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TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. % D	Density (g/ml)	MP (°C)	BP (°C)	$-X_v \times 10^6 @ (°C)$
D-11	Acetone-d <sub>6</sub>	CD <sub>3</sub> COCD <sub>3</sub>	99.9%	1.1	17	56	0.551 (32)
D-120	Acetone-d <sub>6</sub> + 1% TMS		99.9%				
D-13	Acetone-d <sub>6</sub>	CD <sub>3</sub> COCD <sub>3</sub>	99.9%	0.8	94	57	0.460 (20)
D-121	Acetone-d <sub>6</sub> + 1% TMS		99.8%				
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D-14	Chloroform-d	CDCl <sub>3</sub>	99.8%	1.50	-64	62	0.740 (20)
D-21							
D-122							
D-130							
D-28	Chloroform-d	CDCl <sub>3</sub>	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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WCV-XL (S-100)	11" X 26"	HA-100, HA-100J, HA-100D	Gridded-Two Color
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## FORTHCOMING NMR MEETINGS

International Symposium NMR Spectroscopy: Structure and Dynamics of Polymeric Materials in the Solid State, Sponsored by the ACS Division of Polymer Chemistry, **December 5-8, 1989**; Keystone, Colorado; Contact: Mrs. Betty J. Schreiner, E. I. du Pont de Nemours & Co., Experimental Station, Wilmington, DE 19880-0356; (302) 695-4817.

Chemical Frontiers in Magnetic Resonance of Living Systems, Workshop in the Pacific Basin Congress/American Chemical Society, **December 17-22, 1989**, Honolulu, Hawaii; See Newsletter 372, 52.

Spatially Determined NMR, Sponsored by the British Radiofrequency Spectroscopy Group; **December 17-20, 1989**; Cambridge University, U.K.; Contact: Prof. L. D. Hall, Level 4 RTC, Addenbrookes Hospital, Hills Road, Cambridge CB2 2QQ, England: (44) (223) 336805.

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

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

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

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

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No. 375 (December)--17 November 1989

No. 376 (January)-----15 December 1989

No. 377 (February)-----19 January 1990

No. 378 (March)-----16 February 1990

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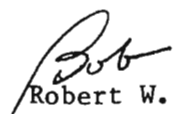
August 31, 1989  
(received 9/2/89)

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

Dear Dr. Shapiro:

Our AM-600 NMR spectrometer has been operational for a year now. The seemingly effortless installation accomplished by a team of experienced engineers (H. Gretler, J. Hatcher and A. Aharon) is still a fond memory. The sensitivity tests produced results which exceeded specifications by a comfortable margin for all nuclei tested. For example,  $^{15}\text{N}$  (90% formamide, proton decoupled, without NOE) S:N was 100:1. This test was run using a 1 kHz spectral width. Months later, when we repeated this test using a 4 kHz spectral width, the S:N was still 101:1 but spurious signals were present, too (Fig. 1b). We have managed to eliminate these undesirable signals and increase S:N another 12% (Fig. 1a S:N 113:1) by installing a low-pass filter (corner frequency 160 MHz) between the preamplifier and the console.

Sincerely,

  
Robert W. Dykstra

RWD/m

a



b

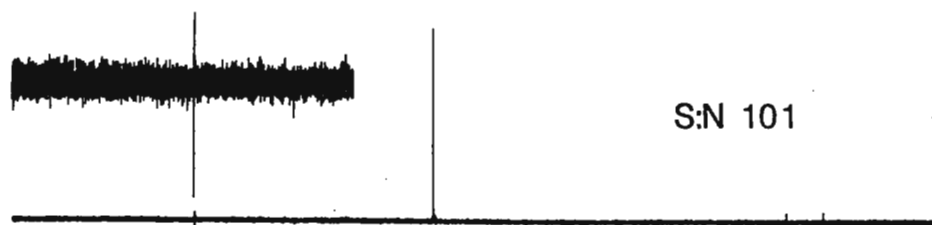
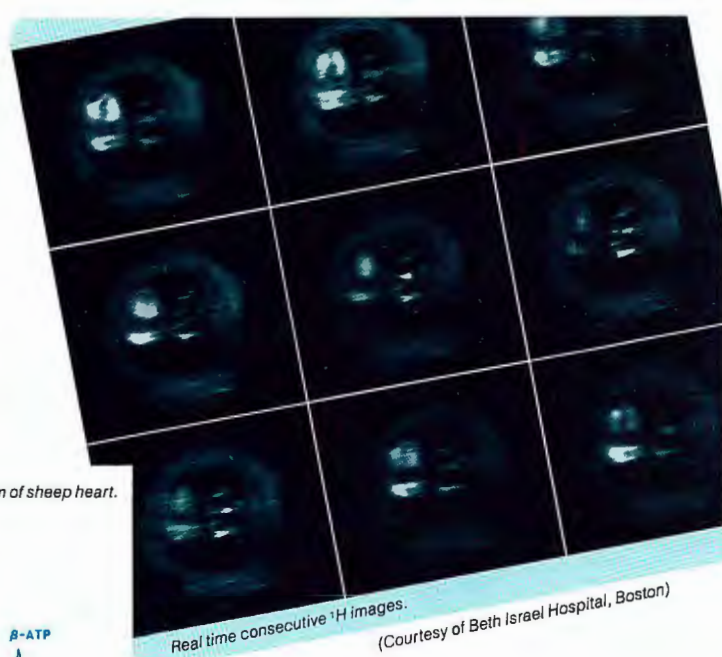


Figure 1



# Real Answers.



Real time consecutive  $^1\text{H}$  images.  
(Courtesy of Beth Israel Hospital, Boston)

## Real time.

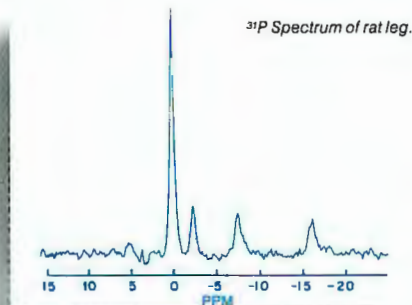
A series of consecutive real time images can be obtained in a single heartbeat using a fast gradient echo technique with phase reversal. Each image can be obtained in only 58 msec. The in-vivo images shown above illustrate the diastole-systole-diastole cardiac cycle in a mouse.

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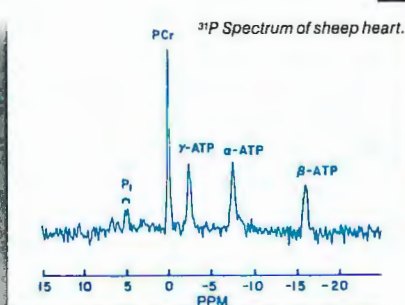
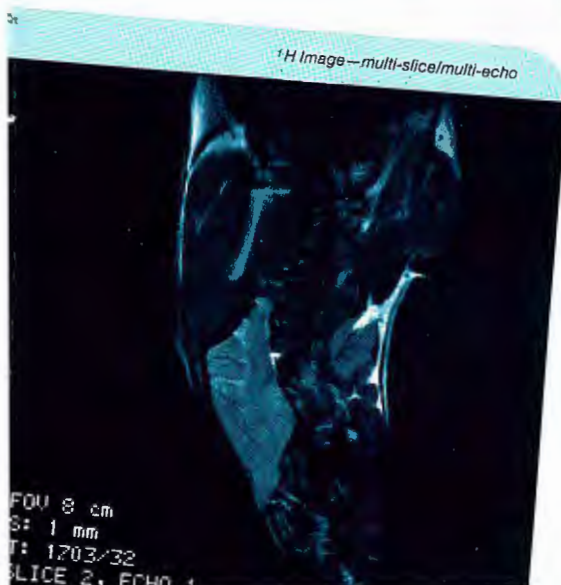


## S/N performance.

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

## Multi-slice/Multi-echo.

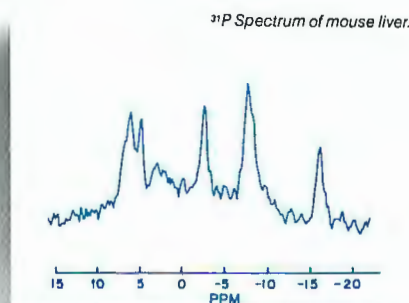
High quality image detail. An MSME image (256 x 256) taken of a rat head (TE = 33 msec, TR = 1700 msec). The slice thickness is 1mm and FOV is 8 cm. Details of the cerebellum, pharynx and the mystacial pads can be clearly discerned.



(Courtesy of Pittsburgh NMR Center)

## Large bore.

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



(Courtesy of Pittsburgh NMR Center)

## In-vivo 7.0 tesla.

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



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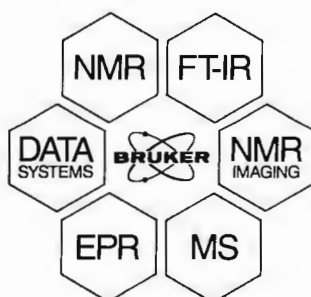
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Department of Physical Sciences

(received 8/30/89)

Professor B.L. Shapiro,  
TAMU NMR Newsletter,  
966 Elsinore Court,  
PALO ALTO,  
California 94303,  
USA

22nd August 1989

Dear Professor Shapiro,

### NMR AND MODELS OF ATHEROSCLEROSIS

We have been using NMR spectroscopy and imaging recently to investigate the development of atherosclerosis in various animal models of the disease. Initially we carried out some ex vivo experiments using water-suppressed (i.e. fat-only) images of rabbit aortas in collaboration with Adrian Carpenter and Laurie Hall at Cambridge University, but we have also looked at these tissues using high resolution  $^1\text{H}$  and  $^{13}\text{C}$  spectroscopy on our AM-360. The first animal model chosen was that where rabbits are fed a high cholesterol diet for 12 weeks and significant build-up of cholesteryl esters occurs in the blood vessel walls. We have looked at both normal and these so-called hypercholesterolemic aortas and can distinguish the normal triglyceride content from cholesteryl esters by making measurements at temperatures at and above normal body temperature (near where the cholesteryl esters undergo a liquid-crystalline to isotropic transition). Interestingly, we can also observe considerable biological variability in the levels of deposited cholesteryl esters.

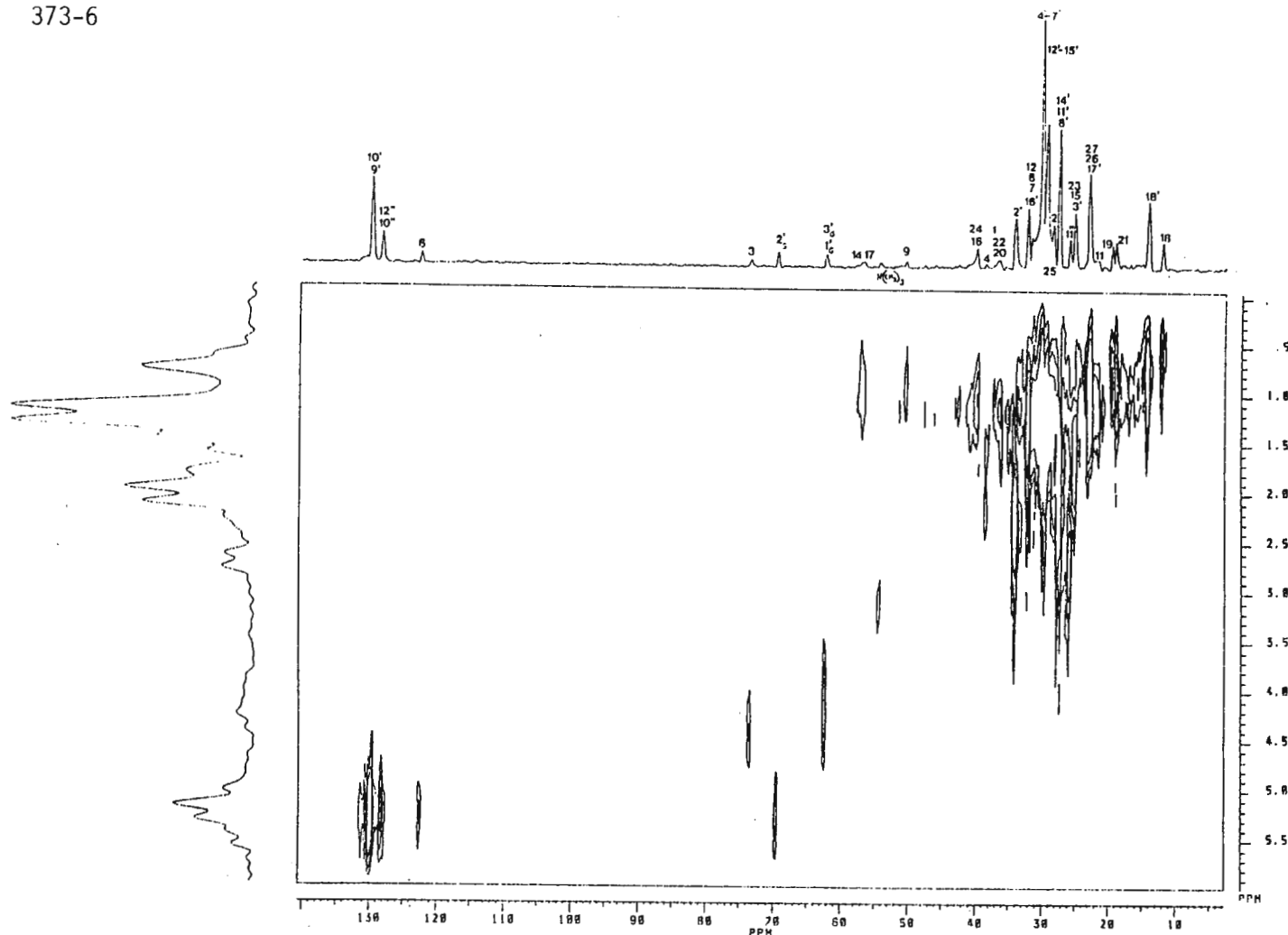
As an example of the information content in the spectra, the figure shows a carbon-proton correlation at 314°K (above the mesophase transition) on a high cholesterol diet rabbit aorta. Assignments in both proton and carbon include many diagnostic resonances of cholesteryl esters and triglycerides including the oleate and linoleate fatty acid moieties plus the choline N-methyl group from phospholipid.

