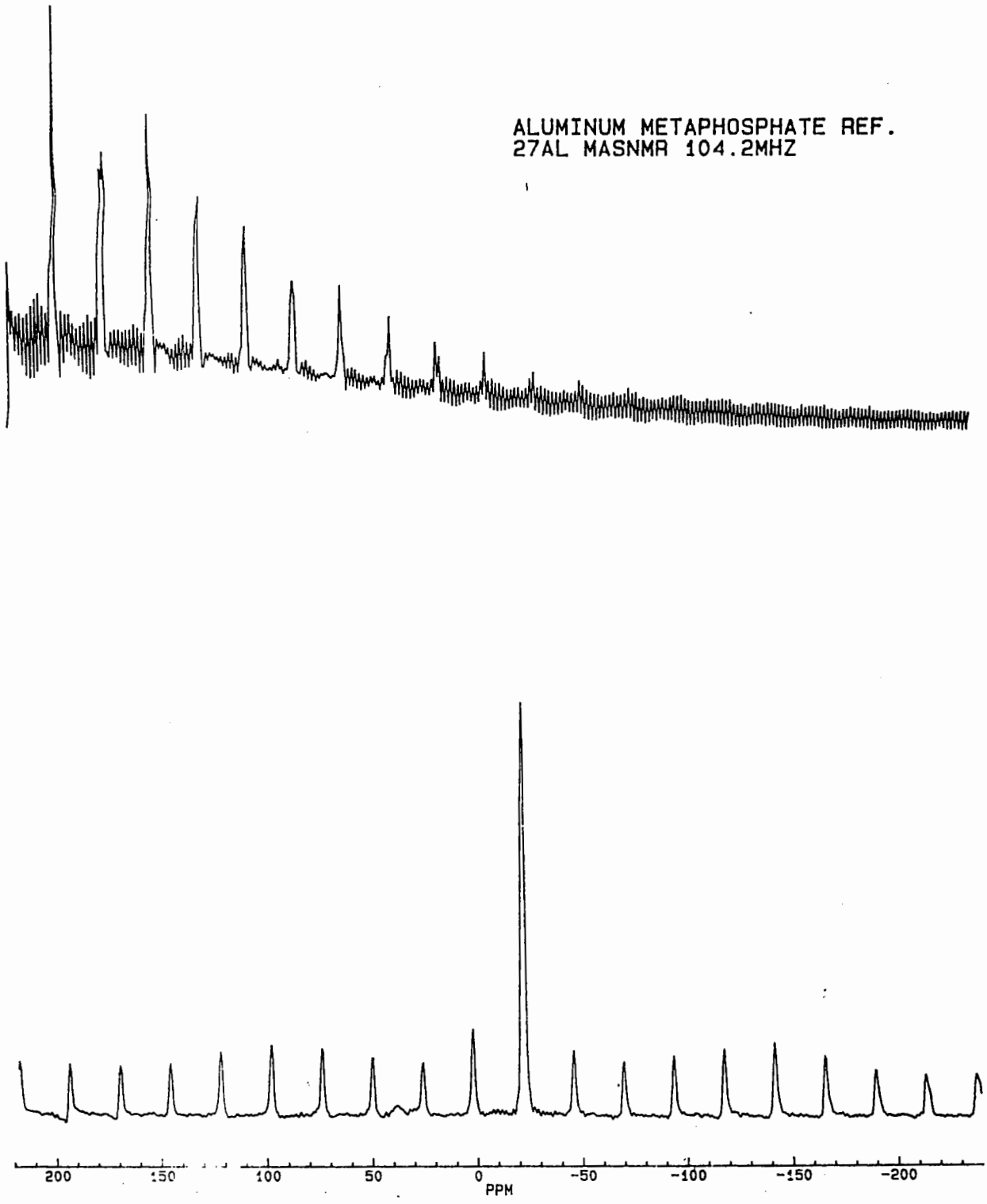
Mysore Narayana

Mysore Narayana
Research Chemist
Shell Development Co.

References:

1. G. Bodenhausen, P. Caravatti, J. Deli, R. R. Ernst and H. Sauter J.Magn.Reson., 48, 143 (1982).

2. J. S. Frye and G. E. Maciel J.Magn.Reson. 48, 125 (1982).



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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).

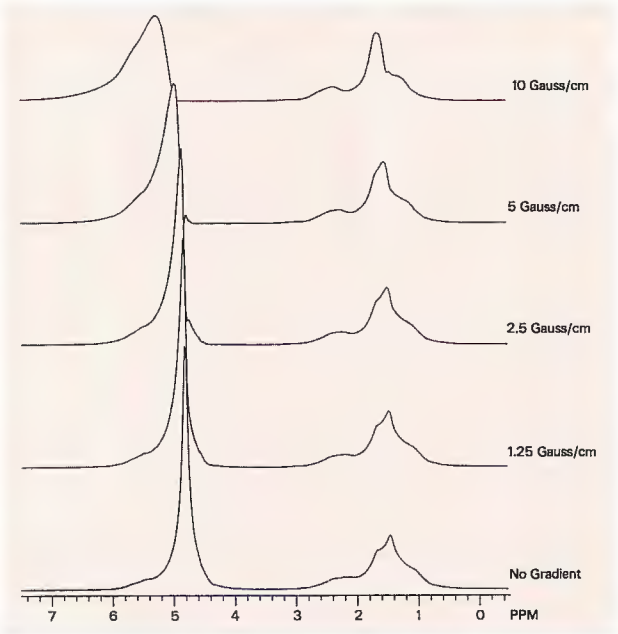

Fig. 1—Using an oil/water phantom, a 10 G/cm gradient will create a water frequency profile extending from 156 KHz to 280 KHz away from normal water resonance. Residual gradient effects of less than 0.01% (50 Hz at 10 G/cm) are observed in a spectrum acquired beginning 1 msec after a 20 msec gradient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

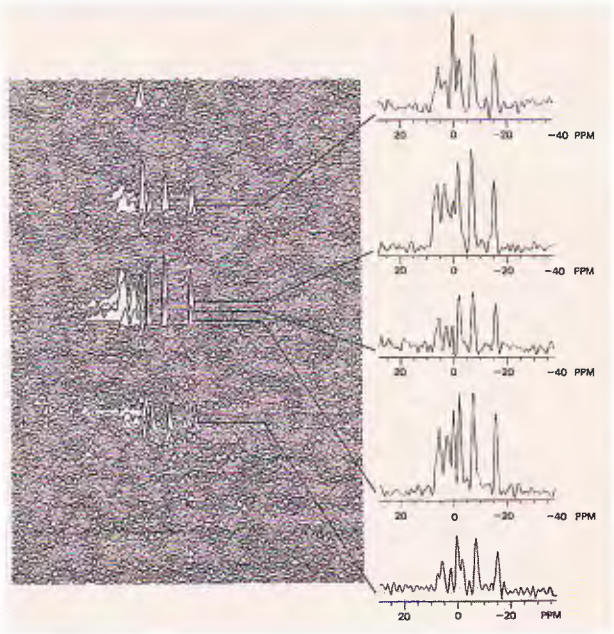

Fig. 2—Stacked plot showing 512 phosphorous spectra from 60 mm cubed region of a live rat. Each trace corresponds to 7.5 mm cubed region (voxel) from within the region of interest. The offset traces clearly show the achievable spectra and spatial resolution of the technique, as well as demonstrating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

GE NMR Instruments

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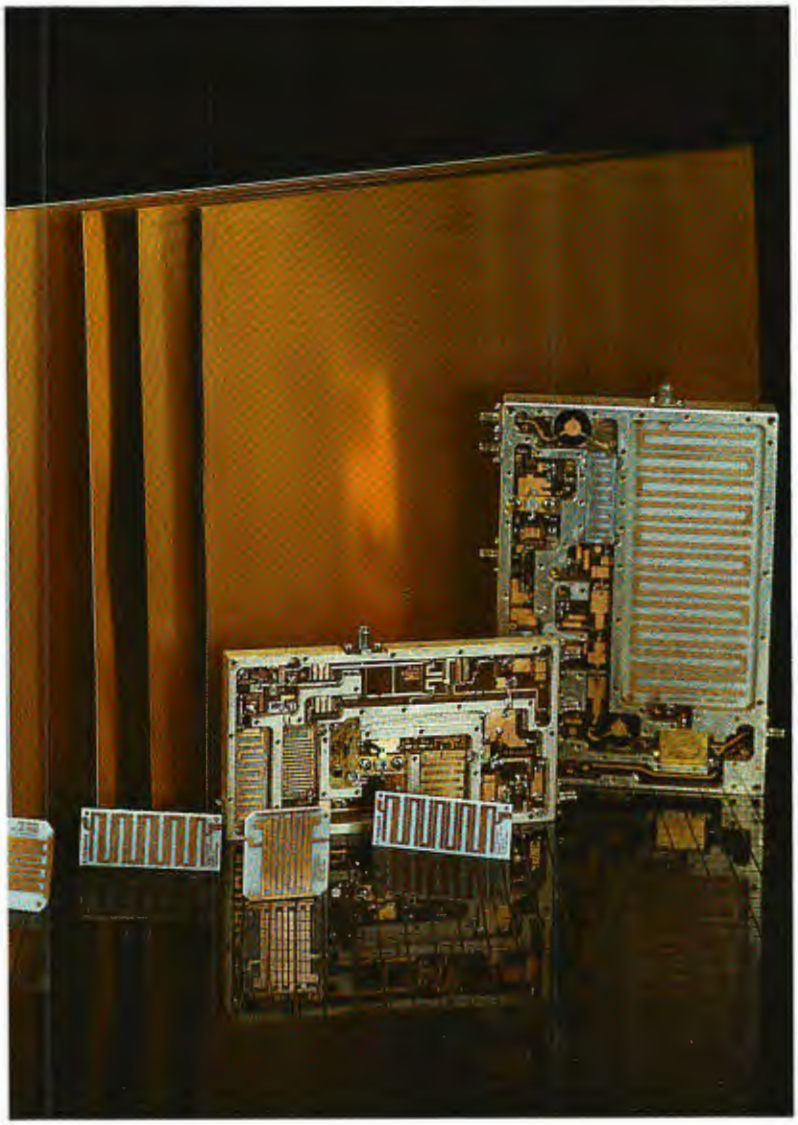

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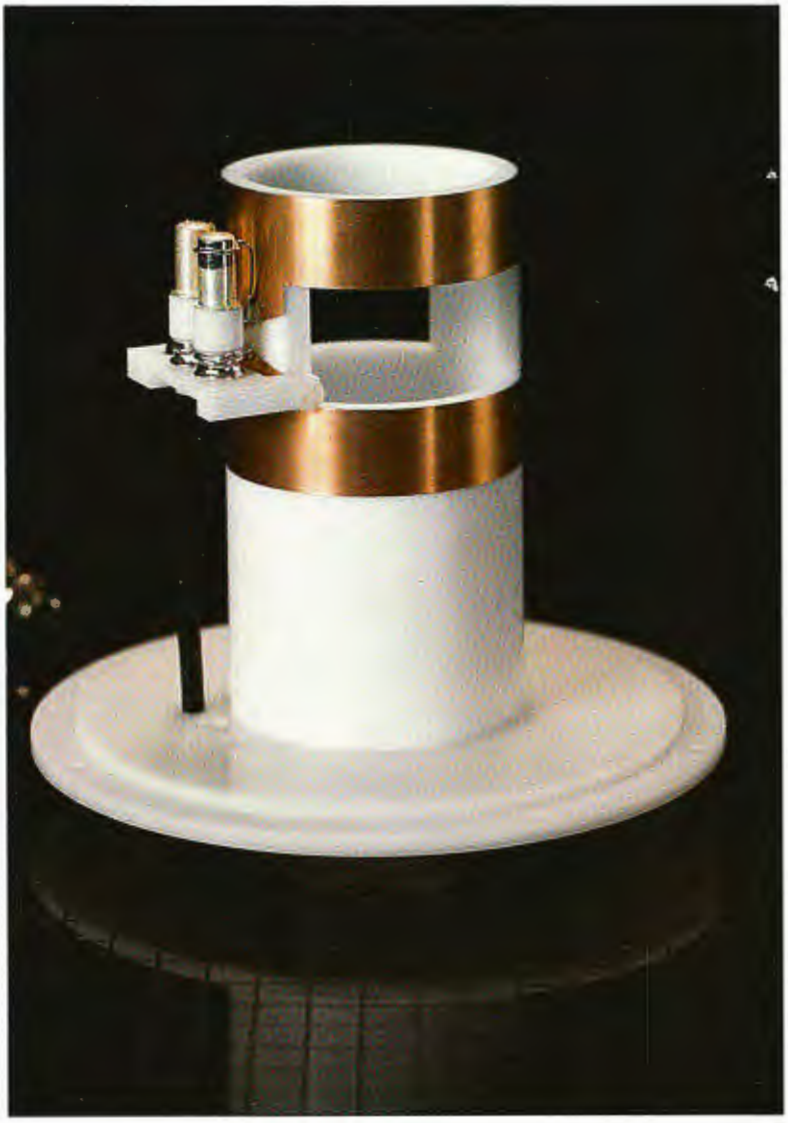

