TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 379

April 1990

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isotopic Purity (%D)	Density (g/cm³)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetic Acid-d_4	CD_3COOD	99.5%	1.1	17	118	0.551 (32)
D-120	Acetic Acid-d_4 TFA		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	Choloroform-d	$CDCl_3$	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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Cat. No.	Paper Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	HA-100, HA-100D	Gridded-Two Color
WCV-60 (A-60C)	11" X 26"	A-60, A-60A	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100	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-600	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4" X 17"	CFT-20, FT-80, FT-80A All Models	Gridded-Two Color Blank

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Continued on page 63

VRIJE UNIVERSITEIT
DEPARTMENT OF PHYSICAL
AND THEORETICAL CHEMISTRY
FACULTY OF CHEMISTRY

De Boelelaan 1083
1081 HV Amsterdam
The Netherlands
dr. J. Bulthuis

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

your ref. | your letter dated | our ref. | date 5th Febr. 1990
(received 2/24/90)

"Mysterious orienting forces"

Dear Dr. Shapiro,

In ^2H relaxation studies of probe molecules in liquid crystals it is possible to determine the contributions from the spin density components J_1 and J_2 to T_1, by applying aselective pulse sequences to the quadrupolar doublet. In many cases the application of a Jeener-Brockaert pulse sequence is the best choice. It allows accurate measurement of the relaxation times of Zeeman and quadrupolar order by a single experiment. This has been convincingly demonstrated and successfully applied by Profs. R.L. Vold and R.R. Vold already years ago [1].
When studying deuterated toluene in a nematic crystal (Merck's Phase V) we found that J_1/J_2 for the ortho- and meta deuterons is smaller than unity, namely about 0.85 and J_1/J_2 for the para- and methyl deuterons are 1.65 and 1.80 respectively. In a normal, isotropic liquid this would mean that the extreme narrowing condition is not fulfilled, although one might not expect ratios smaller than 1. Here, however, the extreme narrowing condition does apply.
To explain the data, we have applied the model of asymmetric rotational diffusion in an orienting potential. This potential is assumed to be fully defined by the two orientation parameters that describe the average orientation of toluene.
Unfortunately no set of diffusion constants $\{D_{xx}, D_{yy}, D_{zz}\}$ could be found that fit the experimental data. It is possible indeed to find a ratio $J_1/J_2 < 1$ for the ortho/meta deuterons, but then the J_1/J_2 values for the para- and methyl deuterons are far too small. This works also the other way around. A set of diffusion constants that gives a compromise between these extremes, and thus a poor fit, is the following one:

	$D_{xx}(10^{10}s^{-1})$	$D_{yy}(10^{10}s^{-1})$	$D_{zz}(10^{10}s^{-1})$	$(J_1/J_2)_p$	$(J_1/J_2)_{o,m}$	$(J_1)_{o,m}/(J_1)_p$
calc.	1.0 (ass.)	0.025 (ass.)	1.25 (ass.)	1.32	1.06	0.46
exp.				1.65	0.86	0.46

The molecular x-axis is taken perpendicular to the aromatic ring and the z-axis is along the C-methyl axis. It should be noted that the anisotropy of the viscosity of the solution has not explicitly been taken into account, but it remains to be seen if this could explain the poor agreement between theory and experiment. A full account of this work has recently been submitted for publication.

Yours sincerely,

Dr. J. Bulthuis

[1] see e.g., R.L. Vold, "Nuclear Magnetic Resonance of Liquid Crystals", Ed. J.W. Emsley, NATO ASI Series, Vol. 141 (Reidel, Dordrecht, 1985) p. 231, and R.R. Vold, Ibid., p.253.

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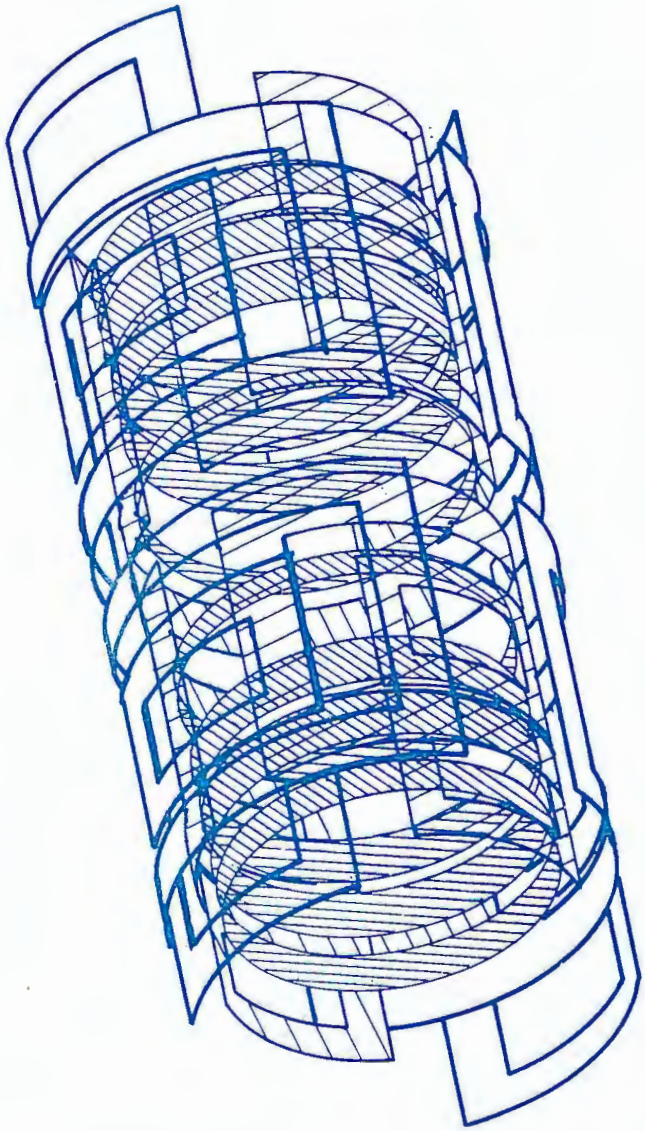

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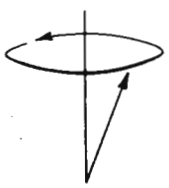
- **1987** First **7 mm 9 kHz** spinning
- **1988** First **6 Kelvin** MAS
- **1988** First **650°C** MAS
- **1989** First **DAS** probe
- **1990** First **?**.



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March 8, 1990
(received 3/12/90)

Dr. B. L. Shapiro
Texas A&M NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

RELAY in the Rotating Frame - Applications

Dear Dr. Shapiro:

Many industrial analytical laboratories are heavily involved with the analysis of competitors' materials. NMR analyses of these materials often present difficult problems, one reason being that the spectroscopist may have no idea of the products' contents. The first step is to obtain ^{1}H, ^{13}C, and DEPT spectra; however these data are not sufficient to identify the components of complex mixtures. 2D methods then become invaluable. One particularly useful technique that is often overlooked is Rotating Frame RELAY[1]. We find it to be more sensitive than the other RELAY versions, and it is especially effective for oligomeric/polymeric materials where short T_2^*'s present problems for pulsed RELAY methods. I include a listing of our Bruker program below, which gives good quadrature detection in F-1 and no axial peaks.

Very truly yours,

Gary P. Juneau
Research Associate
Central Analytical Dept.

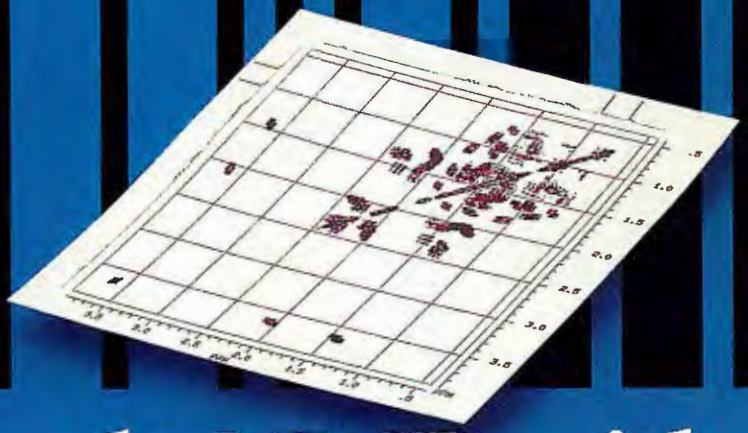
GPJ/skp
Attachment

1. A. Bax, D. G. Davis, S. K. Sarkar, J. Magn. Reson., **63**, 230-234 (1985).

```
LIST TOCHC.AU

------ FILE: TOCHC   .AU

; TOCHC.AU
;  RELAYED COHERENCE SPECTROSCOPY IN THE ROTATING FRAME


1 ZE
2 D1 S1 DO
3 P1:D PH1
3 DO
4 P4 PH4
5 DO
6 D4 S3          ; ALLOW TIME TO SWITCH DCPL POWER
7 (P5 B1 P5 B3 P5 B1 P5 B3 P5 B1 P5 B3 P5 B1 P5 B3):D   ;SPIN LOCK
8 D3 S1
9 P2:D B0 P4 A0
10 D3
12 P3 A0 P1:D PH5              ; XFER H POLIZARIZATION TO C'S
14 D3 S2                  ; ANTIPHASE C MAGNETIZATION COMES IN PHASE
15 GO=2 BB PH6
   D3 DO
16 WR #1
17 IF #1
18 IN=1
19 EXIT


PH1= B0 B3 B2 B1
PH2= B1
PH3= B3
PH4= A0 A0 A0 A0 A2 A2 A2 A2
PH5= B1
PH6= R0 R1 R2 R3

; D4= 400 USEC, S3= 10H, P5 =4000 USEC
; TOTAL SPIN LK TIME = 8*P5
;ND0=2 , P1 AND P2 ARE 90 AND 180 DCPL PULSES
; P3 & P4 ARE 90 &180 C PULSES  D1 = 1-5 T-1'S FOR H
; D3=1/4J OR 2MSEC
; PW & RD =0
```

Start NMR Experiment

^{13}C



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Insert Sample

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enter your user code

USER 1

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experiment-solvent-combination

Experiment/Solvent

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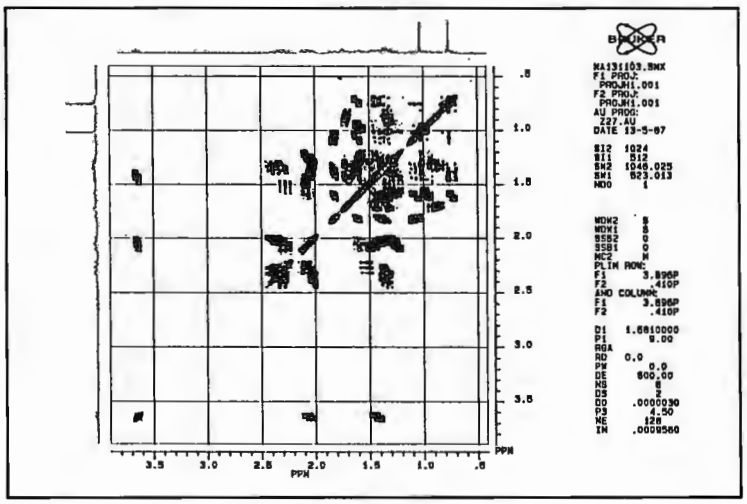

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Prof. B.L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303
U.S.A.

Nijmegen, 27 February 1990
(received 3/8/90)

Ti MAS NMR in solids.

Dear Prof. Shapiro,

Recently, we have registered an increased interest for Ti-NMR in solids. For instance, the possibility to create zeolites containing Titanium has aroused the interest about the capability of Ti-NMR for the identification of these materials. Titanium has two "NMR-active" isotopes, ^{47}Ti (I=5/2) and ^{49}Ti (I=7/2), with a natural abundance of 7.28 and 5.51 % respectively. They both have a low gyromagnetic ratio and a medium quadrupole moment [$Q(^{47}Ti)=0.29\times10^{-28}$ m^2, $Q(^{49}Ti)=0.24\times10^{-28}$ m^2]. This makes them in principle unattractive nuclei for NMR, and it is not surprising that hardly any literature exists on the subject. However, as the Dutch National HF-NMR facility gives us access to a 500 and 600 MHz spectrometer for solid state MAS NMR, we thought it was worthwhile to try to observe Ti in solids. Taking the receptivity and the spinfactor into account, the central line of both isotopes should have approximately the same intensity. However, when the line width is dominated by second-order quadrupolar interactions, the line of ^{47}Ti will be 3.43 times broader than for ^{49}Ti.

Fig. 1 shows the Ti spectrum of $SrTiO_3$ obtained on our AM 500. $SrTiO_3$ has the perovskite structure, with Ti in a perfectly symmetrical octahedral coordination and thus quadrupolar interactions should play a minor role. Indeed, two narrow lines of approximately equal intensity are observed which are assigned to the central transition for the ^{47}Ti and ^{49}Ti (0 ppm) resonance. Furthermore, we see spinning sidebands from the other transitions which are broadened in first order by quadrupolar interactions. The main problem we have met with obtaining this spectrum is the strong acoustic ringing from the probehead. Thus spectra had to be obtained using an echo sequence or a large receiver deadtime. Fig. 2 shows the Ti spectrum of TiO_2 obtained on the AM 600. The commercially available TiO_2 sample consisted of multiple phases. The octahedral coordination in the various TiO_2 phases is no longer perfectly symmetrical, and thus quadrupole interactions will play an important role. The spectrum displays two lines with a two to one intensity ratio and a splitting that does not correspond to the frequency difference of the two isotopes. We believe that the two lines are ^{49}Ti resonances of two different TiO_2 phases, indicating the sensitivity of the Ti chemical shift for small structural changes. The ^{47}Ti resonances are probably lost due to the larger line width in combination with the large receiver

deadtime used. After these first hopeful results, we are currently investigating the possibilities of Ti MAS NMR for the study of Ti containing zeolites.

Sincerely yours,

A.P.M. Kentgens G.H. Nachtegaal

Please credit this contribution to the account of Prof. E. de Boer

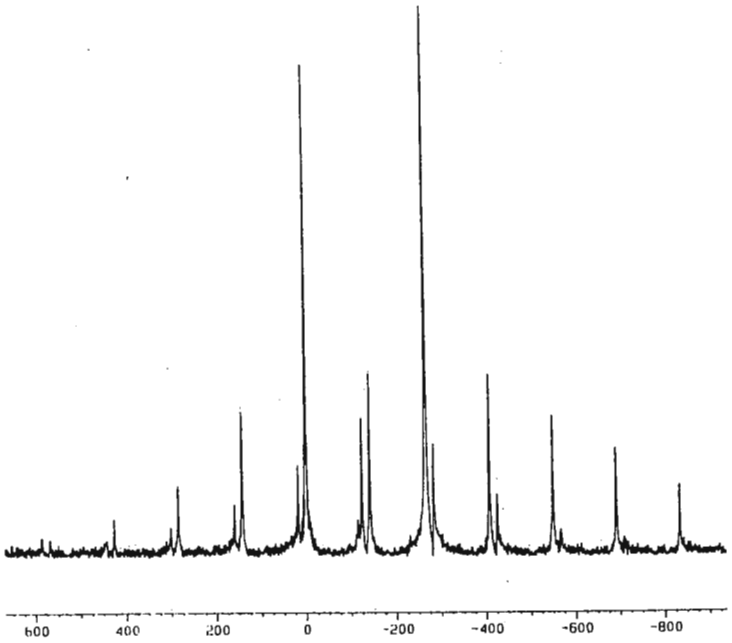

Figure 1. Ti spectrum of $SrTiO_3$ obtained at 28.17 MHz (4000 Hz spinning).

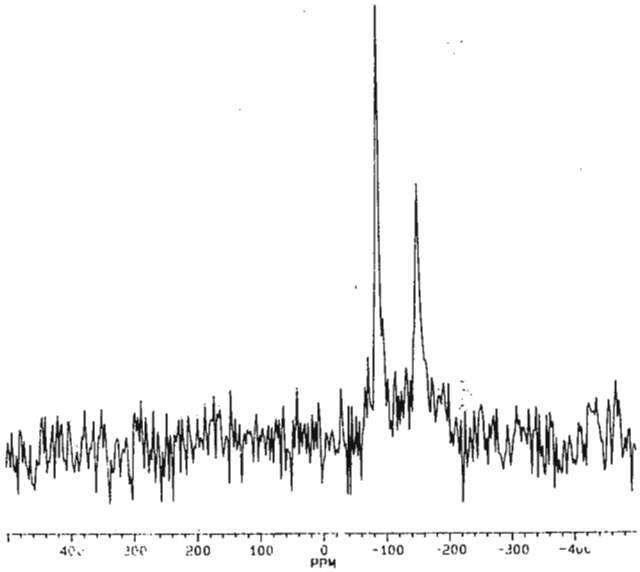

Figure 2. Ti spectrum of TiO_2 obtained at 33.81 MHz (6500 Hz spinning).

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Bruker AM Interface.

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  - New technology to give better performance. Some older probes can be upgraded to give more than a 100 % improvement in sensitivity.
  - Repair or upgrade an older unused probe to serve as a backup for existing used probes.
  - Upgrade an unused probe to be a ^{1}H biological (salt) probe. This will give shorter 90° pulses, better sensitivity and better water saturation performance in ionic water solutions.
  - Upgrade an unused or broken probe to be able to do new experiments. For example FMR can upgrade an older, little used probe to be a ^{1}H {^{31}P-^{15}N} probe for today's powerful inverse 2DFT experiments.

^{31}P / ^{13}C / ^{1}H *QE* Probes

A probe is available for the QE (and other instrument types) which has one port tuned for ^{1}H observation and another port simultaneously tuned for both ^{31}P and ^{13}C observation. This allows the QE console to observe ^{1}H, ^{31}P and ^{13}C spectra without tuning or switching the probe in any way. This is very convenient for sample changer operations.

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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 provides a complete "tool box" full of processing routines for processing 1D files up to 32K words in size and 2D files up to 2K by 2K size. The command line interface provides a fast and flexible interface for the experienced operator. Powerful macro operations and a menu interface allow the inexperienced operator to process data with a minimum of training. This software gives the user the capability to maintain his spectra at his desk and reprocess, expand, integrate and peak pick his spectra at any time. The annotated spectra can be exported to a file for immediate incorporation into word processing and desk-top publishing software which is readily available for the IBM compatible PCs.

Felix/PC software features:

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- Efficient Matrix oriented multi-D processing.
- Contour and "Image" 2D displays.

The NMR user can use his current PC and printers/plotters with Felix and be processing and plotting NMR data for $1000. Alternatively, he 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. If you need desk-top NMR data processing capability, then Felix/PC is for you.