Currently, we have moved on to cardiac gated in vivo NMR imaging on other models of atherosclerosis at Cambridge, which through some probe development work there shows great promise.

Yours sincerely,

J.C. LINDON

R.D. FARRANT



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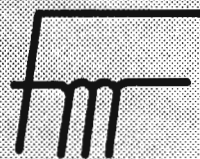
### IN VIVO NMR SPECTROSCOPIST

The Procter & Gamble Company has an opening for an in vivo NMR spectroscopist at the Company's Miami Valley Laboratories located near Cincinnati, Ohio. Applicants should have a Ph.D. in chemistry, biology, physics, or a related field with experience in the area of in vivo NMR. This includes imaging, NMR localization techniques and the biochemistry of energy metabolism. A practical knowledge of RF electronics would also be of value.

This individual will initiate and collaborate on research programs in diverse project areas including drug development, mechanistic studies and safety testing. He/she will also share responsibility for overseeing the operation of an in vivo NMR laboratory now under construction. The laboratory will be equipped with a state-of-the-art 4.7 tesla, 30 cm Bruker Biospec imaging/spectroscopy system.

Applicants must be presently authorized to work in the U.S. on a full-time basis. To apply, send resume and publication list to: Dr. T. J. Logan; Manager, Ph.D. Recruiting; IN VIVO NMR Position, Box T; The Procter & Gamble Company; PO Box 398707; Cincinnati, Ohio 45239-8707. Procter & Gamble is an EQUAL OPPORTUNITY employer.





## NMR Instrument Upgrades

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- Assisting to understand and eliminate instrumental problems.
- Analyzing and improving RF stability.
- Finding work-around solutions for instrumental restrictions.
- Upgrading his existing NMR probes.
- Supplying accessories to expand his NMR's capabilities.
- Providing RF and probe repair services.

### Noise Figure Meters

Knowing your NMR systems noise figure allows you to separate the variables of performance. Is the problem area shimming, preamp or external noise. A five minute test with an inexpensive (\$500) meter can tell you where the problem area lies.

### Indirect Detection

FMR provides accessories to allow excitation of an X nucleus and the observation of  $^1\text{H}$  for indirect detection, 2DFT and 3DFT experiments:

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Upgrade an existing probe to a  $^1\text{H}\{^{31}\text{P},^{15}\text{N}\}$  probe, or buy a new  $^1\text{H}\{^{31}\text{P},^{15}\text{N}\}$  probe.

### Reverse Polarization Transfer Probe $^1\text{H}\{^{31}\text{P},^{15}\text{N}\}$ 5mm Probe

	5mm	10mm
90° Pulse @ 50 Watts	15us	25us
Resolution (ODCB)	0.25	0.4
Lineshape ( $\text{CDCl}_3$ )	10/25	18/40
Gamma H2 (10W)		
$^{15}\text{N}$	2KHz	1KHz
$^{13}\text{C}$	3KHz	2KHz
Sensitivity (0.1%EB)		
200 MHz	60	100
300 MHz	120	200
360 MHz	170	280
400 MHz	200	330
500 MHz	300	500

## Probe Upgrades

Existing probes can be upgraded at less than half the cost of a new probe. An existing unused 20mm probe (or any other probe) can be converted to a 10mm Broadband probe. Or a 5mm  $^1\text{H}$  Probe can be reworked to improved sensitivity or performance with ionic water solutions.

### 5mm $^1\text{H}$ Probe

90° Pulse	10 us @ 50 Watts
Resolution	0.2 Hz (ODCB)
Lineshape	10/20 ( $\text{CDCl}_3$ )
Sensitivity (0.1% EB)	
200 MHz	80:1
300 MHz	160:1
360 MHz	220:1
400 MHz	300:1
500 MHz	400:1

### Broadband Probes

High Band broadband probes cover the range from  $^{31}\text{P}$  to  $^{15}\text{N}$  for solutions up to 0.2 M salt.

5mm	200	300	360	400	500
$^{31}\text{P}$	30	60	85	100	150
$^{13}\text{C}$	30	60	85	100	150
$^2\text{H}$ NA=4	5	10	14	16	25
$^{17}\text{O}$ NA=100	20	40	55	65	80
$^{15}\text{N}$	4	8	10	12	16

### 10mm

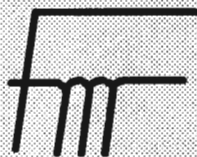
$^{31}\text{P}$	130	260	340	375	450
$^{13}\text{C}$	150	300	360	400	500
$^2\text{H}$ NA=4	15	30	40	50	60
$^{17}\text{O}$ NA=100	110	220	270	300	400
$^{15}\text{N}$	15	30	35	40	50

### 12mm

$^{31}\text{P}$	150	300	400	500	550
$^{13}\text{C}$	180	360	500	600	700
$^2\text{H}$ NA=4	20	40	50	60	75
$^{17}\text{O}$ NA=100	125	250	300	350	500
$^{15}\text{N}$	15	30	40	50	60

### 20mm

$^{31}\text{P}$	250	500	600	750
$^{13}\text{C}$	300	600	700	800
$^2\text{H}$ NA=4	30	60	70	90
$^{17}\text{O}$ NA=100	180	350	450	550
$^{15}\text{N}$	25	50	70	80



## Spin and Non-Spin Shims

Instrumentation Note 12

The actual gradients that shim sets are designed to generate are not purely those indicated by their name. The actual function intended in the design of the common shim sets is shown in the table. As can be seen, the on axis shims can have off axis components and the off axis shims can have on axis components. One useful tool to separate the on axis and off axis components is to shim the on axis shims while spinning and the off axis shims while not spinning. In both the spinning and non-spinning cases, if any shim changes its value significantly, the NMR operator needs to go back and shim the other set again. This process is repeated until no further changes are observed. Remember the functions shown in the table are those intended in the design. They are orthogonal and have a minimum interaction if the actual gradient created is that shown in the equation. This condition is never totally true. The better the shim set design, the closer the gradient will be to the intended function. There is a wide range of the degree of interaction (non-orthogonality) in the shim sets on the market today. The NMR operator needs to get to know his particular shim set characteristics and act accordingly.

Common Shim Name	Function
Z0	1
Z1	z
Z2	$2z^2 - (x^2 + y^2)$
Z3	$z[2z^2 - 3(x^2 + y^2)]$
Z4	$8z^2[z^2 - 3(x^2 + y^2)] + 3(x^2 + y^2)^2$
Z5	$48z^3[z^2 - 5(x^2 + y^2)] + 90z(x^2 - y^2)^2$
X	x
Y	y
ZX	zx
ZY	zy
X <sup>2</sup> -Y <sup>2</sup>	$x^2 - y^2$
Z <sup>2</sup> X	$x[4z^2 - (x^2 + y^2)]$
Z <sup>2</sup> Y	$y[4z^2 - (x^2 + y^2)]$
ZXY	zxy
Z(X <sup>2</sup> -Y <sup>2</sup> )	$z(x^2 - y^2)$
X <sup>3</sup>	$x(x^2 - 3y^2)$
Y <sup>3</sup>	$y(3x^2 - y^2)$

### The Non-Spin Shims

The usual high resolution NMR sample is spun about a cylindrical axis. This process averages the off axis field gradients. The averaging process is avoided during shimming by adjusting these shims with the sample not spinning. Everyone knows that a symptom of poorly adjusted non-spin shims is spinning sidebands. Why does this situation give spinning side bands? Does a condition of mis-adjusted non-spin shims have other effects?? Yes!!

### 1st & 2nd Order Spinning Sidebands

If the portion of the sample which is off axis is passing through different field values as the sample spins, then the resonant frequency of the nucleus is changing at the same rate. This means that the resonance is being frequency modulated and generates sidebands at the rate of frequency modulation. The amount of sample passing through the different fields determines the percentage of SSBs. If the sample passes through one field gradient per revolution it generates a first order SSB. This is the case for X, ZX, Y and ZY gradients in superconducting systems. If the sample passes through two field gradients on the spinning cycle then a second order SSB is generated. This is the case for XY and X<sup>2</sup>-Y<sup>2</sup> gradients. This is a simple process with a straightforward treatment.

First Order SSB      Adjust X, ZX, Y and ZY.  
Second Order SSB      Adjust XY and X<sup>2</sup>-Y<sup>2</sup>.

### Half Order Spinning Sidebands

In some rare cases the NMR operator can see half order spinning sidebands; i.e., a sideband at half the spinning rate. The most common cause of this symptom is the sample hitting the insert during spinning. This can come about from:

- o A bad NMR tube.
- o A damaged spinner.
- o A broken probe insert.
- o Sample lift/bounce from high VT airflow.

Usually half order spinning sidebands cannot be shimmed away. The solutions are obvious for most cases. The high VT air bounce can be cured by either decreasing the VT air flow or increasing the air leaks from the probe to reduce back-pressure. In some NMR systems this can be done by removing the probe tuning sticks from the probe.

### Higher Order Non-Spin Shims

On many supercon NMR systems there are other higher order non-spin gradients. These can include: Z<sup>2</sup>X, Z<sup>2</sup>Y, Z(X<sup>2</sup>-Y<sup>2</sup>), ZXY, X<sup>3</sup> and Y<sup>3</sup>. As a first approximation, X<sup>3</sup> and Y<sup>3</sup> generate spinning sidebands and should be adjusted interactively with X and Y respectively. The Z<sup>2</sup>X, Z<sup>2</sup>Y, Z(X<sup>2</sup>-Y<sup>2</sup>) and ZXY gradients can generate poor low order lineshape problems and/or spinning sidebands. A clue to these particular shims being mis-adjusted is a tendency for the peak to change from good spinning sideband performance with poor low order lineshape to poor spinning sideband performance with good low order lineshape with the adjustment of the spinning shims.



## DEPARTMENT OF HEALTH &amp; HUMAN SERVICES

Public Health Service

National Institutes of Health  
National Institute on Aging  
Gerontology Research Center  
4940 Eastern Avenue  
Baltimore, Maryland 21224

August 25, 1989  
(received 9/12/89)

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

**Human Skeletal Muscle Metabolism During Isometric Exercise**

Dear Dr. Shapiro:

We have been engaged in a series of investigations designed to study skeletal muscle metabolism during isometric exercise as a function of age via in vivo  $^{31}\text{P}$  NMR spectroscopy on our Biospec 1.9 T/31 cm spectrometer. One of the parameters which we monitor during exercise is the ratio of phosphocreatine ( $P_{\text{CR}}$ ) to inorganic phosphate ( $P_{\text{i}}$ ) in the muscle.

Our exercise protocol requires the subject to squeeze a hand dynamometer at 30%, 40%, 50% and 60% of his maximum for 30 seconds followed by a 60 second rest period between contractions. Spectra (4 pulses collected over 15 seconds) of the flexor digitorum superficialis muscle in the forearm are collected throughout the entire protocol using a 4 cm surface coil. Typically the  $P_{\text{CR}}/P_{\text{i}}$  ratio declines during exercise and recovers toward the initial value during the resting portions of the protocol. However, in many of our subjects the initial recovery period following the 30% contraction is interrupted by a drop in the  $P_{\text{CR}}/P_{\text{i}}$  ratio (see Figure 1). This phenomenon is not correlated with the age of the subject.

Examination of  $P_{\text{i}}$  levels indicates that an increase in  $P_{\text{i}}$  (rather than a decline in  $P_{\text{CR}}$ ) is responsible for this effect. In each case the decline in  $P_{\text{CR}}/P_{\text{i}}$  is defined by just one data point. We have considered the possibility of an instrumental artifact but have not been able to identify one.

Sincerely yours,

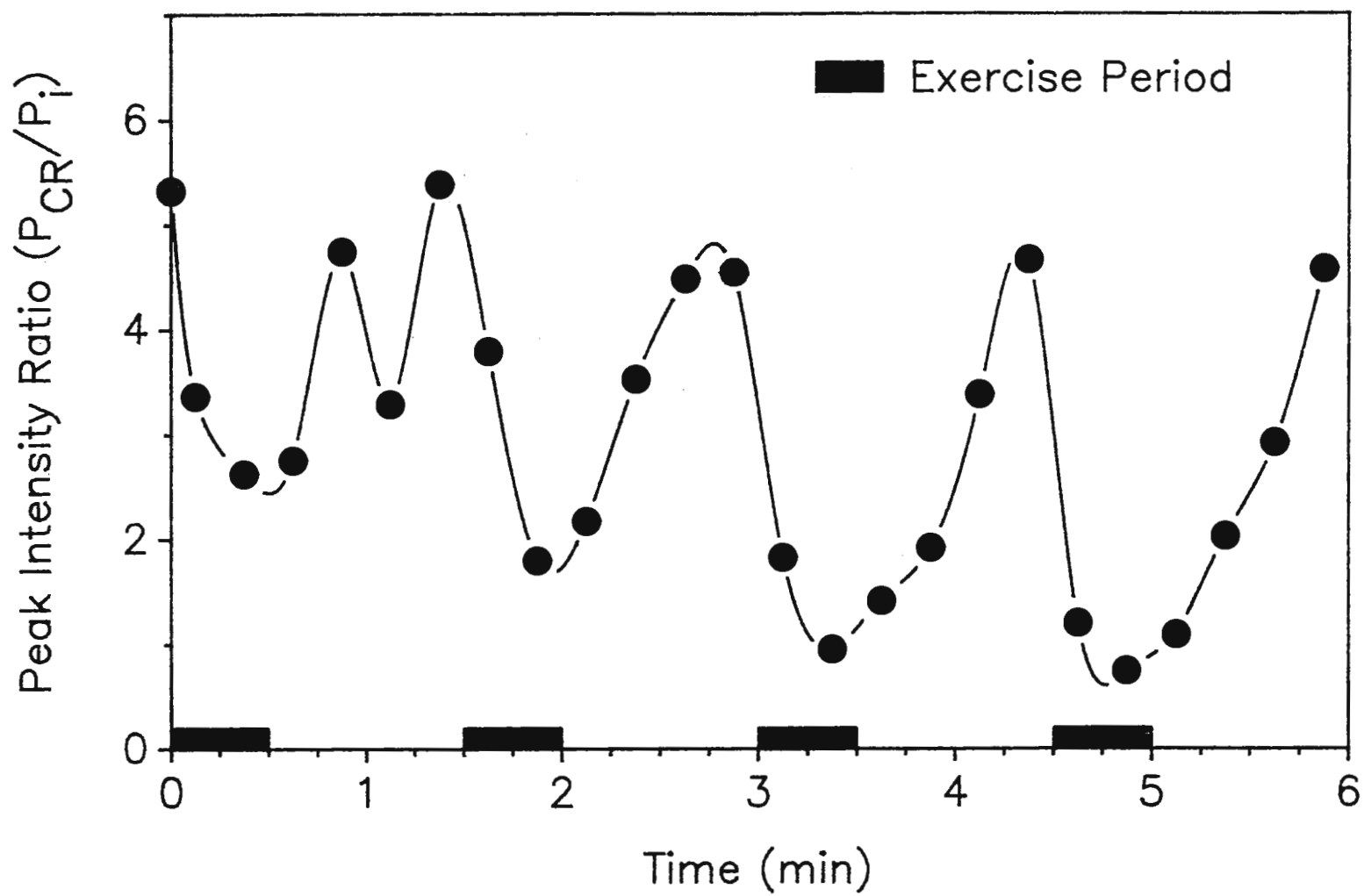
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FIGURE 1