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MR Research Center
Div. of Radiology, Desk L10

February 9, 1990
(received 2/15/90)

^{19}F and ^{31}P MRS of the metabolic response of RIF-1 tumors to 5-fluorouracil chemotherapy

Dear Barry,

We have studied the response of radiation-induced fibrosarcoma (RIF-1) to 5-fluorouracil (5FU) chemotherapy using in vivo 19F and 31P MRS. Ten C3H/HeN mice with RIF-1 tumors received a single dose of 5-FU (260 mg/kg, i.p.). For 19F MRS (subsequent 20 min acquisitions; 45°-pulses with TR=0.5 s) and 31P MRS (400 54°-pulses with TR=3 s) of the tumors a 20 mm φ solenoidal coil was used. Ratios of 31P and 19F signals to, resp., DMP and NaF standards were calculated from peak areas fitted to Lorentzian curves with a Simplex algorithm. To obtain values relating to the levels of NMR visible tumor metabolites, peak ratios were divided by tumor volume.

The level of 5FU in the tumors showed a continuous drop, while the fluoronucleotide (Fnuc, the cytotoxic form of 5FU) signal increased to a plateau level of 50% of the initial 5FU level. The levels of fluoro-β-alanine (FBAL) and of an unknown compound at -2.5 ppm from 5FU were constant. 31P MRS revealed a loss of tumor phosphate and a pH increase over the subsequent days, indicating an increasing necrotic fraction.

Saline solutions of 2 mM Pi and 1mM 5FU (1 ml) were measured by NMR under the same conditions as the RIF-1 tumors. The obtained Pi/DMP and 5FU/NaF ratios indicated a mean pretreatment NTP level of 2.59 mM, a mean initial 5FU level of 211 μM, a plateau Fnuc level of 108 μM and a mean FBAL level of 413 μM.

The changes in phosphate metabolism and tumor pH did not correlate with the detected fluorine levels or tumor response. However, the pretreatment Pi level, the plateau Fnuc level and the 5FU induced decrease in tumor volume showed significant correlation. Our conclusion that vital tumors with a low pretreatment Pi level form high levels of Fnuc and respond well to 5FU chemotherapy, is in agreement with reports that tumors with a low level of anabolism have a low sensitivity to 5FU (3,4).

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3. D. Kessel, T.C. Hall, I. Wodenski. Science **145:** 911-913, 1966.
4. P. Reyes, and T.C. Hall. Biochem. Pharmacol. **18:** 2587-2590, 1969.

Please credit this contribution to Thian C. Ng's account.

Sincerely yours,

Paul E. Sijens

Continued from p.33

and the flow induced phase shift is given by $\Phi = \gamma GTT_dV$. In order to measure slow flow, a sufficiently large first moment m_1 has to be used to give a measurable phase shift. For bipolar gradient pulses this can be accomplished by increasing either the area of the gradient pulses GT or the separation of the pulses T_d. Now for the phase method, with the transverse magnetization evolving for a time T_2 before forming an echo, the bipolar gradient pulse can have a duration of the order of $T_2/2$, and the time between the pulses T_d can be as long as T_1 for stimulated echoes, which implies from Eq. [3] that

$$m_1 = GT_2T_1/2. \qquad [4]$$

Thus, from Eqs. [2] and [4], the minimum velocity measurable by the phase method is

$$V_p = 2\Phi/\gamma GT_2T_1, \qquad [5]$$

where Φ is some minimum phase shift distinguishable from zero.

Because we are not directly considering the effects of the signal/noise ratio, the exact value of Φ is arbitrary in this discussion, just as the value of the minimum spatial resolution R as defined by the half-height was arbitrary. The main reason why V_p is smaller than V_b is because T_2^* is smaller than T_2. Therefore, phase methods are inherently more sensitive to slower velocities than bolus tracking when $T_2^* < T_2$.

A. Caprihan E. Fukushima
Lovelace Medical Foundation,
2425 Ridgecrest Dr., SE,
Albuquerque, NM. 87108.

(received 1/22/90)

SLOW FLOW NMR LIMITS: Bolus tracking versus phase methods.

Two methods for flow measurement which have been widely used are the time-of-flight and the phase methods. The time-of-flight method is based on detecting spin displacement during the time between initial excitation and signal measurement. This displacement is limited by the velocity of the flowing material and the lifetime of the tag. In addition, information outside of the bolus is not accessible without further experiments. The second class of methods detect the alteration of the phase of transverse spin magnetization induced by spin motion along a magnetic field gradient. We have always taken it for granted that phase methods could measure lower velocities than the bolus tracking methods but had not looked into this question. Here we discuss the circumstances when phase methods can measure slower velocities than the bolus-tracking methods. The discussion is in terms of stimulated echo experiments where the lifetime of the tag is T_1, but the conclusion is equally applicable to spin echo and gradient-echo experiments.

The difference arises because the position of the bolus is defined in the frequency encoding direction and its accuracy depends on the effective T_2^* caused by static field homogeneity but not the applied gradient whereas the velocity phase-encoding is analogous to the spatial phase-encoding in spin warp imaging experiments, and is not affected by T_2^* but is affected by T2. In a given voxel, T_2^* will be smaller than T_2 because of inhomogeneous magnetic field within a voxel caused by susceptibility changes in the sample and imperfect shimming.

First, we consider the bolus-tracking method in which the velocity V_b has to be greater than R/T_1, where R is the resolution of the imaging experiment, in order to resolve the bolus displacement. For a given T_2^* the half-height bandwidth of the broadening is $2/T_2^*$ and if the read-out gradient is G, this corresponds to a resolution of $R = 2/\gamma G T_2^*$, which leads to the lower limit on the measurable velocity of

$$V_b = 2/\gamma G T_1 T_2^*. \qquad [1]$$

Here we are assuming that, because of broadening, the bolus has to move a distance R before it can be accurately distinguished from its original position.

The phase methods depend on the phase shift of the transverse magnetization of spins moving with a constant velocity and a component V along the velocity encoding gradient G being given by

$$\Phi = \gamma m_1 V, \qquad [2]$$

where m_1 is proportional to the first moment of the gradient ($m_1 = \int tG(t)\,dt$ with $\int G(t)\,dt = 0$). A simple velocity encoding method consists of a bipolar gradient pulse, with gradient amplitude G, duration T, and the time between the pulses T_d. Then

$$m_1 = GTT_d \qquad [3]$$

Continued on p.32

Abbott Laboratories
Abbott Park, Illinois 60064

February 1, 1990

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

Re: Disk interface problems on a GN500, Disk Drive Wanted.

Dear Dr. Shapiro:

DISK INTERFACE PROBLEMS

For several years we have had an ongoing problem with the communication (or lack thereof) between the Nicolet 1280 computer on our GN500 and the 227 Mbyte Piram disk drive. After working fine for a period of days to weeks, we will suddently get a disk error 240 ("Access denied") or error 220 ("Illegal Inode"). These errors not only cost us time and data, but they force us to reformat the disk every few weeks.

In an effort to fix this problem we have changed both the disk drive and the SMD interface board. We have removed the backplane and connected the drive directly to the SMD board. At GE's suggestion we have tweaked the voltage from the power supplies of both the disk drive and the 1280. Nothing seems to help. We are writing to ask if any of the TAMU readership has had similar problems, and, if so, how were they fixed. Any help or suggestions sent to Peter Fruehan or Dave Whittern would be greatly appreciated.

DISK DRIVE WANTED.

We are currently seeking a second hand CDC 96 Mbyte disk drive. Anyone interested in selling one should contact either Peter Fruehan or Rich Stephens.

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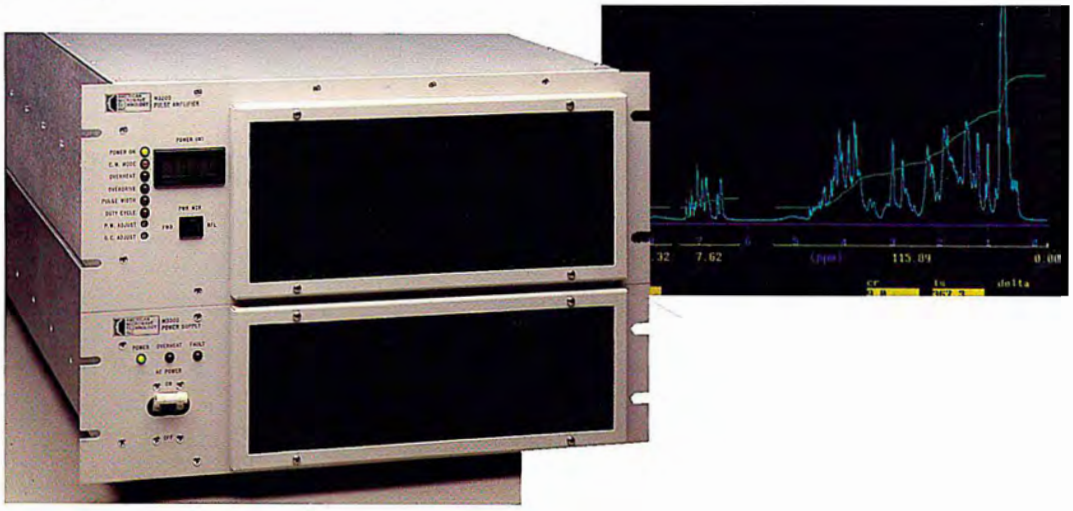

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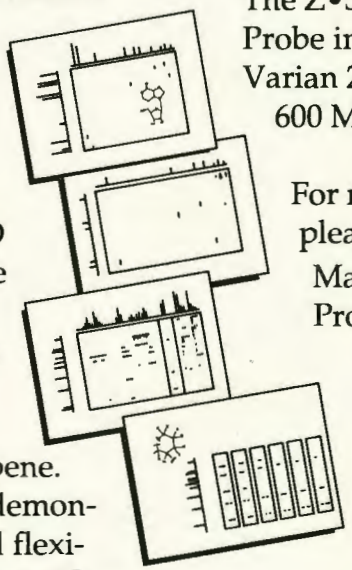

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For more information, please contact Toby Zens, Manager of the Z•Spec Products Group.

