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TABLE 1 DEUTERATED SOLVENTS

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D-120	Acetone-d ₆	CD ₃ COCD ₃	99.5%	1.17	-17	56	0.551 (32)
D-13	Acetone-d ₆	CD ₃ COCD ₃	99.8%	0.87	-34	57	0.460 (20)
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D-14							0.611
D-21							
D-122							
D-130							
D-28	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

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FORTHCOMING NMR MEETINGS

- 1991 Pittsburgh Conference & Exposition, Chicago, Illinois, **March 4 - 8, 1991**; NMR sessions and symposia on March 5 and 6; Information: Pittsburgh Conference, Dept. CE, 300 Penn Center Blvd., Suite 332, Pittsburgh, PA 15235.
- In Vivo Magnetic Resonance Spectroscopy Tutorial and Participatory Workshop, St. Louis, Missouri, **April 4 - 7, 1991**; See Newsletter **385**, 58.
- 32nd ENC (Experimental NMR Spectroscopy Conference), St. Louis, Missouri, **April 7 - 11, 1991**; Contact: ENC, 750 Audubon, East Lansing, MI 48823; (517) 332-3667.
- 1991 Keystone Symposia on Molecular & Cellular Biology, Keystone, Colorado: **April 8-14, 1991**, *Frontiers of NMR in Molecular Biology; Proteolysis in Regulation and Disease; Protein Folding, Structure and Function*; See Newsletter **384**, 46.
- Fifth Wasington University-ENI/Emerson Electric Co., Symposium on NMR, St. Louis, Missouri, **May 20, 1991**; See Newsletter **388**, 52.
- Contrast-Enhanced Magnetic Resonance, a workshop of the Society of Magnetic Resonance in Medicine, Napa, California, **May 23-25, 1991**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.
- Tenth International Meeting on NMR Spectroscopy, St. Andrews, Scotland, **July 8-12, 1991**; Contact: Dr. John F. Gibson, Secretary (Scientific), The Royal Society of Chemistry, Burlington House, London W1V 0BN, England; See Newsletter **387**, 69.
- Gordon Research Conference on Magnetic Resonance, Brewster Academy, Wolfeboro, NH, **July 15-19, 1991**; Chairman: R. Griffin; Information from Dr. A. M. Cruickshank, Gordon Research Center, Univ. of Rhode Island, Kingston, RI 02881-0801; Tel.: (401) 783-4011 or -3372; FAX (401) 783-7644.
- Tenth Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, San Francisco, **August 10-16, 1991**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.
- International Conference on NMR Microscopy, Heidelberg, Germany, **September 16 - 19, 1991**; See Newsletter **385**, 28.
- Eleventh Annual Scientific Meeting and Exhibition, Society of Magnetic Resonance in Medicine, Berlin, Germany, **August 8-14, 1992**; Contact: S.M.R.M., 1918 University Ave., Suite 3C, Berkeley, CA 94704; (415) 841-1899, FAX: (415) 841-2340.

Additional listings of meetings, etc., are invited.



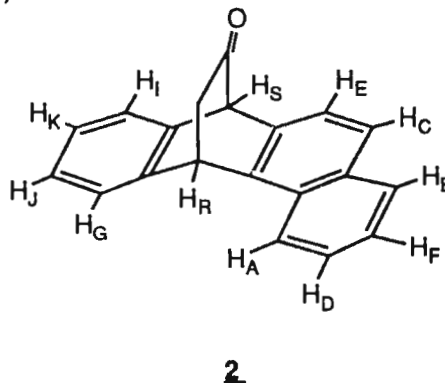
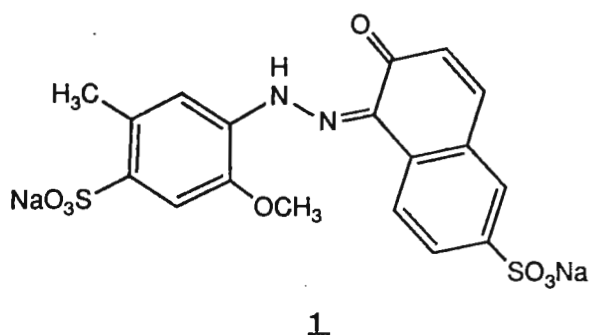
Nov. 19, 1990
(received 11/23/90)