BXR Data Transfer & Translation Software

BXR is a set of programs that transfers data files from the Bruker Aspect computers to PC computers. BXR stores the data in translated files that Felix/PC can read. Parameters related to data processing are transferred for use by Felix. Transfer rates of up to 19200 baud are usually routine (> 100 KBytes per minute). In normal operation the PC and the Aspect are connected with a communication cable and the BXR transfer program started on the PC. The unattended PC then waits for files to be transferred by the Aspect. There is no need to halt the PC program. You can start and stop the transfer program on the Aspect without stopping and restarting BXR on the PC. Under this condition, the PC waits for additional files from the Aspect until you halt it. This is for convenient data transfers to an unattended PC.

FELIX/PC Data Translation Software.

Both Nicolet/GE and Bruker provide Kermit data transfer software for their spectrometers. This software can easily communicate with a PC running any one of the many software communication packages using Kermit transfer protocol at transfer speeds up to 38K Baud.

Once the data is transferred by Kermit or any other means, data translation software packages are available to convert the Nicolet/GE 1280 20 bit word or the Bruker Aspect 24 bit word into floating point Felix/PC words. Key paramaters are also converted from the Nicolet/GE and Bruker integer and floating point header parameters into the respective file headers for Felix/PC:

- Spectrometer Frequency
- Sweep Width
- Data Table Size
- Non-Quadrature / Quadrature Data

This capability to process both manufacturers' data with a single software package can make life in a mixed instrument laboratory simpler for the user.

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.

PLOT allows the chemist to remove his spectra from the NMR instrument and still be able to plot, replot or do expansions at any later time. No more finding out later that you did not expand a critical region of the spectrum while on the instrument. PLOT is also an ideal method for NMR service facilities to distribute their spectra without tying up valuable instrument time.

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22 February 1990 (received 3/2/90)

Dr. Bernard L. Shapiro
966 Elsinore Court
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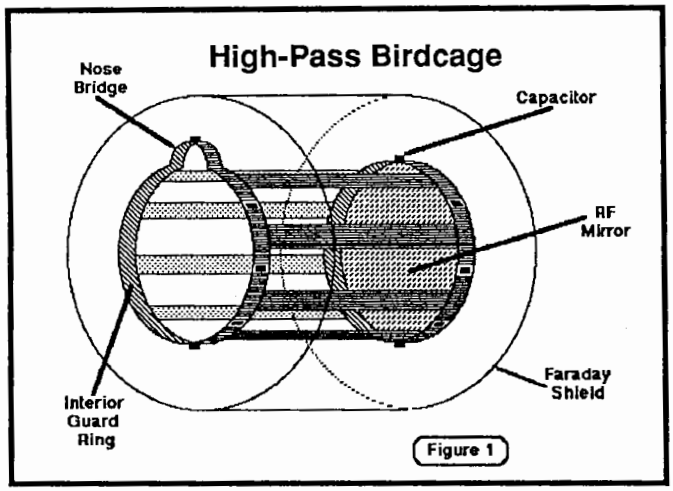
MAGNETIC RESONANCE UNIT
University of California Service
Veterans Administration Medical Center
4150 Clement Street (11D)
San Francisco, California 94121
(415) 750-2146

Gilding the Birdcage

Dear Dr. Shapiro,

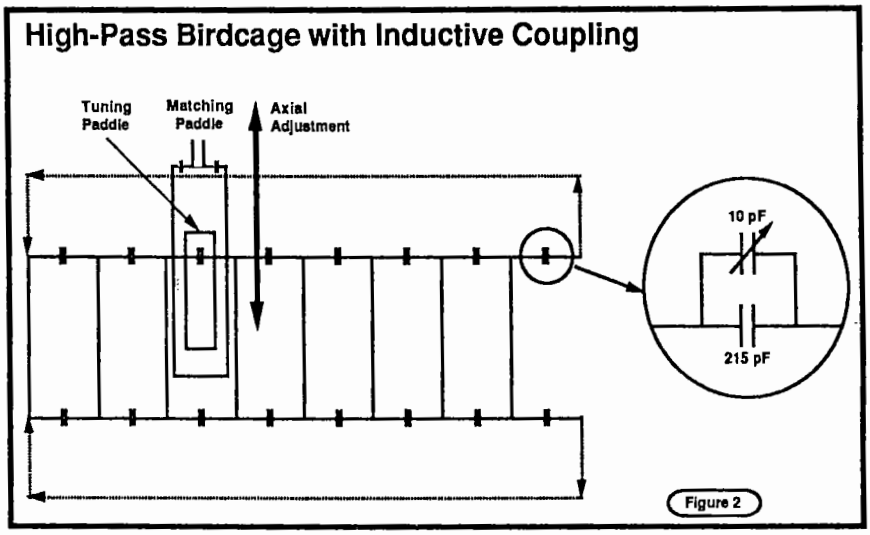
We have been developing rf birdcage coils for the human head to provide homogeneous excitation and detection and improved sensitivity in ^{31}P spectroscopy studies of the brain. In particular, we are using birdcage coils for ^{31}P three-dimensional MR spectroscopic imaging studies in patients with stroke or brain tumors and in normal volunteers [Ref 1]. Our original birdcage was not optimized for sensitivity, homogeneity, or ease of use. Therefore, we decided to gild the birdcage with a number of small improvements.

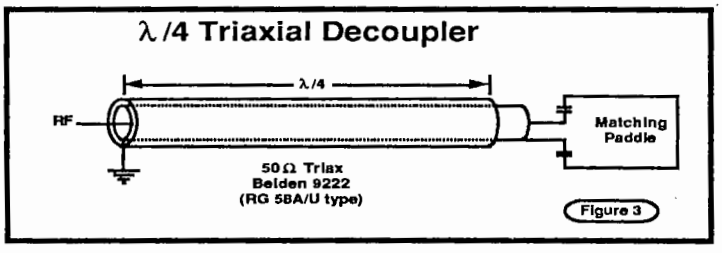


Our current ^{31}P head coil is a high-pass birdcage with 8 rungs, designed to resonate at 34.8 MHz for use in our 2 Tesla Philips Gyroscan whole body spectrometer. The coil is constructed on a 23 cm long acrylic cylinder with an outer diameter of 25 cm. The rungs and end rings are constructed of 50 μm copper foil. Each rung is electrically connected to its nearest neighbors at gaps in the end rings bridged by 215 pF fixed capacitors and 10 pF parallel variable capacitors for precise tuning (Figs 1 & 2). Guard rings of 25 μm copper foil, electrically isolated from the rf coil, are placed inside the cylinder to provide Faraday shielding around the capacitors, whose large E-fields would otherwise excite large dielectric losses in the patient. The guard rings are interrupted by a small gap bridged by two 620 pF chip capacitors. This much of the design is standard for birdcages [Refs 2 & 3].

Now the **gilding**: To accommodate larger patient heads, we have designed a "proboscis accommodator" – a protuberance in the lower ring with a complimentary cutaway of the acrylic cylinder (Fig 1). The additional length of copper foil in the lower ring adds some inductance, slightly breaking the birdcage symmetry. But the neighboring variable capacitors are simply adjusted slightly lower in capacitance to restore rf tuning.

Matching and tuning are accomplished by inductively coupling to the birdcage. The matching device consists of an inductive loop (6 cm x 18 cm) of copper foil mounted on a polycarbonate paddle which slides into a pocket on the circumference of the birdcage. The signal cable is connected in series with two balanced 110 pF capacitors to the matching loop (Fig 3). The pocket is located exterior to one loop of the birdcage, that is between two rungs, and matching adjustment is made by moving the paddle axially to vary the overlap of the matching loop with the birdcage loop. The matching range of the coil is sufficient to match empty or with a

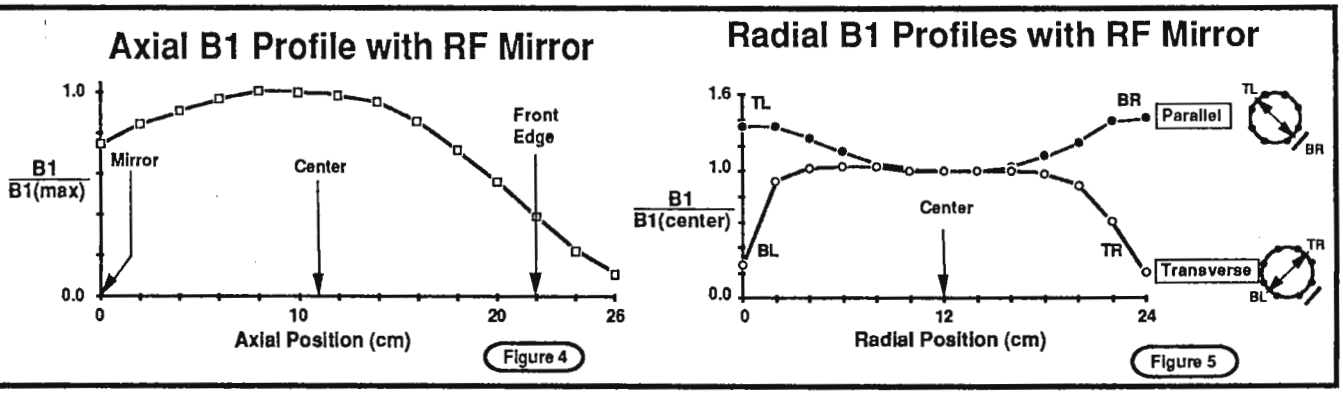




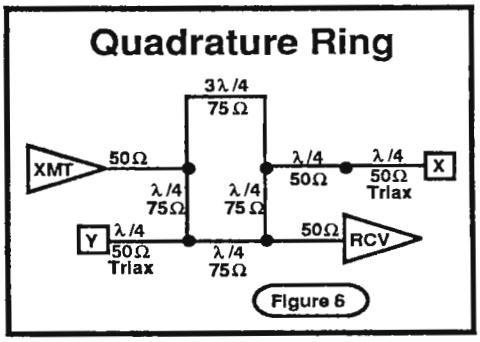
phantom which loads more than a large human head. Tuning adjustment is similar; a second pocket located at the same birdcage loop allows insertion and axial movement of a tuning paddle, which consists of a loop (4 cm x 18 cm) of copper foil mounted on a polycarbonate paddle. Tuning adjustment is made by moving the paddle axially to vary the overlap of the tuning loop with the birdcage loop. Matching and tuning adjustments are virtually independent. These gildings were inspired by Refs 4 & 5.

To reduce interaction of the signal cable with the bore of our superconducting magnet we use a quarter-wave triaxial decoupler (Fig 3, Ref 6) to suppress unwanted antenna currents excited on the cable shield. To reduce interaction of the coil with the magnet bore (or with the experimenter at the lab bench during development) we constructed a cylindrical Faraday shield to surround the coil. It is made of 10 cm wide strips of 20 μm aluminum foil which overlap slightly, but are electrically isolated from each other to suppress gradient eddy currents. These strips are mounted on the exterior of a 30 cm long acrylic cylinder with an outer diameter of 40 cm (Fig 1). This Faraday shield also raises the Q and resonant frequency of the coil.

Measurements of rf spatial distribution revealed significant drop-off of B1 along the axis within about 6 cm from the coil center. Therefore, a conductive rf "mirror" of 20 μm aluminum foil was placed at the upper end of the birdcage coil to substantially improve the B1 homogeneity and thus enlarge the sensitive volume of the coil (Figs 4 & 5).



The tuned birdcage resonates at a single frequency in its symmetric, linearly polarized mode, thus facilitating its use for circularly polarized (quadrature) mode applications. Plans include driving the birdcage coil in quadrature by installing a second set of pockets two birdcage loops away (1/4 circumference, or $\pi/2$) from the first set. A convenient quadrature circuit, now being tested, is shown in Fig 6 [Refs 5 & 7].



1. JW Hugg, et al, SMRI Paper #P-098 (1990).
2. JC Watkins & E Fukushima, Rev Sci Instr 59 (1988) 926.
3. J Tropp, J Mag Res 81 (1989) 1.
4. PL Kuhns, et al, J Mag Res 78 (1988) 69.
5. K Yoda & M Kurokawa, J Mag Res 81 (1989) 284.
6. WI Orr, Radio Handbook , 23rd Ed (1989) 21-21.
7. VJ Sank, et al, J Mag Res 69 (1986) 236.

James W. Hugg

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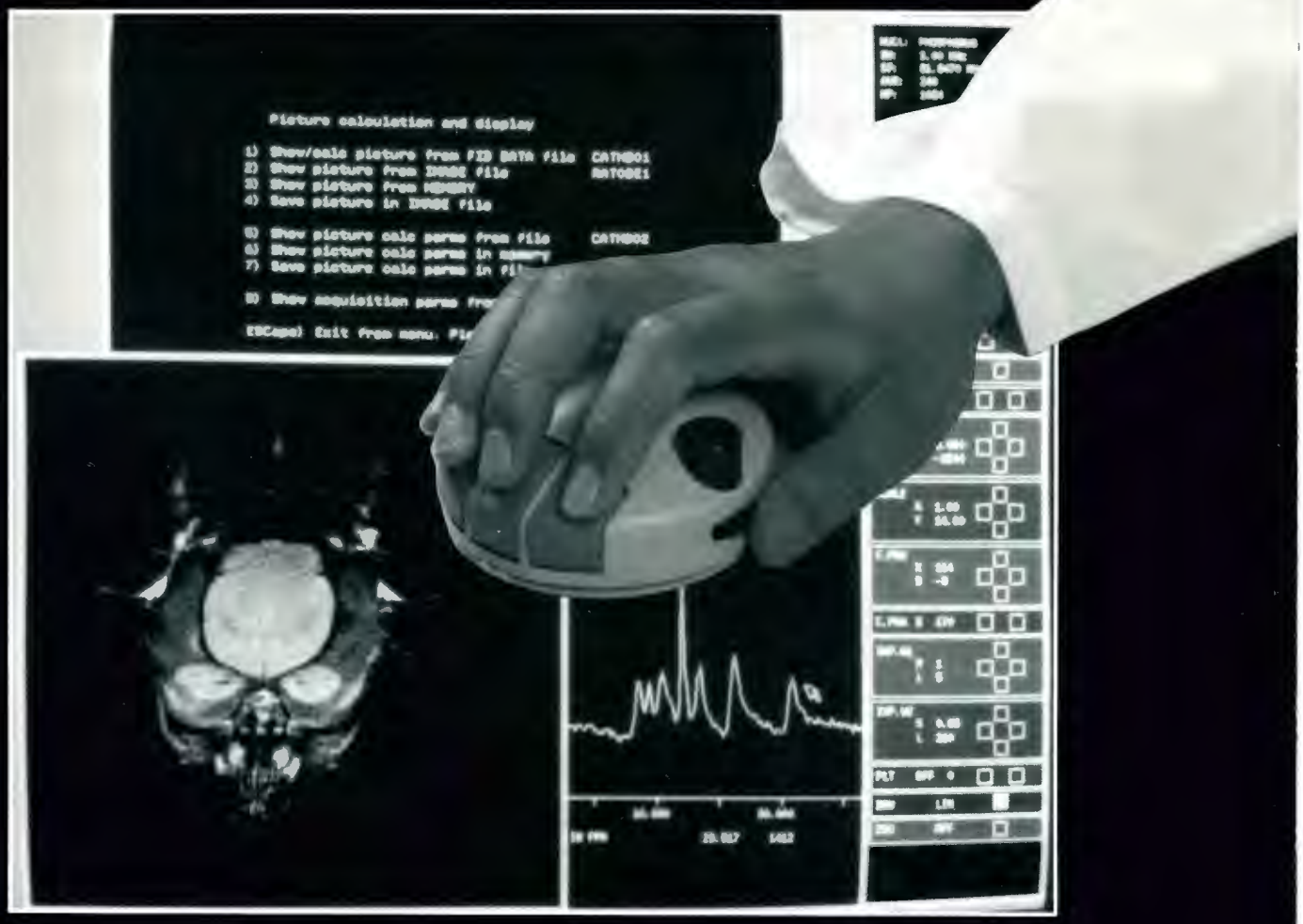