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## NMR AND DATA PROCESSING LABORATORY

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August 28, 1989 (received 9/1/89)

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

## Two New Services at the Syracuse University NIH Research Resource

Dear Barry:

Over the past two years we have been developing capabilities for determination of 3-dimensional solution structures of DNA and RNA molecules, as well as peptides or small proteins. In particular, we are combining 2D NOESY experimentation with initial structure calculation and refinement using restrained dynamics, distance geometry and cross-relaxation matrix approaches. Our work follows pioneering studies of the Wuthrich, Reid/Hare, Kaptein, James and other laboratories, but there are a few differences.

1. As a result of our collaborations with New Methods Research, Inc., we are able to increasingly automate the primary 2D FT processing and cross peak quantitation.
2. A continuing collaboration with Milligen/Biosearch and University of Colorado scientists has provided us with sources for RNA precursors and molecules.
3. 2D contrast enhancement methods, such as maximum likelihood, have shown themselves to be invaluable for clarifying heavily overlapped cross peak regions needed to determine NOE connectivity.
4. One of our graduate students, Mark Roggenbuck, and two of our collaborators, Hans Grahn (Syracuse University, and now Umea University) and Frank Delaglio (New Methods Research) [along with some additional contributions from Tim Hyman, Karl Bishop, Sophia Wang and other students], have been developing new methods, including multivariate pattern recognition techniques and automated (NOESY) path analysis, to help automate NOESY spectrum analysis.
5. Sophia Wang, Pankaj Kumar and Sandor Szalma of our laboratory are developing the tiny beginnings of an expert system for 3D structure determination from NOESY data.

Over the past two months, our first refined structures are beginning to appear, for molecules ranging to 8000 Daltons (DNA-12mer duplex). We are now in an active phase of technique and algorithm development and evaluation and would welcome particularly availability of small peptides with "fixed" structure (to 20mers) to help us test our methods.

CENTER FOR SCIENCE AND TECHNOLOGY / SYRACUSE, NEW YORK 13244-4100

315-443-1021 / FAX 315-443-1022

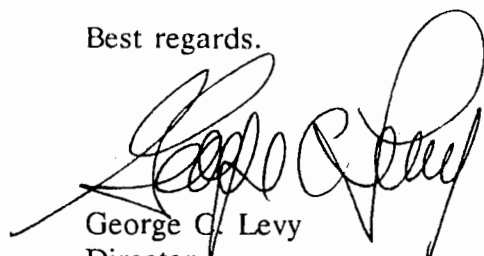


One of our biggest surprises has been the significant assist provided by maximum likelihood constrained deconvolution coupled with a symmetrization operation. One example is shown below for the RNA octamer duplex, (CACAUGUG)<sub>2</sub> [van den Hoogen, et al. (1988) *Nucleic Acids Research*, **16**, 2971]. This small region of its NOESY spectrum contains H'-H2',3',4',5',5'' connectivities, with the intranucleotide H1'-H2' peaks marked in the enhanced spectrum shown on the right. The spectrum was produced with a cosine bell apodization (left) then enhanced using the DECO2D program [Ni & Scheraga, (1989) *J. Magn. Reson.*, **82**, 413] with 5Hz linesharping in F1 and F2, followed by diagonal symmetrization (right). The large marked peaks and most of the small peaks are easily identified with expected features in the spectrum.

Starting in January we expect to perform 3D structure determination, as a service for Resource users, using high-field data obtained at our Resource or with data obtained at other facilities (which we can accept in a wide variety of formats). A service that is available immediately to all Resource users is MLM contrast enhancement of 2D arrays to 2Kx2K complex data points. (We just require a UNIX 1/4" or 1/2" TAR Tape).

Please write or call us for more information.

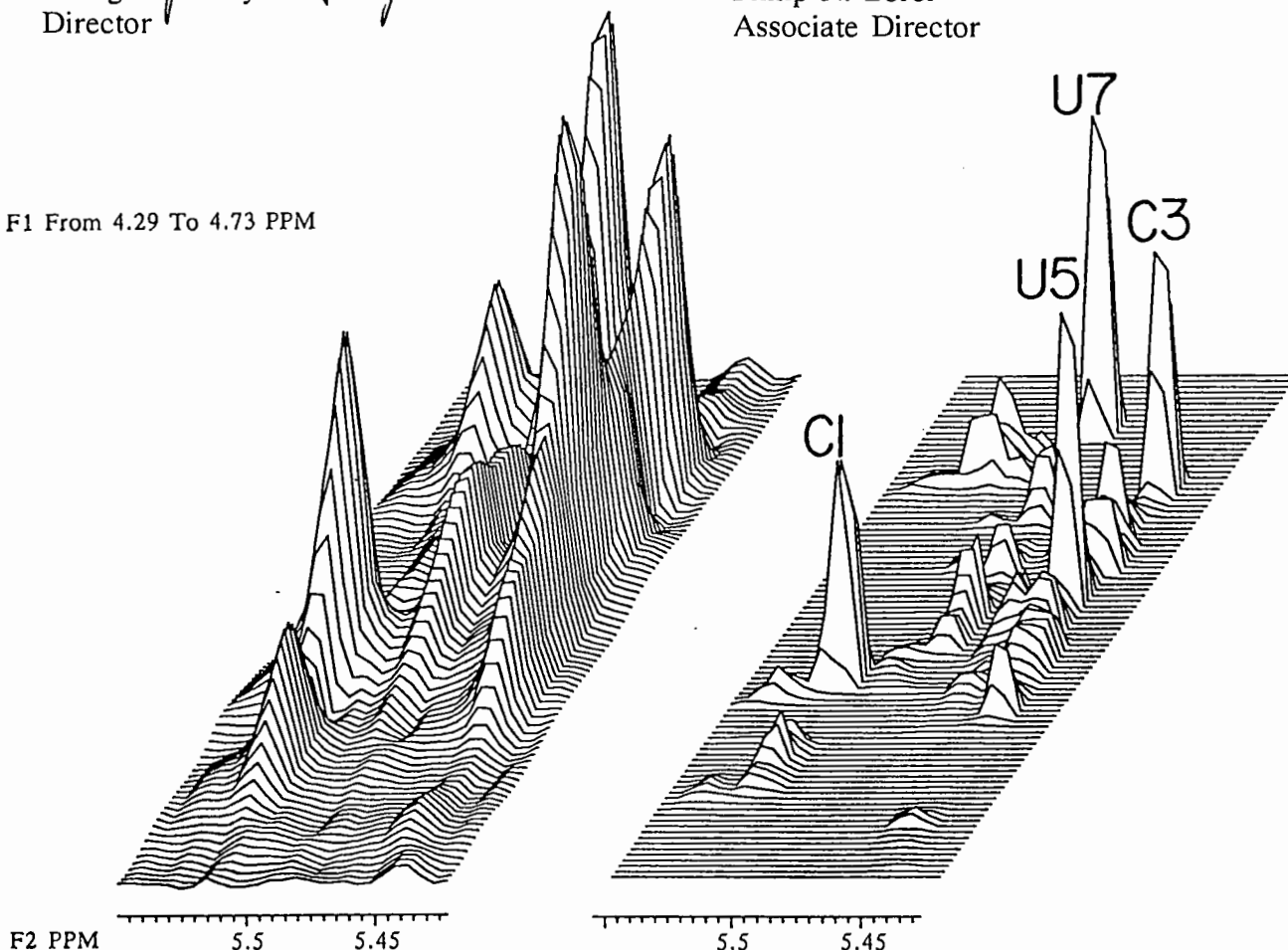
Best regards.

  
George C. Levy  
Director



Philip N. Borer  
Associate Director

F1 From 4.29 To 4.73 PPM





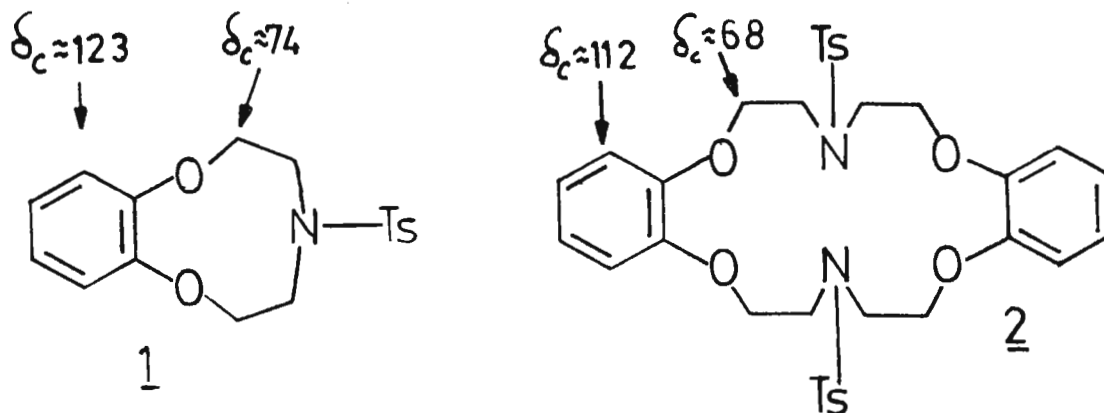
Carleton University  
Ottawa, Canada K1S 5B6

Title: **C-13 NMR Shift Differences in Crown Ether Segments.**

Aug. 29/89

(received 9/2/89)

During the course of his MSc. work, Denis Landry has prepared the 9 and 18-membered ring materials with structures **1** and **2** respectively as depicted below. In both the solid phase and solution C-13 spectra, two notable chemical shift differences are present, specifically at the ortho aromatic carbon and at the CH<sub>2</sub>-O sites. In each case, **1** is deshielded, with the differences being 11 and 6 ppm respectively at the aforementioned carbons.



We believe these effects arise primarily from torsional angle differences in the aromatic C-O bonds. For the 18-membered case, X-ray data on the related (1) dibenzo-18-crown-6 indicate that the CH<sub>2</sub>-O carbon is in the plane of the aromatic ring. A strong n.o.e. was observed between the CH<sub>2</sub>-O and the ortho aromatic proton in **2**, in support of such a geometry in the present case. (For n.o.e. results on another related system see reference (2)). By contrast in **1**, ring constraints of the 9-membered heterocycle (likely in an extended chair conformation) force the O-CH<sub>2</sub> carbon into a geometry in which the critical torsional angle appears from models to be ca. 60°. As a result, the CH<sub>2</sub>-O protons are much more distant from the ortho aromatic proton and a negligible n.o.e. is observed. Concomitantly, the expansion of this torsional angle would be expected to lead to deshielding at both the ortho aromatic carbon and the CH<sub>2</sub>-O site in **1** vs. **2**. We are attempting at present to grow X-ray quality crystals of these materials.

G.W. Buchanan  
Professor of Chemistry

1. Bush and Truter J.Chem. Soc.B, 1440 (1971).
2. Kleinpeter, Gabler and Schroth. Mag. Res.Chem. 26, 380 (1988).



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# CSI 2T Applications

## Shielded Gradients and Localized Spectroscopy

Eddy current effects are the leading cause of errors and lack of consistent results in gradient localization methods. It is not surprising, then, that actively shielded gradients, which have dramatically reduced eddy currents, represent a significant technology advance for all forms of  $B_0$  gradient volume localization and spectroscopic imaging methods. The fast rise time and high gradient strength characteristics of the coil used in these experiments are also important.