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Z·SPEC Indirect Detection Spectra

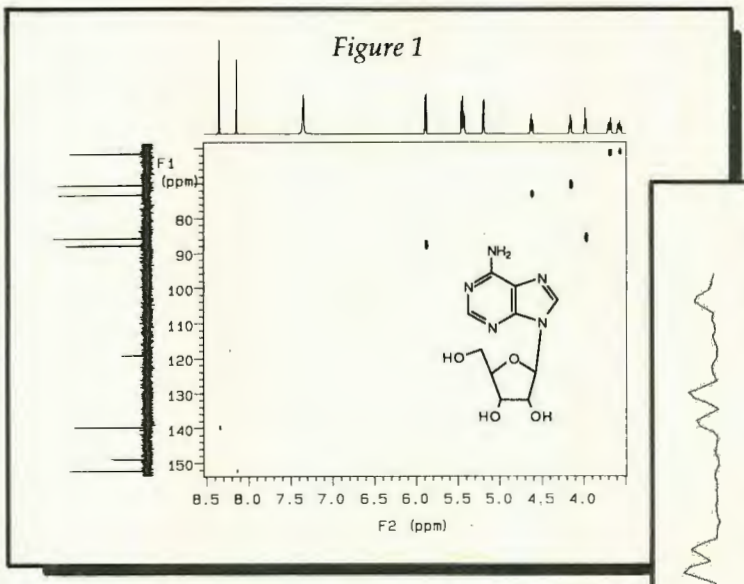
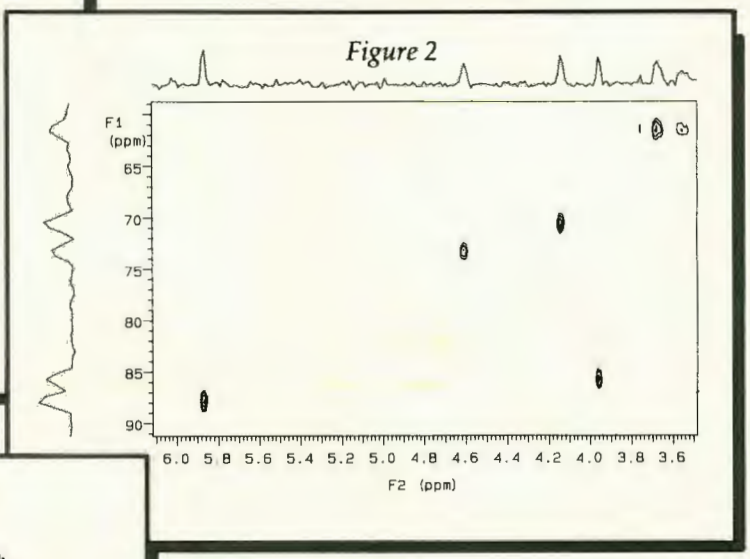


Figure 1: Z•Spec ID probe, HMQC experiment run on a 1 mg/ml $DMSOd_6$ solution of Adenosine. Full chemical shift region, PW90=10 μ sec ^{13}C, 8 transients/FID, total time 19 minutes.

Figure 2: HMQC Ribose region Adenosine, 125 μg/ml. Total acquisition time 2.2 hours, nt = 224.



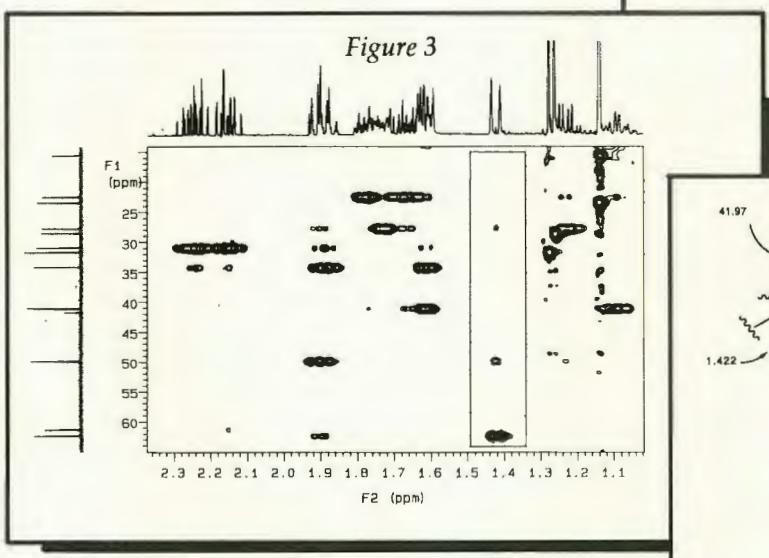


Figure 3: HMQC/TOCSY experiment run on a 8 mg/ml solution of a Marine Sesquiterpene decomposition product. Total acquisition time 2 hours, 10 minutes.

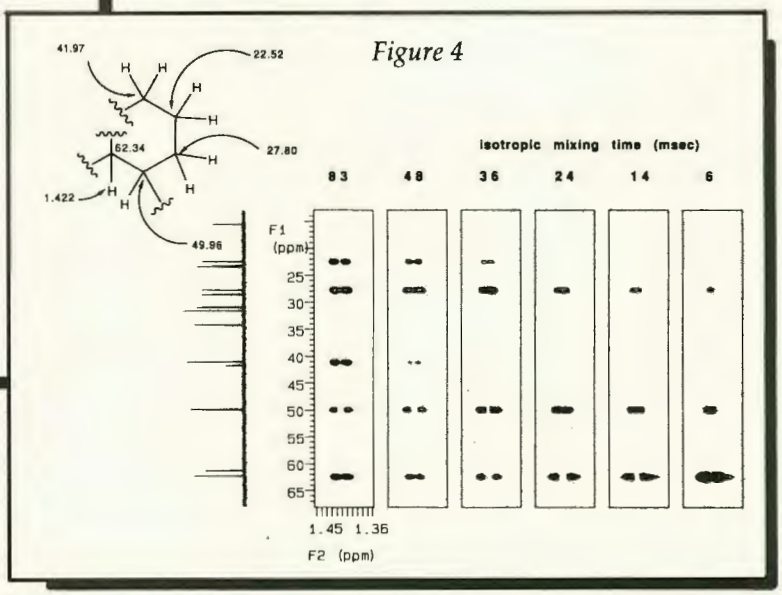


Figure 4: Expansions at the 1.4 ppm proton resonance showing the propagation of magnitization to the carbons with increasing mixing time. Note the excellent F1 phase characteristics throughout.

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FOX CHASE
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Department of Nuclear Magnetic Resonance
and Medical Spectroscopy

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

February 16, 1990 (received 2/19/90)

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

User-Friendly Display of Mg^{2+} Levels in Human Brain.

Dear Dr. Shapiro:

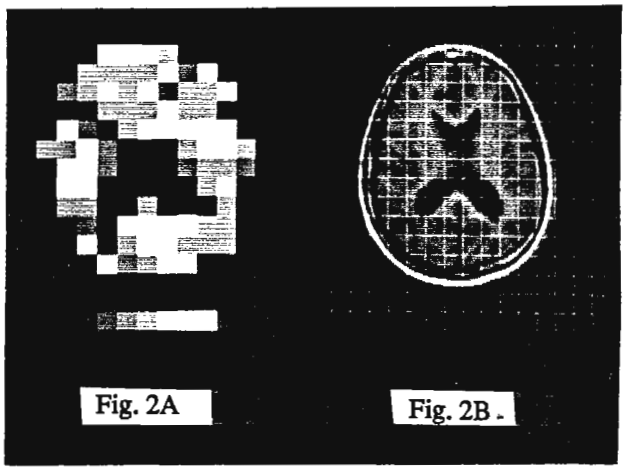
Our laboratory has developed 3D chemical shift imaging techniques (3D CSI) on a standard 1.5T clinical magnetic resonance imager (Siemens Magnetom) for *in vivo* spectroscopic studies of the brain. The recent design and construction of a ^{31}P quadrature birdcage resonator (1) enabled us to survey the brain with excellent S/N and spatial resolution of 12-27 $(cm)^3$. Spectra localized to a 3-dimensional grid of 8x8x8 contiguous 27 cm^3 voxels spanning the brain were acquired in 35-70 min. Planes of 64 spectra may be selected from the 3D dataset(Fig. 1). The sheer number of spectra and the necessity of making correlations with a three-dimensional object (here, the brain) pose interesting problems of data analysis and display. Data analysis techniques developed here enable us to represent the spectral data in the form of metabolite images which display the spatial distribution of individual metabolites to allow correlations with anatomy and pathology. An image display is much more readily and rapidly processed by the human mind for such correlations.

In studies of healthy volunteers, we observed that a significant fraction of observable intracellular ATP in brain is not complexed to Mg^{2+}. The β-ATP chemical shift in a series of 5 volunteers was -19.07 ± 0.084 ppm(mean, SEM), referenced to the brain phosphocreatine peak as -2.52 ppm. This corresponds to approximately 70% MgATP and 30% free ATP, and $[Mg^{2+}_i]$ of 0.2 mM(2). This fraction free ATP is higher, and the Mg^{2+} lower, than the values reported previously in studies of animal brain(3).

The β-ATP shift, and hence $[Mg^{2+}_i]$, may be measured in each voxel across the brain. The standard deviation of the chemical shift measured in a series of 6-10 voxels in an individual was 0.2-0.3 ppm. The variations in Mg level from voxel to voxel across the brain may be represented on a gray-level image as deviations above(lighter) and below(darker) the individual's mean β-ATP shift to create a user-friendly display of these data that facilitates comparison with the anatomical proton image of the brain obtained in the same examination(Fig 2 A and B, respectively, below). A color overlay of 2A on 2B is even better.

Sincerely,

J.S. Taylor, J. Murphy-Boesch, D. Vigneron, S.J. Nelson and T.R. Brown



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2. Gupta, R., P. Gupta, W. Yushok, Z. Rose (1983). Physiol. Chem. Phys. and Med. NMR 15, 265.
3. Vink, R., T. McIntosh, P. Demediuk, M. Weiner, A. Faden (1988). J. Biol. Chem. 263, 757.

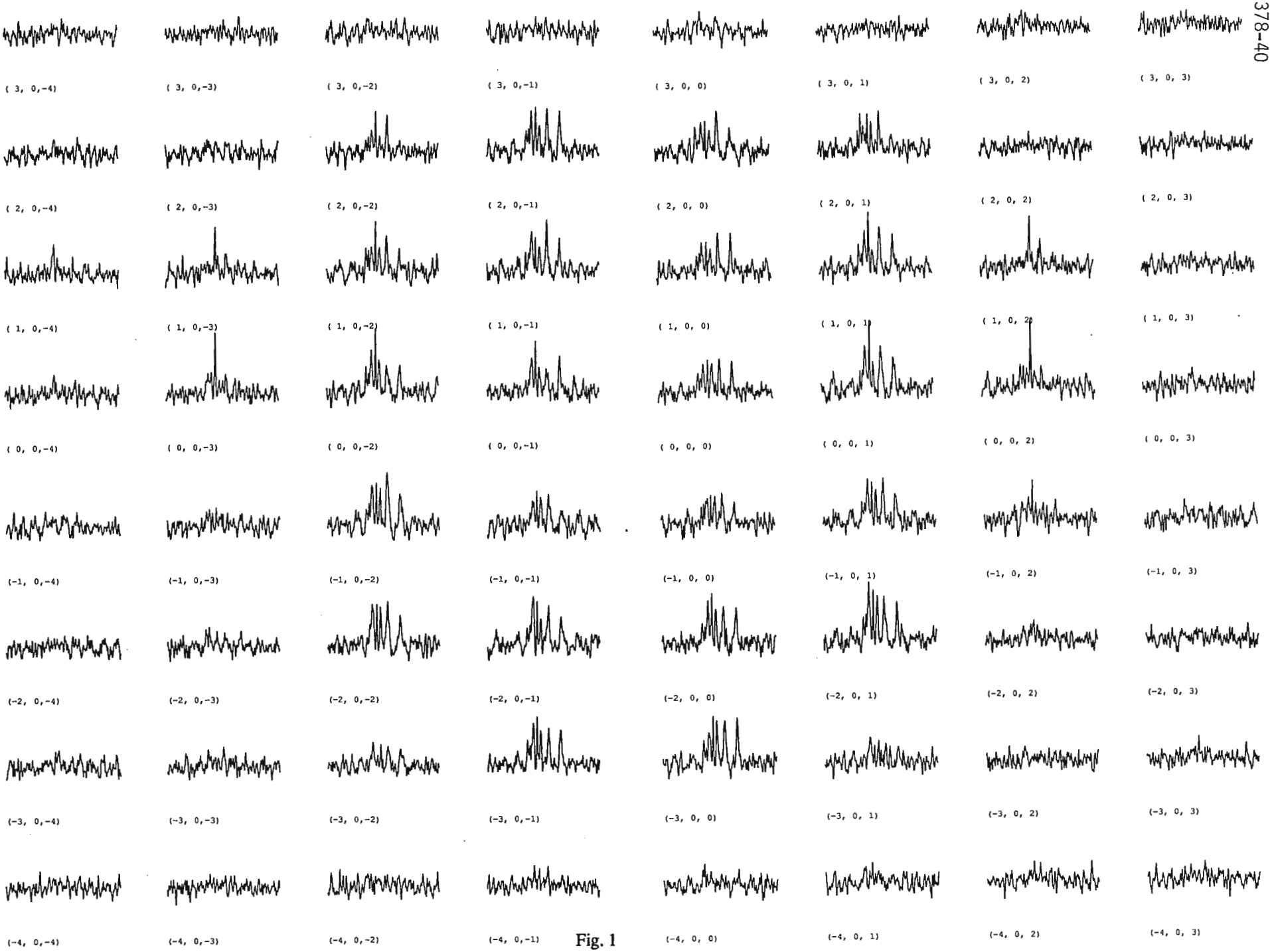
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Fig. 1

PENNSTATE

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Department of Radiology

P.O. Box 850
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February 15, 1990
(received 2/16/90)

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

Dear Barry:

Ionic Strength Effects on ATP Chemical Shifts

Systematic in vitro studies such as those conducted by Pettegrew et. al. for the determination of pH from the γ-α ATP chemical shifts (1) are necessary to accurately evaluate in vivo results. We have investigated the ATP shifts in more detail and in this communication would like to summarize our assessment of the effect of ionic strength.