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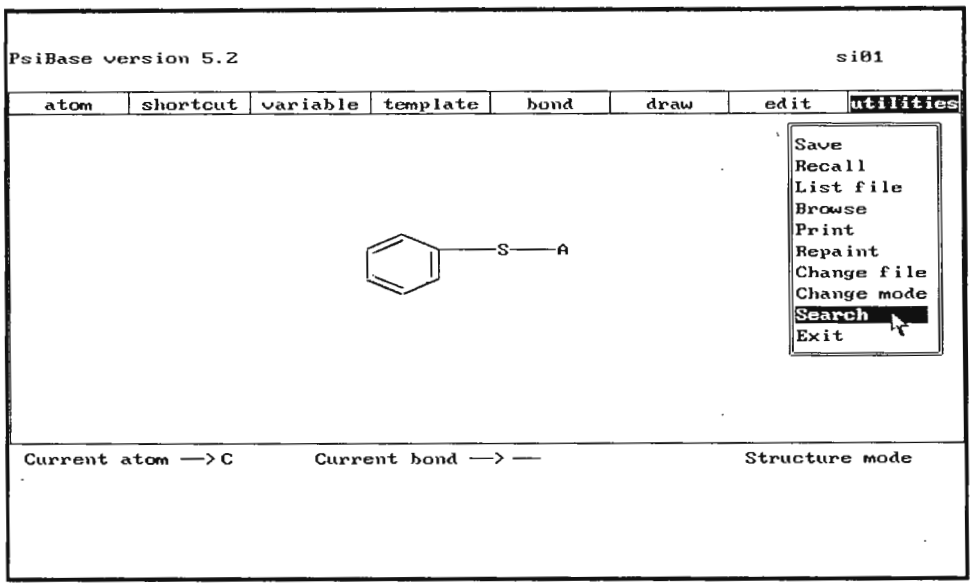
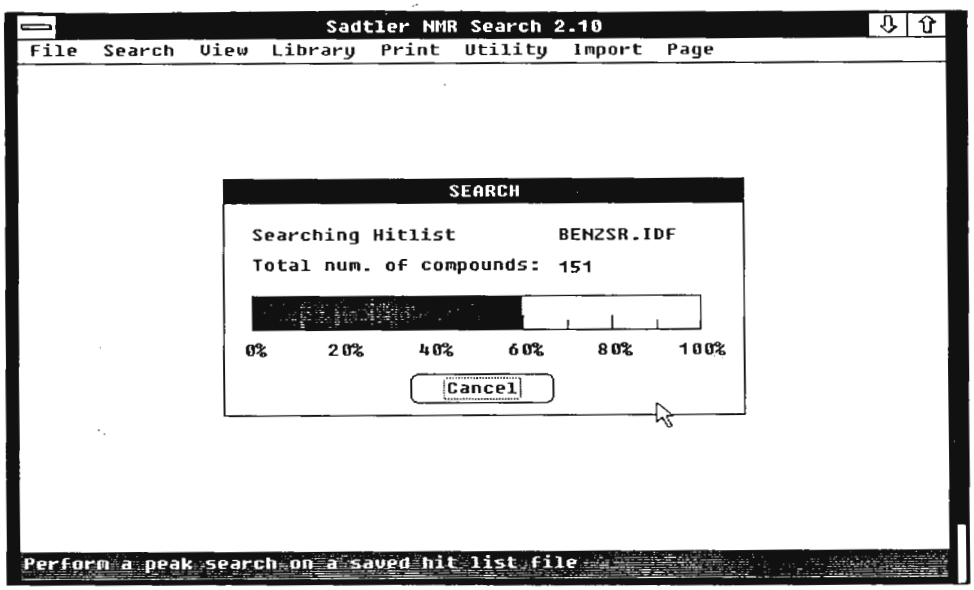
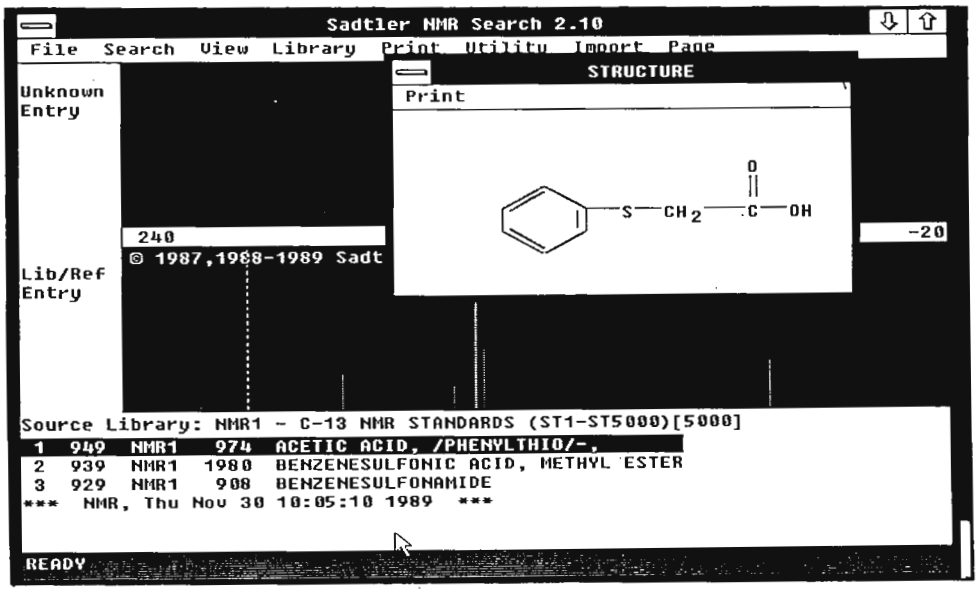


Step 1: Search for all thiophenol derivatives.



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



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Department of Chemistry

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Wednesday, May 23, 1990

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The symposium will feature invited papers by:

* A.M. Gronenborn, National Institutes of Health
"Protein Structure Determination by 2- and 3D-NMR"

* R.G. Shulman, Yale University
"High Resolution ^{13}C NMR Studies of Glucose Metabolism in Humans"

* C.P. Slichter, University of Illinois
"NMR Studies of Catalytic Surfaces"

* R.L. Vold, University of California at San Diego
"Solid State Deuterium NMR of Molecular Complexes"

* G. Wagner, University of Michigan
"Use of NMR for Protein Design"

For additional information, contact the departmental secretary, Mary Ann Wiegers, at (314) 889-6530.

CHEMICAL CENTER
UNIVERSITY OF LUND
PHYSICAL CHEMISTRY 2
Prof. Sture Forsén

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

February 20, 1990
(received 2/26/90)

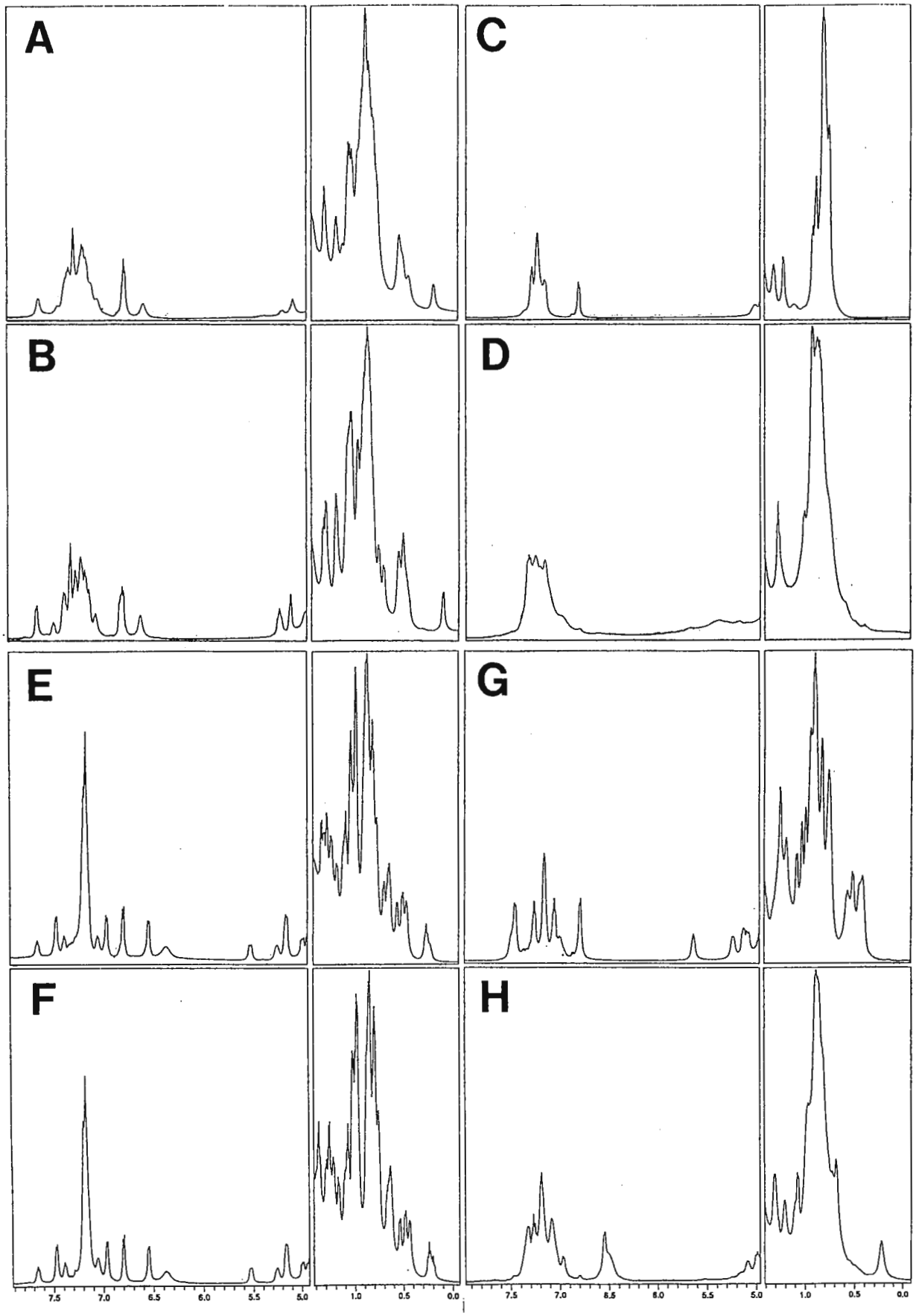
Cleaved calbindin retains its structure

Dear Barry,

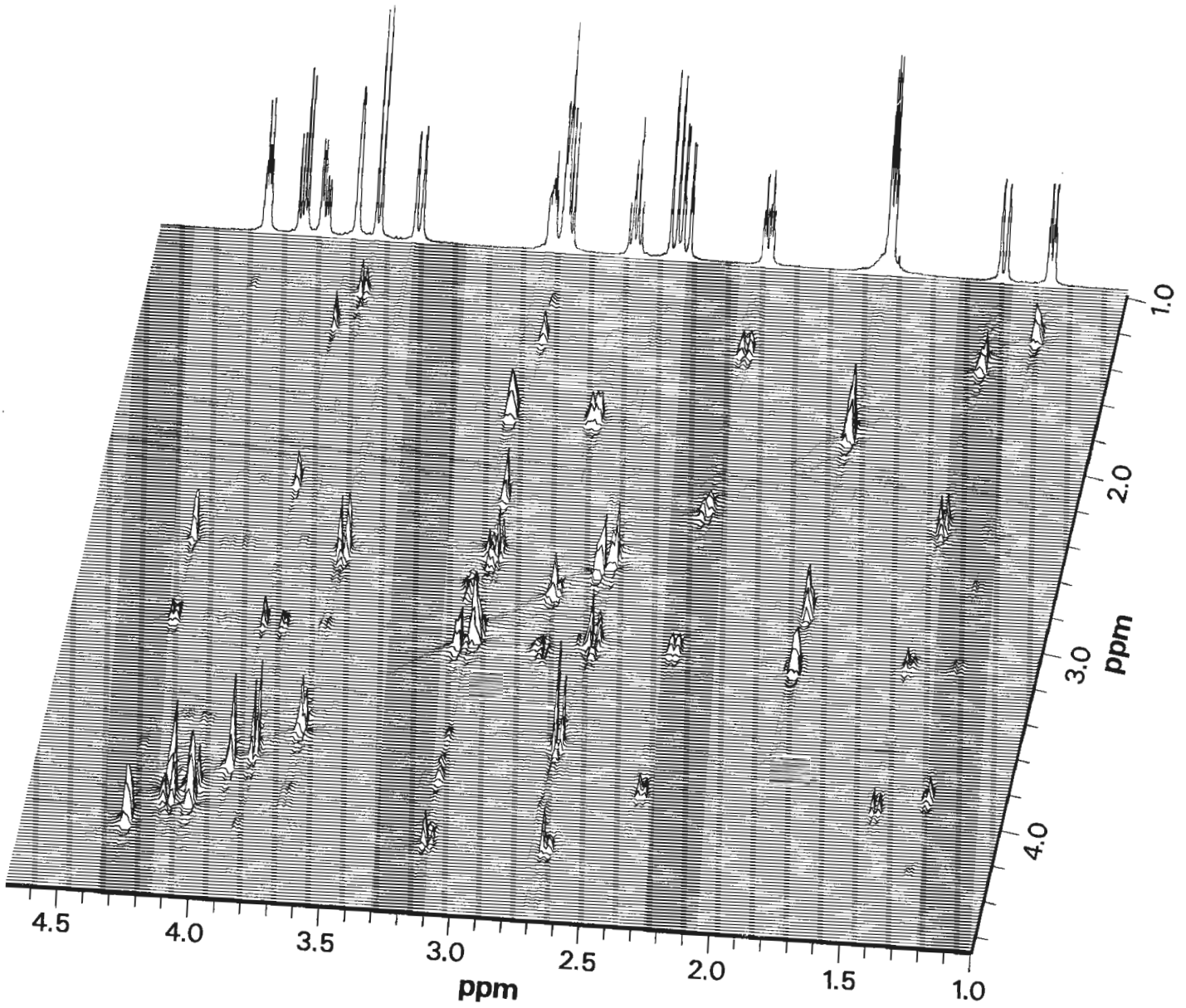
We have during the last few years studied the calcium-binding protein calbindin using various NMR techniques. One of the questions we have tried to adress is that of the mechanism behind the positive cooperativity in the binding of the two calcium ions. We have therefore manufactured a mutant protein, Pro43→Met, that allows us to cleave the protein and subsequently isolate the two intact calcium-binding sites. Both halves bind calcium with a binding constant in the order of 10^5 M^{-1}. To our surprise we find that the characteristically shifted ^1H NMR signals in intact calbindin also appear in the calcium-saturated isolated fragments, although not with exactly the same chemical shifts (fig. F-H). In the calcium-free form, however, hardly any similarities can be seen (fig. B-D). Now, if we mix the two fragments, whether calcium-loaded or not, the ^1H NMR spectra are almost indistinguishable from those of intact calbindin(fig. A-B, E-F). We have independent evidence for a complex between the two fragments and these NMR studies show that the conformation of intact calbindin is retained in the complex. Preliminary studies of calcium affinity to the mixture of the two fragments indicate that calcium is bound with positive cooperativity also in the complex!

Yours sincerely,

Johan Kördel Torbjörn Drakenberg Sture Forsén



^{1}H NMR spectra of calcium-free (A-D) and calcium-loaded (E-H) calbindins: The mutant Pro43→Met (B, F), a mixture of fragment I (residues 1-43 of the mutant) and fragment II (44-75) (A, E), fragment I (C, G), and fragment II (D, H). The spectra were recorded from 3-5 mM D_2O samples at pH 6.0, 300 K on a GE Ω-500 spectrometer.



HOHAHA of Strychnine on an Omega 600

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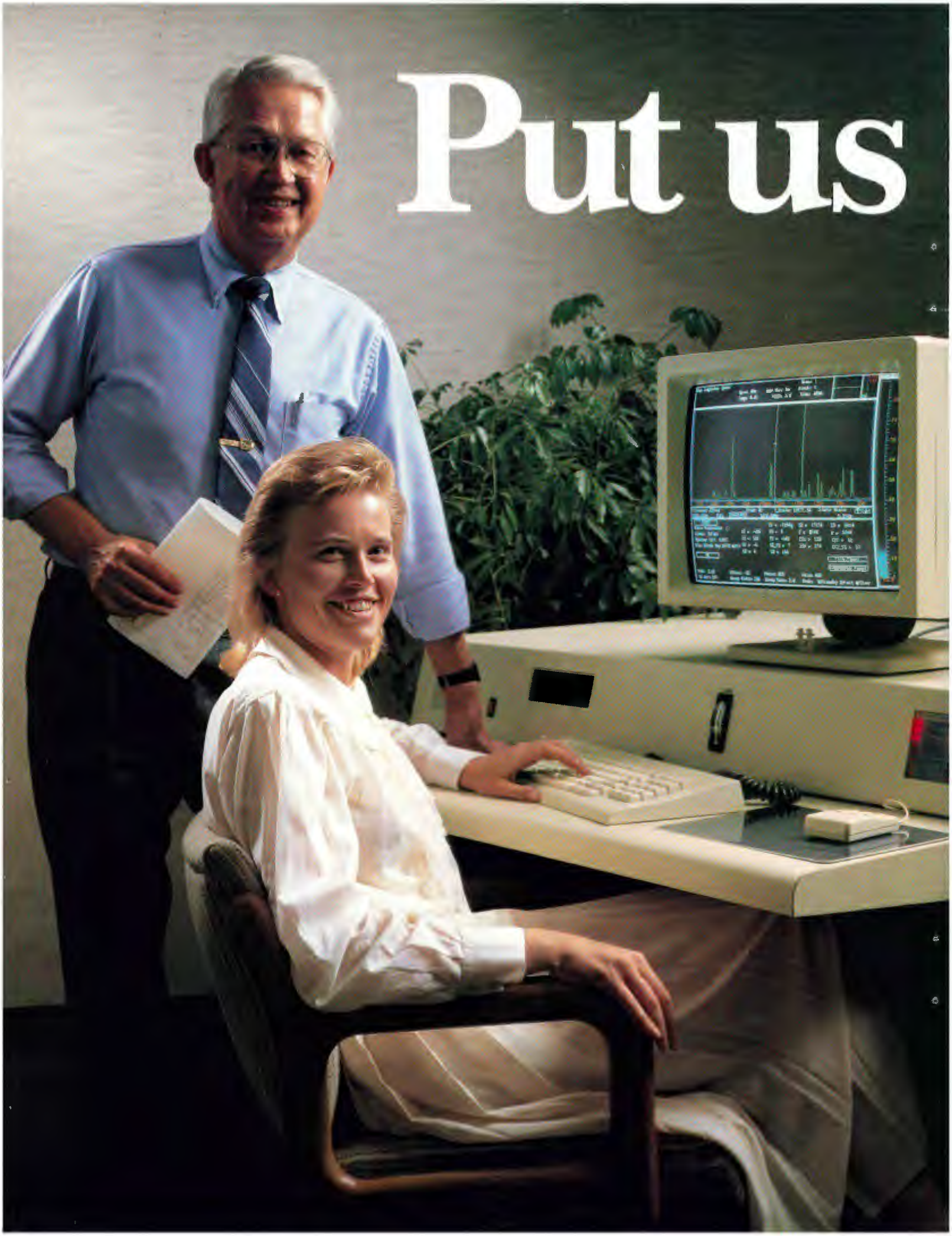
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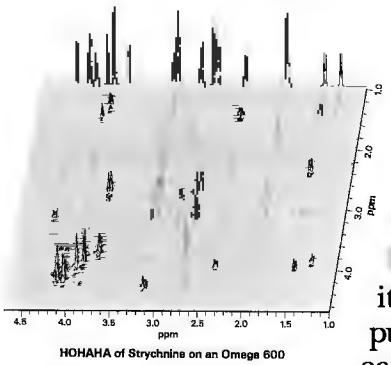

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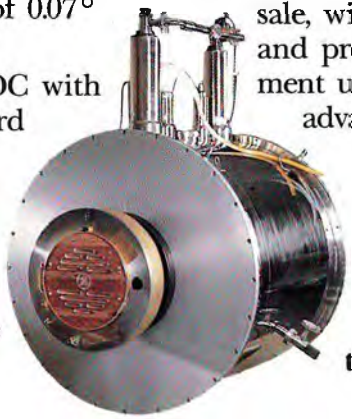

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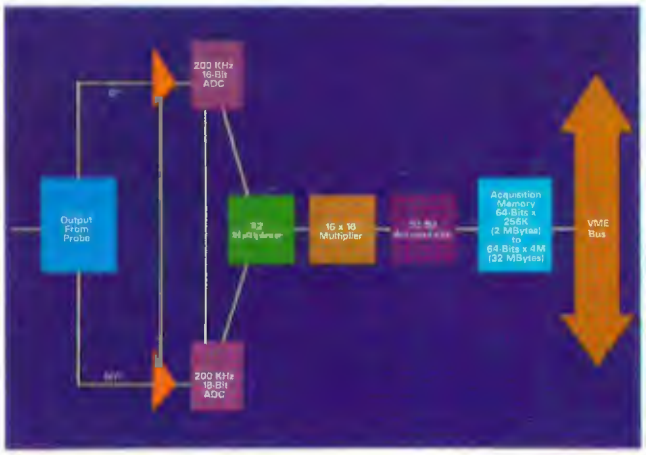

Fig. 1

The Alpha HDR digitizer.