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

Application of the FLOCK Experiment in Structural Elucidation. II

Dear Barry,

An unusual manufacturing impurity of the food colorant FD&C Red No. 40 (**1**) was recently isolated and identified. A variety of nmr experiments have shown it to be a bicyclo[2.2.2]octanone (**2**). The bridgehead protons (R,S) appear at 5.41 and 5.04 ppm, respectively, due to their doubly-benzylic nature, *viz.* being simultaneously located in the planes of two aromatic rings. Moreover, the former is more deshielded, perhaps, because of a *peri*-type of interaction. Lacking an inverse-detection probe, Bill Reynolds' FLOCK experiment¹ permitted unambiguous determination of the structure of **2** by means of 2- and 3-bond C-H connectivities. We believe that **2** is produced in the sulfonation synthesis of 5-sulfo-2-naphthol. It likely arises from a Diels-Alder reaction of a dehydratively-formed "naphthyne" derivative (the dienophile) of 2-naphthol with the parent aryl alcohol (the diene).



Sincerely,

Sandra J. Bell

Eugene P. Mazzola

1. Reynolds, W. F. *et al.*, *Magn. Reson. Chem.* 27, 162 (1989).

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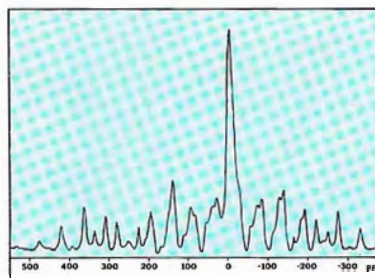
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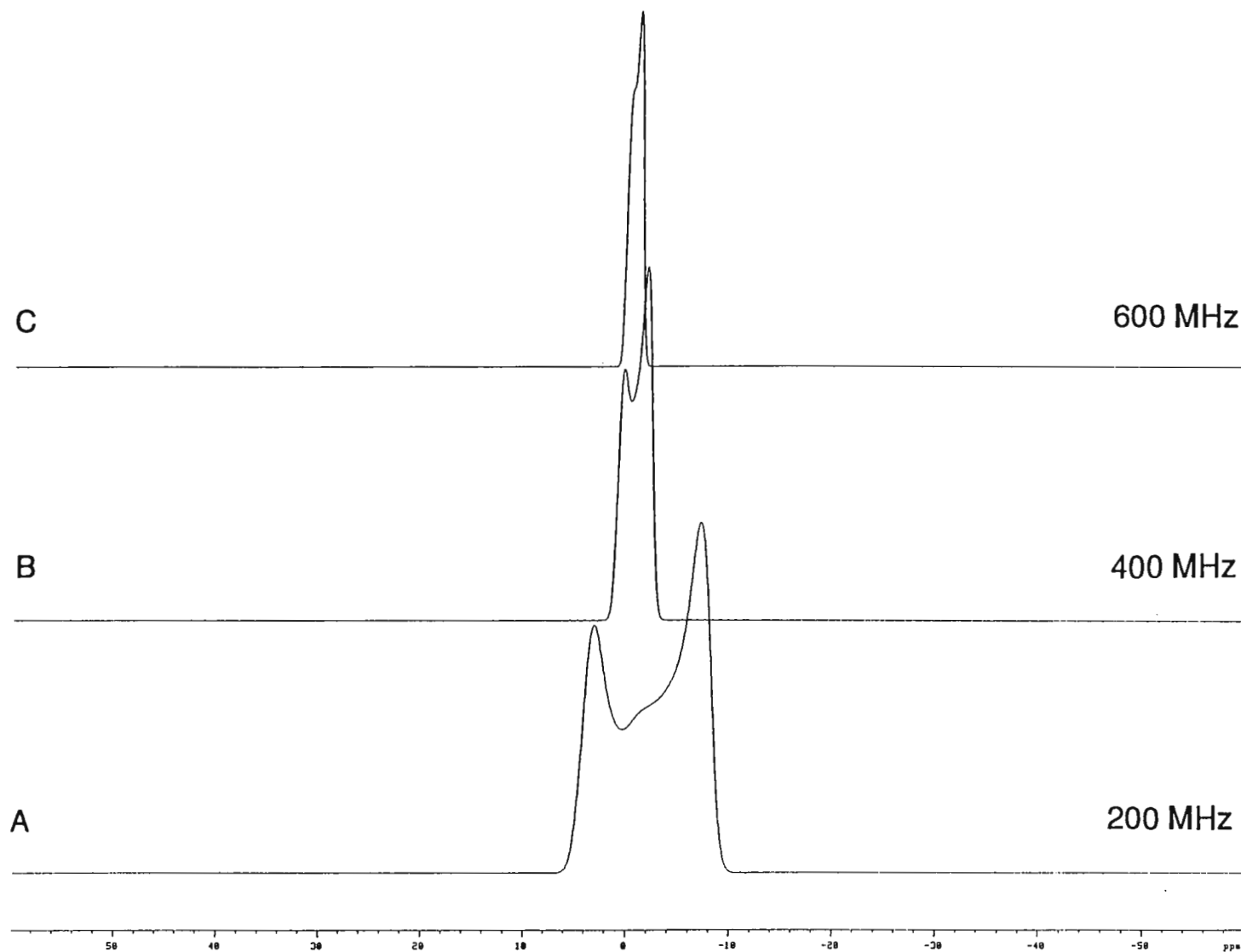
NMR WITH A FUTURE