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

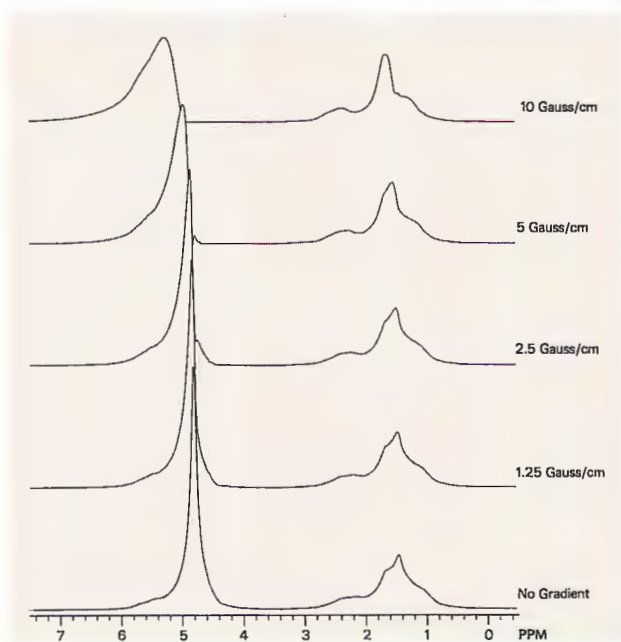


Fig. 1—Using an oil/water phantom, a 10 G/cm gradient will create a water frequency profile extending from 156 KHz to 280 KHz away from normal water resonance. Residual gradient effects of less than 0.01% (50 Hz at 10 G/cm) are observed in a spectrum acquired beginning 1 msec after a 20 msec gradient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

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

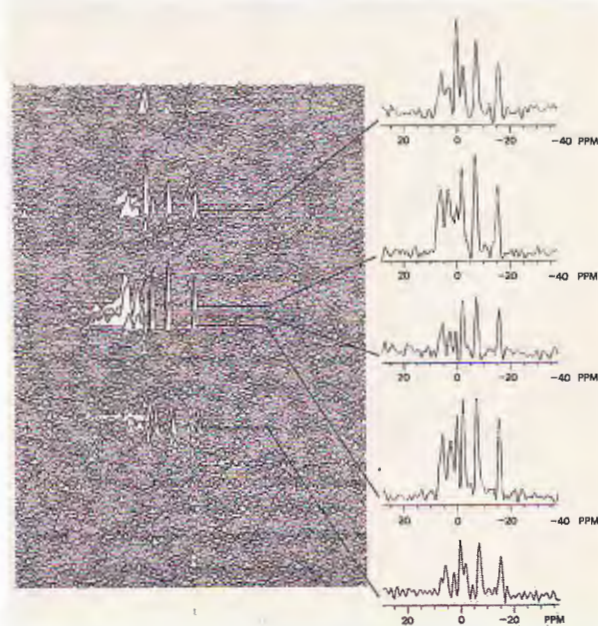


Fig. 2—Stacked plot showing 512 phosphorous spectra from 60 mm cubed region of a live rat. Each trace corresponds to 7.5 mm cubed region (voxel) from within the region of interest. The offset traces clearly show the achievable spectra and spatial resolution of the technique, as well as demonstrating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



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As may be seen from the Editorial Board, "Applied Magnetic Resonance" will be a truly international journal and the intention is to apply the highest standards to choice of the material to be published in it. It is expected that manuscripts will be published within six months of their being accepted.

Special editions will be published under the guidance of guest editors.

**Further information for those interested in contributing materials can be obtained from the editor:**

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RESEARCH AND DEVELOPMENT

September 6, 1989

NMR Position  
Wha-13-89

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

Dear Barry:

We currently have a position open for an NMR spectroscopist in the Analysis Branch in the Research and Development labs of Phillips Petroleum in Bartlesville, Oklahoma. The individual would be responsible for undertaking NMR research in support of Phillips' products, including hydrocarbons, polymers and chemicals. The individual would be expected to work closely with other researchers in R&D and to propose, and carry out, projects on their own. A strong background in interpretation of NMR spectra and computer programming are assets.

Interested individuals should contact me at the address below. Phillips is an equal opportunity employer and offers a competitive salary and benefit package.

Sincerely yours,

A handwritten signature in dark ink, appearing to read "S. M. Wharry". The signature is fluid and cursive.

S. M. Wharry  
Analysis Branch  
144 PL, Research Center  
(918) 661-9793

SMW:rk



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The individual will be responsible for the identification, selection and conduct of corporate and cooperative research programs contributing to the use and enhancement of SISCO products. Maintaining an awareness and working knowledge of diverse applications, current and future, will be needed. He or she will supervise a staff of experienced Ph.D. scientists in the development of advanced system capabilities and testing of hardware and software innovations. Close interactions with multiple user groups will be very important, requiring routine travel.

This is also an opportunity for a bright, motivated person who is interested in promoting the advantages and uses of magnetic resonance imaging and spectroscopy to the research community. Scientific presentations and system demonstrations will be part of the role. The individual will provide advanced training, technical reference and scientific support to users.

Spectroscopy Imaging Systems (SISCO) is known worldwide for our innovative and reliable NMR Imaging Spectrometers, and for providing excellent technical support. Outstanding facilities and resources are available in our Fremont headquarters as well as with our parent companies, Varian Associates and Siemens Corporation.

Our Applications Laboratory is currently equipped with an SIS 200/330 NMR Imaging Spectrometer, utilizing a Sun 4/150 TAAC workstation and including a new Passively Shielded 4.7T/ 33 cm bore magnet with Actively Shielded Gradient Set and high performance 18 cm Auxiliary Gradient Set. The lab incorporates full animal facilities for *in vivo* studies. Access to SIS 300/183 (7.T) systems and other advanced Sun workstations will be routine. Collaborative access to both Siemens and Varian facilities is ongoing.

SISCO is now completing a 4T/whole body system in collaboration with Siemens for research in clinical imaging and spectroscopy. Applications development using this facility in Fremont will be a position responsibility jointly with Siemens.

The position is now open. It offers an attractive salary, comprehensive benefits and excellent growth potential. Applicants should submit a *curriculum vitae*, list of publications, references and any other information pertinent to their candidacy to the Vice President, Marketing and Applications, at the following location:

**Spectroscopy Imaging Systems Corporation**  
**1120 Auburn Street**  
**Fremont, CA**

**94538**

September, 1989

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SCHOOL OF BIOCHEMISTRY

PLEASE QUOTE

12 September, 1989

(received 9/22/89)

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

Dear Professor Shapiro,

## PROTEIN STRUCTURES DOWN UNDER

As a newcomer to the Newsletter, I thought a brief outline of our research interests might be in order.

My group is interested principally in studies of protein structure and structure-function relationships using high-resolution  $^1\text{H}$  (and, more recently,  $^{13}\text{C}$ ) NMR. We routinely use the full range of 2D (soon also 3D) experiments to make resonance assignments, and are running distance geometry (DISGEO, DISMAN) and molecular dynamics (GROMOS) program packages to derive structures. The main focus of our interest is a series of small proteins from sea anemones, which act by binding to the sodium channel of excitable tissues, thereby prolonging the action potential. We have published a low resolution structure of one of these proteins, anthopleurin-A, and Rasmus Fogh, a Ph.D. student in the lab, has just completed the structure of a second, *Stichodactyla helianthus* neurotoxin I. In addition to these proteins, we are working on the structures of other toxins of marine and terrestrial origin, as well as other proteins of pharmacological interest. My senior co-worker in most of these studies is Dr. Bridget Mabbutt, a research fellow in the lab.

We also collaborate with Dr. Keith Cross, who is in charge of the the University's NMR Unit. Keith is working on the structure of another sea anemone toxin. As well, he has a strong interest in the analysis of ring current shifts in porphyrins and proteins.

Our NMR facilities consist of a Bruker AM-500, 1986 vintage, and an aging but very reliable CXP-300. The AM-500 is linked by optical fibre to an Aspect 2000 data station equipped with a tape drive. In my lab we have a Silcon Graphics IRIS 4D/70 workstation which we employ for molecular graphics and NMR data processing (using Dennis Hare's program FTNMR). This is linked to an excellent campus-wide optical fibre network which operates under the TCP/IP protocol. Our AM-500 is not yet connected to this network, and we are currently exploring the best way of achieving this. Installation of a Bruknet unit in the Aspect 3000 will (we trust) permit transfer of data to a VAX cluster on the network from which we can then transfer data to the IRIS. A "direct" transfer (Aspect to IRIS) could be achieved by modifying source code or via an IBM PC. We would be most interested in the experience of any of your contributors who have solved a similar problem.

I trust this brief introduction will suffice as our initial contribution.

Yours sincerely,

Raymond S. Norton

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---

Dr. Barry Shapiro  
TAMUNMR Newsletter  
966 Elsinore Ct.  
Palo Alto, CA 94303

Dear Barry:

### **Position Available**

I have an immediate opening for a postdoctoral student in the field of Lab Automation. We are looking for a candidate with strong workstation programming abilities in C under DOS, OS/2 or Unix and an interest in laboratory data gathering and manipulation. The project I have in mind can definitely lead to one or more significant papers in the field. Those interested can contact me directly for information, but to apply need to request application materials from Scott Moore, Corporate Ph.D. Recruiting, at the above address, mentioning this position. IBM is, of course, an equal opportunity employer.

Best regards,

A handwritten signature in cursive script, appearing to read 'Jim Cooper'.

James W. Cooper,  
Manager, Laboratory Automation

---

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September 19, 1989 (received 9/22/89)

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

Dear Barry:

We wish to describe the development of perhaps the first potential agent for adjuvant cancer therapy derived as a result of NMR studies. We had previously shown that multi-drug resistant (mdr) human MCF-7 breast cancer cells (the ADR cell line) exhibit an enhanced rate of glycolysis compared to their parent wild-type (WT) cell line (1). This led us to test the effect of an inhibitor of glycolysis, 2-deoxyglucose (2-DG), on these cells. We have observed a selective toxic effect of 2-DG on the ADR cells, which is more than 15-fold greater than for WT cells (2).

Using  $^{31}\text{P}$  NMR of perfused MCF-7 cells we continuously monitored the accumulation of 2-deoxyglucose-6-phosphate (2-DG-6P) together with concomitant changes in other phosphate-containing metabolites. Kinetic measurements demonstrated that ADR cells accumulated 2-DG-6P faster and to a greater extent than WT cells, while their depletion of high energy compounds (ATP, PCr) was more pronounced and became irreversible earlier.

The phosphorylation of 2-DG could be followed more effectively by the use of  $^{13}\text{C}$  NMR of 2-DG enriched with  $^{13}\text{C}$  at the  $\text{C}_6$  position (3). This is advantageous since the signals of 2-DG and 2-DG-6P are clearly resolved and, unlike  $^{31}\text{P}$  MRS, there are no other interfering signals. Employing this technique with ADR and WT cells the rates of phosphorylation of 2-DG were found to be different (2).

The results of these studies indicate that differences in the energy metabolism of resistant cells may make them targets for energy anti-metabolites. We do not mean to imply that 2-DG itself could be an anti-cancer drug, but that combinations with other drugs, such as adriamycin (2), or other inhibitors of energy metabolism (such as rhodamine) might be useful adjuvants to chemotherapy.

Yours sincerely,

A handwritten signature in black ink, appearing to read "Jack S. Cohen".

A handwritten signature in black ink, appearing to read "Ofer Kaplan".

Jack S. Cohen and Ofer Kaplan

1. Lyon *et al.*, *Cancer Research* 48, 870, 1987.
2. Kaplan *et al.*, *Cancer Research*, in press.
3. Navon *et al.*, *FEBS Letters*, 247, 86, 1989.

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Telephone 03 560 7066  
Fax 03 561 6709

14 September 1989 (received 9/20/89)

Dr Bernard L. Shapiro  
966 Elsinore Court  
Palo Alto  
CALIFORNIA 94303  
UNITED STATES OF AMERICA



Dear Dr Shapiro,

New Applications for a Bruker pc-120 "Minispec"

In addition to our high resolution instrumentation (WP-200 and CXP-200) we also measure hydrogen content of liquids with a Bruker pc-120 'minispec'. For the uninitiated, this is an extremely versatile 20 MHz pulsed instrument most often associated with routine applications in the food industry. Our 'minispec' is used in fuel science research<sup>1</sup> and to analyse hydrocarbon products from our coal hydroliquefaction plant. The general methods for "wt.%H" measurements have been described<sup>2</sup>, and we have developed rapid routine procedures which give results of particularly high accuracy (s.d. = .025%), which enables us to extend our range of monitoring and research applications for "%H", with confidence.

In our coal hydroliquefaction studies we use a disposable iron oxide catalyst in the presence of sulfur, where the active catalyst species is pyrrhotite,  $\text{Fe}_{1-x}\text{S}$ , generated *in situ*. The sulfur/catalyst ratio is critical for process efficiency: if the ratio is low, hydrogen transfer to coal will be reduced; if it is too high, increased  $\text{H}_2\text{S}$  production/ $\text{H}_2$  consumption results with no benefit. Results from a series of microautoclave experiments on hydrogenation of creosote oil, using different sulfur/catalyst loadings, are shown in Fig. 1. Product weight %  $^1\text{H}$  (by NMR) is plotted against sulfur/catalyst ratio. The fact that observable trends may be measured by NMR within such a narrow band of %H is in itself remarkable. The trends correlate with XRD analysis of the solid residues and are also confirmed by G.C. analysis of product gases, which enables calculation of hydrogen transfer and prediction of product oil %H. Correlation between calculated and NMR values is again excellent ( $r^2=0.983$ ).

Furthermore, combination of NMR data with other experimental data allows estimation of an optimum sulfur/catalyst ratio, which can then be applied to our kg/hr scale continuous coal hydroliquefaction pilot plant.

Potential applications for 'minispec' throughout industry are innumerable and we hope that other high-resolution users might be encouraged to apply themselves to this less glamorous, but very interesting, corner of the NMR world.

A handwritten signature in black ink, appearing to read "P. J. Barker".

Philip J. Barker

A handwritten signature in black ink, appearing to read "A. A. Awadalla".

Albert A. Awadalla

- 1) e.g. D.J. Cookson, C.P. Lloyd and B.E. Smith, Energy Fuels, 1988, 2, 854.
- 2) e.g. M. Bouquet, Fuel, 1985, 64, 226.

P.S. Please credit this contribution to Ian Rae's account.