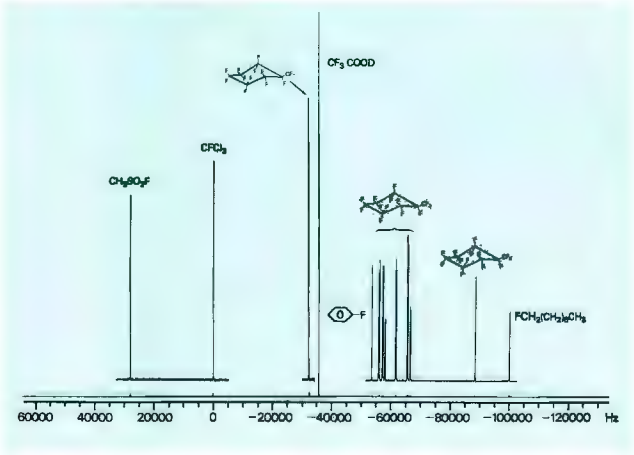

Fig. 2

200 KHz spectral width ^{19}F spectrum acquired on a GN-500 Omega System. Note the extremely flat baseline obtained with the Alpha HDR.

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

12th March 1990
(received 3/17/90)

Dear Barry,

We have recently introduced a new technique for volume localisation by saturation of the spins outside the region-of-interest (1). The method is limited by longitudinal relaxation times, as opposed to the transverse relaxation dependance of techniques relying on selective excitation, and takes the form of a pre-pulse sequence containing multiple applications of a noise-modulated pulse where the field gradient is in a different direction for each pulse. Pseudo-noise modulated pulses (2) aim to excite magnetisation with a random phase relationsip across the bandwidth of the pulse, except over a narrow band of frequencies, typically about the centre of the spectrum, which receive (ideally) no excitation. In the 2-dimensional version of our experiment, (Fig. 1) the field gradient vector describes a circle, stepped after each noise pulse, so that coincidence of the null slices for the noise pulses defines a central cylindrical region of undisturbed spins, while spins throughout the rest of the sample lie in the transverse plane with random phase. Once this column has been selected, it may be examined with an imaging sequence, or a slice selective excitation pulse to acquire a spectrum from a part of the column. We have had some success with this technique both for zoom imaging and also for localised spectroscopy, where the smoother gradient shape appears to produce less eddy currents in the magnet construction. The selected region of interest may be moved to any part of the sample by changing the frequency offsets for each of the noise pulses, and arbitrary convex ROI shapes are available by modification of the gradient waveforms.

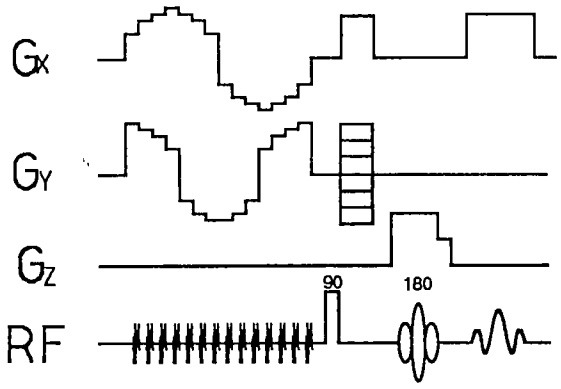

Fig. 1 2D ROISTER imaging sequence.

All the experimental work on this project has been carried out using a standard ORS Biospec I consol which does not normally allow the use of arbitrary gradient waveforms. To get the gradient control necessary to do this experiment we therefore need to load the gradient waveform memories explicitly from the forground using a combination of PASCAL and machine code software prior to starting the acquisition program, thus overwriting the gradient values loaded by the existing pulse program complier. During the acquisition sequence, values are simply clocked out of the waveform memory to the DACs each time a new gradient level is required; there is no looping until the start of the next acquisition cycle, when the pointer is reset to the beginning of the memory. This means that when loading the values for the read gradient,

the dephase and read gradient values must also be loaded (with appropriate intervening zeros), after the shaped part of the sequence, Fig 1. In the case of the phase encoding gradient however, for NS scans in the experiment, NS copies of the shaped gradient waveform plus the phase encoding step must be loaded into memory at the start. The waveform memories supplied with the machine can contain up to 32k values, which is quite adequate for most ROISTER experiments, but we have found it insufficient when trying to use 2D dimensional selective pulses (3) where the gradient shape is typically defined by 256 points in each channel. One way to evercome these memory constraints is to dynamically load the waveform memories.

The DISNMR programme on our spectrometer supports a degree of multi-tasking in the form of 3 jobs, so that processing can occur in one job while data is being acquired in another. A pascal program, running continuously in the second job, monitors the scan counter in the first job from which the acquisition program was started. This counter is incremented at the end of each scan, and the program is triggered by this change to load the new value of the phase encoding gradient into the correct location in the waveform memory, ready to be clocked out next time around. This process occurs during the relaxation delay in the experiment and takes about 2ms. However if the delay allowed is too short and the next scan begins before the loading process is complete, then the resultant gradient waveform shape can be unpredictable ! Also, since the computer on the spectrometer (an ASPECT 3000) is involved in the process, it is important that there are no other concurrent tasks requiring processing, such as a data transfer over ethernet. The flow chart in Figure 2 shows how this programme works, the whole process is controlled by a short forground interpretive program (EXE file), so that the operator is just required to enter the filename for the result. The same dynamic loading process could also be applied to soft RF pulses, if a different shape is required on each scan for example.

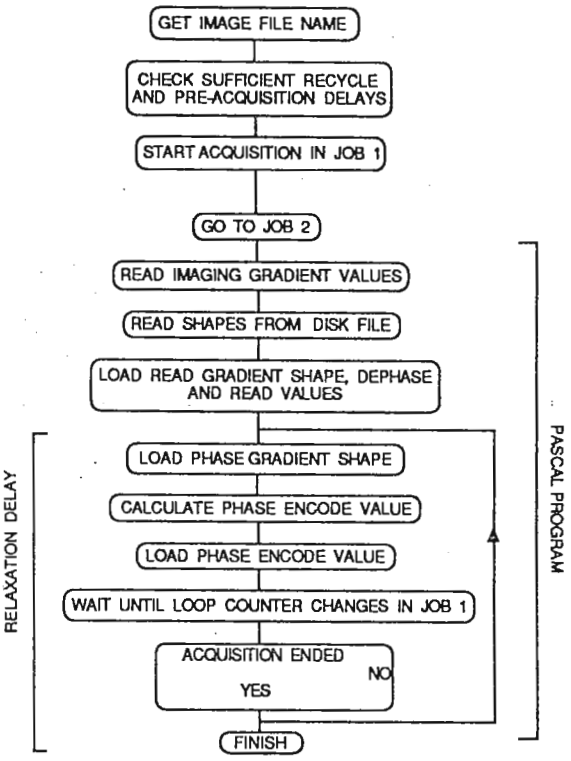

FIG. 2 Dynamic waveform memory loading

This has enabled us to use sequences on our machine without any hardware changes which would have otherwise necessitated more modern equipment.

Yours sincerely,

Laurie Hall, Adrian Carpenter, Alex de Crespigny.

(1) A. de Crespigny, T.A. Carpenter, L.D. Hall, J.Magn.Reson. **85**, 595 (1989).
(2) R. Ordidge , Magn. Reson. Med. **5**, 93 (1987).
(3) J. Pauly, D. Nishimura and A. Macovski, J.Magn.Reson. **82**, 571 (1989).

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March 1, 1990
(received 3/5/90)

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

RF Tuning with PACMAN (Phase Adjustment with Cartesian MApping for NMR)

Dear Barry:

Recently, while tuning quadrature RF phases and amplitudes on our Bruker CXP-100 Spectrometer, we stumbled upon a display technique which we feel greatly simplifies the RF tune-up process. An offshoot of the commonly employed method of using Lassajous figures for receiver channel adjustment, this technique projects the RF amplitudes and phases onto points in the rectangular (cartesian) plane. This essentially allows direct measurement and noninteractive adjustment of both the RF phase angle and amplitude. A brief outline to the technique with the appropriate background material follows.

Generally, when using Lassajous displays for receiver channel amplitude and phase balance adjustments, a CW signal is input into the receiver and the receiver audio outputs (U, V) are displayed on an oscilloscope XY display. The X and Y amplitudes are then expressed as

$$X = A \sin(\omega t + \alpha)$$
$$Y = B \sin(\omega t + \alpha + \Theta)$$

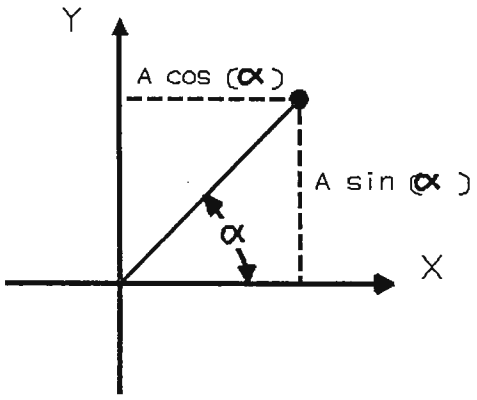
where omega and alpha are the offsets from the receiver reference frequency and phase, respectively, A and B are the receiver channel amplitudes, and theta is the phase difference between channels. In the limit of perfect receiver channel quadrature adjustment, A=B and theta=90°. Thus, Y reduces to

$$Y = A \cos(\omega t + \alpha)$$

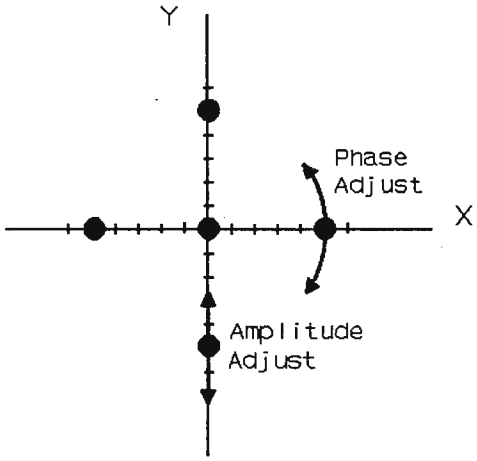
and the figure displayed progresses from an ellipsoid to a perfect circle. As the input frequency approaches the receiver reference frequency, circular precession ceases ($\omega \rightarrow 0$). X and Y are then simply expressed as

$$X = A \sin(\alpha)$$
$$Y = A \cos(\alpha)$$

and the figure displayed becomes a point in the rectangular (cartesian) plane.



This method was implemented on our spectrometer to adjust RF phases and amplitudes in multipulse decoupling experiments. RF transmitter channels were adjusted by applying a four pulse sequence (X, Y, -X, -Y) and connecting the appropriate RF generated pulse from the pulse modulator to the receiver input. The visual display of amplitude and quadrature phase-adjusted pulses is shown below.



We have found this technique to be particularly robust due to its ease of application for both conventional quadrature RF adjustments, as well as its application to nonconventional amplitude and phase angle (45°, 60°, etc.) adjustments. Perhaps in this last application, this technique will have its greatest utility.

Sincerely,

Steve Dieckman

Sami

N. Gopalsami

Robert Botto

Z•SPEC
Four Nuclei Probe

FEATURES:

^{1}H, ^{19}F, ^{31}P and ^{13}C observe capability without retuning the probe. The four spectra shown on the back were obtained using this probe. The only thing the NMR operator does is change the observe frequency of the spectrometer. The probe contains no internal switches and thus cannot wear out from repeated observe nuclei changes.

APPLICATIONS:

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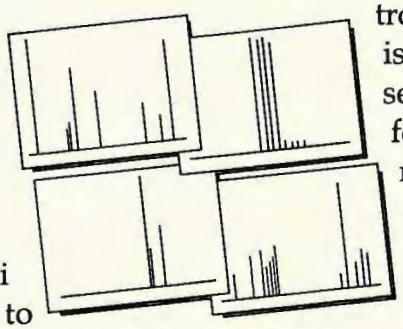

TECHNICAL:

The Z•SPEC Four Nuclei Probe interfaces directly to any Varian 200, 300 or 400 MHz NMR Spectrometer. The probe is capable of observing any of the four nuclei without retuning the observe frequency or changing 1/4 wavelength cables.

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

NCC

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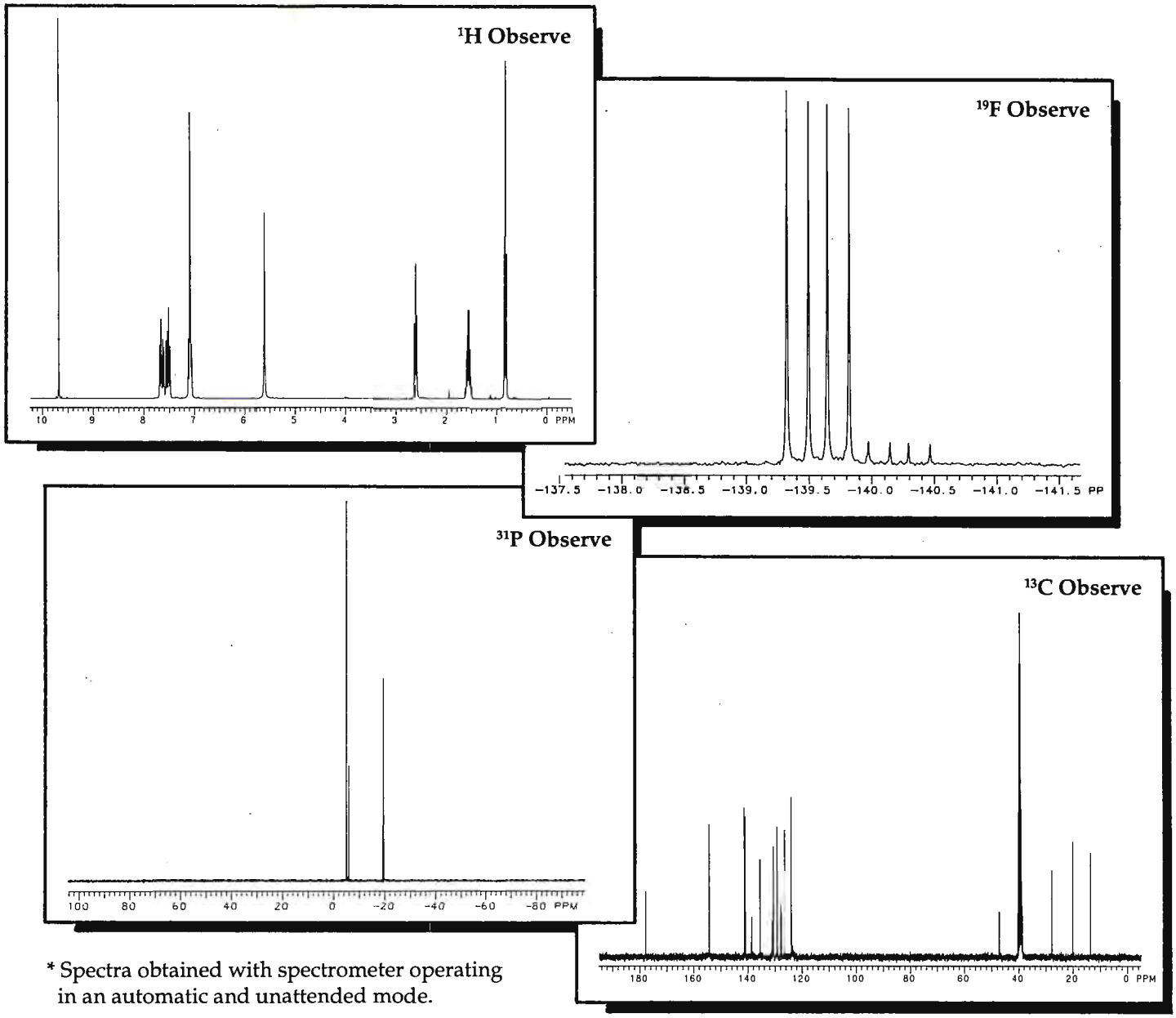

* Spectra obtained with spectrometer operating in an automatic and unattended mode.

NALORAC CRYOGENICS CORPORATION
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Washington
WASHINGTON·UNIVERSITY·IN·ST·LOUIS

Department of Chemistry

March 2, 1990
(received 3/8/90)

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

The Phosphocreatine Proton-Decoupled ^{31}P Doublet: Rediscovering the Deuterium-Phosphorus Isotope Effect

Dear Barry,

In recent high resolution ^{31}P NMR studies of RIF-1 murine tumor extracts (pH 9, 4°C, D_2O) on a Varian VXR-500 spectrometer, we observed a single resonance for phosphocreatine as one would expect. However, to make correlations with our *in vivo* NMR tumor data, we lowered the pH of two of the tumor extracts to 7. Surprisingly, we then observed a 0.09 ppm (18 Hz) splitting of the phosphocreatine ^{31}P resonance into two lines. Raising the pH back to the original value revealed that this phenomenon was a reversible one, as the phosphocreatine ^{31}P signal was once again observed to be a single peak.

Before searching the literature, we prepared a solution of 0.1 M phosphocreatine in a mixture of D_2O and H_2O and varied the pH to examine the splitting profile and chemical shift dependence on $[H^+]$. At extreme pH conditions, there was only one peak observed; whereas two peaks were observed for intermediate pH values (Fig. 1). Though the chemical shift values of the two resonances did show a change for pH values of less than 6, the splitting remained fairly constant at 0.09 ppm over the pH range where the two peaks were well-resolved.

To confirm that an exchange process was responsible for these observations, variable temperature studies were performed with a solution at pH 8.3. The sample temperature was varied from -5°C to 23°C. As the temperature increased, the two lines were observed to come closer together and finally merge at a temperature of about 20°C (Fig. 2).

A literature search turned up an article by V. A. Saks and coworkers [*Biochem. Biophys. Res. Commun.*, **114**, 1117 (1983)] that presents an earlier study of this effect and attributes the splitting to a deuterium isotope effect upon the ^{31}P chemical shift resulting from H^+/D^+ exchange at the nitrogen directly bonded to phosphorus. This appears to be the only published report of a deuterium isotope effect on a ^{31}P NMR spectrum [P. E. Hansen, *Progress in NMR Spectroscopy*, **20**, 225 (1988)]. As D_2O is a common solvent for high resolution ^{31}P NMR studies of tissue extracts, those involved in such research should be aware of the potential for this effect, as other phosphorus metabolites of interest may have exchange labile hydrogen sites.