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POWDER PATTERN SIMULATION OF THE CENTRAL TRANSITION OF NON INTEGER QUADRUPOLES

Given the spin quantum number, the quadrupolar coupling constant, and the asymmetry parameter, the Solids Analysis Software calculates the FID for the resulting central transition static powder patterns of non integer quadrupoles. For many half-integer spin nuclei, the quadrupolar interaction is large and only the central transition is observable. The fine structure observed in the powder pattern is due to the field

dependent second-order quadrupolar effects which are no longer negligible compared to the Zeeman interaction. The spectra below illustrate the magnetic field dependency of the powder pattern, at spectrometer frequencies of 200, 400 and 600 MHz, for a quadrupolar coupling constant of 1.0 MHz. The spin quantum number was 5/2; the asymmetry parameter was 0.





Koninklijke/Shell-Laboratorium, Amsterdam

Shell Research B.V.

Dr. B.L. Shapiro
966, Elsinore Court
PALO ALTO, CA 94303

U.S.A.

388-5

(received 11/26/90)

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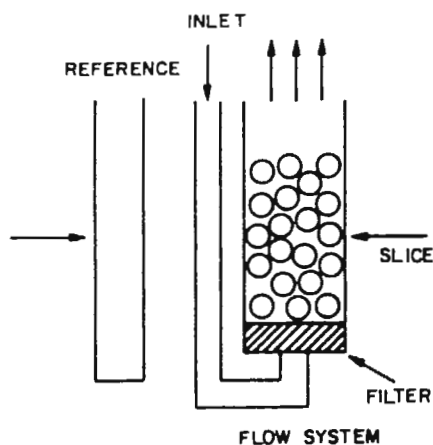
Dear Barry,

'NMR Imaging and Image-analysis Techniques'

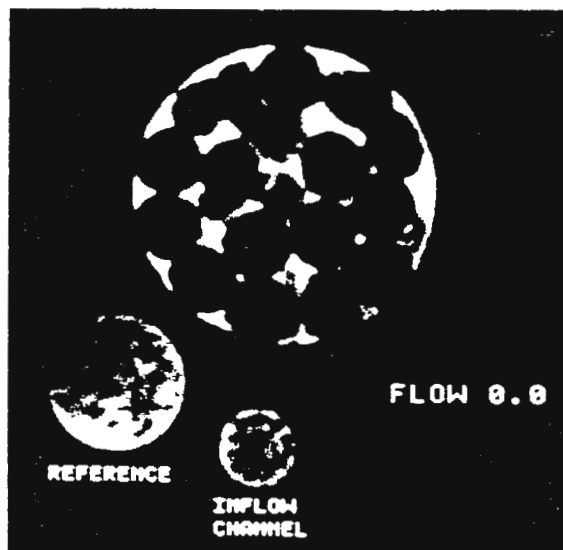
Further to our previous instalment (and promise), concerning the application of image-analysis techniques to NMR microscopic data, we enclose a contribution to stave off the latest DREADED ULTIMATUM. The figure shown below demonstrates some remedial steps towards quantifying the behaviour of fluid flowing in a porous-media model.

The investigation of fluid in porous media represents a particularly difficult problem; by definition, the abundance of the NMR probe may be small (typically 1 to 50% by weight). Further, the data can be accompanied by dispersive effects, non-coherent motional artifacts, local susceptibility effects or, in the case of immobilised fluids, a contribution to the T2 value. This means that the calculated resolution parameters of the experiment are invalid, and more importantly, a non-representative sample of the total spin population is acquired. Detection and quantification of errors such as these is not a trivial procedure, requiring the development of data acquisition and manipulation methodologies to ensure the integrity of the data.