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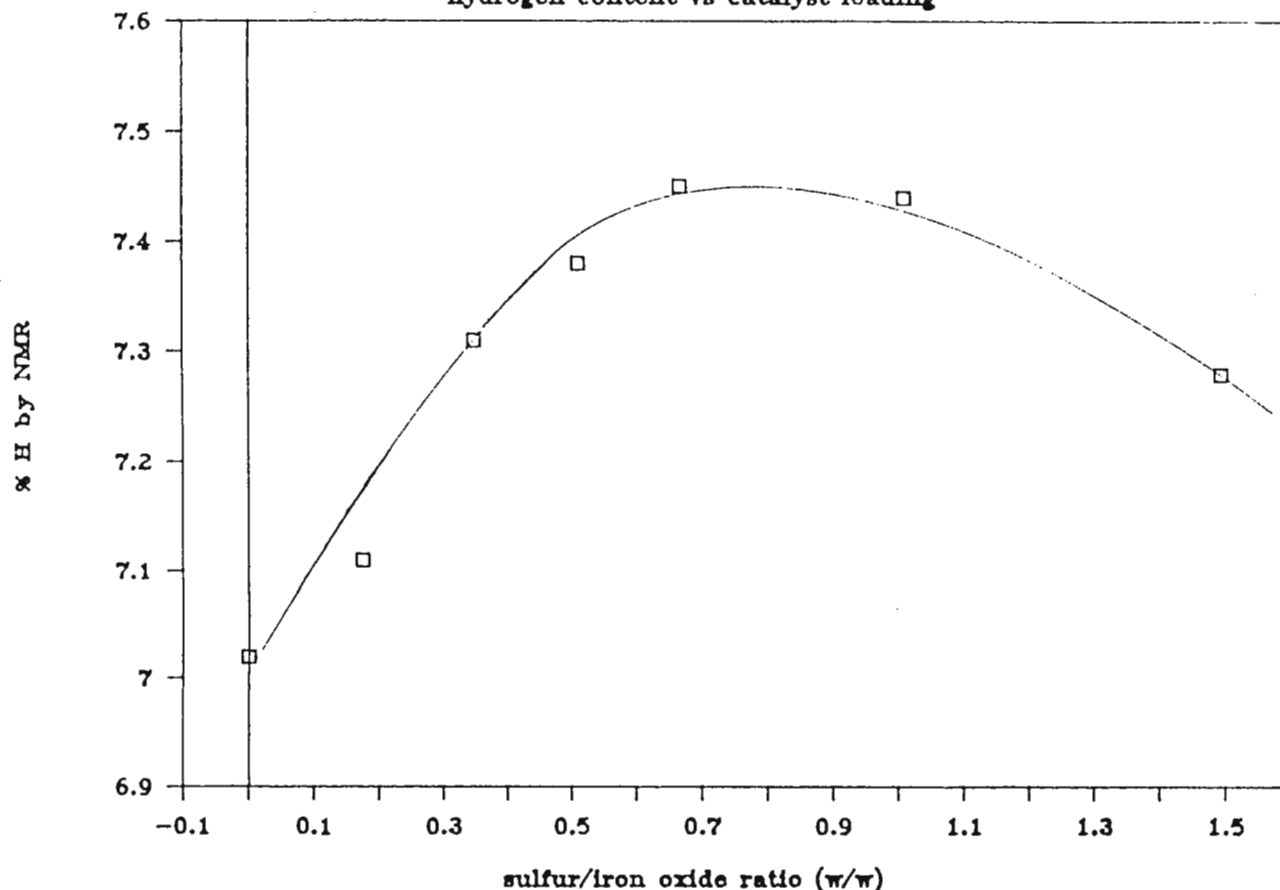
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Figure 1

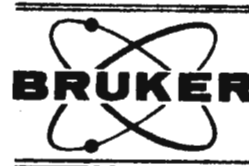
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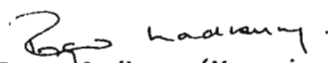
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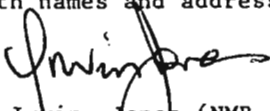
Bruker Spectrospin Limited have recently moved their United Kingdom N.M.R. applications and marketing operation into a new purpose-built laboratory and office facility alongside new manufacturing premises. It is intended to expand the scientific and technical staff in order to create a major centre for N.M.R. applications back-up, which will be able to complement our existing laboratories in West Germany, Switzerland, France and the United States.

We are currently seeking a high-calibre applications scientist, whose duties will include implementing and demonstrating novel techniques on Bruker high field N.M.R. spectrometers, conducting lectures, seminars and training programs, and interacting with Bruker electronics and software design engineers.

The position requires a Ph.D level chemist with considerable N.M.R. experience, probably including a period with a major N.M.R. research group. A keen interest in new hardware developments and N.M.R. techniques is desirable together with the practical and theoretical background necessary for these techniques.

Interested applicants should submit c.v, with names and addresses of two referees, to either of the undersigned.

  
Roger Ladbury (Managing Director)

  
Irwin Jones (NMR Applications Manager)



THE ROYAL  
INSTITUTE OF  
TECHNOLOGY  
*Dept. of Inorganic Chemistry*  
*Dr. Julius Glaser*

373-29

Stockholm, August 21, 1989  
(received 8/26/89)

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

## "Complicated ionic equilibria"

*Dear Prof. Shapiro,*

It seems to be difficult to find limits for multinuclear NMR. Currently, we are working on equilibria in the four-component system  $\text{Tl(III)} - \text{H}^+ - \text{CN}^- - \text{Cl}^-$  in aqueous solution using a combination of  $^{205}\text{Tl}$ - and  $^{13}\text{C}$ -NMR and a small portion of potentiometry. The system contains 18 soluble species: all combinations of  $\text{Tl}(\text{CN})_m\text{Cl}_{n-3-m-n}$  with  $(m+n) \leq 4$ ,  $\text{HCN}$ ,  $\text{CN}^-$  and in some solutions also  $\text{TlCl}_5^{2-}$  and  $\text{TlCl}_6^{3-}$ . NMR spectra contain an interesting mix of slow and fast exchange on the actual timescales (90 and 400 MHz spectrometers): roughly, most of the chlorides exchange fast<sup>1</sup> and most of the others slower (see e.g. Fig.1.). Certainly, it took us some time to figure out what is what in the spectra. But having done that, by using the vast amount of information contained in the NMR spectra and a suitable computer program,<sup>2</sup> we are able to calculate the stability constants and the individual chemical shifts for all the species, and spin-spin coupling constants  $^1\text{J}(\text{Tl}-\text{C})$  for most of them. In fact, we have had some help from our previous investigations of the simpler systems  $\text{Tl(III)} - \text{chloride}$ <sup>3</sup> and  $\text{Tl(III)} - \text{cyanide}$ ,<sup>4</sup> but even without this help we would probably be able to crack the four component puzzle. Figure 2. shows an example of the distribution of the complexes.

*Sincerely,*

*Johan Blixt*

*Julius Glaser*

<sup>1</sup> E.g., this *Newsletter*, 362 (1988) p.24.

<sup>2</sup> Program "LAKE". See *K.Holmström. Thesis. Univ. of Umeå, Sweden. 1988.*

<sup>3</sup> J.Glaser and U.Henriksson. *J.Am.Chem.Soc.* 103 (1981) 6642.

<sup>4</sup> J.Blixt, B.Györi and J.Glaser. *J.Am.Chem.Soc.* 111 (1989). *In press.*

Fig.1.

Some typical Tl-NMR spectra for solutions containing 50 mM  $\text{Tl}^{3+}$  and varying concentrations of  $\text{H}^+$ ,  $\text{CN}^-$  and  $\text{Cl}^-$ .

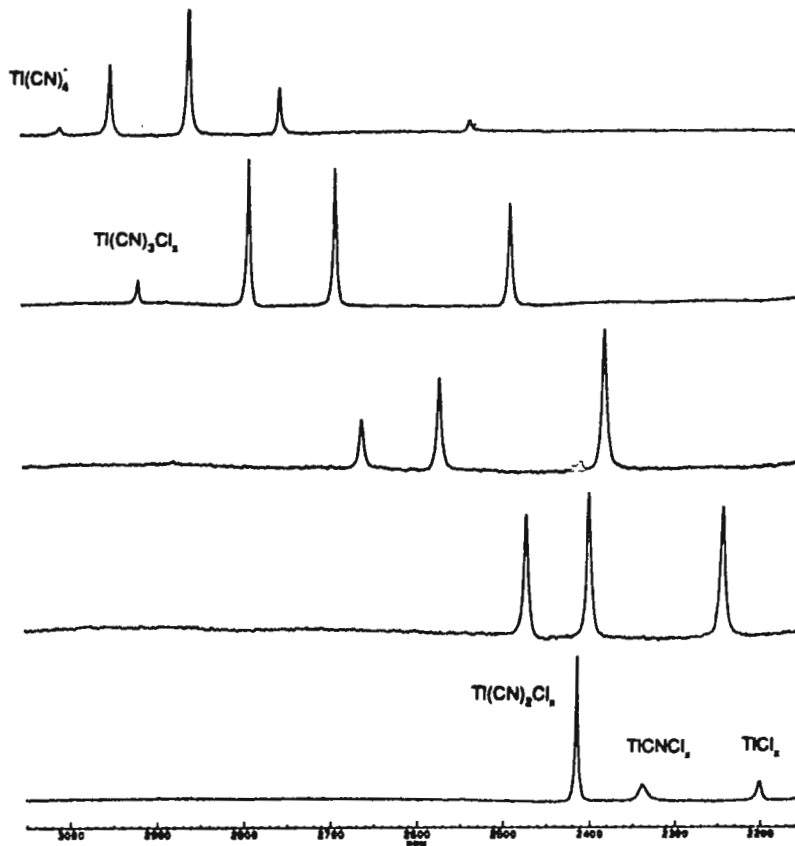
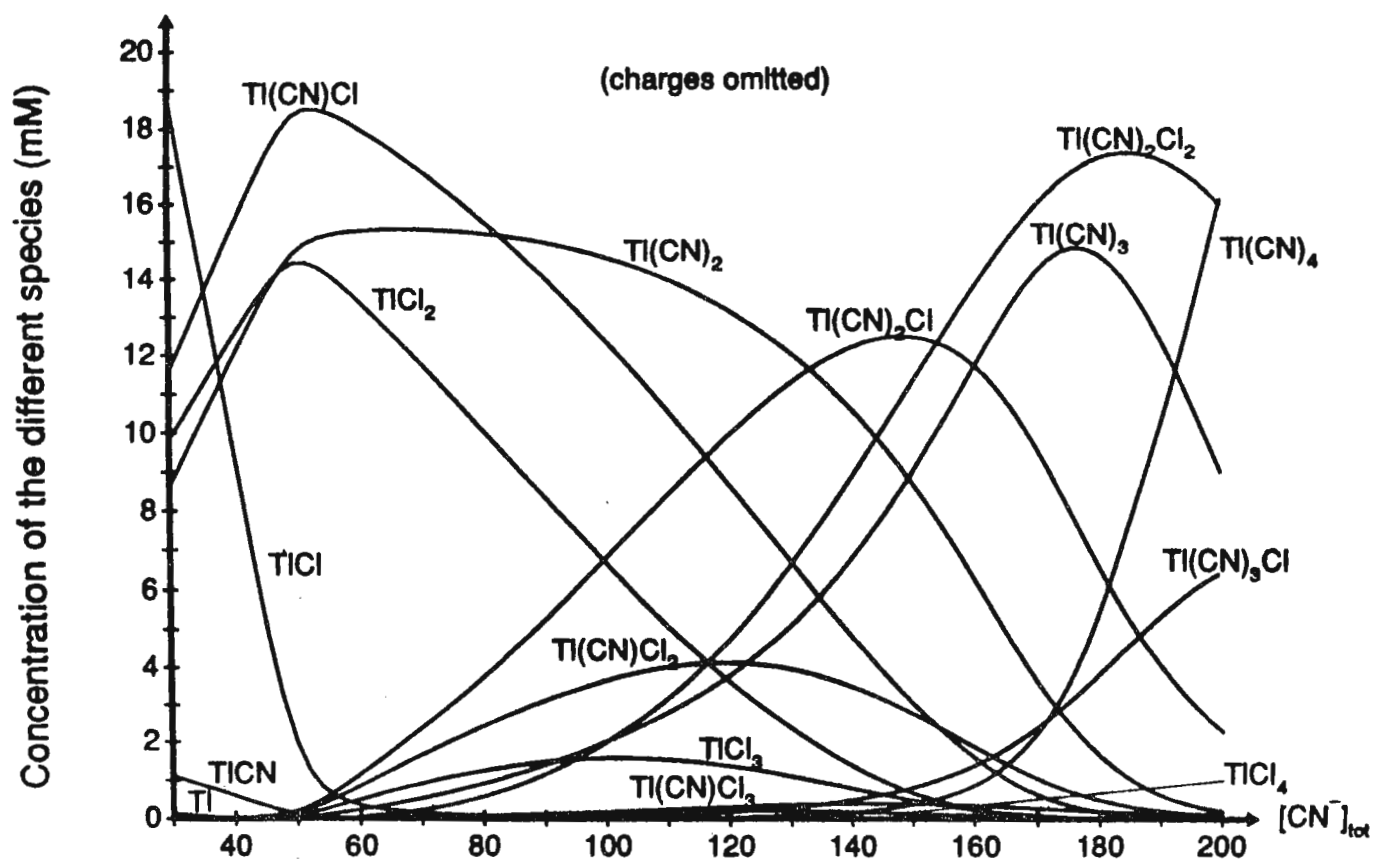


Fig.2.

Complex distribution for the  $\text{Ti/CN/Cl}$ -system



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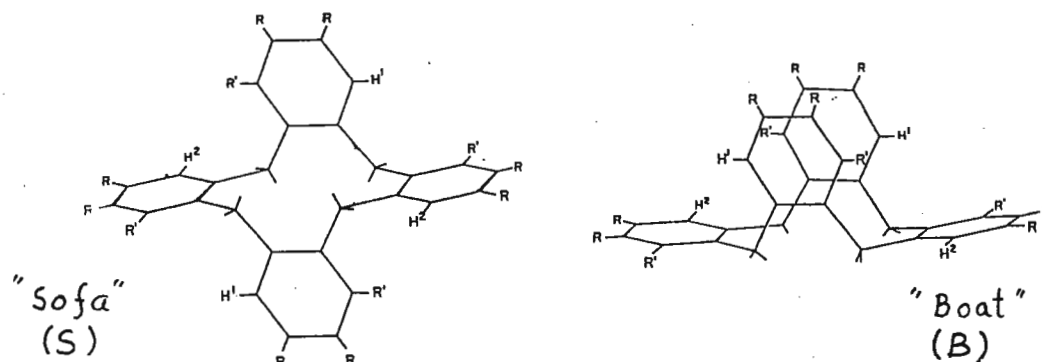
August 28, 1989  
(received 9/6/89)

מחלקת איזוטופים  
טלפון ישיר: (08) 48

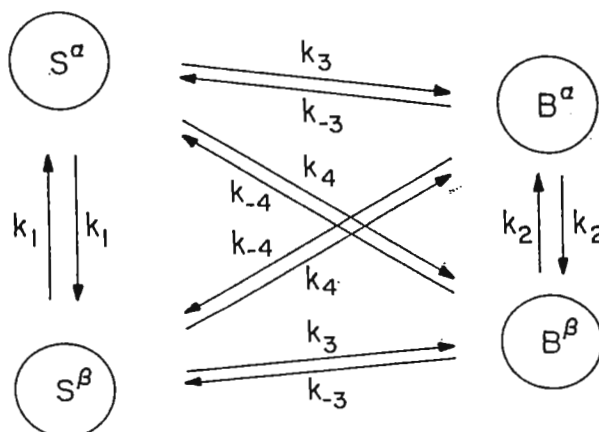
Dr. B.L. Shapiro  
TAMU NMR Newsletter  
966 Elsinore Court  
Palo Alto, CA 94303  
U.S.A.