Sincerely,

Tedros Bezabeh
Tedros Bezabeh

Joseph J.H. Ackerman
Joseph J.H. Ackerman

Washington University
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One Brookings Drive
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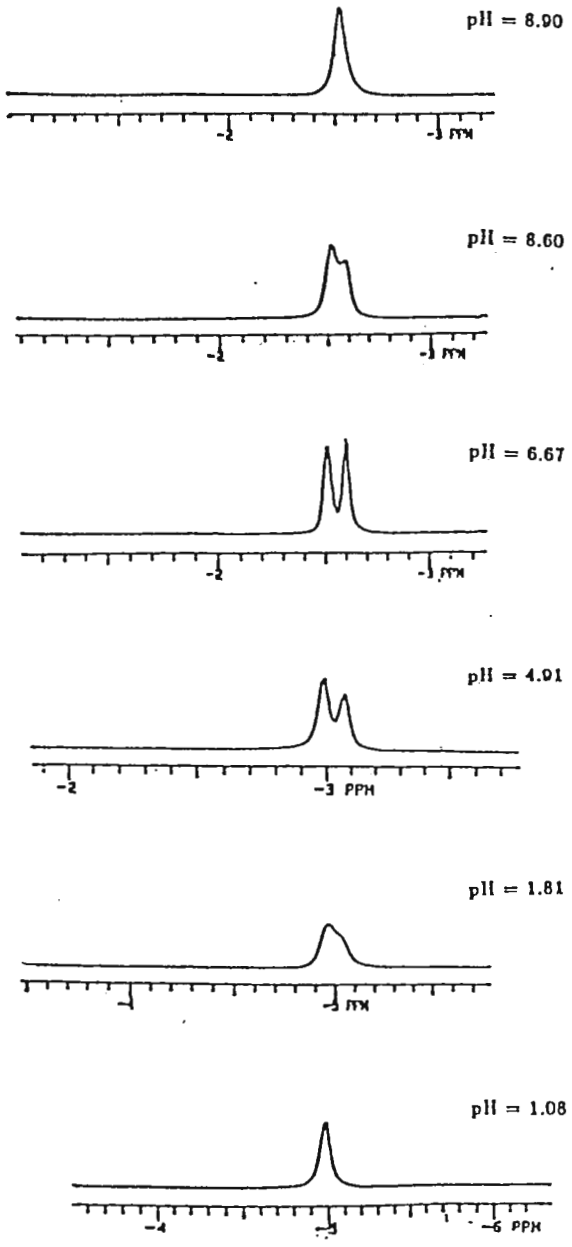

Figure 1. Proton decoupled ^{31}P NMR Spectrum of Phosphocreatine in mixture of D_2O and H_2O showing splitting as a function of pH at a temperature of 4.0°C.

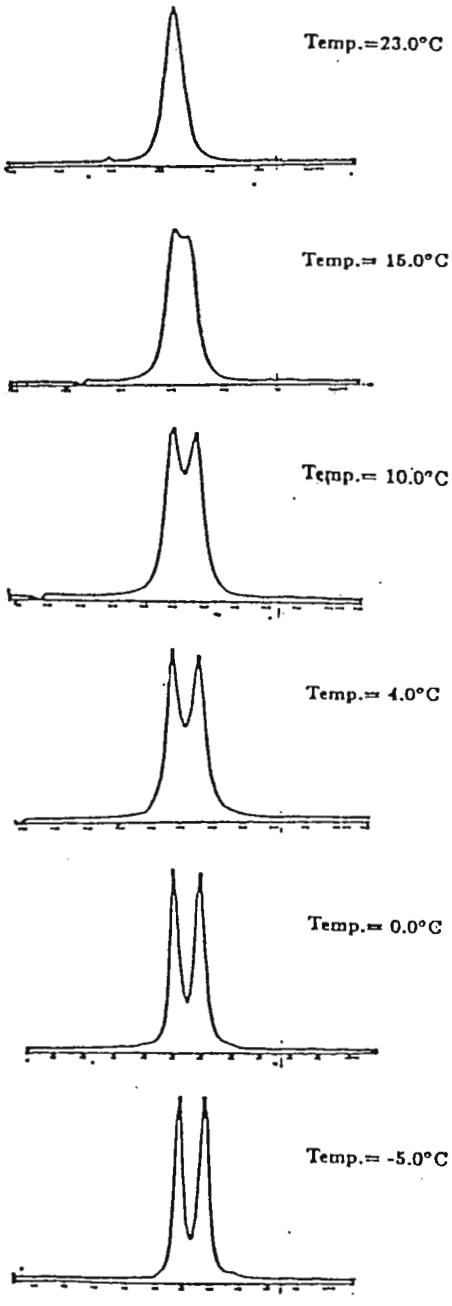

Figure 2. Proton decoupled ^{31}P NMR Spectrum of Phosphocreatine in a mixture of D_2O and H_2O showing splitting as a function of temperature at a pH of 8.30.

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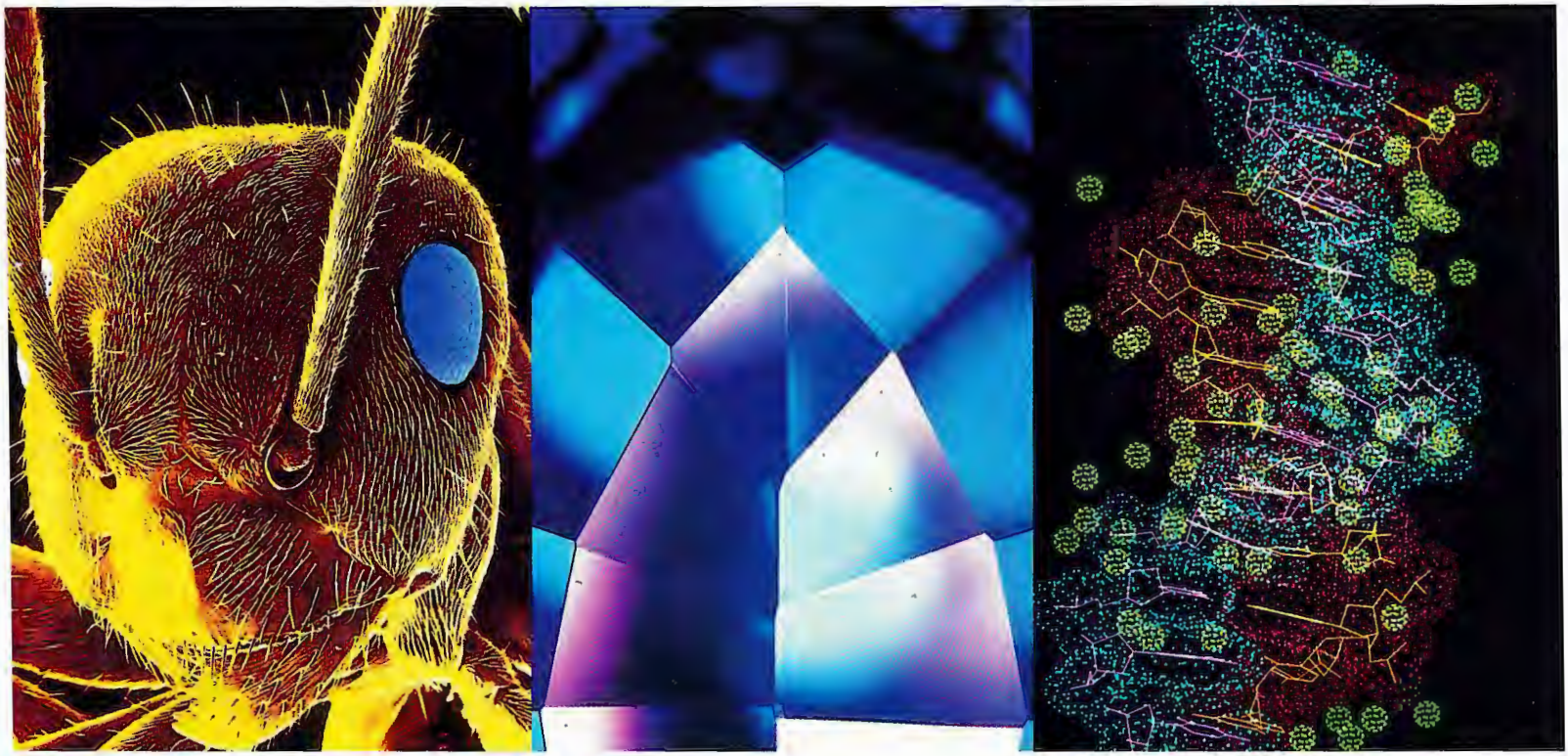

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January 29, 1990
(received 2/20/90)

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

OBSERVATION OF NOVEL PENTACOORDINATE-BICYCLOSILANES

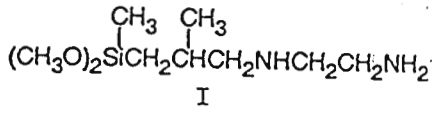
Dear Dr. Shapiro,

We have recently looked at several aminofunctional alkoxysilanes by Si-29 NMR that were previously reported by Speier, Roth, and Ryan (1). These compounds form cyclic azasilanes with loss of methanol on slow distillation. To our surprise Si-29 NMR spectra of the distillation product of compound I showed not only the resonance expected for the pentacyclic compound II previously reported but also the octacyclic compound III and two unexpected resonances to higher field, Figure 1. All four of these compounds revert back to the original silane I on addition of a stoichiometric amount of MeOH. These upfield resonances are assigned to the new pentacoordinate bicylosilanes, IV and V. Pentacoordinate silicon compounds are rarely observed but are implicated as intermediates in nucleophilic substitution reactions of silicon. Silatranes are the most common examples of pentacoordinate silicon complexes and all have N,O linkages to silicon. IV and V are the first examples of pentacoordinate silicon we are aware of that have C,N and N,N linkages to silicon. We are currently examining a series of these compounds which will be reported in more detail later.

Regards,

Maris J. Ziemelis Richard B. Taylor

1) J.L. Speier, C.A. Roth, and J.W. Ryan; J. Org. Chem.,36 (1971) 3120-26



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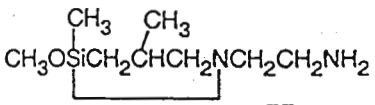

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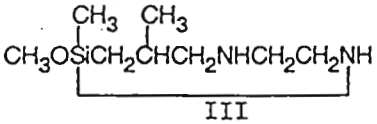

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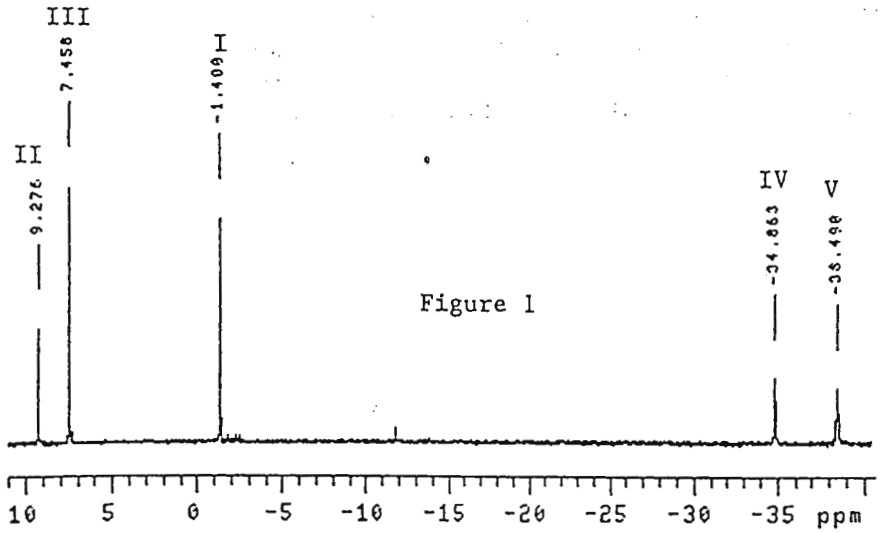
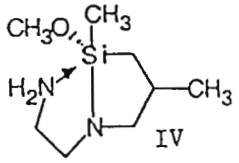
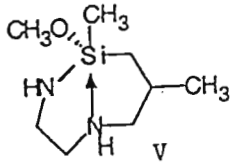


Figure 1



IV



V

379-36

University of Nottingham

Department of Chemistry

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TEL. NOTTINGHAM 484848

28th December, 1989
(received 3/17/90 [sic])

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

Dear Dr Shapiro,

Migration Reactions in Organometallics

We have had substantial success detecting unexpected migration reactions in straightforward variable temperature ^1H nmr runs. For example in the compound $[(azulene)Mo_2H(CO)_6]^+$ migration of the hydride to the ring occurs at subambient temperatures to a specific position on the five-membered ring. However, with the related guiazulene complex migration occurs to two adjacent positions at a roughly equal rate and this is followed by a subsequent isomerisation to the thermodynamically most stable isomer.[1]

Curently, we are investigating a series of arene-ruthenium complexes, such as $[(cymene)RuH(terpinene)]^+$ which undergo a related migration reaction. However in this case there appears to be little regioselectivity in the site of migration.

The migrations are not restricted to hydrogens, we are also currently following competitive migrations between an isocyanide ligand and a cyclopentadieny ring in a cobalt complex. In this instance the final product involves loss of the substituted ring from the coordination sphere of the cobalt.

Yours sincerely

Tony Wright

Dr A.H. Wright

1. K.E. Clode and A.H. Wright, J. Chem. Soc., Chem. Commun., (1988), 1463.

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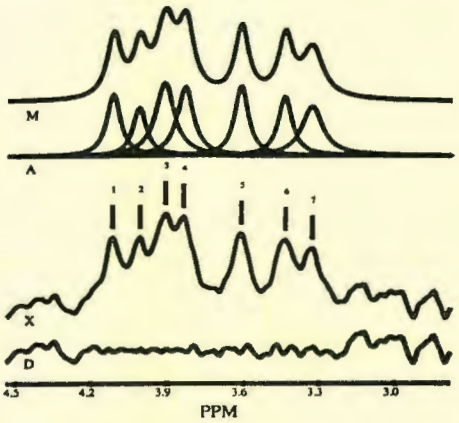

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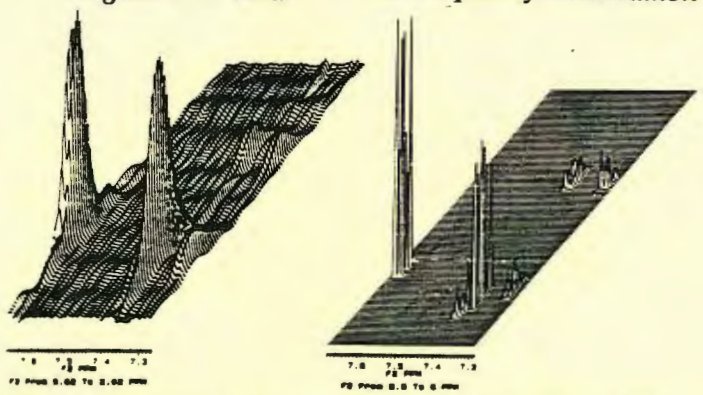
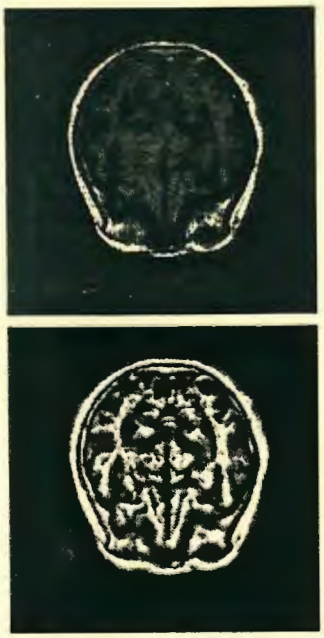
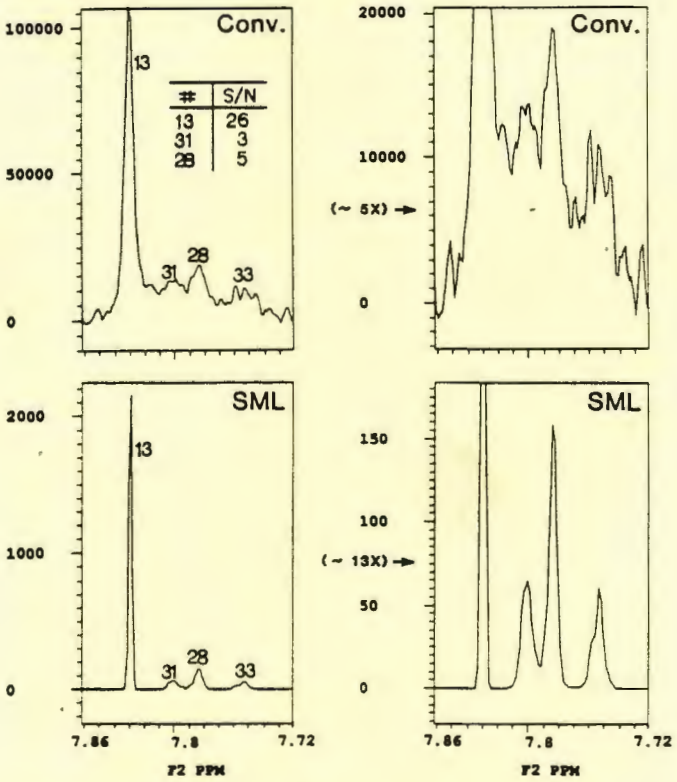


MLM (Maximum Likelihood) Spectral Deconvolution for 2D NMR. Zoom region of DNA duplex (500 MHz NOESY spectrum). Quantitatively resolve NOESY cross peaks and observe spin–spin couplings.



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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55 21 78 Dr. A. Sebald
Dr. L. Merwin

March 12th, 1990
(received 3/16/90)

please credit: Angelika Sebald, Larry Merwin

COSY revisited: application to ^{119}Sn CP MAS NMR spectroscopy

Recently, the homonuclear COSY experiment, combined with CP MAS, has been reported for ^{13}C, ^{29}Si, ^{31}P [1-3]. The problem with these examples is: either the experiment was not done on "real" solids (i.e. adamantane or Q_8M_8 were used) or, for ^{31}P, unrealistically high S/N ratios are obtained in comparison to most other solids applications. Especially for ^{119}Sn, the homonuclear CP-COSY experiment could be particularly useful, as it would provide valuable additional long-range J-coupling information with regard to $^{n}J(^{119}Sn\ ^{119}Sn)$ versus $^{n}J(^{119}Sn\ ^{117}Sn)$. This additional information from the CP-COSY experiment is highly desirable as in most normal ^{119}Sn CP MAS spectra the $^{119/117}$Sn-satellites are not well enough resolved for unambiguous assignments.