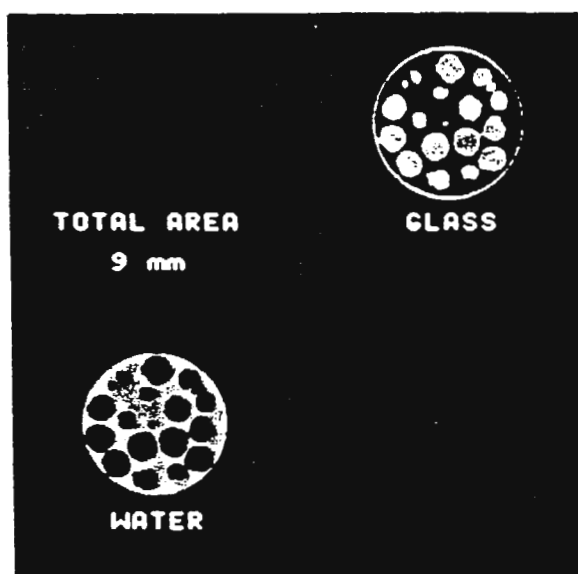
Figure 1b shows a cross-sectional NMR image of the cell shown in Figure 1a, in the absence of flow, as represented by image-analysis software. Following a masking procedure to remove the background from the statistics, pixels in the cell were assigned to be either water or glass by applying first a smoothing algorithm (convolution with a Gaussian kernel and local noise reduction via Sigma filtering), followed by a discrimination procedure based on a local minimum in the grey level histogram of the image. This resulted in the complementary binary images shown in Figure 1c. The binary image corresponding to the glass area, derived from the zero-flow image, was used as a mask for other NMR images which were recorded under flow conditions. By counting the intensity in the resulting histograms the properties of a model may be quantified under a variety of flow regimes. The result is shown in Figure 1d.



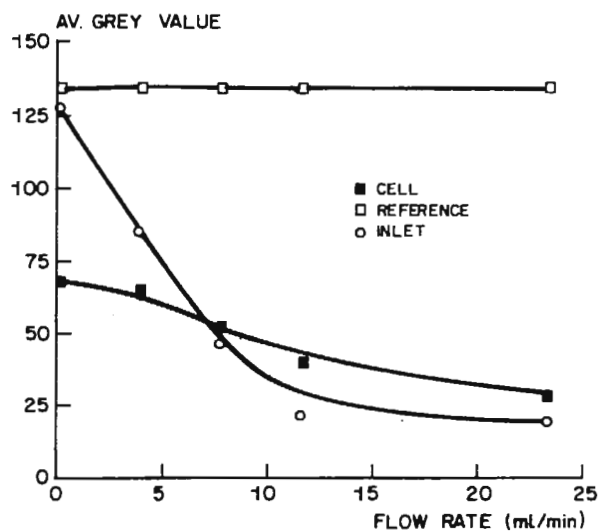
A



C



B



D

Figure 1

The results presented here are simple and of a preliminary nature. With the use of more discriminating experiments estimates of local velocities, irreducible saturation, and permeability will be possible in porous media, even where more than one fluid is involved. As this technique matures it could provide a direct method of validating computational models of systems such as catalytic reactors or oil/gas reservoir rock, which are not yet well understood. These results will be reported more fully at the meeting on porous media to be held in Bologna, November 14-16, 1990.

Sincerely yours,

G.J. Nesbitt

A. de Groot



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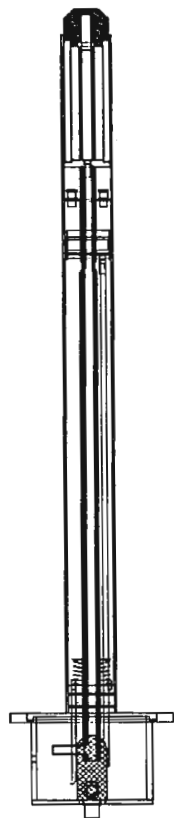
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DEPARTMENT OF CHEMISTRY

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Dr. Hellmut Eckert

11/26/90
(received 11/29/90)Prof. B. L. Shapiro
968 Elsinore Court
Palo Alto, CA 94303**Heteronuclear CW Decoupling in Lithium Salts.**

Dear Prof. Shapiro:

In connection with our dipolar NMR studies of glasses we have recently taken a look at the effect of heteronuclear cw decoupling upon dipolar second moments. Few experiments of this nature have been reported and have been restricted mostly to ^1H and ^{19}F decoupling [1-3]. Capitalizing on new heteronuclear double resonance capabilities available to us at UCSB, we have extended these decoupling studies to ^7Li and report here results for some cubic lithium salts.