It is known that Mg^{2+} has a large effect on the β-ATP and γ-ATP resonances, while the effect of pH is mostly on the γ shift. However, the influence of ionic strength has not been carefully characterized. For our studies we prepared NMR solutions containing unbuffered 5 mM ATP and KCl concentrations varying from 0 to 250 mM. There were 14 pH values from 2.5 - 10 pH units for each ionic strength to define titration curves. We used a four parameter non-linear least squares fit to model δ_{ss} vs. pH using the following equation:

$$pH = pK + (1/N)*\log[(\delta_{ss} - \delta^{+})/(\delta^{-} - \delta_{ss})]$$

where δ_{ss} is the measured ATP chemical shift, δ^{+}, δ^{-} are the acid and base endpoints,
N is a Hill coefficient which corrects for cooperativity of the dissociation,
pK is the log of the apparent dissociation constant K.

Figure 1 shows plots of chemical shift relative to PCr vs. pH at each KCl concentration for the case of the γ-ATP resonance. In Figure 2, the fitted pK's from the γ-phosphorus titration curves are given. Note that within the physiological range of 100-200 mM KCl, pK varies by less than .15 units. Typically one uses a solution of 155 mM KCl to approximate intracellular osmolarity. Therefore, small changes, in ionic strength, such as those that occur with tissue hypoxia or ischemia should not significantly affect the determination of intracellular pH using the γ-α ATP chemical shifts with in vivo samples.

FIGURE 1

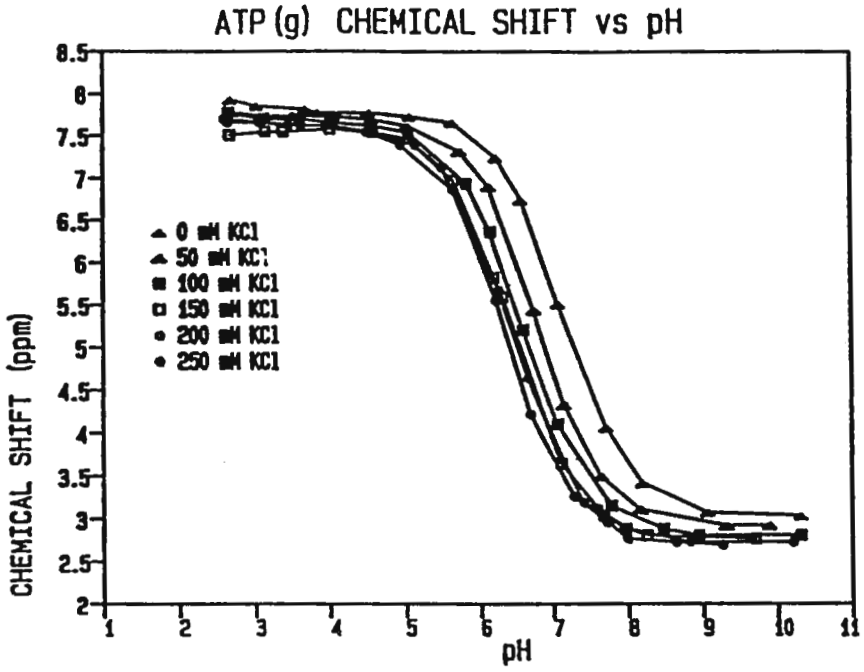
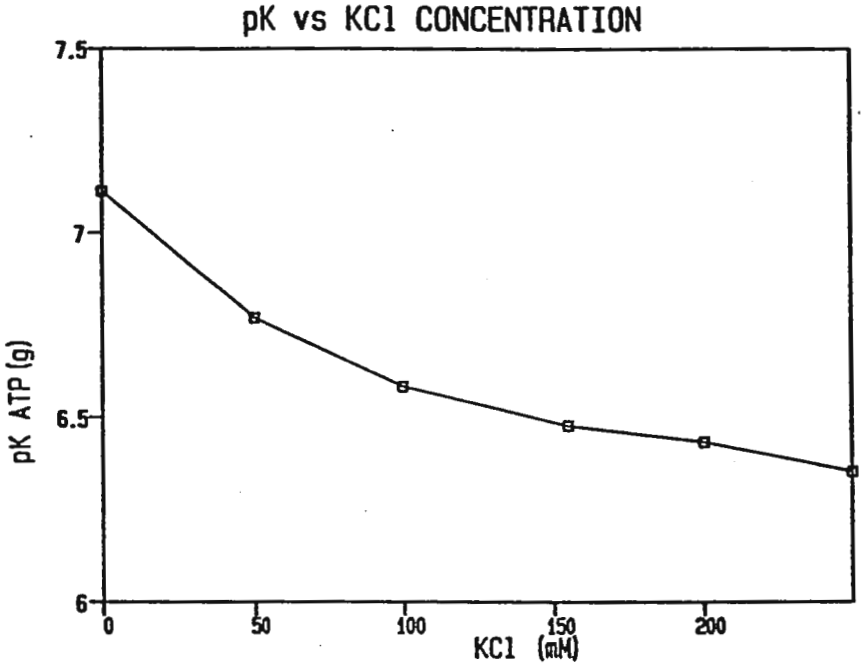


FIGURE 2



(1) Pettegrew J.W., Withers G., Panchalingam K. and Post, J.F.M. Magn. Reson. Imaging 6 135-142, 1988.

Sincerely,

Timothy J. Mosher, M.D.
Research Fellow
Bitnet: TIM@PSUHMED

Michael B. Smith, Ph.D.
Associate Professor

Gerald D. Williams, Ph.D.
Radiology Facility Mgr.

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JAB132.001
DATE 30-11-88

SF     500 135
SY   83.0
O1    7900 000
SI   32768
TD   32768
SW    5000.000
HZ/PT     .305

PW        5.0
RD        0.0
AQ          3 277
RG         32
NS         72
TE        297

FW       6300
O2       7925.000
DP     10H PO

LB          0 0
GB          0.0
CX         36.00
CY          0 0
F1          2.700P
F2            401P
HZ/CM      31.950
PPM/CM       .064
SR     5426 67
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(give catg. no. and name of manufacturer)
Type of NMR equipment used in the above evaluation: BRUKER AM-500
Date Nov 30, 1988 Name SCOTT VIRGIL Title GRADUATE STUDENT
Address 12 OXFORD STREET # 342
CAMBRIDGE, MA 02138
Telephone: (617) 495-4034
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Comparison and evaluation carried out at Harvard University by Scott Virgil on a Bruker AM-500 NMR unit, on November 30, 1988.

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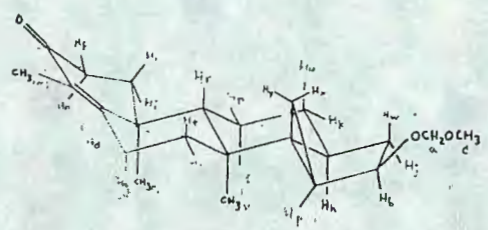

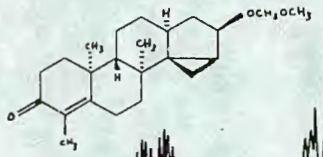

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i.d. 4.20 ± 0.025mm (0.001 in.);
camber ± 0.025mm (0.001 in.).
For additional length,
add $0.16 per cm ($0.40/in.).

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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.

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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.
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26 +.....$9.00 ea.

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

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Standard length: 178mm (7 inches);
o.d. 20.00 ± 0.01mm;
i.d. 17.70 ± 0.02mm;
camber ± 0.03mm.
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SMIS Cuts Upgrade Costs!

London- Surrey Medical Imaging Systems will unveil a modular, upgradeable NMR Spectroscopy System at ENC available as a complete system or console only. David Taylor, President, said, "Just remove the RF In and RF Out from the existing probe, and plug in a new multiprocessor console."

By simplifying the installation to this extreme, the SMIS console option can compete favorably with upgrades to existing systems. The delivered system is itself upgradeable. Modularity guarantees that future enhancements will be cost effective as well.

SMIS is shipping a *MULTIPROCESSOR* system with **100 MIPS** of dedicated RF control and **75 MFLOPS** of dedicated vector and array processing -- a processor per NMR function. A PC/386 host platform with Microsoft Windows provides a point and click user interface, and an interface to standard utilities.

Field Tests Successful

Since last summer, the company has been testing the new consoles at several sites around Europe. The flexibility of a modular system has allowed quick configuration for a variety of system upgrades.

Field engineers for SMIS noted the ease of connecting a new console to an existing magnet. They indicated that their past experience trying to resurrect a dead computer console was often fruitless, and could be costly. In the end, the newly polished out-dated computer was acceptable based on familiarity, not performance.

The new multiprocessor systems being installed are easily upgraded. By adhering to AT/386 standards, SMIS takes advantage of the enormous popularity of the AT/386, and wide variety of off-the-shelf components.

SMIS plans to have a demonstration system installed in Cambridge, MA before the summer.

SMIS ENC Address: (April 1-5)
EMBERS Living Room
demonstrations and refreshments

Multiprocessor Systems

Using a backplane approach, SMIS has been able to surpass standard computer systems such as SUN and VAX. Each module on the backplane is highly optimized for its function, has its own memory, and operates without host intervention.

All the processors operate in *parallel*. Typical systems which *multitask* a CPU for several tasks can't keep up.

Hardware:
3x VECTOR 8500 75 MFLOPS
2x MR3020 Pulse Prgmr 100 MIPS
1x Host PC/386-20 4 MIPS

ref-Sun SPARC- 10 MIPS, 2 MFLOPS

Not a Typical Backplane System
Another innovation is SMIS's private busses for data acquisition and waveform store. Each processor can access its peripherals concurrently, without using the backplane. The host is a 19-inch rack-mount, 15 slot unit.