With respect to the $^{119/117}$Sn-satellites problem in the solid state, some time ago we used the compound $(Me_2SnS)_3$ as a guinea-pig: in the solid state this molecule has a twisted boat conformation and, therefore, shows two ^{119}Sn resonances in a 2:1 ratio. The intramolecular nature of this splitting could be shown (independently from the X-ray crystallographic data) by a ^{119}Sn CPMG-spin echo experiment, combined with CP MAS and rotor-synchronised data acquisition[4].

This time we have used the same compound, $(Me_2SnS)_3$, to test the practical limits for the ^{119}Sn CP-COSY experiment, as this compound represents a "real" solid with dilute spin-1/2 nuclei. We have to admit, however, that for most other ^{119}Sn cases in the solid state one will be confronted with much larger anisotropies. The initial 90°-pulse in the COSY pulse sequence was replaced by the cross polarisation $^{1}H \rightarrow ^{119}Sn$ sequence, and the rotation rate was chosen so that there is no overlap between "real" cross peaks and cross peaks caused by the spinning sidebands.

The result of this experiment on $(Me_2SnS)_3$ is shown in figure 1, together with the conventional ^{119}Sn CP MAS spectrum. As can be seen, there is no intrinsic problem with the ^{119}Sn CP-COSY experiment: the desired information is clearly visible as cross peaks in the contour plot. In general, however, we feel that one will suffer from severe practical limitations using the homonuclear CP-COSY experiment for

^{119}Sn-work. There will generally be signal-to-noise problems, (i.e. the experiment will be very time-consuming) and quite often it could be difficult ... impossible to adjust the rotation rate so that there will be no overlap problems with the numerous spinning sideband cross peaks (well, in theory one could synchronize the data acquisition with the rotor period).

To cut the long story short: the CP-COSY experiment works quite well for "real" solids - but we can only recommend it as a "last resort", unless you have problems with your spectrometer sitting idle ....

Best Wishes
Larry Merwin
Angelika Sebald

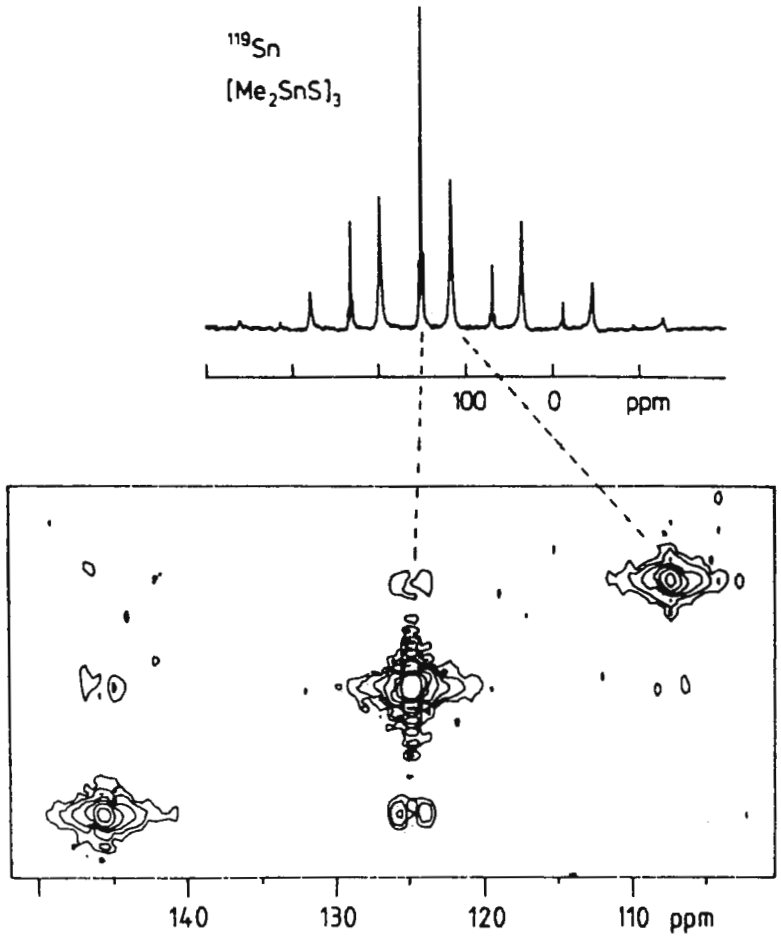

Figure 1: Conventional ^{119}Sn CP MAS spectrum of $(Me_2SnS)_3$ (top), together with an expansion (centre band region) of the ^{119}Sn CP-COSY contour plot.

References

1) R. Benn, H. Grondey, C. Brevard and A. Pagelot; J. Chem. Soc. Chem. Commun. 102 (1988)

2) C.A. Fyfe, H. Gies and Y. Feng; J. Am. Chem. Soc. 111, 7702 (1989)

3) T. Allman; J. Magn. Reson. 83, 637 (1989)

4) R.K. Harris and A. Sebald; Magn. Reson. Chem. 27, 81 (1989)

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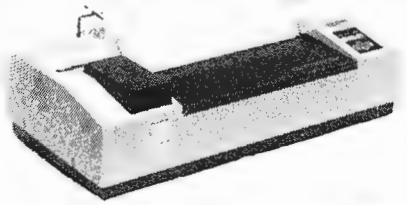

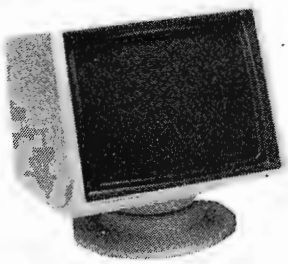

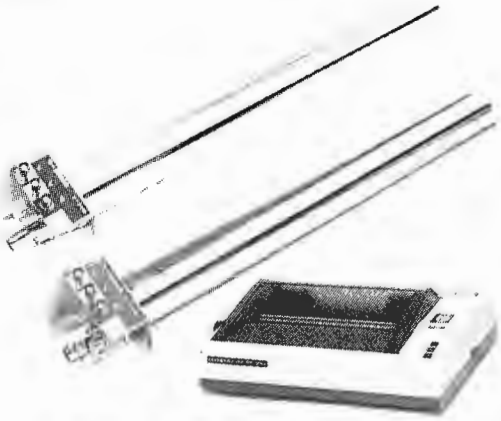

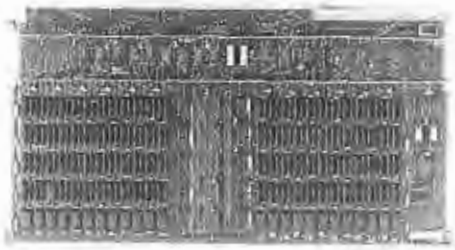

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IN REPLY REFER TO:

6120-103:KJM
20 February 1990
(received 2/24/90)

Professor Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 9430

CRAMPSI of Aluminosilicate Glasses

Dear Barry,

Recently we have been interested in studying the coordination of aluminum in an assortment of ground aluminosilicate glasses using ^{27}Al NMR with magic angle spinning. Unfortunately, all initial attempts at getting the samples to spin at anything greater than a few hundred Hertz on our MSL-300 were fruitless. A variety of techniques were used in our efforts to induce higher spin rates, including grinding of the samples to finer particle size, mechanical mixing with sulfur powder and other materials, changing rotors, changing/repositioning rotor caps, varying bearing and drive pressures, etc., all to no avail.

Finally, we resorted to the technique of **C**ontingent **R**otation through **A**queous **M**odulation of **P**rofluentless **S**ilicates, **CRAMPSI**. We simply mix our finely ground sample with a small quantity of water (distilled works best) to form a thick paste with the viscosity of spackling compound. The sample is then loaded into the rotor and spun up as usual. Our experience has been that normal spin rates (1-5 KHz) are usually attainable within the first few tries using this technique; without **CRAMPSI** we were unable to get satisfactory spinning at all!

We believe that the water imparts sufficient flow to the sample and thereby allows the contents of the rotor to distribute itself uniformly, resulting in balanced rotation. Although we experienced no difficulties with this technique, we do caution that excess water content in the paste could result in high pressure on the rotor cap during spinning and subsequent detachment from the rotor. Also, this technique is obviously limited to samples which are unaffected by the presence of water, although other liquids may give equally successful results.

We are now in the process of converting our MAS probe over to an aluminum-free version and will have background free ^{27}Al spectra in the near future.

Please credit this entry to the Naval Research Laboratory.

Ken

K. J. McGrath
Code 6120, Chemistry Division

C. Merzbacher

Texas A&M University NMR Newsletter - Book Reviews

Book Review Editor:
William B. Smith, Texas Christian University, Fort Worth, Texas

"Quantum Description of High-Resolution NMR in Liquids"

by

Maurice Goldman

Oxford University Press, 200 Madison Ave. New York 10016, U.S.A.;

xiv + 268 pp.; 1988; $65.00; ISBN 0-19-855639-X

It is the author's intent to provide a "self-contained description" of one- and two dimensional NMR experiments for readers without an adequate background in quantum mechanics. The first chapter (22 pages) is an overview of NMR including classical and quantum descriptions. The next four chapters provide the background for the use of the operator form of the statistical density matrix. This is the method of choice in describing the more popular 2D methods in Chapters 6-8. Chapter 2 (29 pages) emphasizes vectors and operator algebra and Chapter 3 (41 pages) is concerned with angular momentum and their relation to rotation operators. The author makes a point of not using matrix forms except for qualitative purposes. The properties of the density matrix are introduced in Chapter 4 (21 pages). The formalism developed in the previous chapters is brought together in Chapter 5 ("Quantum Description of NMR," 24 pages). Chapter 6 (20 pages) includes a description of the double Fourier transform method before describing the topics of homonuclear and heteronuclear correlation spectroscopy. This is followed by the longest chapter (Chapter 7, 51 pages) explaining heteronuclear and homonuclear shift Correlation Spectroscopy, and polarization transfer. This is followed by a chapter (Chapter 8, 39 pages) introducing applications of multiple quantum coherence for multi-Q-filtered COSY and INADEQUATE methods. The NOESY technique is also described in this chapter. The final chapter (40 pages) is concerned with the fundamentals of relaxation theory, emphasizing dipolar relaxation in two spin-½ systems. About five pages are concerned with other mechanisms.

Goldman's book is a welcome addition to the many books now available on 1D and 2D NMR. This semester I am using it in a graduate course in magnetic resonance theory for students who are not generally specializing in NMR. In the past it was necessary to use selected chapters from texts on quantum mechanics, angular momentum, and magnetic resonance. Even though two semesters of quantum mechanics are prerequisites for the course, it is unlikely that the students would have been exposed to topics such as density matrices and angular momentum operators as used for NMR applications. For the purposes of this review, I had hoped to have some feedback from the students. Because the book seems to be difficult to get from the publisher, the students have only had it available for 2 - 3 weeks.

Mike Barfield
University of Arizona

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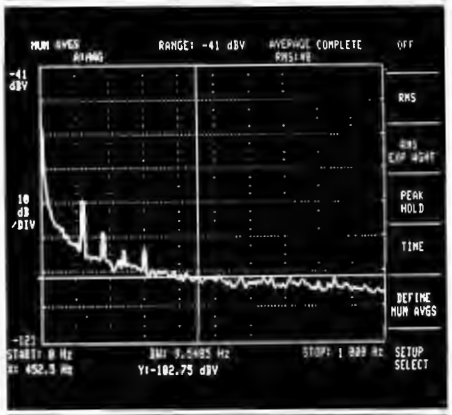

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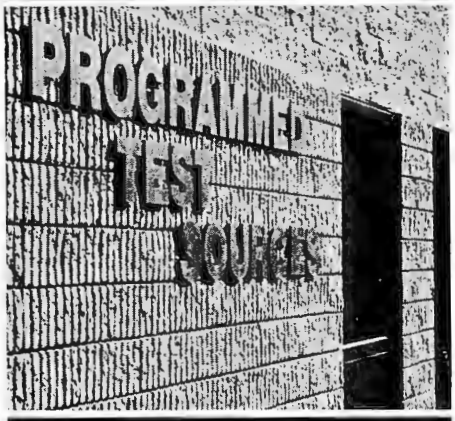

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March 12, 1990
(received 3/16/90)

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

Dear Barry:

One of the more annoying aspects of most presentations of density matrix theory in NMR is that explicit forms of the various spin operators one needs are usually not given explicitly. Usually, a rather annoyingly abstract presentation of their construction is about the best one can hope for. The most commonly occurring problem is that most authors assume that the reader is already an expert in the density matrix theory and quantum mechanics needed to understand the theoretical background of NMR. From my experience, this is rarely the case.

In particular, the presentation of rotational operators corresponding to ordinary rectangular pulses is more often opaque than not. This is puzzling since the basic recipe for the generation of such operators is really quite simple. All that one really needs is an *explicit* presentation of how to generate the operators for rotation by an arbitrary angle, θ, about the x– and z–axes in the rotating frame. Given these, everything else falls in place quite rapidly.

To be more explicit, the rotational operators for a single spin are

$$\mathbf{R}_x^{\pm}(\theta) = \begin{bmatrix} \cos(\theta/2) & \mp i\sin(\theta/2) \\ \mp i\sin(\theta/2) & \cos(\theta/2) \end{bmatrix} \tag{1}$$

for rotation about the x–axis (*i.e.*, a θ_x pulse) and

$$\mathbf{R}_z^{\pm}(\theta) = \begin{bmatrix} \exp(\mp i\theta/2) & 0 \\ 0 & \exp(\pm i\theta/2) \end{bmatrix} \tag{2}$$

for a similar rotation about the z–axis. In the above expressions, $\mathbf{R}^+$ is the regular form ($\mathbf{R}$) of the rotation operator and $\mathbf{R}^-$ is its inverse (*i.e.*, $\mathbf{R}^{-1}$). These operators work in pairs on the density matrix, which we shall call $\mathbf{D}$. For example, rotation of the nuclear magnetizations by 90° about the x–axis would be calculated by $\mathbf{R}_x^{-1}(90°)\cdot\mathbf{D}\cdot\mathbf{R}_x(90°)$.

Where many presentations become unclear is where one wishes to represent a pulse with an arbitrary phase angle, ϕ, in the xy–plane. (For example, $\phi = 0°$ for rotation about the x–axis and $\phi = 90°$ for rotation about the y–axis.) This is quite easily done if one remembers one's elementary *classical* mechanics. Rotation about an arbitrary axis in the xy–plane can be decomposed into rotation about the x–axis by the pulse angle, θ, and

rotation about the z–axis by the phase angle, ϕ. That is, such an arbitrary rotation can be easily represented as

$$\mathbf{R}_{\phi}(\theta) = \mathbf{R}_z^{-1}(\phi)\cdot\mathbf{R}_x(\theta)\cdot\mathbf{R}_z(\phi). \tag{3}$$

For example, if we let $\phi = 90°$ and perform this operation, we get the familiar form

$$\mathbf{R}_y^{\pm}(\theta) = \begin{bmatrix} \cos(\theta/2) & \pm\sin(\theta/2) \\ \mp\sin(\theta/2) & \cos(\theta/2) \end{bmatrix}. \tag{4}$$

for rotation by an angle, θ, about the y–axis. (This is easily seen if one remembers that $\exp(\pm i\phi/2) = (1 \pm i)/\sqrt{2}$ when $\phi = 90°$; all that is required is a little algebra to get this.)

We now rewrite eqn (3) in a more explicit form:

$$\mathbf{R}_{\phi}(\theta) = \begin{bmatrix} \cos(\theta/2) & -i\exp(+i\phi)\sin(\theta/2) \\ -i\exp(-i\phi)\sin(\theta/2) & \cos(\theta/2) \end{bmatrix}. \tag{5}$$

The inverse of this operator can also be obtained by replacing $\mathbf{R}_x(\theta)$ by its inverse in eqn (3) to give

$$\mathbf{R}_{\phi}^{-1}(\theta) = \begin{bmatrix} \cos(\theta/2) & +i\exp(+i\phi)\sin(\theta/2) \\ +i\exp(-i\phi)\sin(\theta/2) & \cos(\theta/2) \end{bmatrix}. \tag{6}$$

(It should be noted here that eqn (5) is a *much easier* way to derive eqn (4) than direct evaluation *via* eqn (3)!)

Having these explicit rotational (pulse) operators for a single spin, the operators for multiple–spin systems are then easily constructed using "Kronecker" matrix products. This is left as an exercise since if we let this go on too long, space restrictions would become prohibitive. (Of course, one could now accuse me of being less than perfectly frank in giving explicit expressions for multiple–pulse operators. My only defense is that I am restricting myself to single spins in this writeup!)

Sincerely yours,

Milton D. Johnston, Jr.
Prof. of Metaphysical Chemistry

Suggested title: Pulse operators and arbitrary phases

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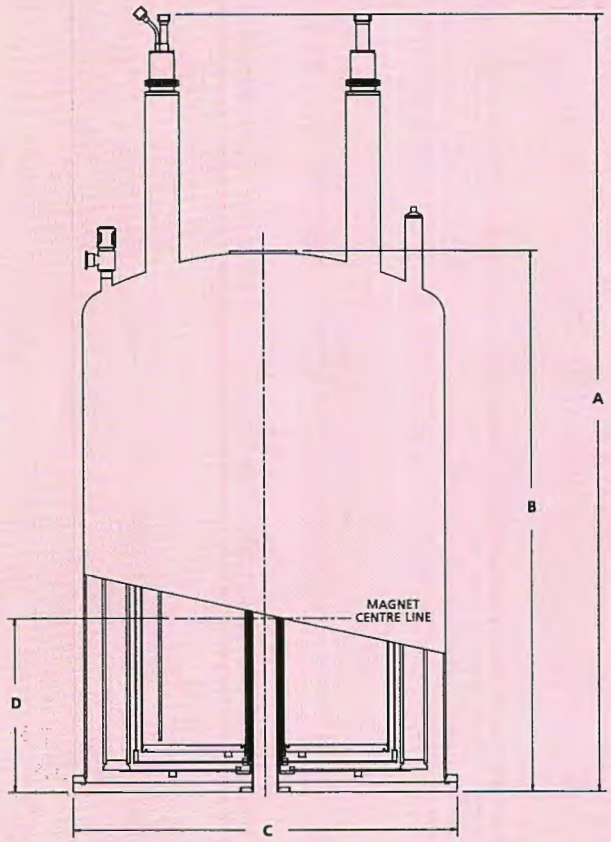
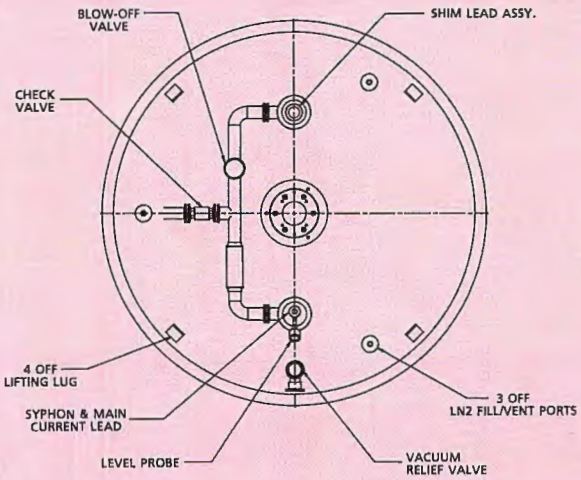
NMR MAGNET SYSTEMS





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

(received 3/1/90)

Cardiac Massage Inside the Magnet

Dear Barry,

In our P-31 and Na-23 NMR studies of heart preservation using the model of perfused rat heart we compare NMR data with haemodynamic parameters monitored simultaneously (see e.g. Schwalb et al. JMCC **19**, 991 (1987). In these studies, during the stage of post ischemic reperfusion, a certain fraction of the hearts fails to beat spontaneously. If this sad event would have happened in a standard physiological experiment, or, say, during an open heart surgery, you would give the heart a good hand massage or an electrical shock, and if you (and the patient!) are lucky the heart will beat again. However, in our case this happens while the heart is inside our AM-360WB NMR spectrometer. By loosing the heart we may loose a whole day's work and, even worse, heavily distort the statistical analysis by removing from the cohort those hearts that did not recover. In the following we describe our solution to the problem: a mechanical massage apparatus.