In this simple case (absence of chemical shift anisotropies and quadrupolar interactions) the static lineshapes are broadened only by dipole-dipole couplings, which can be calculated from structural information using the van Vleck equation. We were interested to see whether application of a strong on-resonance ^7Li decoupling field would be able to eliminate the dipolar broadening due to the ^7Li spins and hence yield second moments that were predicted from the residual interactions present. To this end, we studied the ^6Li , ^{35}Cl , ^{77}Se , ^{79}Br , and ^{127}I resonances in LiCl , LiBr , LiI , and Li_2Se respectively. For example, Figure 1 shows the ^{79}Br second moments in LiBr determined from the width at half height, as a function of decoupling field strength ω_2^2 . The inset compares this data with the calculated second moment only due to ^{79}Br - ^{79}Br and ^{79}Br - ^{81}Br interactions. Even very weak irradiation results in profound sharpening and it is in this regime, that decoupling sidebands are observable (Figure 2).

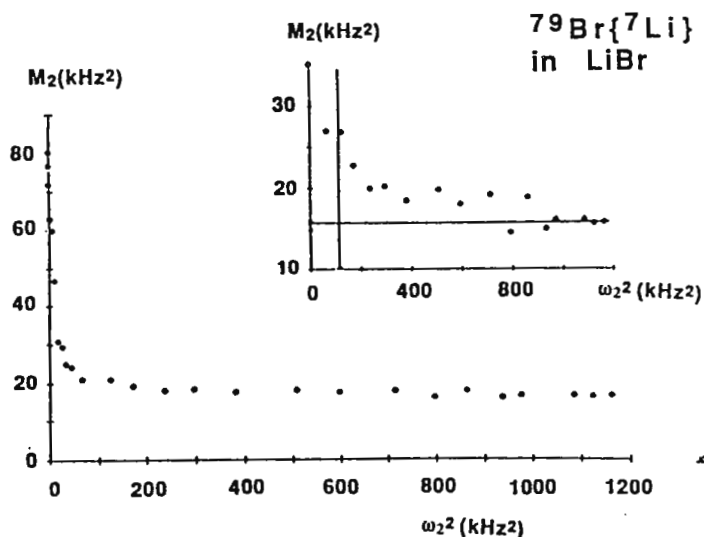


Figure 1

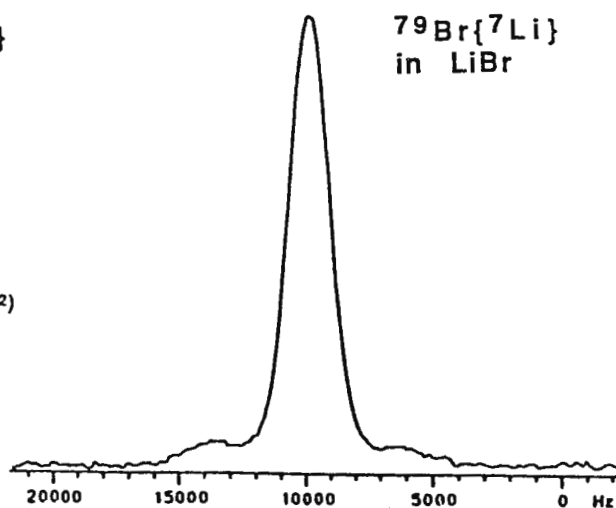


Figure 2

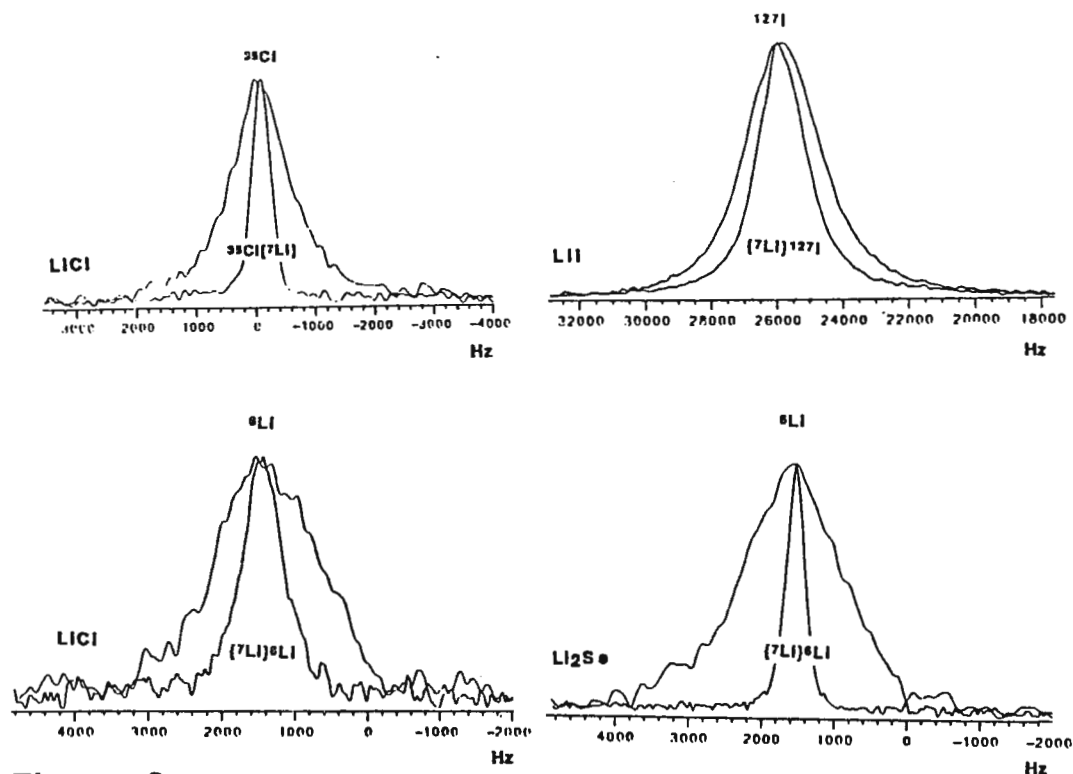


Figure 3

However, as previously predicted [1], the theoretically expected M_2 is only attained in the limit of $\omega_2^2 \gg M_{2I}$ and M_{2S} , where S refers to the observed spin and I refers to the irradiated spin. In the present case $M_2(^7\text{Li}-^7\text{Li})$, which exceeds $M_2(^7\text{Li}-^{79}\text{Br})$, is shown as the vertical line in the inset of Figure 1.