SMIS Welcomes New Faces

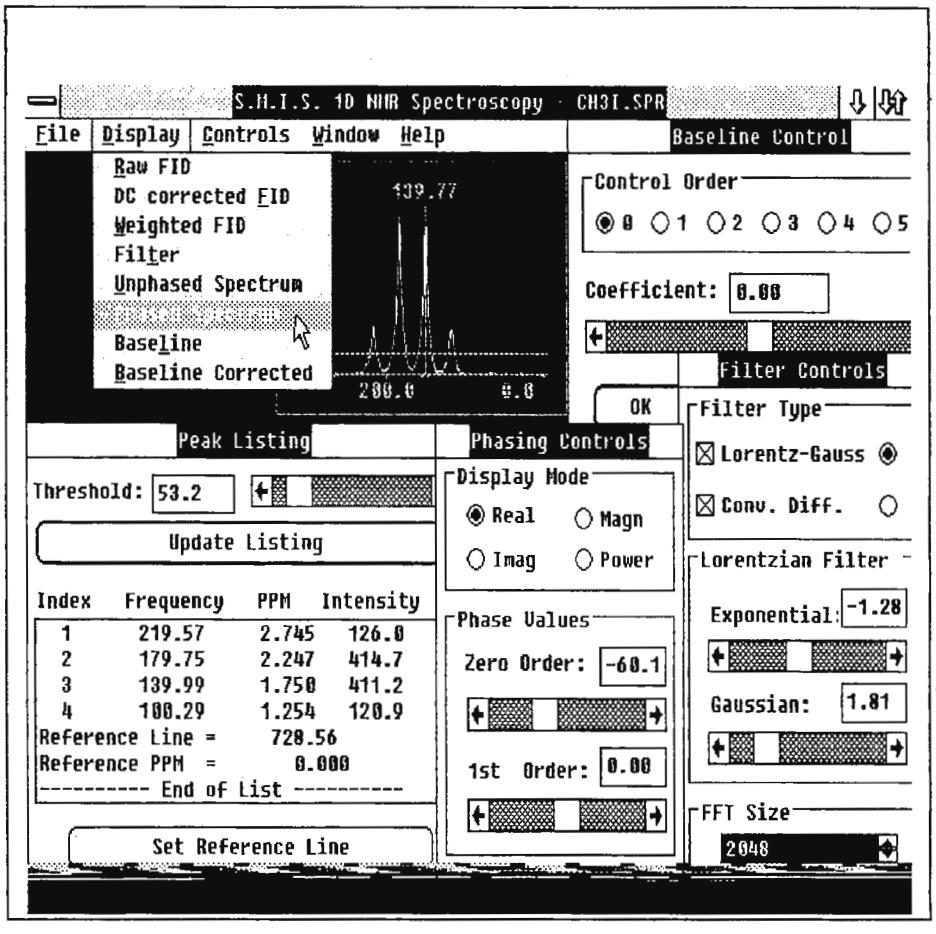
Graham Naylor - *NMR Scientist*
PhD in Composite Pulse NMR under Ray Freeman and Gareth Morris. 10 years NMR experience, including Picker International (MR cardiac flow imaging software).

Nick Bolas - *NMR Scientist*
D. Phil in NMR Spectroscopy with Geroge Radda, Oxford. 8 years NMR spectroscopy, specializing in *'in-vivo brain biochemistry'* and neonatal brain metabolism.

Bruce Schulman - *US operations Mgr.*
5 years Signal Processing experience, including lectures at major universities and industrial sites. Fellow, MIT-CAES, on-going NMR studies.

Windows Software Features

- efficient data presentation
- *pull-down* menus
- fill-in *dialog boxes*
- Dynamic Data Exchange to other windows and spreadsheets (reports)
- Windows Toolbox - write, paint, note communications
- Print Spooler (background print)

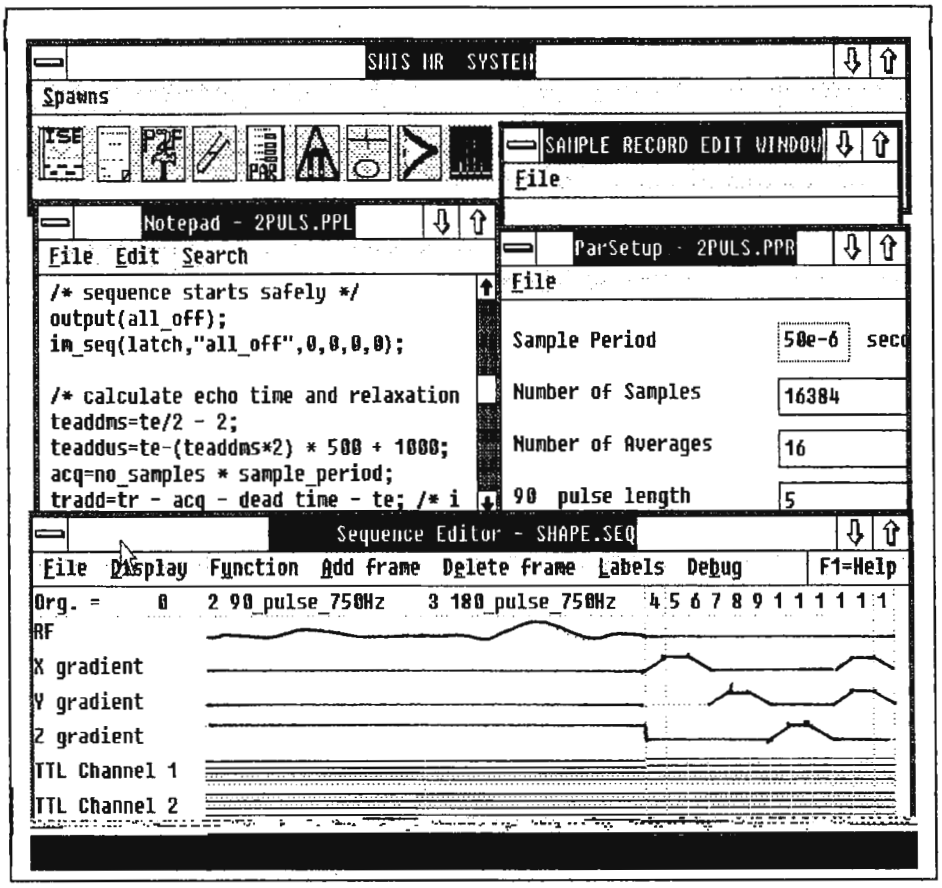


SMIS Announces a New Era in Sequence Control

Real-time 'LOGICAL' sequence control is accomplished through graphic frame editing and *C-like* macro programming. Some sample screens:

Details of the MR3020 and Peripherals

- Point/click user interface
- Graphical display of Pulse sequences
- 10 MIPS cont/ 50 MIPS peak RISC
- private bus to 4 waveform stores



VECTOR 8500 Array Processor and A/D board

- 25 MFLOPS performance (IEEE compatible- single precision)
- DSP-Link data acquisition interface (5 MHz sample rate)
- FFTs to 16k - 1D, 1kx1k - 2D complex floating point
- 8.5 MBytes RAM capacity

The VECTOR 8500 provides high speed data acquisition without interfering with PC operation. The data is gathered and averaged in real-time.

The user only has to press Window's buttons to activate the processor. Integration, windowing, scaling, type conversion, all with the same speed and floating point accuracy.

The FFTs are lightening fast--256x256

complex floating point FFT in 1.5 seconds. In 1-D, FFTs keep up with the screen update speed, so you can't tell that its recalculating, not just scaling the screen data.

SMIS wrote and maintains the array processor library, so new functions are easily added. A direct user interface is scheduled for introduction summer/fall of 1990.

Sequences are defined using the Pulse Programming Language. Waveform segments are graphically edited. Once the sequence is defined, the compiled program and waveforms can be downloaded to the MR3020 Pulse Programmer--Frame Sequencer.

Once loaded, the host only starts things happening, the MR3020 runs independently, out of its own program memory, signaling the host when the experiment is finished.

While traditional waveform store boards clock out a stored waveform, SMIS plays a programmed sequence of "Frames" which can loop (with parameter update), and vary time intervals in a wide variety of ways. Also, .225° phase control with any arbitrary value or sequence is possible. This advanced control is ideal for new experiments.

Pulse Programming Language

- subroutine calls (w/ parameters)
- loop, conditionals (nest to 128)
- arrays (phase-cycling, t_r stepping)
- logical operations
- simple arithmetic
- parameterized timing (delay, pulse)

MR3020 Pulse Programmer

- 500 kBytes (32K std) program RAM
- 8 TTL in
- 16 TTL out
- 100 ns time resolution (no jitter)
- 4 kBytes PC dual-port RAM

MR3030 Waveform Store

- 2 x 12-bit D/A (12-bit multiplier DAC for range)
- 8 TTL out (sync to D/A data)
- 32k x 32-bit RAM
- Arbitrary sequencing from MR3020

Data Acquisition

- 2 x 12-bit 100 kHz A/D (2 x 16-bit 100 kHz optional)
- sync 2 chan S/H -- 100 ps jitter
- self-calibration S/H circuits
- MR3020 triggered coherent sampling

SMIS USA Inc.
MIT Room 9-468
77 Massachusetts Ave.
Cambridge, MA 02139
Tele: 800-USA-SMIS

SMIS Ltd.
Alan Turing Road
Surrey Research Park
Guildford, England GU2 5YF
Tele: (0483) 506611
Fax : (0483) 63114

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8 February 1990
(received 2/17/90)

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

Dear Barry

Rapid P-31 Measurements in Perfused Hearts

Many thanks for your gentle reminder and herewith our attempt to stave off any more vigorous ones.

Frustrated by our attempts to get our pharmacologically-minded colleagues interested in drug discovery targets in the form of small, soluble protiens, we turned our minds to how we could nevertheless use NMR to say something about drug-receptor binding, even when the receptor was decidedly unobliging from a high resolution point of view. As a result, we decided about 3 years ago to enter the perfused organ NMR field with the purchase of a 8.43T 89mm magnet, to time share with an existing high resolution Bruker AM360. Peter Morris (Biochemistry Department, Cambridge) gave us a flying start in the hitherto mystical realms of probe construction and before long, to our great surprise, we were producing usable P-31 data acquired in ca. 10s time windows. This temporal resolution allowed us to characterise the dramatic but extremely brief depletion of phosphocreatine in Langendorff-perfused rodent hearts when they were challenged with the adrenergic agent isoprenaline (= isoproterenol). (Unitt J. F. et al., Biochem. J. 262, 293-301 (1989)). We have since performed a number of determinations of high energy phosphate (HEP) content in perfused hearts challenged with other inotropes (drugs which increase myocardial contractile force), and ischaemia. Emboldened by our success, we recently decided to see if we could measure the rate of interconversion of ATP and PCr on similar short time scales, during inotropic intervention. The results are typified by the spectra in the Figure. They were all acquired with 8 60° pulses, an interpulse delay of 1.2s giving a net acquisition time for a full f.i.d. of 11s, taking account of disk write time. Saturation of the Y-ATP signal was achieved with a DANTE sequence. The reductions in intensity of the PCr signal as a result of saturation transfer are quite obvious and we have been able to show that flux in both directions through creatine kinase increases about three fold during stimulation of the heart by isoprenaline.

Professor B L Shapiro

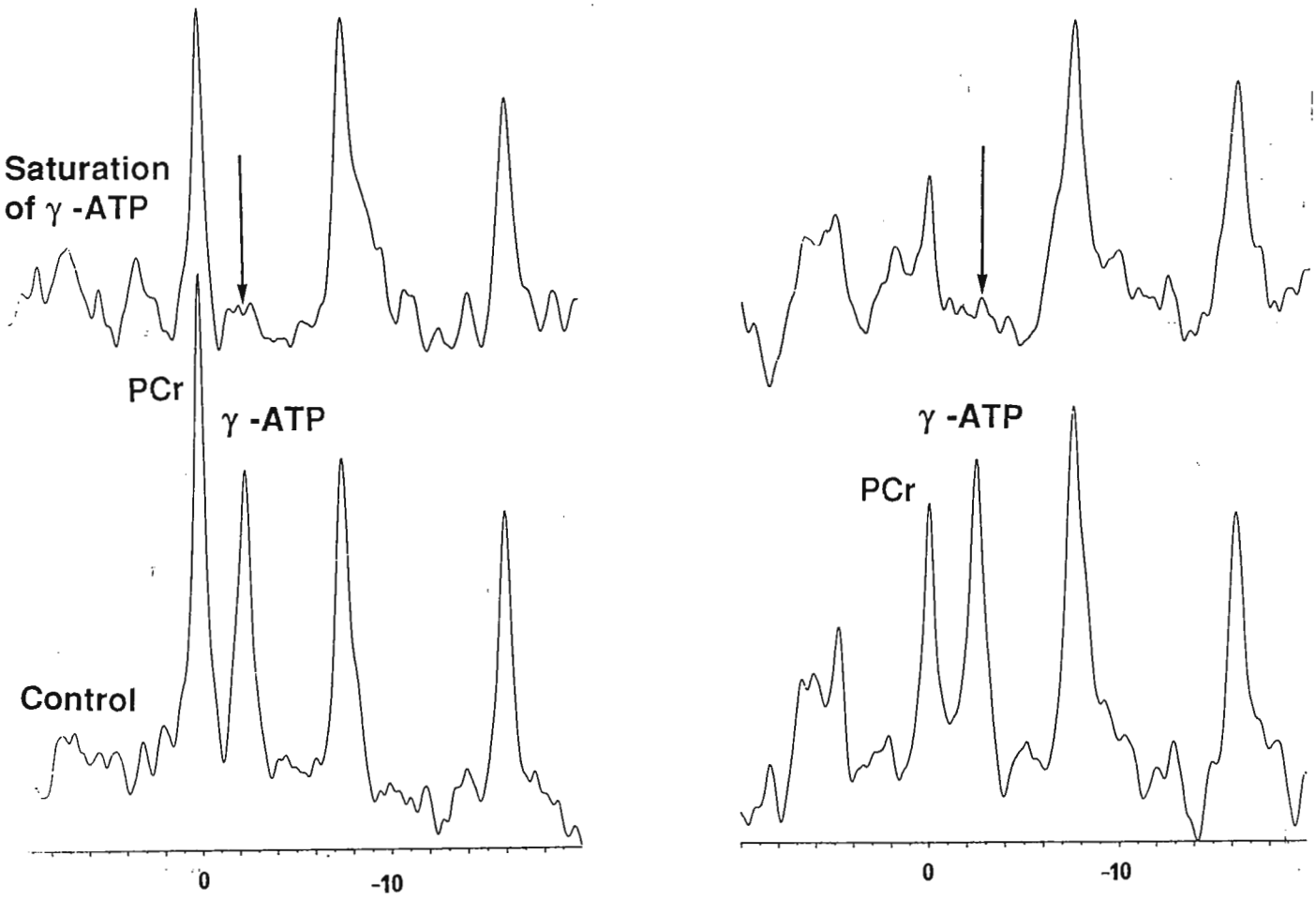
8 February 1990

Having conquered hearts, we are now starting on minds and assisting CNS pharmacologists with HEP and pH measurements in intact brains. We are indebted to our colleagues Paul England for pharmacological input and Dave Harrison, John Hare and Steve O'Connor in our Research Engineering Labs for knocking up and tuning probes.