Fig. 1 is a technical sketch of the apparatus, which is inserted into a 20mm NMR tube at the beginning of the experiment. By pulling a silk wire A through the tube B part D presses the bottom of fingers C which rotate and squeeze the heart which is placed between them. A spring placed under the fingers pulls them back when the wire is loose.

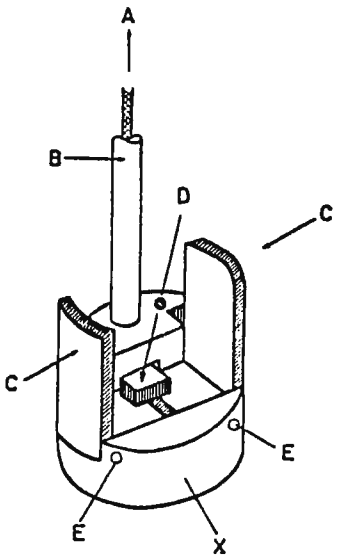
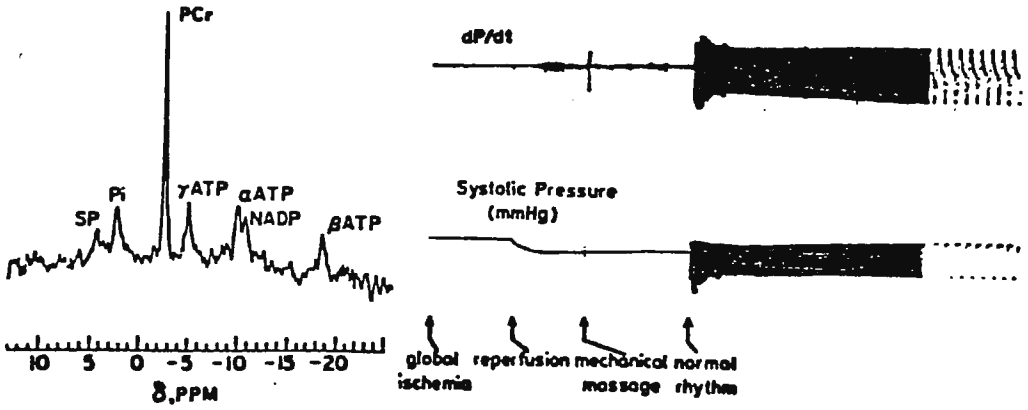


Fig. 2 demonstrates an experiment in which the haemodynamic functions: left ventricular peak pressure, (LVP) and dP/dt were recorded during the activation of the heart (see arrows) by the apparatus inside the NMR spectrometer. The massage was applied to a rat heart during the reperfusion period without any interference to the NMR measurement and the field homogeneity. A more detailed description of the experiment will appear in the one of the forthcoming issues of Magn. Reson. Medicine.

Sincerely yours,

Tammar Kushnir Gil Navon

Tammar Kushnir Gil Navon



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February 26, 1990

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

GE TEMPERATURE CONTROL

Dear Barry:

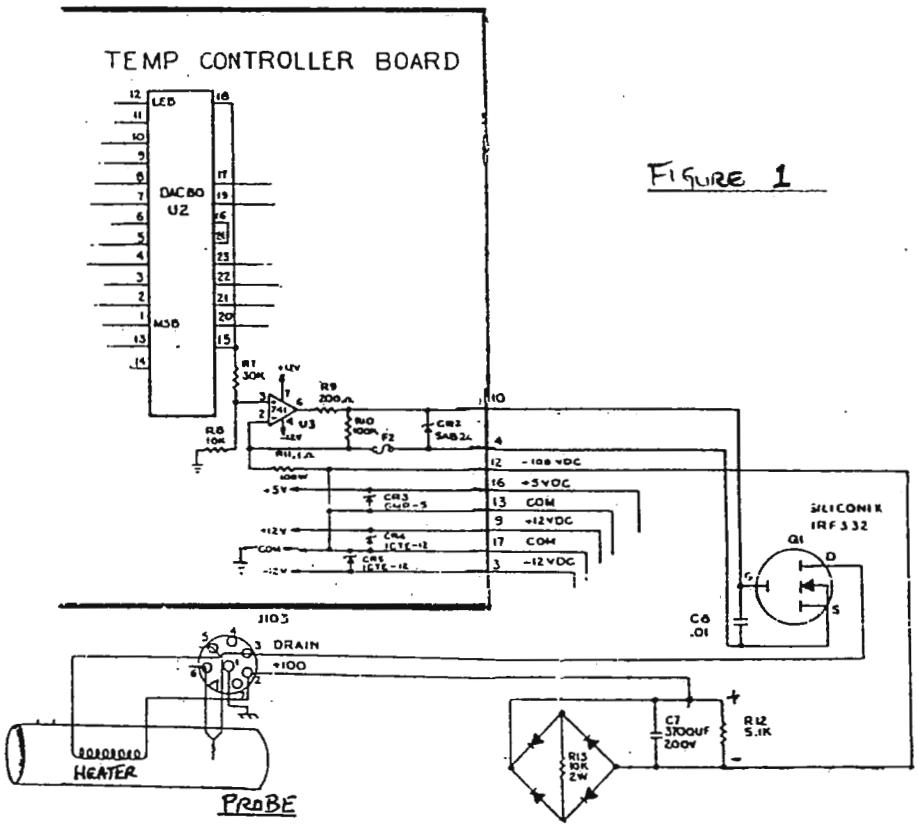

FIGURE 1

Referring to Figure 1: As designed, (at least on older Temperature Controllers, like ours) if the control circuitry U2, U3, etc. on the temp. controller board malfunctions and the gate (G) is driven high on the power FET (Q1), a thermal runaway will exist until fuse F2 blows, breaking the

probe heater circuit to ground. One solution G.E. made early on our system was to add a 20 OHM, 100 watt limiting resistor in series with the +100 VDC output.

On the other hand, if the power FET (Q1) shorts out or develops resistance between the gate (G)/drain (D) and/or between drain (D)/source (S), as happened to us recently, not only will fuse F2 blow, because the heater sees +100 VDC to ground, but another +100 VDC current path is developed through the FET's drain (D) and Gate (G) by way of R9, U3 and R8 to ground. Once the fuse (F2) opens, these 1/4 watt components literally explode. Of course we also lost all the "chips" associated with the +12 VDC.

We may be wrong, but it seems the circuit placement of the fuse (F2) is doing more harm than good. Instead of having the fuse on the minus side of the +100 VDC, wouldn't it make more sense to put it on the plus side? Then when an overcurrent exist due to a bad controller board or a shorted FET, the fuse opens and the +100 VDC is out of the circuit completely.

Sincerely,

Jim Loo

Duke University

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SUNBOR

February 26, 1990
(received 3/1/90)

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

Dear Barry:

An Affordable Alternative to Better Variable Temperature Control

Those of us working with samples in H_2O solution, where data are required for the labile backbone amide, amino or imino resonances in proteins and nucleic acids, are aware of the need to maintain a constant temperature throughout the entire period of data acquisition. While I (MGZ) was at Columbia University, we had Bruker AM consoles and frequently many of our 2D runs were spoiled due to fluctuations of the temperature within the probe. These problems were particularly acute for ^{31}P or ^{1}H spectra of DNA samples run in H_2O solution where only a slight (0.2° C) temperature change would produce severely distorted cross peaks in 2D spectra. The variable temperature (VT) units on the Bruker AM consoles were (in my opinion) terrible. In a previous TAMU article by Bax & Tschudin, better temperature control of the Bruker VT unit was obtained by keeping the room temperature (near the VT unit) constant, since the magnitude of the displayed "error signal" was the result of differences between the room temperature and the desired probe temperature, the latter signal being displayed on the VT unit and set by the operator. I have found that in addition to maintaining a constant room temperature, better control of the probe temperature can be obtained by maintaining a constant VT air temperature. In fact, this is really nothing new, as you can buy special devices to keep the air temperature constant (the NIH regional NMR Center at University of Wisconsin has such a device), but these devices generally cost around $4,000-5,000. I also believe that now Bruker has available a similar device for keeping the air temperature more constant.

Alternatively, I bought a cheaper ($1,200) Neslab cooling bath equipped with an analog temperature controller. Immediately before entering the probe, the VT air line was interrupted with a copper coil, which was kept immersed in the Neslab cooling bath. The temperature of the bath is usually kept at -25° C using MeOH as the bath coolant. The air line which leaves the bath and enters the probe was wrapped several times with special pipe insulation tape (usually obtainable for a low cost at most hardware stores). The bath is kept running continuously at -25° C. In addition, despite the bath being only 5 feet away from the magnet (with the bath compressor motor clicking on and off at irregular intervals), no additional noise in either 1D or 2D spectra were apparent; i.e. the bath is constructed of stainless steel. The improvements in the quality of 2D data from using the bath were quite dramatic.

I am now working with Dr. Takashi Iwashita at the Suntory Institute for Bioorganic Research (SUNBOR) in Osaka, Japan (Professor Koji Nakanishi is Director of SUNBOR) and we are engaged in several projects pertaining to the structure elucidation of proteins in solution. The spectrometers at SUNBOR are General Electric instruments (GN-500 & GN-300) and we have encountered similar problems with maintaining constant temperatures during the measurement of 2D spectra. The VT unit on the GN console seems to function better than the VT unit on the Bruker AM console, although still slight variations exist. As before, these temperature fluctuations were partly removed by using a cheap VT cooling bath. The only problem with using this method at SUNBOR is that the air is not 100% dry and therefore the small amounts of water in the air can freeze in the copper coils. Thus, we generally keep the bath temperature at -5° C.

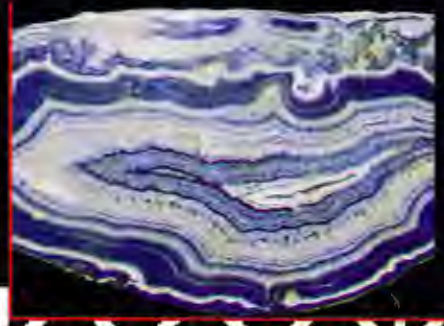
Kindest regards,

T. IWASHITA
Takashi Iwashita

Mike
Michael G. Zagorski

Spectral Data Services, Inc.

NMR Analysis



818 Pioneer
Champaign, IL 61820 USA
Telephone: (217)352-7084 Telefax: (217)352-9748

Our Background and Philosophy

Spectral Data Services, Inc., was founded in 1985 in order to provide rapid, first class NMR data acquisition capabilities and data analysis to industrial, university and governmental clients who either do not have modern high-tech Fourier transform NMR spectrometers, or who are plagued by extremely long in-house turnaround times.

We started with a 270 MHz spectrometer, then added a wide-bore 360 MHz spectrometer, followed by a second wide-bore 360 instrument in 1989. We have capabilities for all modern 1D and 2D experiments, as well as solid-state, liquid-state and gas phase (e.g. Xe-129) experiments. Spectra are typically recorded in less than one working week after sample receipt, and results are sent via overnight mail, or by FAX. For those requiring data in a hurry, our *Spectral Express* service provides a 24 hour turnaround service capability.

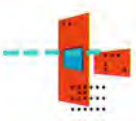
Spectral Data Services, Inc. was established by Dr. Gary L. Turner, Ph.D., who is currently assisted by five B.S. level instrument operators and administrative staff. Gary has over ten years of experience in solid and liquid-state NMR and is dedicated to solving *your* NMR needs. As required, additional consultants can be added to provide extra insight into solving your particular type of problem—from patent disputes, to difficulties with pilot plant runs.

Our philosophy is simply to be the best, friendliest, most helpful, rapid turnaround NMR service company available. We want to help solve *your* problems!

We look forward to working with you on:
Polymers • Fossil Fuels • Pharmaceuticals • Zeolites • Catalysts • Food Science and Technology • Oil Recovery • Patent Infringement • Advanced Ceramics • Superconductors • Agrochemicals

Geri, Margaret, Gary, David and Brenda

"We provide a total NMR service—from sample preparation and data acquisition, to data interpretation and report preparation. Our level of commitment to hard work and high quality service is the same—whether you're talking about the President, our instrument operators or our support staff. It's our people that make the difference."



The Services We Offer

Liquids NMR All three of our instruments: two 360 MHz spectrometers with wide-bore (89mm) magnets, and a 270 MHz standard (54mm) bore spectrometer, routinely perform liquid-state NMR experiments.

SDS has 5mm, 10mm, and 20mm sample capabilities, investigating anything from a few micrograms (H-1) to very large, dilute spin systems (e.g. C-13, N-15).

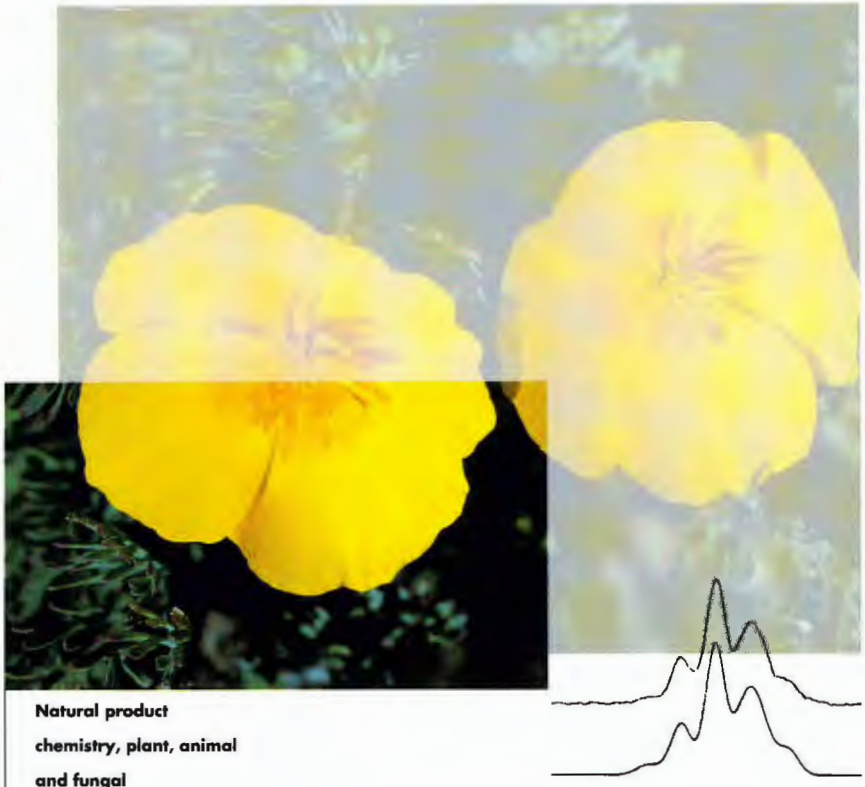
We carry out all routine 1D and 2D pulse sequences, such as DEPT, APT, COSY, NOESY, etc.

Spectral Data has full variable temperature capabilities—from -100°C to 140°C, and frequently carries out detailed polymer characterizations using VT equipment. Many questions about polymer structure can usually be answered from a simple C-13 spectrum, and a complete VT analysis can often give important information on polymer dynamics.

We also have routine wet chemical capabilities, enabling us to make your samples up for you.

All normal relaxation (T_1, T_2, $T_{1\rho}$) measurements are available, as are special decoupling schemes (for NOE measurements, or for quantitation).

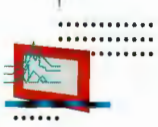
We are not limited to H-1/C-13 analyses—other solution state nuclei we frequently investigate include: N-15, O-17, Al-27, Si-29, and P-31.



Natural product chemistry, plant, animal and fungal biochemistry–we have experience in a variety of such areas.

"All you have to remember can be summed up in a few words: commitment to excellence and personal service, at a reasonable price."

Brenda setting up for proton solution data acquisition.



Solids NMR Each of our instruments can perform solid-state NMR experiments. We have magic-angle spinning and cross-polarization magic-angle sample spinning capabilities, with Al free probes, and fast MASS rates—up to over 10kHz. Where necessary (e.g. Si-29 NMR) we can provide spectral simulations of your samples.

SDS personnel have extensive experience in quantitative solid-state NMR, of e.g. Cs-133 and Al-27, as well as qualitative C-13 CPMAS of polymers and other organic species.

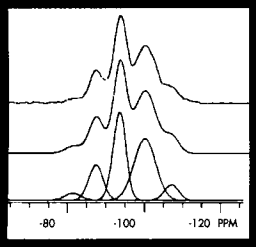
SDS also routinely performs static spin-echo experiments on e.g. H-2, O-17, and other "wide-line" nuclei.

Also available is a full variable-temperature range, from -100°C to 140°C, with or without magic-angle spinning, and "variable-angle" sample spinning, suitable for NMR of some quadrupolar nuclei.

We have also recently offered a "sealed-sample" service, for air or moisture sensitive compounds, as well as gas-phase (Xe-129 NMR) characterization of catalyst samples using the technique developed by Fraissard (*Zeolites*, *8*, 350 (1988)).

Gary discusses NMR project with attorney Traci Nally-Harris.