Figure 3 shows representative single- and double resonance spectra in the limit of strong decoupling. In the majority of cases, the second moments obtained from the decoupled lineshapes are close to those calculated. A special case is the ^{35}Cl spectrum in LiCl. Here, the single-resonance spectrum reveals ^7Li self-decoupling, resulting in a near-Lorentzian lineshape that is much sharper than predicted. Irradiation of the ^7Li resonance quenches this mechanism, resulting in the theoretically predicted second moment.

Marked deviations occur for the compound LiI, which exhibits an excess ^{127}I NMR line width both in single and double-resonance experiments. In contrast, the ^6Li spectra are much sharper than predicted. We suspect that this observation is due to strong ^{127}I - ^{127}I exchange interactions, which broaden the ^{127}I spectrum and function as a mechanism of self-decoupling on the ^6Li resonance.

[1] L. R. Sarles and R. M. Cotts, Phys. Rev. 111, 953 (1958).

[2] B. D. Mosel, W. Müller-Warmuth, and G. W. Schulz, Z. Naturforsch. A, 23, 1224 (1968).

[3] M. Mehring, G. Sinnig, and A. Pines, Z. Physik B24, 73 (1976).

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Sincerely,

Timothy Luong

Hellmut

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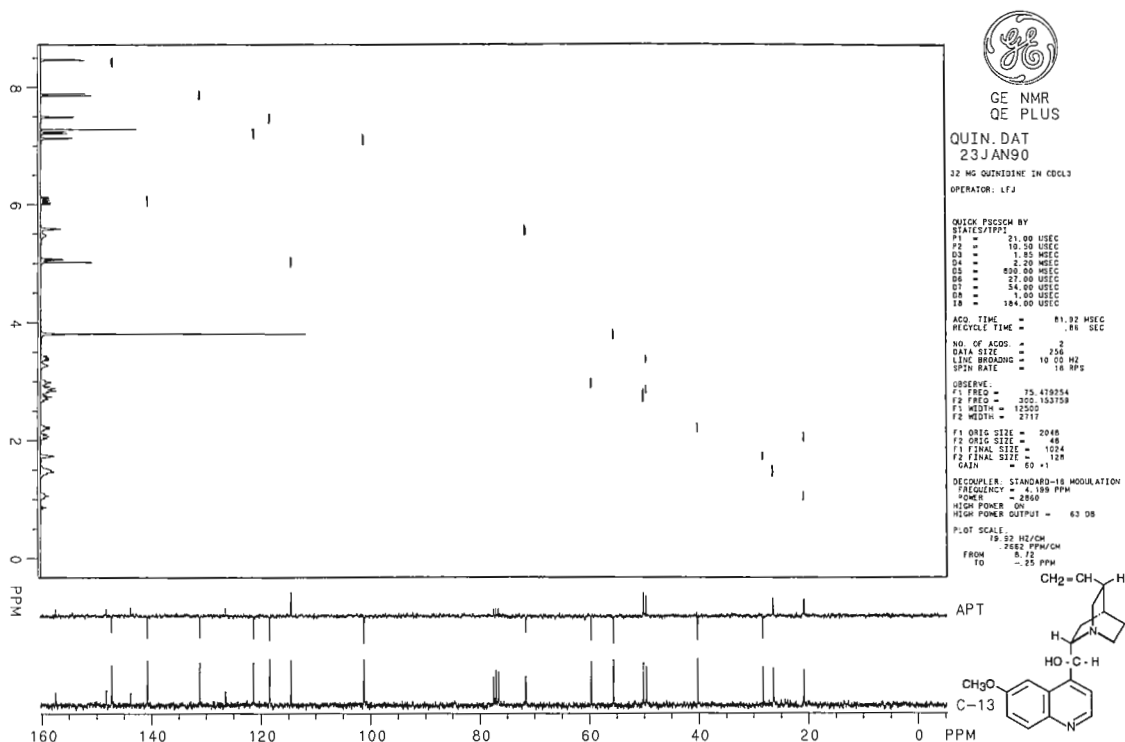
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PLEASE POST
MSL GROUP