Best wishes.

Dave Reid - Simon Robinson Lesley MacLachlan

David G Reid Simon Robinson Lesley MacLachlan



CONTROL HEART

STIMULATED HEART
(100 nM Isoprenaline)

ROCHE PRODUCTS LIMITED

RESEARCH CENTRE

30 January 1990
(received 2/6/90)

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

Dear Barry,

Dynamics of Potassium Channel Activators

We have reinvestigated the dynamic behaviour of the potassium channel activator cromakalim (I) together with some related compounds (II→IV), to attempt to elucidate the receptor-bound conformation. Previous nmr studies of (I) suggested that rotation about the bond joining the pyrrolidone ring to the benzopyran is prevented[1]. Evidence for rigidity was based on lack of temperature dependence of the ^{1}H spectrum of (I) in $CDCl_3$, n.O.e. difference experiments, and similar ^{13}C relaxation times in both rings. We believe that one conformation is overwhelmingly predominant in $CDCl_3$, but that rotation about this single bond is fast at ambient temperature.

We examined (I) in CD_3OD, in which two rotamers in the ratio 97.5:2.5 were observed at -60°C (Fig). Chemical shifts of the protons H-3 and H-4 show that the major isomer is (IA), with an energy difference $\Delta G°$ of 2.2 kcals/mole between (IA) and (IB) and an approximate energy barrier to rotation $\Delta G^{\#}$ of 11.5-13.5 Kcals/mole. This small energy difference does not preclude the higher energy rotamer from being that bound by the receptor. The barrier to rotation is raised to ~16 Kcals/mole in (II) where again rotamer (IIA) predominates (100% in $CDCl_3$, 88% in CD_3OD and d_6-dmso, and 91% in D_2O). The methyl group in (III) raises the barrier further, but not sufficient to stop rotation completely at 20°C. Finally compound (IV), a highly potent antihypertensive and bronchopulmonary agent being developed in our laboratories provides an interesting case of enantiomorphic behaviour. The methyl groups are prochiral and provide a good probe for studying the barrier to rotation, which is 13.0 Kcal/mole in CD_3OD solution.

This is a good example of a case where n.O.e. difference experiments and ^{13}C T_1 values do not provide criteria for defining conformational dynamics in small molecules. Variable temperature behaviour in more than one solvent is much more reliable.

Yours sincerely

W A Thomas

I W A Whitcombe

1. F Cassidy, J M Evans, D M Smith, G Stemp, C Edge and D J Williams Chem. Communications, 1989, 377

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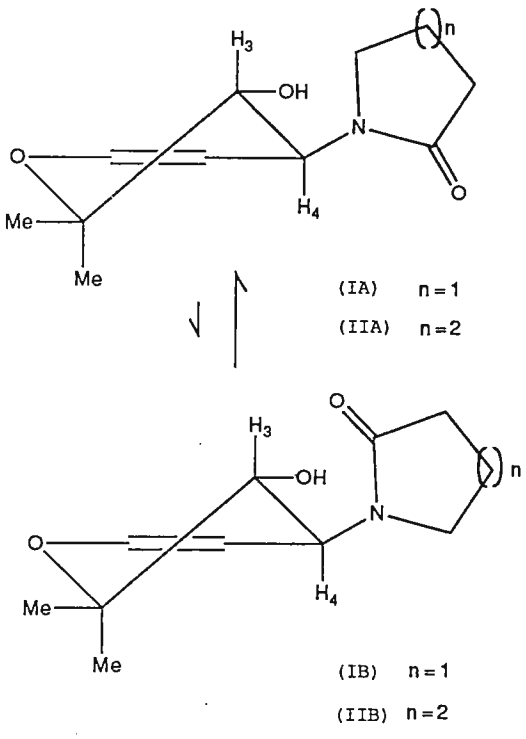
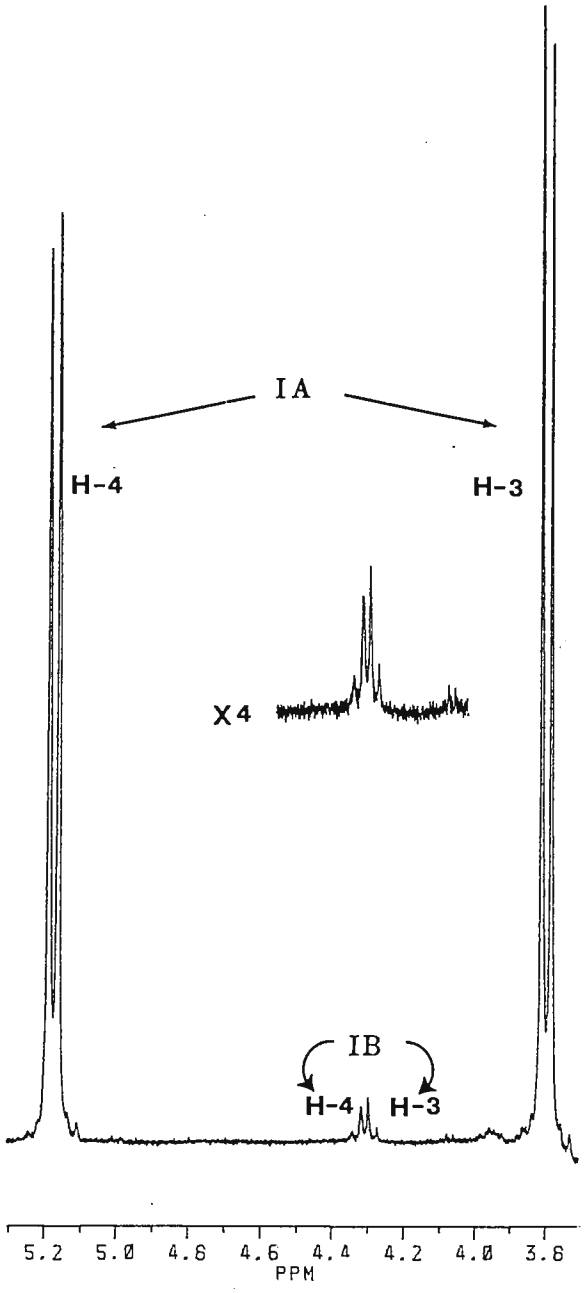
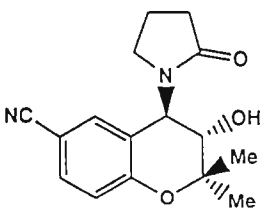
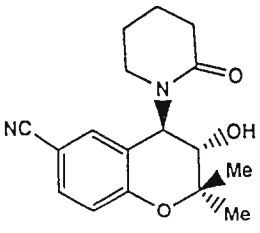




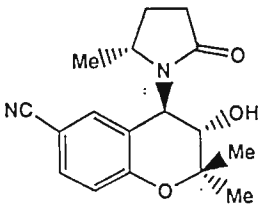
400 MHz spectrum of (I), CD_3OD, 213^0 K



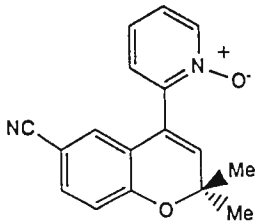
(I)



(II)



(III)



(IV)

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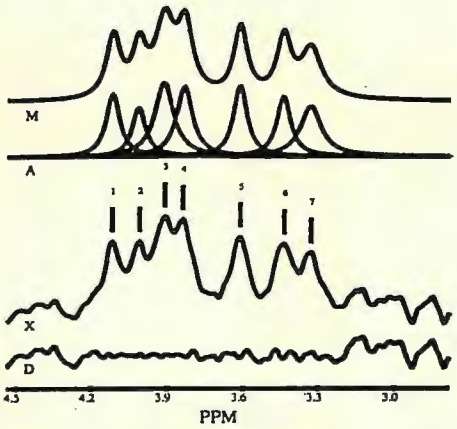
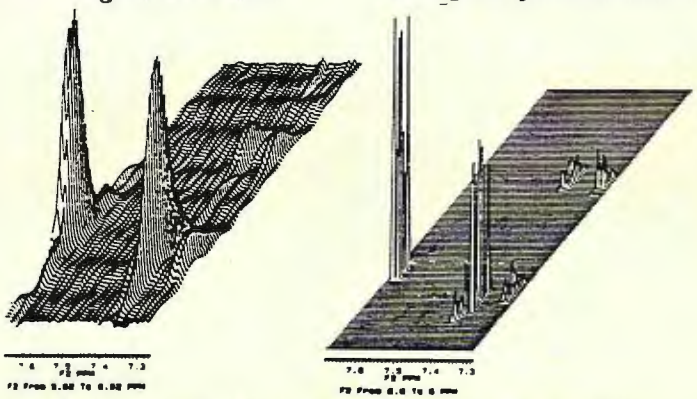
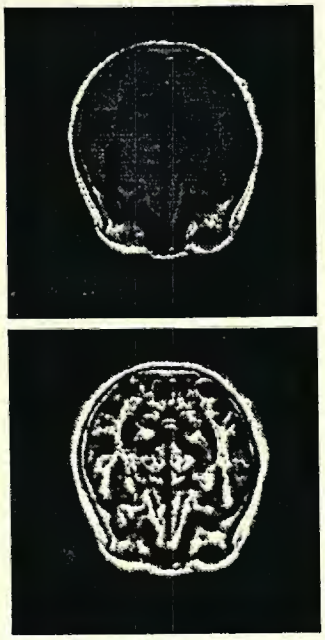


Totally automatic curve fitting

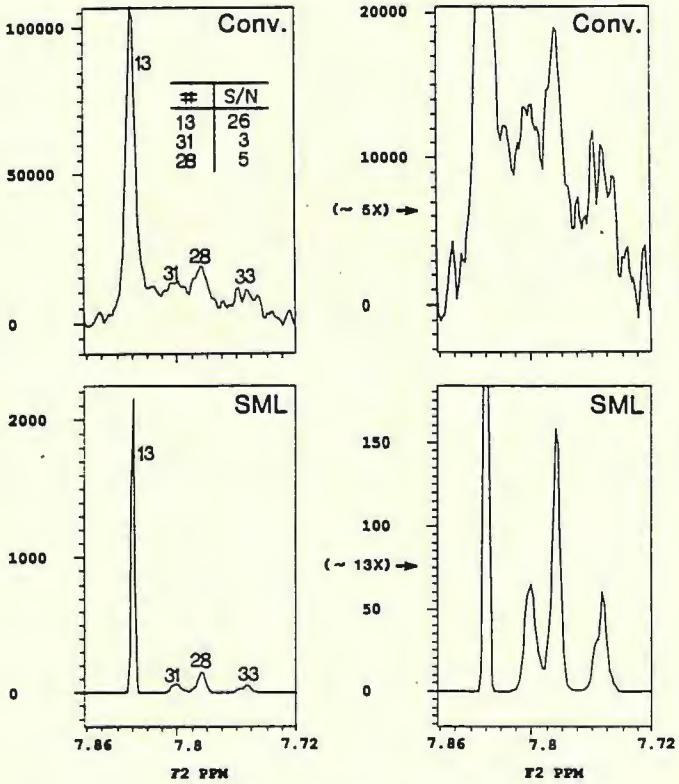
Original FFT data *MLM plus symmetrization*



MLM (Maximum Likelihood) Spectral Deconvolution for 2D NMR. Zoom region of DNA duplex (500 MHz NOESY spectrum). Quantitatively resolve NOESY cross peaks and observe spin–spin couplings.