"We have over ten years experience in solid-state NMR... and specialize in Al-27 and Si-29 NMR of catalysts."



Si-29 MASS + SIMULATION, Linde Y

We use both custom designed solid-state NMR spectrometers as well as multinuclear commercial solution state instrumentation, together with individually-tailored pulse sequences for your particular needs.

Price Schedule

270 MHz Instrument: $45/hr
360 MHz Instruments: $65/hr
Spectral Simulations: $65/hr
Surcharge for VT or Air-sensitive: $10/hr
*Spectral Express: $100/hr**
FAX transmission: $1/min

Included in the above are all normal operating expenses, sample preparation, and routine interpretation.

Personal Attention, High Quality

We provide NMR data services that are fast, reliable, and of high quality. We encourage our clients to call and discuss their projects, and to ask questions concerning the results. Data are routinely sent using overnight delivery, free of charge.

For more in-depth studies, we welcome on-site collaboration, data presentation, experiment design, data discussion and consultation—at your location, Spectral Data Services, Inc., or a third party location.

For further details contact Gary Turner at (217) 352-7084 or FAX (217) 352-9748.

**Express overnight data service. Minimum 12 hours. Subject to availability. Please inquire.*

S D S

The University of Leeds LEEDS LS2 9JT

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Telex 556473 UNILDS G Fax (0532) 336017

School of Chemistry

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

1st March 1990 (received 3/5/90)

3D NMR!

Dear Dr. Shapiro,

In the course of our protein NMR work, we are sometimes interested in observing the relative intensities of cross peaks in 2D spectra. These can be displayed using a "stacked plot". However many such plots are often necessary, since peaks that are subsequently found to be interesting often tend to be hidden behind larger ones. Ideally a 3D surface is required (or continuous access to computer graphics facilities), but in the absence of such a facility a limited solution is the use of "stereo pairs". Using Bruker software, stack plots can be drawn which differ in slant (ie. X entry to the OP command). Two such plots can approximate a stereo pair. The greater the difference in X settings for the two plots, the greater is the apparent depth of the 'image'. Strange effects such as being able to see a peak 'through' a larger one become apparent using a stereo viewer. An example of such a stereo pair is given below. The result is purely a curiosity as of course only relatively small areas of spectra can be accommodated.

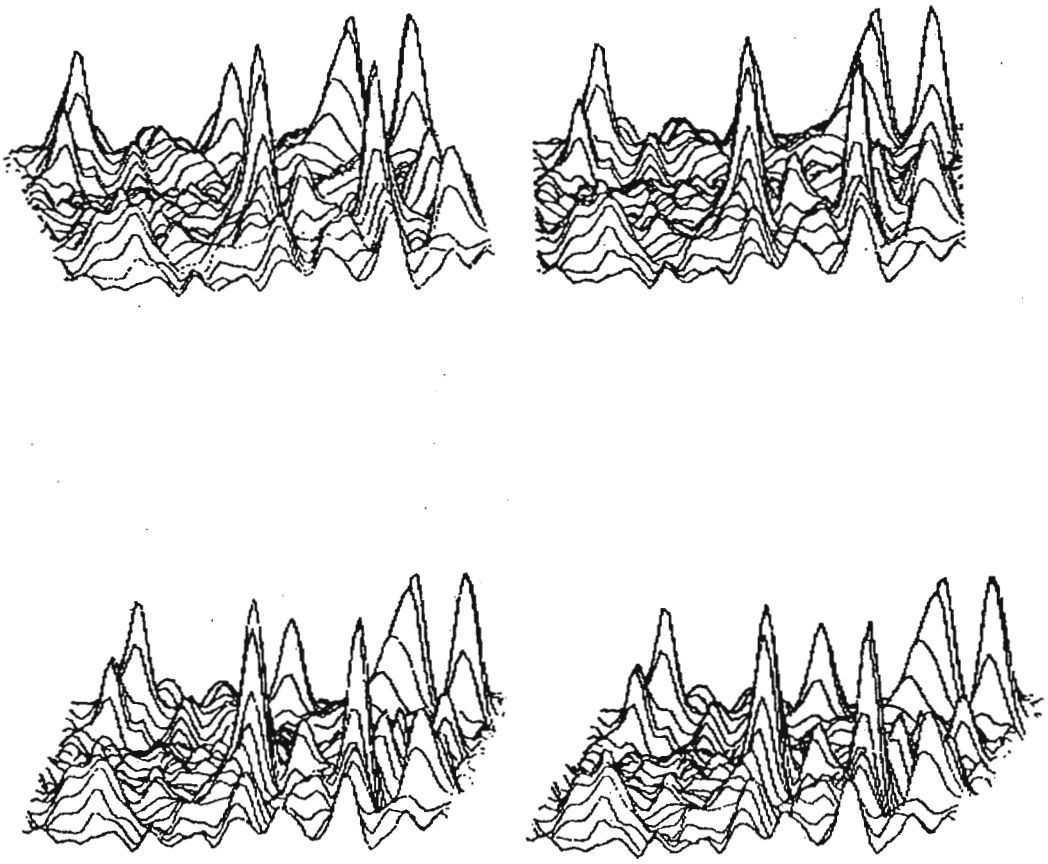
Please credit this contribution to the account of Dr. J. Kennedy.

John. Arnold

Julie Fisher

Dr. J.R.P. Arnold
(Biological NMR Centre ,Leicester)

Dr. J. Fisher
(School of Chemistry, Leeds)



STEREO PAIR

Postdoctoral Research Associate

Applicants are invited to apply for the position of Postdoctoral Research Scientist at the Suntory Institute for Bioorganic Research (SUNBOR) in Osaka, Japan. SUNBOR is a non-profit organization and is funded under the auspices of the Ministry of Education of Japan. All of the research work done at SUNBOR pertains to Basic Research and the official spoken and written language of the Institute is English. The person will be involved in several ongoing research projects which involve studying protein structure and function in solution using NMR and computational techniques. Collaborations with other well known academic scientists in Japan will be included as part of the research work. The facilities at SUNBOR include: GN-500, GN-300 and JEOL-400 MHz NMR spectrometers, Sun 3/60 and Iris 4D/25 graphic workstations. In addition, exposure to other new instrumentation at SUNBOR is possible: i.e., new JEOL-HX 110 tandem high resolution mass spectrometer for MS-MS applications as applied for peptide sequencing. Roundtrip airfare, subsidized housing and company fringe benefits such as health care will be provided. Salary is approximately $24,000 and will be paid in yen (no U.S. tax will have to be paid). If needed, additional overseas travel time (min. six weeks) will be provided for subsequent job interviews. SUNBOR is a 25 minute trainride outside of beautiful, downtown Kyoto and a 40 minute trainride from downtown Osaka. Interested persons should contact Professor Koji Nakanishi, Department of Chemistry, Columbia University, New York, NY 10027 USA (tel. 212-854-2169).

ROYAL INSTITUTE OF TECHNOLOGY

Department of Physical Chemistry

Dr. Ulf Henriksson

Prof. B. L. Shapiro
TAMU Newsletter
960 Elsinore Court
Palo Alto
California 94303 USA

February 26, 1990
(received 3/3/90)

Dear Professor Shapiro:

Dynamics of surfactant micelles with cryptand-complexed counterions.

It has been reported from fluorescence quenching measurements (Evans *et al. J. Phys. Chem.* **1988**, *92*, 784.) that sodium dodecylsulphate (SDS) micelles decrease their aggregation number considerably when the sodium counterions are complexed with the cryptand *C222*. We were curious whether NMR relaxation could detect any difference between these micelles and normal micelles. Multifield ^{2}H relaxation combined with ^{13}C T_1 and NOE at a few fields have been shown to give both detailed and accurate information on the molecular dynamics in surfactant aggregates (Söderman, Henriksson and Olsson *J. Phys. Chem.* **1987**, *91*, 116.). Figure 1 shows that the ^{2}H relaxation of α-deuterated SDS is faster in the complexed micelles in the whole frequency range 2-61 MHz which of course means slower molecular motions in spite of the reported low aggregation numbers. However, the correlation time that can be obtained from the relaxation data is sensitive to the *size* of the micelle and not directly to the aggregation number and the NMR and fluorescence results can be reconciled by assuming that the complexed hydrophobic $C222Na^+$-counterions are partly solubilized in the micelles. From the ^{13}C relaxation data it is possible to evaluate correlation times for the alkyl chain motions τ_c^{ch} for the individual methylene segments along the chain (figure 2). As seen the counterion complexation has a clear effect on the dynamics in the upper part of the chain which of course is most affected by the presence of partly solubilized counterions.

Sincerely,

Molly Ginley

Ulf Henriksson

PS Please credit this contribution to Peter Stilbs subscription.

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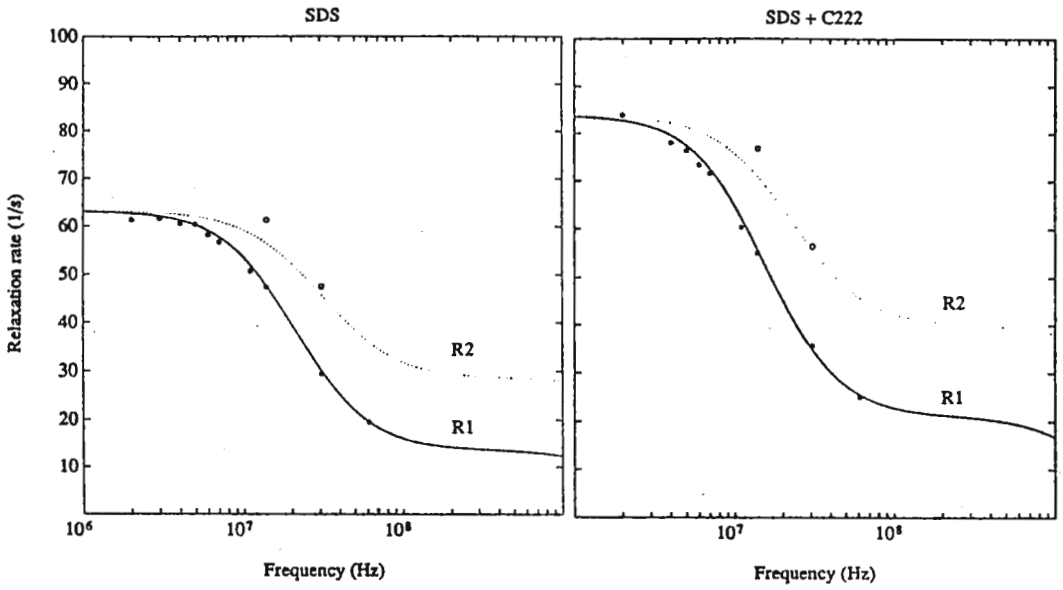


Fig. 1 ^{2}H-relaxation rates for α-deuterated SDS.

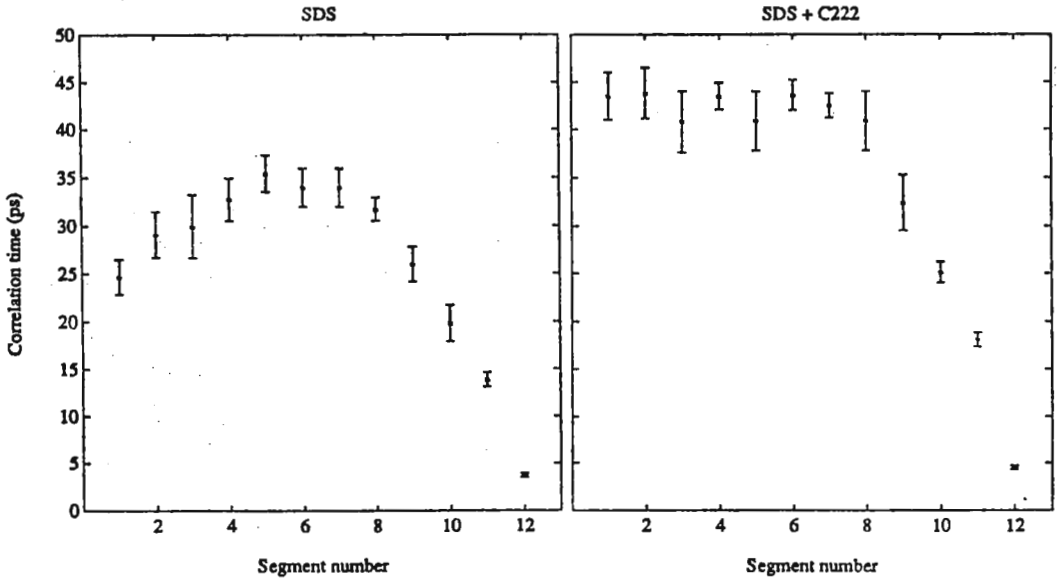
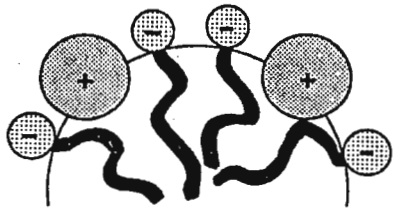


Fig. 2 Correlation time τ_c^{ch} for the local motion of individual methylene segments.

Schematic picture of the binding of hydrophobic $C222Na^+$-ions to spherical SDS micelles.



Continued from page 1:

FORTHCOMING NMR MEETINGS

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

Gordon Research Conference: Magnetic Resonance in Biology and Medicine, **July 16-20, 1990**; Tilton School, Tilton, NH. Chairman: R. G. Bryant. Contact: Dr. A. M. Cruickshank, Gordon Research Center, Univ. of Rhode Island, Kingston, RI 02881-0801.

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. 381 (June) ---------- 18 May 1990
No. 382 (July) ---------- 15 June 1990
No. 383 (August) ---------- 20 July 1990
No. 384 (September) ----- 17 August 1990

* * * * *

Mailing Label Adornment: Is Your Dot Red ?

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

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Attention overseas subscribers: If you must use paper which is longer that 11", *please* take care that nothing appears below 10" (25.5 cm) on your pages. It is costly to make reductions. Thank you.

* * * * *

✱ ***(415) 493-5971 is my home (and only) telephone number. It would be greatly appreciated if all calls to this number could be restricted to the hours 8:00 a.m. to 10 p.m., Pacific Coast Time. Thank you for your kind cooperation.***

B.L.S.

* * * * *

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.

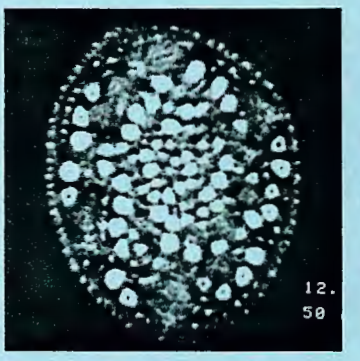

***Fig. 1**—Agapanthus bud*
Matrix 256 × 256, TR 200
Slice 2 mm, TE 30
FOV 12.8 mm, NEX 4,
45° Tip Angle DEFT
Sequence

***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.*

GE NMR Instruments

255 FOURIER AVENUE, FREMONT, CA 94539
(415) 683-4408, TELEX 910 381 7025 GE NMR FRMT
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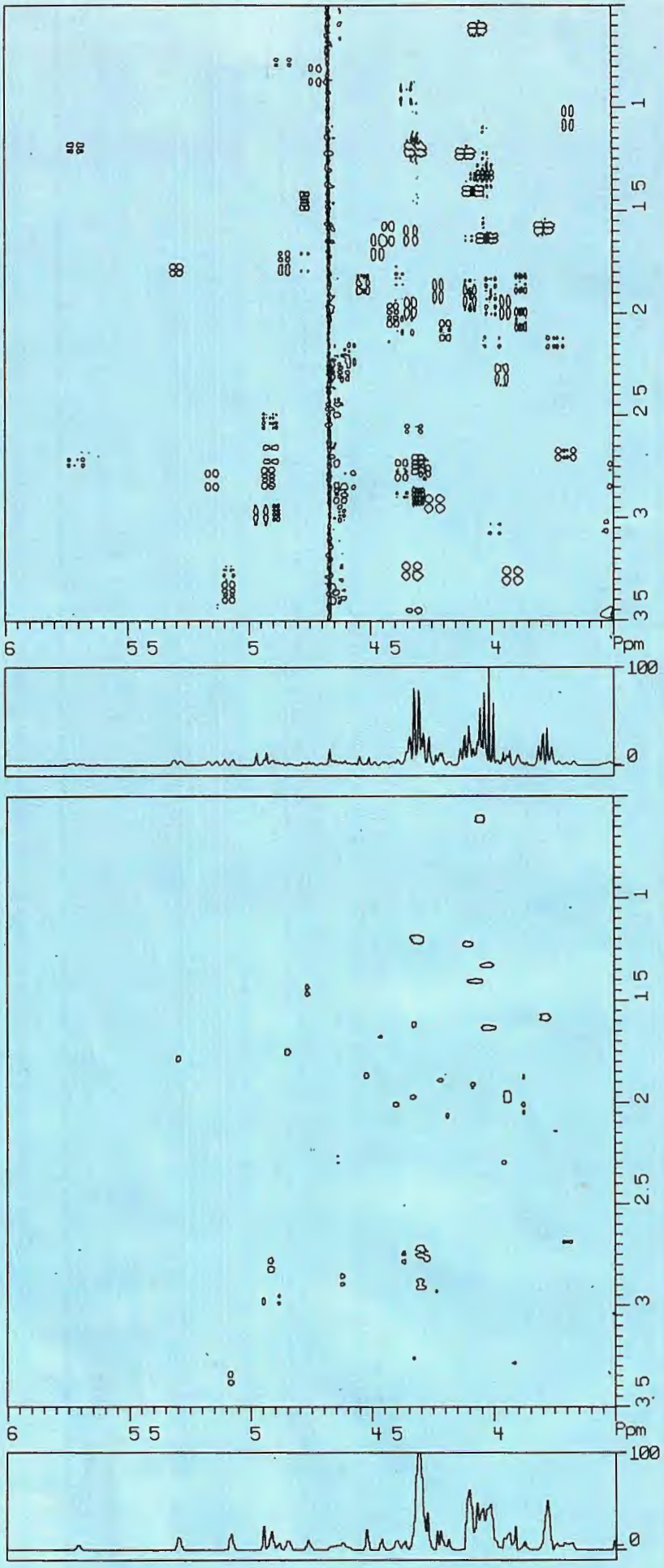
VPLX

A GSX UPGRADE

JEOL USA introduces the VPLX data processing package, our latest upgrade to the GX and GSX NMR spectrometers. When used with the latest network options of Multi-PLEXUS, VPLX provides the power and speed of a VAX™ and eliminates the need to learn a new set of software commands.

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.



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).