Dear Barry,

In response to your yellow reminder we would like to report on an application of the 2D exchange method to elucidate the various rearrangement mechanisms in solutions of the compound dodecamethoxyorthocyclophane (DCP). Its 1D NMR spectra in organic solvents indicate that in solutions it exists in two mutually exchanging conformations identified as the "sofa" and "boat" forms,



Several rearrangement mechanisms may be involved in the exchange process, including direct pseudorotations of the sofa and boat forms as well as two different sofa-boat interconversion reactions. The overall rearrangement scheme can be summarized by the following diagram



In principle the various rate constants in this scheme can be obtained by a complete lineshape analysis of the 1D spectra, but in practice this did not prove feasible. We have therefore performed 2D exchange experiments on solutions of DCP and examples of results obtained in nitrobenzene- $d_5$  for the aromatic protons are summarized in Fig. 1. Quantitative analysis of these results indicate that while the sofa-sofa pseudorotation is fast the direct boat-boat pseudorotation is slow and most likely involves a sofa form as an intermediate. The two sofa-boat interconversion reactions appear to have similar rate constants. Best-fit plots to the experimental intensities at 30°C of the diagonal and cross-peaks using the following rate constants,  $k_1=6.7$ ,  $k_2=0$ ,  $k_3=4.5$ ,  $k_4=4.7 \text{ sec}^{-1}$ , are shown in Fig. 2. The results demonstrate the power of the 2D exchange method to elucidate mechanisms of complex rearrangement processes.

With best regards,

*A. Maliniak*      *R. Poupko*      *Z. Luz*      *H. Zimmermann*  
 A. Maliniak      R. Poupko      Z. Luz      H. Zimmermann

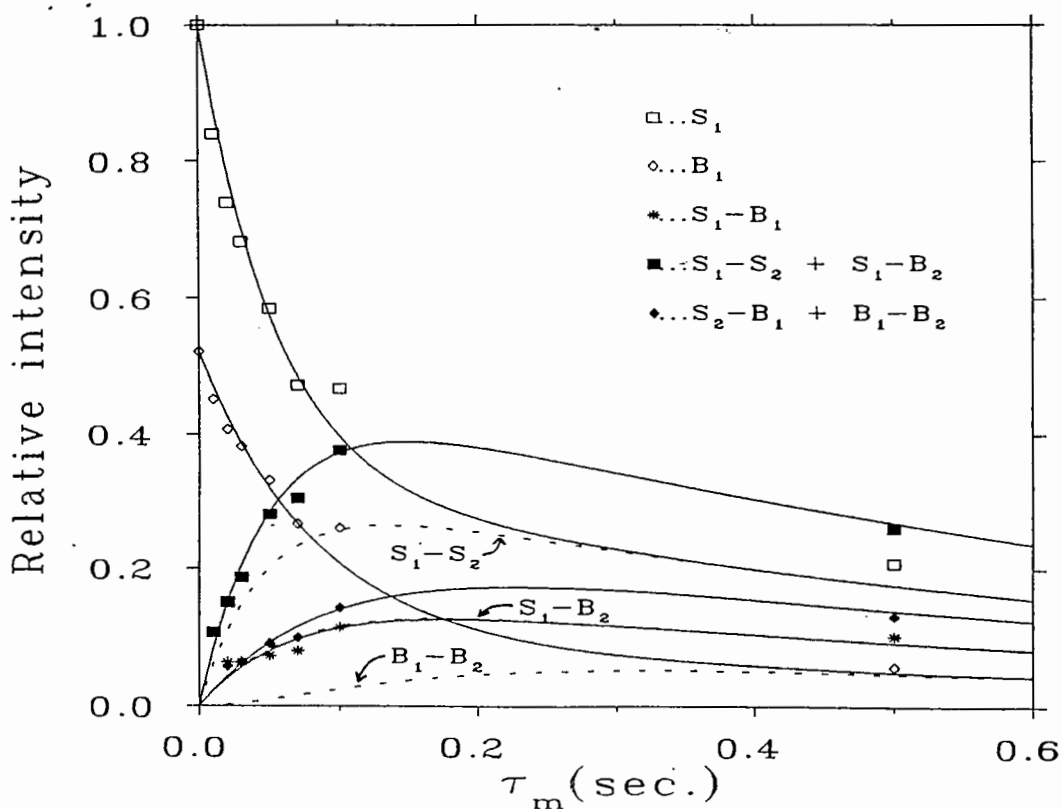
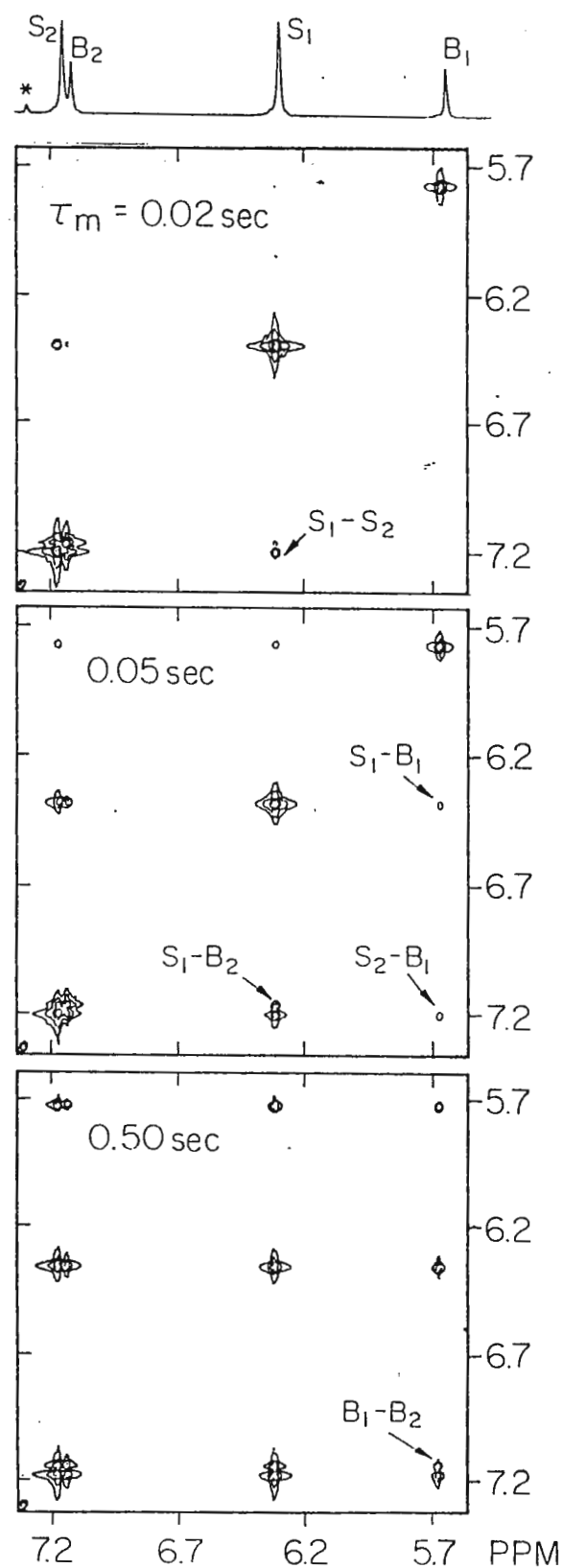


Fig. 2. Best-fit plots of the integrated intensities of the diagonal and cross peaks obtained in 2D spectra of the type shown in Fig. 1. The best-fit rate constants used in the plots are given in the text of the letter.

Fig. 1. Two dimensional exchange spectra of the aromatic region of a DCP solution in nitrobenzene at 30°C at three different mixing times,  $\tau_m$ . The labelling of the cross peaks are according to the 1D spectrum at the top of the figure and are indicated upon first appearance. S and B correspond to "Sofa" and "boat" respectively.



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Cornell University

Department of Chemistry  
Baker Laboratory  
Ithaca, New York 14853 USA

August 20, 1989

(received 9/12/89)

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

### Single Frequency Decoupling of $^{15}\text{N}$ from $^6\text{Li}$

Dear Barry,

In our studies of lithium dialkylamide solution structures, we often find that NMR spectroscopy on  $^6\text{Li}$  and  $^{15}\text{N}$  doubly labelled samples is a powerful probe of atom connectivities and aggregation states (equation 1 and figure 1a,b). For complex cases wherein many species can coexist, correlating the  $^6\text{Li}$  and  $^{15}\text{N}$  multiplets with specific connectivities can be difficult. We have now found that single frequency decoupling of  $^{15}\text{N}$  from  $^6\text{Li}$  can be achieved with only minor adaptations of a generic high field spectrometer.



On the Varian XL400 equipped with a 30-105 MHz broadband probe, the procedure is as follows. The  $^2\text{H}$  lock channel is tuned to the frequency of  $^6\text{Li}$  to function as the 'observe' channel. Using a PTS 160 radio-frequency synthesizer, irradiation at the frequency of any given  $^{15}\text{N}$  resonance can be achieved using the broadband coil. Filters are necessary to minimize excessive noise at the frequencies of  $^6\text{Li}$ . Single frequency decouplings of  $^{15}\text{N}$  resonances corresponding to dimeric and monomeric lithium tetramethylpiperidide are illustrated in Figure 1c,d.

We repeated the experiment with similar results using a Brüker AC300 spectrometer. To observe  $^6\text{Li}$  via the  $^2\text{H}$  lock channel, the probe was modified by adding a capacitor and removing the  $^1\text{H}$  filter in the probe lock circuitry.

We would like to thank Jim Simms of MIT and Brian Andrew of Brüker for several very helpful discussions.