UNIVERSITY OF ST. ANDREWS

388-13



From: Dr. F. G. Riddell

DEPARTMENT OF CHEMISTRY,
THE PURDIE BUILDING,
THE UNIVERSITY,
ST. ANDREWS,
FIFE KY16 9ST
SCOTLAND

Tel: (0334) 76161 Ext. 8364
FAX (0334) 78292

FGR/EF

21st November, 1990.
(received 11/26/90)

Professor F. Shapiro,
966 Elsinore Court,
Palo Alto,
CA 94303,
U.S.A.

Dear Barry,

MSL 500 Spectra

This letter is to reopen my account with you since my move from Stirling to St. Andrews two years ago. Since arriving here we have had a Bruker MSL 500 installed - the first one outside the Bruker applications laboratory in Karlsruhe. We share the instrument on a roughly 2:1 basis with David Tunstall in Physics.

We are now, we hope, getting to the top of the learning curve in operating the instrument and it is producing fine spectra from a wide variety of applications.

Joe Crayston and his student Rob Lee have been amongst the first users of the spectrometer to study the reactions of NbCl_5 in benzene with alcohols. As progressive stoichiometric amounts of methanol are added the ^{93}Nb resonance moves to progressively lower frequency and resonances corresponding to replacement of the chlorines in turn by an alkoxide can be seen.

The figure shows spectra of the same sample run on our AM 300 and MSL 500 spectrometers. The increase in dispersion improved baseline linearity and much better spectral excitation of broad lines in the MSL spectrum are immediately obvious. The resonances are $\text{NbCl}(\text{OMe})_4$ ca 1000 ppm and $\text{Nb}(\text{OMe})_5$ ca 1150 ppm.