An example from MRI/IMAGE™. Automatic adaptative histogram equalization (bottom) enhances image contrast.



Another view of 2D MLM. These pictures show FFT (conventional) and symmetrized MLM (SML) protocols on a 2D spectrum. These are views of a single representative slice with four closely spaced peaks (right side–vertical expansions).

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 February 1990

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February 6, 1990 (received 2/12/90)
Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

^{19}F MAS-NMR OF Sb-DOPED FLUOROAPATITE PHOSPHORS

Dear Barry:

Four new Varian NMR spectrometers and a Sun SPARCI datastation were installed at Michigan State this year in time for the dedication on October 20 of the Max T. Rogers NMR Facility. We have generally been pleased with the performance of the instruments, the software, and the usefulness of computer networking; recently we have added on a 600Mbyte optical disk from Pinnacle. I thought it might be interesting to show some results we have obtained from the VXR-400 spectrometer devoted to solid state studies, obtained with a 5mm high-speed ^{19}F/^{1}H MAS probe from Doty Scientific.

Many useful materials have the apatite lattice structure: bones, teeth, and the phosphors in most fluorescent lights! In addition to continuing interest in the former, we are collaborating with Dr. Jefferey Berkowitz of GTE Products to investigate the location of the antimony "activator" in Sb^{3+}-doped fluoroapatite ($Ca_5F(PO_4)_3$), which emits visible light upon UV-irradiation in a lamp. Although such phosphors have been used for decades, the site of the Sb substitution has remained controversial.

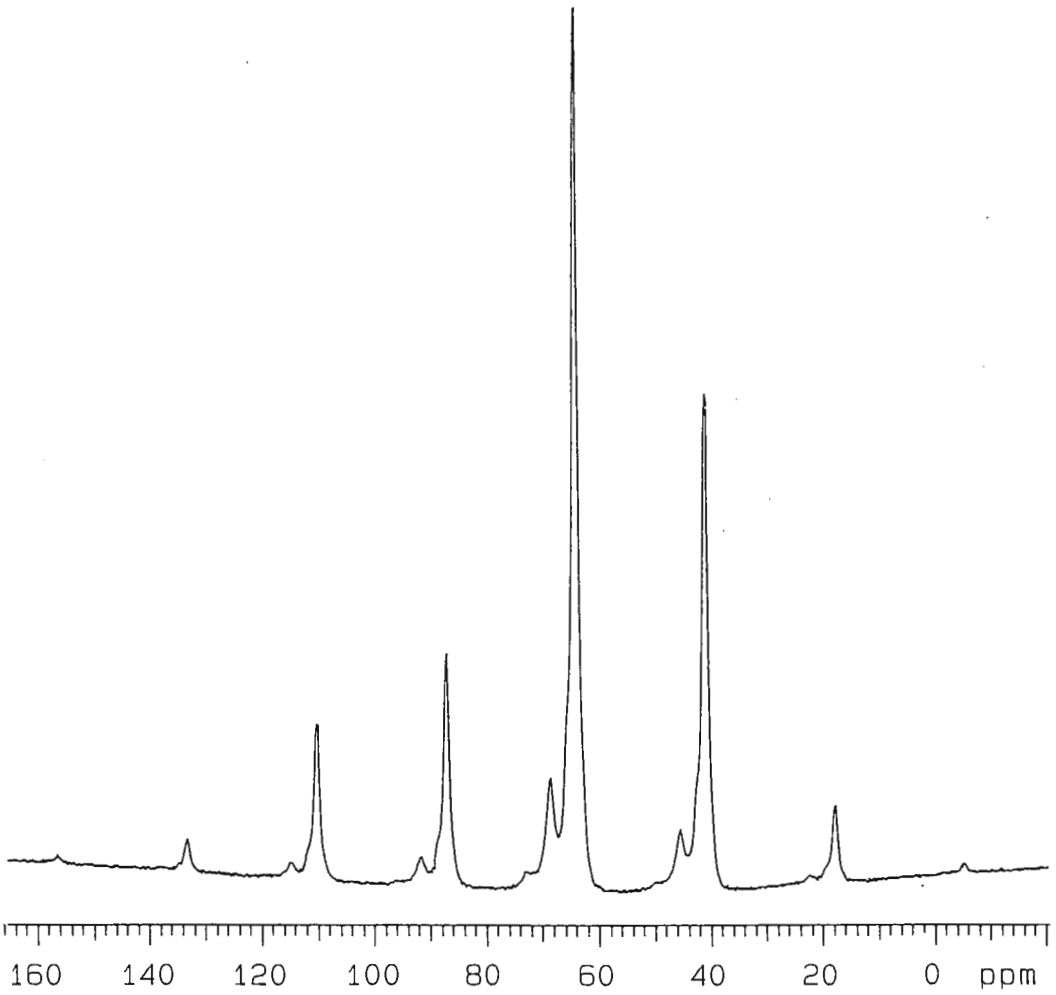
^{19}F MAS-NMR spectra of these samples, containing 0.18% to 3.1% Sb, are very revealing. First of all, the half-height linewidth of the main (unperturbed) fluoroapatite peak in these samples is only 0.7ppm, five-fold sharper than previously observed for fluoroapatite samples, suggesting that crystallite size effects or defects may influence the linewidths. The presence of an infinite linear chain of fluorine spins in the apatite structure means that magic-angle spinning alone, without CRAMPS, produces sharp spectra. Secondly, a distinct downfield peak at 68.6ppm, as well as a weaker peak around 73ppm and shoulders on the main peak, are observed with varying intensities in the Sb-doped apatites (see Figure).

We believe that the 68.6ppm peak arises from two fluoride ions in the apatitic lattice that are perturbed by the Sb substitution, based on relative intensity measurements. The T_1 relaxation times of the 68.6ppm peak are identical to those of the main 64ppm peak in a given sample, implying that spin-diffusion along the linear chain is equalizing the T_1 values (which are minutes). Surprisingly, doping with Sb results in *longer* T_1 values, perhaps by producing fluorine vacancies along the linear chain that inhibit spin diffusion to the rare paramagnetic impurities. The absence of significant perturbations in the ^{31}P MAS-NMR spectra tend to support a model in which an $Sb(III)O_3^{3-}$ group replaces a PO_4^{3-} group, but one would then expect to see at least two types of perturbed fluorine atoms in a 2/1 ratio. Broadening of the 68.6 ppm peak by the quadrupolar Sb nucleus with its large NQCC does not seem to be significant, perhaps because of rapid quadrupolar relaxation. Quantitative "spin-counting" experiments can provide additional information, but it is difficult to obtain results reproducible to within a few percent. We are currently measuring T_2 values using Hahn spin echoes and the rotor synchronization feature of the VXR-400 to see whether changes are produced by Sb substitution.

Sincerely yours,

James P. Yesinowski

Liam Moran



^{19}F MAS-NMR Spectrum at 376 MHz of $Ca_5F(PO_4)_3$ Doped with 2.1% Sb (Spinning Speed = 8.8 kHz). The strong central peak at 64.0 ppm (from C_6F_6) and its spinning sidebands are characteristic of fluoroapatite; the weaker peak at 68.6 ppm arises from fluorines perturbed by Sb incorporation.

POSTDOCTORAL POSITION. Available immediately to study molecular structure of plant cuticle polyesters. Requires Ph.D. in biochemistry or chemistry, with experience in one or more of the following areas: modern spectroscopy (NMR in solution and solid state, FT-IR); biosynthesis, biodegradation, and isolation of plant materials; analytical methods (GC-MS, column chromatography). Send a *curriculum vitae*, two letters of reference, and copies of relevant publications to Dr. Ruth E. Stark, Department of Chemistry, City University of New York, The College of Staten Island, 50 Bay Street, Staten Island, NY 10301. EO/AA Employer.

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Massachusetts General Hospital
NMR Center
149 13th Street
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February 12, 1990
(received 2/16/90)

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

Multinuclear MRI of Inorganic Solids

Dear Barry:

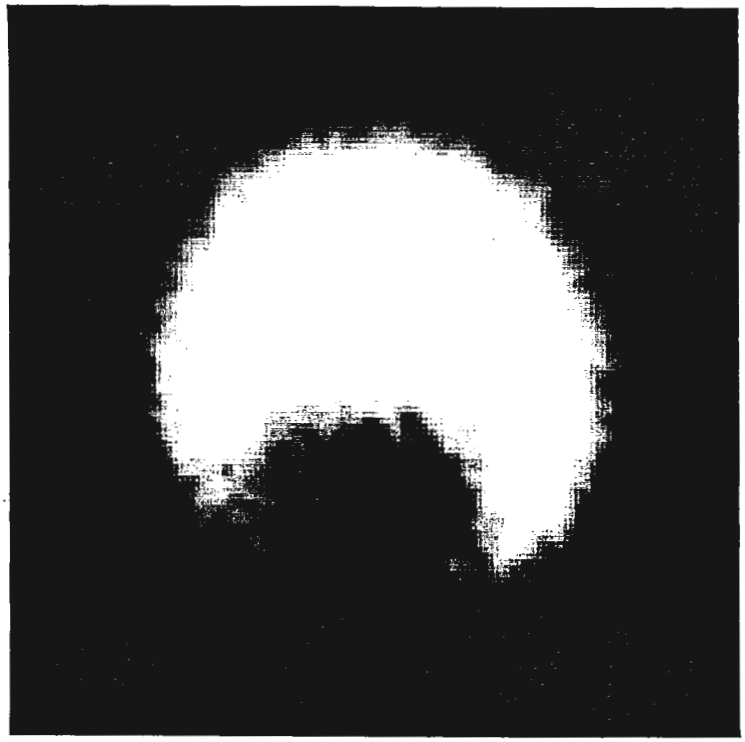
We are continuing our development of magnetic resonance imaging of polymeric and ceramic materials, and have extended our efforts to encompass the hard inorganic phases of the ceramics.

Using small, efficient and low inductance magnetic field gradient coils, it is a straightforward process to generate echo times of 200 μs, and field gradients above 200 G cm^{-1}. It now becomes possible to examine a wide variety of nuclei—both spin 1/2 and quadrupolar—in the solid state. For example, we have produced complete two-dimensional images of bioceramic materials (^{31}P resonance in hydroxyapatite, $Ca_{10}(OH)_2(PO_4)_6$, and brushite, $CaHPO_4{\cdot}2H_2O$), aluminum oxides (^{27}Al resonance in γ-Al_2O_3 and an aluminosilicate), and borosilicate glass (^{11}B resonance).

The figure shows the ^{27}Al image in a 5 mm tube packed with γ-Al_2O_3 powder, into which an empty 1 mm tube has been inserted to provide a visual feature. No slice selection was used. The pulse sequence was a TE = 200 μs spin echo with pulsed comp and readout gradients of 225 G cm^{-1}. The image was reconstructed by backprojection from 32 views, and took a little under 2 hr to acquire. Taking into account the linewidth of the central transition of this quadrupolar nucleus (roughly 13 kHz at our 6.0 T field strength), the true spatial resolution is about 500 μm.

The great advantage MRI holds over other methods of nondestructive evaluation is its exquisite sensitivity to chemistry, of course, and we endeavor to develop pulse sequences to highlight chemical distinctions within these materials. In the bioceramics, by comparing cross-polarized with non-CP sequences, we can produce cleanly separated images of the PO_4^{-3} and HPO_4^{-2} distributions. In the quadrupolar systems, we can base chemical discrimination upon the differential response to varying pulse lengths due to differences in nutation frequencies.