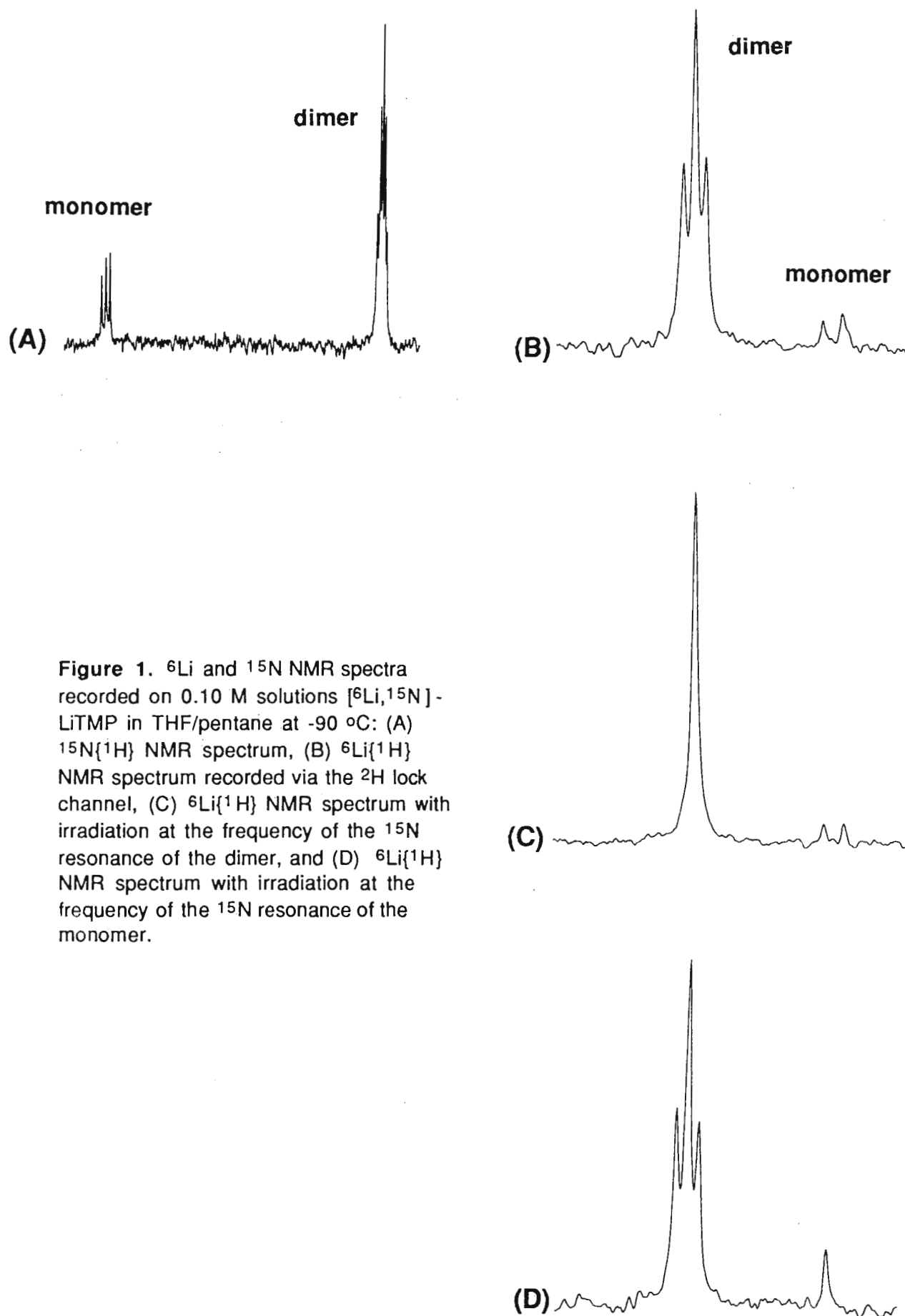
Sincerely,

Aidan Harrison

David Fuller

James Gilchrist

David Collum







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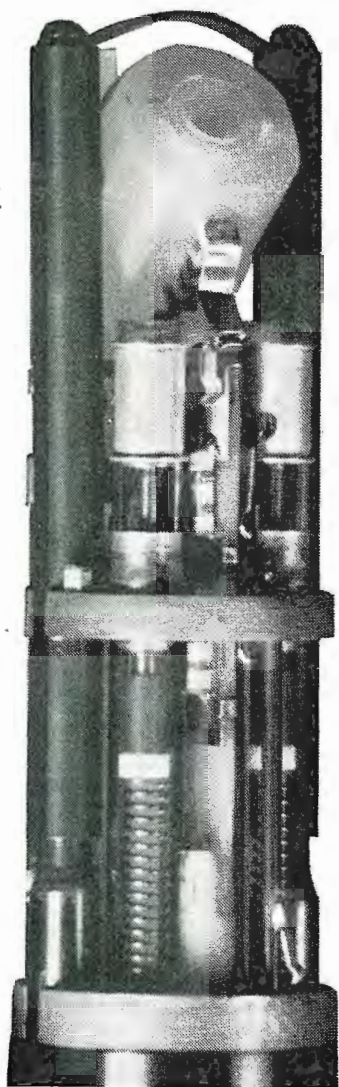
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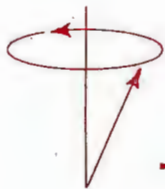
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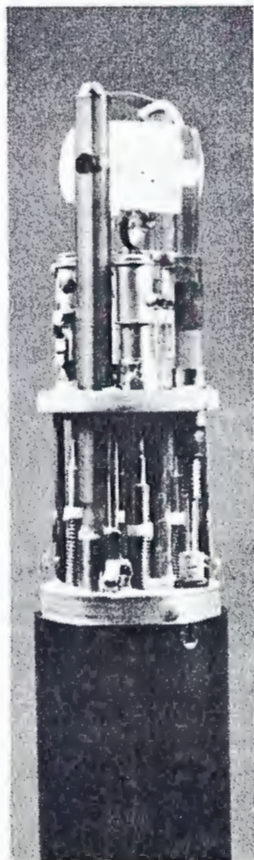




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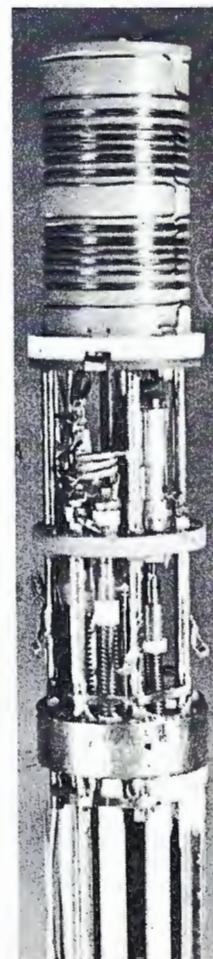
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Head of Department:  
Professor J.H.P. Utley  
BSc, PhD, DSc, CChem, FRSC

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

31st August 1989  
(received 9/18/89)

Dear Barry,

$^{29}\text{Si}$  N.m.r. of opals and flints

This is to open our new (ULIRS) subscription from Queen Mary College, and I would like to start by summarising our function: ULIRS (University of London Intercollegiate Research Service) provides wide-ranging instrumental facilities to Departments of Chemistry and related disciplines in London University. London is a federal University and although its constituent colleges are widely separated (in 2-dimensional Euclidian space), ULIRS is a strongly unifying force. The individual instruments are located in colleges where there is considered to be sound local expertise, and as far as n.m.r. is concerned we have a Bruker WH-400 (located here at Q.M.C., managed by myself, installed in 1980), Bruker WM-250 (Kings College, Jane Hawkes, 1980), Bruker MSL-300 (Royal Holloway & Bedford New College, Chris Groombridge, 1985), JEOL GSX-500 (Birkbeck College, Harry Prkes, 1988), and CISCO 4.7 T 33 cm. bore spectroscopy and imaging system (Q.M.C., Steve Williams, 1989). Other ULIRS instrumental facilities include e.s.r. mass spec., ORD/CD etc., etc. There are, in addition, somewhere around 12 other supercon systems in the University for individual (chemistry) departmental or research group use, and an increasing number of medically oriented systems.

For a technical contribution I would like to mention some  $^{29}\text{Si}$  solid state results we have obtained on samples of powdered opals and flints. We have looked at ca. 30 samples of widely different geographic origin by three main methods: (a) chemical analysis with emphasis on quantification of the water content; (b) X-ray diffraction patterns to calssify the mineralogical phases; (c)  $^{29}\text{Si}$  n.m.r. The  $^{29}\text{Si}$  spectra were run with MAS only, MAS with  $^1\text{H}$  decoupling, and CP-MAS with  $^1\text{H}$  decoupling. As shown in Figure 1 for beltane opal the  $^1\text{H}$  decoupling did sharpen up the spectra. We have assigned the resonances as shown to kaolinite  $Q^3$  (OAl), quartz  $Q^4$ , tridymite  $Q^4$ , silanol  $Q^3$ , and possibly a shoulder on the tridymite resonance is due to cristobalite. The bands assigned to kaolinite and silanol  $Q^3$  were the only ones visible in the CP-MAS spectrum and we believe that essentially all the water present (ca. 4.7%) is associated with these components - principally the kaolinite. In this case the X-ray powder pattern also indicated kaolinite, quartz, cristobalite, and tridymite, but for some other opals the powder patterns were too ill defined to be of use whereas the  $^{29}\text{Si}$  spectra did give useful phase information. Figure 2 shows our attempt to correlate the width of the major  $^{29}\text{Si}$  band (from the MAS only spectrum) with total water content. There are clearly two regions with the sharper resonances coming from these samples with lower water content. A similar appearance is found for the correlation between  $^{29}\text{Si}$  chemical shift and total water content.

This work is ongoing and is in collaboration with Stu Adams (Geochemistry here at Q.M.C.), and Eirian Curzon (now with Bruker, formerly of Warwick University). We thank Chris Groombridge for the MSL-300 spectra. Best wishes from Ed. Randall.

Yours sincerely,

Dr. G.E. Hawkes

$^{29}\text{Si}$  MAS/NMR spectra (MSL - 300) of Beltane opal (S3); (a) with MAS only and (b) with both MAS and high power  $^1\text{H}$  decoupling.

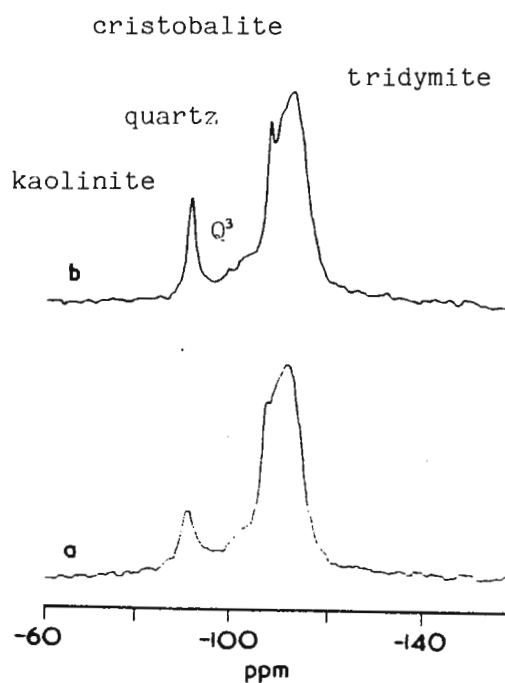
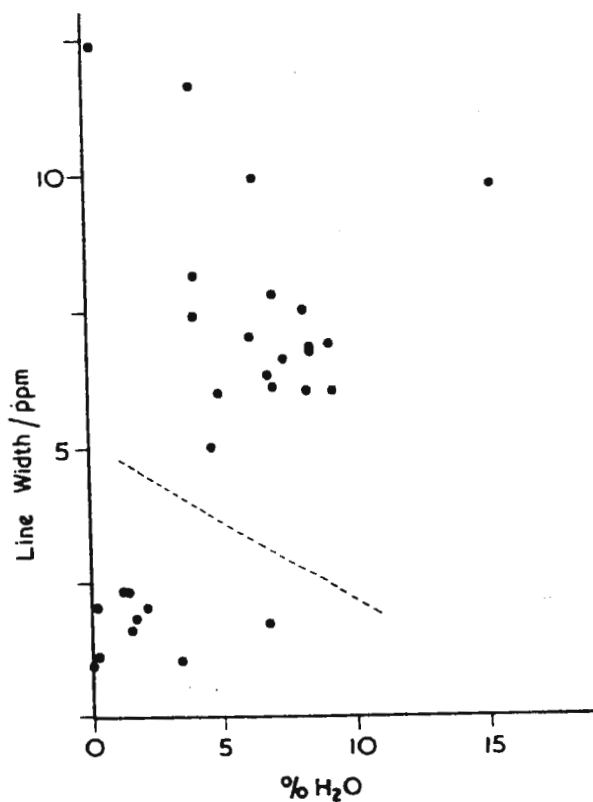


Figure 2.

Correlation between the full width at half height of the major resonance in the  $^{29}\text{Si}$  MAS only spectrum and the total water content of the opal samples. The broken line arbitrarily divides the correlation into two regions.



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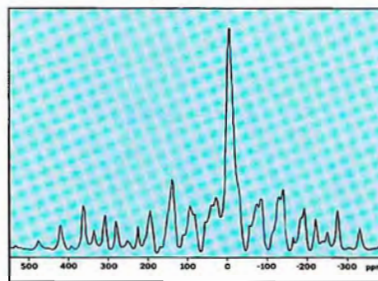
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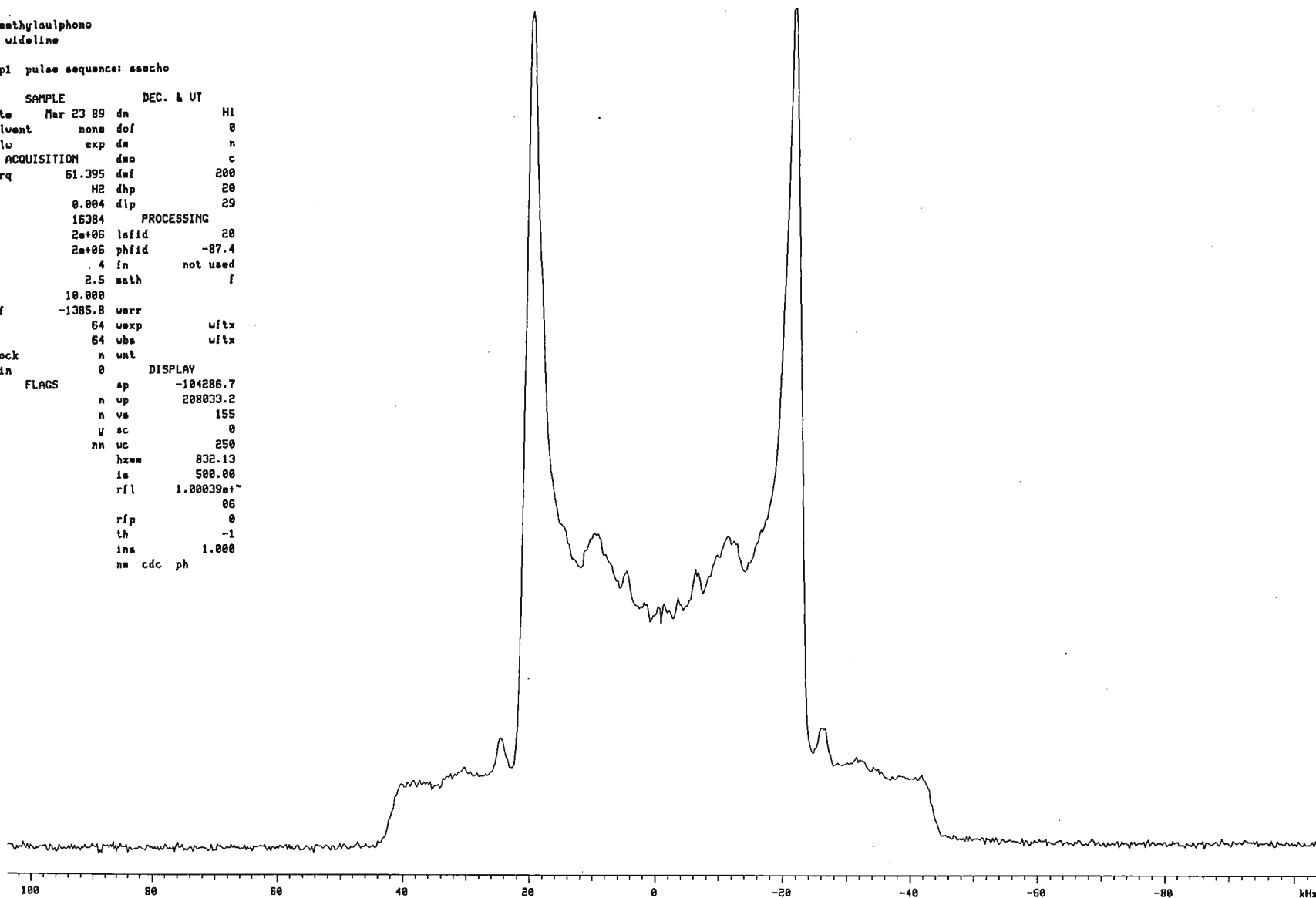
# **<sup>2</sup>H WIDELINE DIMETHYL SULPHONE**

Run on a UNITY 400, this spectrum, transformed with no window function, shows the effects of macroscopic crystallinity. The sample was packed in the tube, raised to its melting point in the probe, and then allowed to slowly cool back to room temperature before the wideline spectrum was obtained.

Dimethylsulphone  
2H wideline

expl pulse sequence: sscho

SAMPLE		DEC. & UT	
date	Mar 23 89	dn	H1
solvent	none	dof	0
file	exp	dm	n
ACQUISITION		dwa	c
afreq	61.395	dof	200
tn	H2	dhp	20
at	0.004	dip	29
np	16384	PROCESSING	
su	2e+06	lsfid	20
fb	2e+06	phfid	-87.4
bs	.4	in	not used
pu	2.5	math	f
d1	10.000		
tof	-1385.8	verr	
nt	64	wexp	u1tx
ct	64	wba	u1tx
elock	n	unt	
gain	0	DISPLAY	
FLAGS	ap	-104286.7	
il	n	up	208033.2
in	n	vs	155
dp	y	sc	0
hs	nn	uc	250
	hzmm	832.13	
	is	500.00	
	rfl	1.00039e+~	
	rfp	06	
	th	-1	
	ina	1.000	
	nm	cdc	ph





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Pasadena, California 91125

Division of Chemistry and Chemical Engineering  
Gates and Crellin Laboratories of Chemistry

John D. Roberts  
Institute Professor of Chemistry, Emeritus

September 26, 1989  
(received 9/28/89)

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

Dear Barry:

*LAOCOON Revisited.*

One would believe that multidimensional FT-NMR would solve all problems in spectral analysis, but there actually are some tightly coupled, or otherwise unsuitable, cases which are better handled by more old-fashioned methods. Lately, we have been encountering some quite complex  $^{15}\text{N}$  spectra with proton couplings which are not very amenable to multidimensional FT-NMR procedures. For spectral analysis in olden times (olden times = 20-30 years ago), we used the FORTRAN LAOCOON programs I, II and finally III (cf. LAOCN3, A. A. Bothner-By and S. M. Castellano in D. F. DeTar, Editor, *Computer Programs for Chemistry*, Vol. I, pp. 10-53, W. A. Benjamin, Inc., 1968). We still have drawers and drawers of LAOCOON IBM cards, but have no card readers to assist in getting the program into usable shape with our current computers.

I assume that there have been plenty of alternative spectral analysis programs developed for PC's and, of course, many of these PC's are easily the equivalent of the IBM 704's we used with LAOCOON. But I was in a hurry and, rather than scouting the field, I decided to port LAOCOON over into the PC world on my own. With no card reader, it was fortunate that I had a listing of the program at least in the form of a poor xerox copy. So scanning followed by optical-character recognition and heavy editing converted the text to source code, although with probably no more efficiency than a complete retyping. With that investment of time, I was committed and it was a bit of a cultural shock to realize that the olden days of using cards was not all that bad. You punched your input data (especially the multitude of observed frequencies you were trying to fit to the calculated values) on cards, ran them through, then just repunched the few cards that needed to be changed and ran them again. In the modern PC world, you wouldn't want to reenter 50 transitions for each run and you need to be able to store all that stuff in a file which can be easily edited. But to do this, you need to write endless grudgy file-modifying routines so that the inputs can be saved in useful reusable form on disk.

I decided to convert the FORTRAN LAOCN3 to *TRUE BASIC* because *TB* is highly structured, uses no line numbers, no GO TO's, has strong graphics capabilities and, in compiled form can be converted into runtime packages which, with only IO modifications, can be used on either IBM (MS-DOS) or Macintosh.

I won't bore you with problems of converting an almost wholly undocumented heap of spaghetti code, full of fancy FORTRAN integer arithmetic, scratch tapes and all, to something that appears to run reliably, has pretty solid error traps and also has those editable data files, as well as choices of stick or line plots that can be output to a laser printer. Amusingly, the original 640 lines of FORTRAN code has swollen to a 130-Kbyte file of almost 3,200 lines of *TRUE BASIC* in the transformation. It would never have fitted in an IBM 709!

A test of Bothner-By's sample run on the proton spectrum of pyridine gave a slightly different and better fit on iteration, probably because the arithmetical precision used by the Mac II is higher than single precision on the IBM 704-709 machines.

If your readers are interested, I may consider some form of distribution. With all good wishes,

Very truly yours,

Jack



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

September 19th, 1989  
 (received 9/29/89)

### Rf Phase Switching on Bruker AM Spectrometers

Dear Dr. Shapiro:

The timing scheme of pulse and phase commands on Bruker AM spectrometers constitutes the subject of this communication. Many Bruker users may be unaware of a very important subtlety in the relative timing of rf phase changes and pulse execution, so we thought it might be helpful to discuss this point briefly. In those models of the AM spectrometer which have a process-controller and digital phase-shifting hardware, for a pulse command such as P1 PH1 there is a fixed delay of about 2 microseconds between when the phase of the rf is changed to PH1 and when the pulse is actually generated; this delay is designed to allow time for the phase to change and settle to the new value. On the other hand, if only a phase change is desired, a command such as D1 PH1 will cause the phase to be switched at the start of the delay D1. It is very important to realize that if two pulse commands are placed back-to-back, or a delay with phase change command immediately follows a pulse command, the phase will be changed approximately 2 microseconds before the end of the first pulse. We have observed pulse distortions when such a delay is absent in these situations, and they are eliminated by the insertion of an additional delay of 2 microseconds between the consecutive pulses, or between the pulse and the phase change command.

We were able to observe this timing difference clearly using an oscilloscope and a test pulse sequence on our AM-600 spectrometer. Our set-up allowed us to simultaneously monitor rf pulses and their corresponding relative phase. The pulse sequence consisted primarily of two consecutive pulses, each with a different phase program as follows:

- 1 (P1 PH1)
- 2 (P1 PH2),

where PH1 and PH2 are phase shifted by some amount. The phase of the first pulse was observed to switch to the next phase program about 1-1.5 microseconds prior to the end of this pulse. If the phase transients produced at the beginning of the next pulse are also taken into account, the total actual distortion time introduced by the phase switch is an estimated two microseconds. Based on this, the test sequence was rewritten to include a delay between the two phase and pulse commands as follows:

- 1 (P1 PH1)
- 2 (D6 P1 PH2) ----> D6 is approximately 2 microseconds.

Both pulses then appeared to be undistorted and unaffected by the change to the second phase program. Thus, the delay between consecutive pulses or between a pulse and a phase reset command is necessary to avoid distortions.

It is often essential to know that one is delivering a set of consecutive, undistorted pulses in a given experiment. The errors from these distortions vary from experiment to experiment. In other situations, the ability to reinitialize the phase of either the observe or decoupler channels of the spectrometer prior to signal acquisition without affecting the phase of the previous pulse is important. We have specifically encountered the latter situation when using an "ADC" instead of a "GO" command to trigger acquisition, for example. With the addition of an extra delay prior to the receiver phase command and directly after the previous pulse and phase commands, the problems caused by this distortion were remedied.

Please credit this to the account of P.E. Wright.

Sincerely,

Pearl Tsang

Mark Rance



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August 22, 1989  
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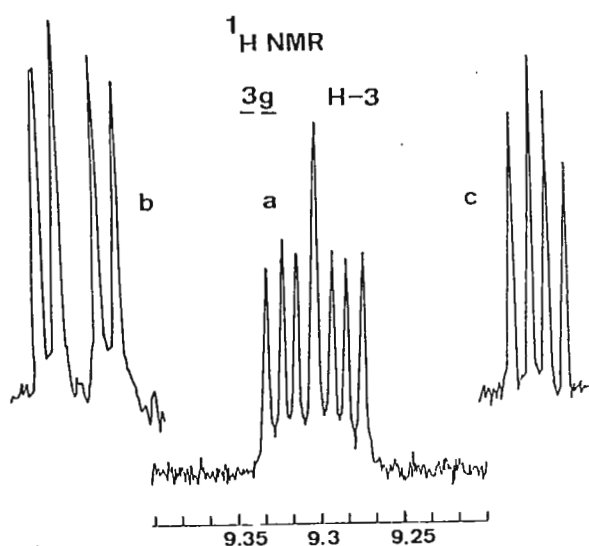
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Prof. B.L. SHAPIRO  
966 Elsinore Court  
Palo Alto, California 94303  
USA

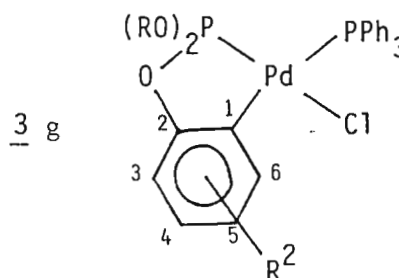
Dear Barry,

Aryl phosphite and phosphine complexes of transition metals are useful catalysts and reagents so that their  $^1\text{H}$ -spectra occasionally receive careful scrutiny. Interestingly, complexes such as 3g routinely show  $^4J$  values to H-3 of *ca.* 7 Hz (see figure) as well as  $^5J$  values to remote protons (e.g.,  $^5J(\text{P}', \text{H}-5) = 2 - 3 \text{ Hz}$ ).

We find it useful to recognize these interactions since the interpretation and recognition of selective aryl protons can provide hints as to which of these are proximate to the metal, and potentially readily attacked.



Best wishes



$^1\text{H}$ -NMR of H-3 in complex 3g: a) conventional spectrum showing splittings arising from three spins b) with phosphite  $^{31}\text{P}$ -decoupling and c) with  $\text{PPh}_3$   $^{31}\text{P}$ -decoupling (WM-250,  $\text{CDCl}_3$ ).  $^3J(\text{H}-3, \text{H}-5) = 2.7 \text{ Hz}$ ,  $^4J(\text{P-phosphine}, \text{H}-3) = 8.0 \text{ Hz}$ ,  $^4J(\text{P-phosphite}, \text{H}-3) = 4.9 \text{ Hz}$ .

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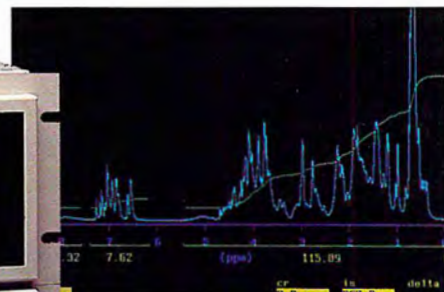
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August 31, 1989

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

**At Last, a "Pen" that Writes.**

Dear Barry,

The variety of computer peripherals is increasing every day. If the NMR operator keeps his eyes and mind open, these peripherals can provide solutions to some of the oldest problems for the operator.

At Du Pont, there is an HP7550A plotter on the Bruker AM 600. This plotter is used to produce hard-copy plots of 1DFT spectra and 2DFT contour plots. It is an amazing machine which can change pens for different colors and "throw" the paper into, around and out of the plotter with tremendous force. The problem was that the plots were infrequent enough that the pen and paper status of the plotter was usually unknown. A particular problem (the age-old one) was that the pen sometimes skipped when drawing lines or failed to write at all. When the pens worked, they plotted best on a glossy (and expensive) paper. The result was that to get high quality plots, the plots often had to be redone after fixing the paper or cleaning the pen. The struggle to keep the pen and plotter working gave the users enough of a hassle that plots were delayed or foregone entirely.

One manufacturer has brought out an inexpensive (list \$495, but can be purchased for under \$300) cartridge for the HP Laserjet Series II which allows it to emulate an HP Plotter.

Pacific Data Products  
*Plotter in a Cartridge*  
(619) 552-0880

The HP Laserjet Series II printer can be purchased with the required 2Mbytes of additional memory about \$2000. When combined with the above cartridge and plugged in to the Aspect computer plotter port, the user realizes faster high quality plots on standard (and cheap) copy paper. *And, there are no pens to not write.* The lines are very sharp and clear and the plots are on a convenient 8 1/2" X 11" paper. The communication parameters are:

9600 Baud  
7 Bits  
Odd Parity  
2 Stop bits

The Laserjet/Plotter Cartridge has a substantial cost advantage over flat-bed plotters, and can be used with any computer with HPGL output. Further details can be supplied upon request.

Regards,

Peter J. Domaille  
E. I. du Pont de Nemours & Company  
Central Research  
Experimental Station  
Wilmington DE 19880

Woodrow W. Conover  
Fremont Magnetic Resonance

# NMR

## NEWSLETTER

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\* \* \* \* \*

### POST-DOCTORAL FELLOW - BURROUGHS WELLCOME CO. Research Triangle Park, North Carolina

The NMR Section of the Organic Chemistry Division has a postdoctoral position available immediately. Responsibilities involve performing state-of-the-art two-dimensional NMR experiments and participating in the development of UNIX based software to extract data and to augment existing display software. This individual will also be expected to assist in the networking of four existing superconducting NMR spectrometers and several data stations are well as a new spectrometer which will be added to the network in the coming year.

Candidates must have a Ph.D. in chemistry and experience with modern 2D-NMR techniques. Experience with the UNIX programming language and computer networking required. Other factors to be considered include: Technical knowledge and proficiency; exposure to or familiarity with 3D NMR and/or NMR imaging; Ph.D. with a specialization in physical chemistry; and experience with NMR software packages.

To apply, submit your curriculum vitae by November 20, 1989 to:  
Burroughs Wellcome Co., Scientific Recruiting and Staffing, Position #61288-4,  
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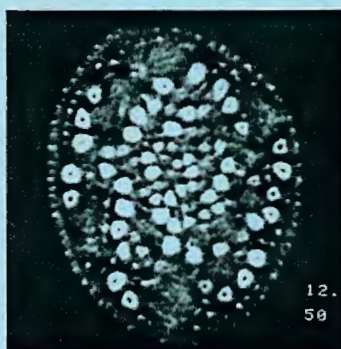
# CSI 2T Applications

## Shielded Gradients and NMR Microscopy

In spin warp imaging, there is a trade-off between minimum TE and maximum resolution. Even if rise and fall times were zero and phase encoding occurred during the entire echo delay, a  $\pm 2$  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  $256 \times 256$  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  $32 \times 256 \times 256$  DEFT data set.



**Fig. 1**—Agapanthus bud  
Matrix  $256 \times 256$ , 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

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# VPLX

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

For more information, contact JEOL.

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\*VAX is a trademark of Digital Equipment Corporation

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