378-56 A more detailed report will appear in Ceramic and Engineering Science this summer.



James R. Moore

Leoncio Garrido

Jerome L. Ackerman

Call for Papers

Advanced Tomographic Imaging Methods for the Analysis of Materials

Symposium "U" at the 1990 Fall Meeting of the Materials Research Society
November 26 - December 1, 1990
Boston, Massachusetts

This is the first symposium by MRS specifically devoted to new tomographic imaging techniques applied to the study of materials. Contributions in all aspects of magnetic resonance, computed tomography and holography (including theoretical papers) in which materials science applications are important are welcomed. Contributions are solicited in the following general topics areas, as well as in areas not listed but which are appropriate to the theme of the Symposium:

- **Magnetic Resonance Imaging**
- **X-ray (Including Synchrotron Radiation), Neutron, Isotope Computed Tomography**
- **Ultrasound, Electron Microscopy, Thermal, Laser CT**

Invited speakers include **H. Deckman** (Exxon) *High Resolution Synchrotron X-Ray Tomography;* **A. Devaney** (Northeastern University) *Diffraction Tomographic Imaging Using Ultrasound, Coherent Light and Soft X-Rays;* **W. Edelstein** (General Electric) *NMR Imaging/Analysis of Oil Cores;* **L. Garrido** (Massachusetts General Hospital/Harvard Medical School) *NMR Imaging of Polymeric and Composite Materials;* **A. Garroway** (Naval Research Laboratory) *Line Narrowing Approaches for Solid State NMR Imaging;* **J.Greenleaf** (Mayo Clinic) *Acoustic Tomography;* **J. Kinney** (Lawrence Livermore Laboratory) *Microtomography Using Synchrotron Radiation;* **J. Koenig** (Case Western Reserve University) *NMR Imaging of Adhesives;* **R. Komoroski** (University of Arkansas) *NMR Imaging of Elastomers and Porous Media;* **D. Kupperman** (Argonne National Laobratory) *Neutron Tomography;* **M. Vannier** (Mallinckrodt Institute of Radiology) *3D X-Ray Imaging.*

Abstracts (an original and two copies) must be received at MRS Headquarters, marked "ABSTRACT ENCLOSED," 9800 McKnight Road, Pittsburgh, PA 15237, not later than July 1, 1990. Abstracts should be prepared according to the new MRS format. Full length camera-ready manuscripts will be refereed at the meeting and will be published in the MRS Symposium Proceedings Series.

Symposium Cochairs

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TAMU NMR Newsletter

Instructions to Contributors: Practical Considerations

1. Technical contributions should be on the *minimum* (NOTE!!) number of 8.5 x 11" (21 x 27.9 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 do not routinely do reductions of figures or tables.

2. 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" (25.4 cm) from top to bottom.*

3. Significant savings of Newsletter pages and total space (and hence, subscription costs) can be made by exercising close control over the formatting and type sizes of the contributions. **Please consider the following:**

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

b) **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.

c) **AVOID DOUBLE SPACING BETWEEN LINES 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.

4. '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.

5) Please provide short titles of all topics of your contributions, so as to ensure accuracy in the table of contents. This will also avoid titles created on the run by me, frequently without much serious or solemn thought.

Contributions which fail to meet any of these specifications may be returned for re-working.

Thank you! Your cooperation in making the Newsletter economical and tidy-looking will be appreciated, as will your efforts to keep my work to a reasonable level.

B. L. Shapiro
1 March 1990

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

Continued from page 1:

FORTHCOMING NMR MEETINGS

Workshop on *In Vivo* Magnetic Resonance Spectroscopy III, **March 29 - April 1, 1990,** San Francisco, CA.; Contact: Dr. M. W. Weiner or Dr. G. B. Matson, Magnetic Resonance Unit, Veterans Administration Medical Center, 4150 Clement Street (11D), San Francisco, CA 94121; (415) 750-2146.

31st ENC (Experimental NMR Conference), **April 1-5, 1990;** Asilomar Conference Center, Pacific Grove, California; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667; Attendance: 1,200.

Industrial Applications of MRI, a one-day tutorial/workshop ", Boston, Mass., **April 20, 1990;** See Newsletter 378, 9.

Frontiers of Polymer Characterization by NMR Spectroscopy, Symposium at the American Chemical Society National Meeting, **April 22-27, 1990,** Boston, Mass.; See Newsletter 372, 54.

10th European Experimental NMR Conference, **May 28 - June 1, 1990;** Veldhoven, The Netherlands. Contact: M. J. A. de Bie: see Newsletter 376, 26.

Workshop of Special Topics in Medical Magnetic Resonance, sponsored by the Society of Magnetic Resonance in Medicine and the National Research Council of Canada, **July 23-27, 1990;** Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Expanding Frontiers in Polypeptide and Protein Structural Research, sponsored by the National Research Council of Canada, **July 23-27, 1990;** Whistler Mountain, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Tenth International Biophysics Conference, sponsored by the International Union of Pure and Applied Biophysics and the National Research Council of Canada, **July 29 - August 3, 1990;** Vancouver, BC, Canada. Contact: L. Forget - see Newsletter 374, 46.

Bat-Sheva Workshop on New Developments and Applications in NMR and ESR Spectroscopy, **October 14-24, 1990,** Israel; Contact: Dr. D. Goldfarb, The Weizmann Institute of Science, Rehovot, Israel. See Newsletter 377, 10.

Advanced Tomographic Imaging Methods for the Analysis of Materials, Symposium at the Fall Meeting of the Materials Research Society, Boston, Mass., **Nov. 26 - Dec. 1, 1990;** See Newsletter 378, 57.

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence

Should Be Addressed To:

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

(415) 493-5971 *

DEADLINE DATES

No. 380 (May)---------- 20 April 1990
No. 381 (June) ---------- 18 May 1990
No. 382 (July) ----------20 June 1990
No. 383 (August) ----------21 July 1990

* * * * *

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B.L.S.

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CSI 2T Applications

Shielded Gradients and NMR Microscopy

In spin warp imaging, there is a trade-off between minimum TE and maximum resolution. Even if rise and fall times were zero and phase encoding occured during the entire echo delay, a ±2 Gauss/cm gradient range and a TE of 2 msec would provide best case resolution of 0.32 mm. This translates to a 7 cm field of view in a 256 × 256 matrix. To improve resolution by a factor of 10, TE may be increased by a factor of 10 (which is not acceptable in a sample with short T2 values) or gradient strength may be increased by a factor of 10. The long echo times required for T2 weighted images create an undesired loss of signal in many non-T2 weighted image experiments. These effects, however, are tolerable at 2 Gauss/cm for resolution at the 100-200 micron level.

Clearly, added signal that would be available with a shorter TE would be useful. The current practical limits of high signal-to-noise NMR micro imaging are greatly reduced by high strength shielded gradients. A 50 micron resolution image of an Agapanthus bud is shown in Figure 1. Unlike very high field (>7 Tesla) micro NMR imaging, magnetic susceptibility effects at 2T do not compromise the 50 micron digital resolution obtained during these gradient strengths.

In a second example, (Figs. 2 and 3), 25 micron resolution is achieved in a small phantom by using a moderate access (5 cm) rf coil. The phantom consists of seven small capillary pipets in a 5 mm NMR tube. Data was collected as a 32 × 256 × 256 DEFT data set.

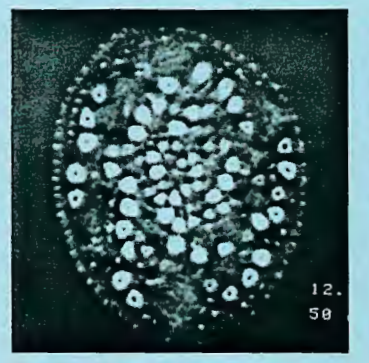

Fig. 1—Agapanthus bud Matrix 256 × 256, TR 200 Slice 2 mm, TE 30 FOV 12.8 mm, NEX 4, 45° Tip Angle DEFT Sequence

Fig. 2—16 contiguous 1 mm slices FOV 6.4 mm, NEX 4. TR 150 msec, Field Strength 2T, TE 14 msec

Fig. 3—Expanded view of four of the 16 slices shown in Fig. 2.

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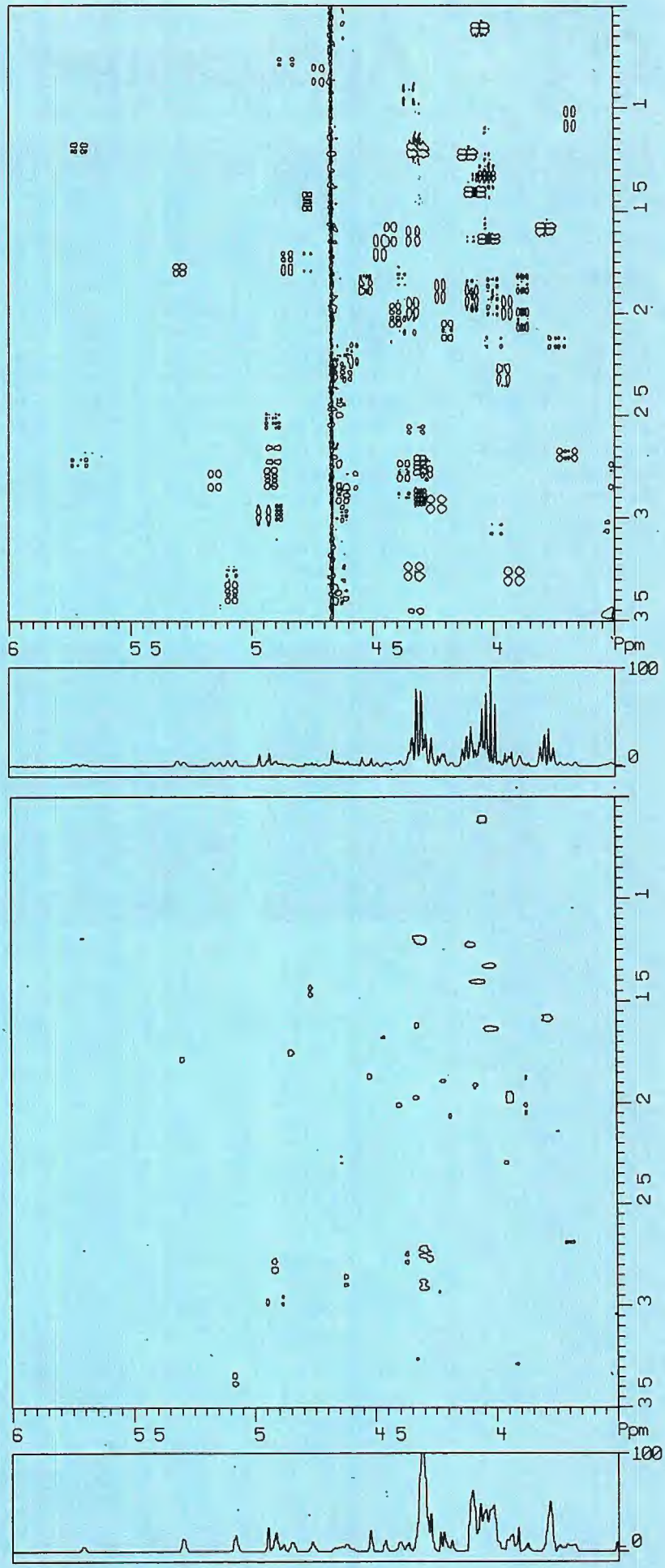
In addition to allowing for off-line processing, VPLX offers advanced functionality such as MEM/LPZ and Symmetry Filtering. The top data shows the normal NH to alpha region in a double quantum filtered COSY of BPTI in water. This matrix was produced on a GSX-400, processed on VPLX, and printed on a laser printer. The bottom data is identical to the first with the exception that a symmetry filter has been applied to the matrix. This symmetry filter discriminates on the basis of the known phase relationship of true COSY peaks. Each of the COSY peaks that passes through the filter is reduced to a centroid representation.** This filtering allows for the rapid elimination of spurious cross peaks and is the first step necessary for computer based spectral interpretation.

For more information, contact JEOL.



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