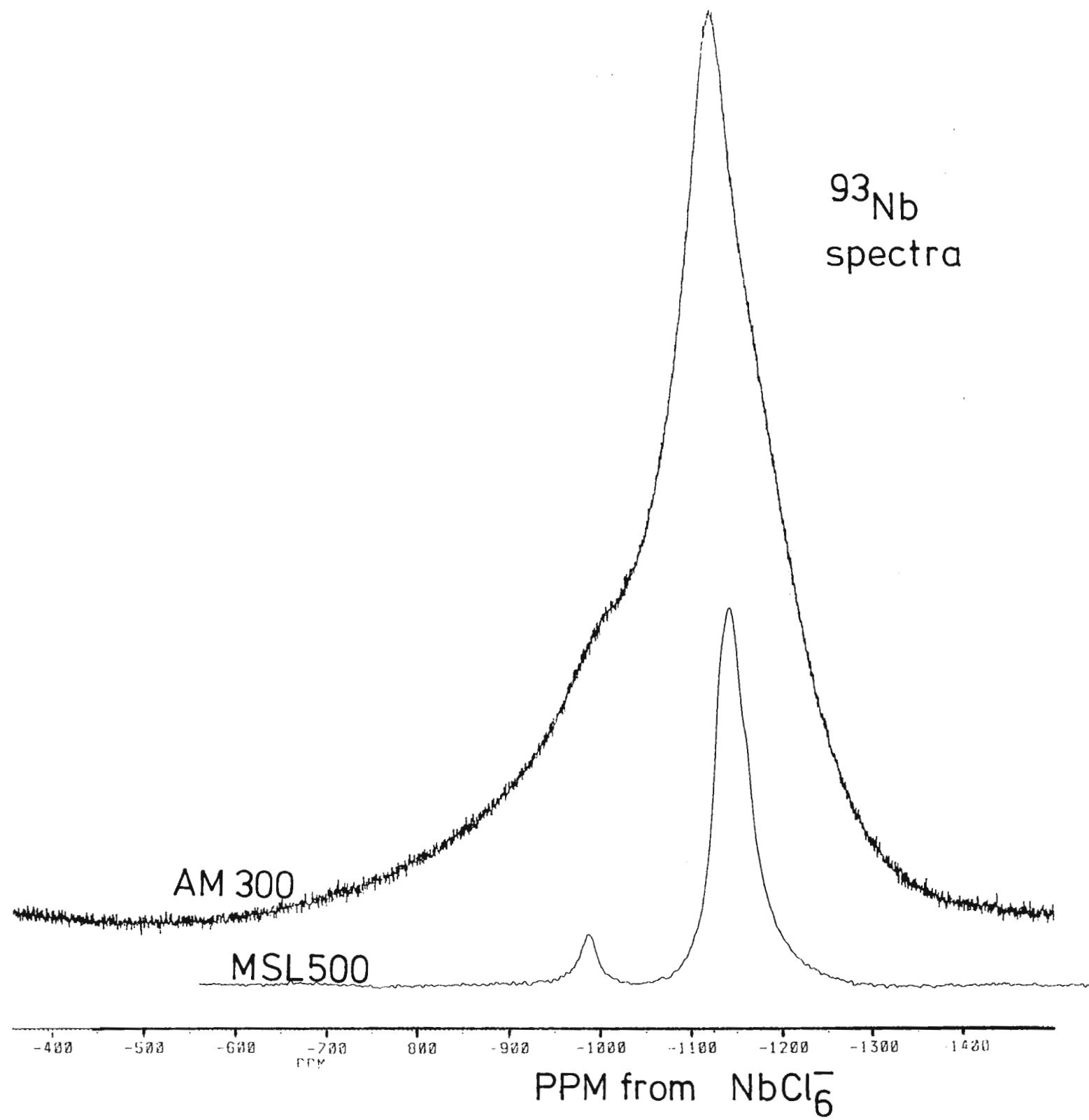
It seems clear that our new toy will keep us happy for a long long time to come.

Best wishes.

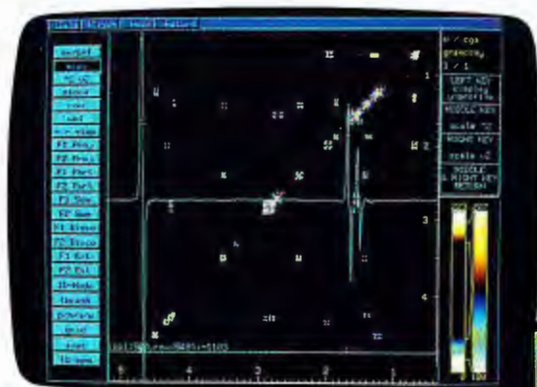
Yours sincerely,

A handwritten signature in cursive script, appearing to read 'F. G. Riddell'.

F.G. Riddell.

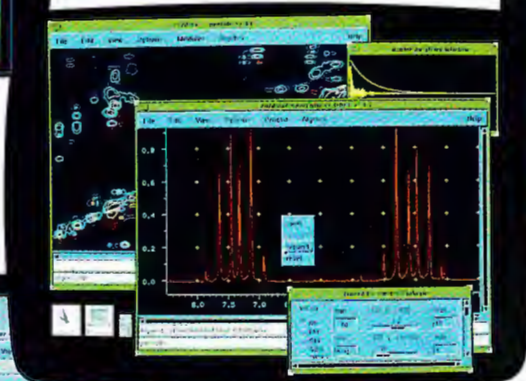


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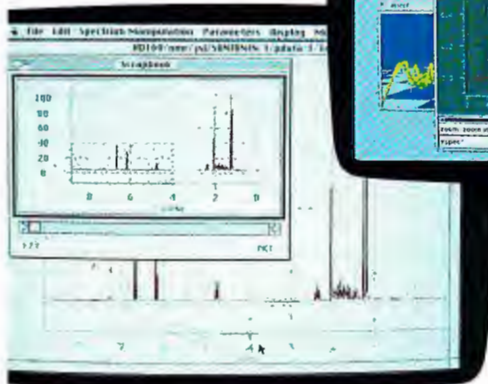
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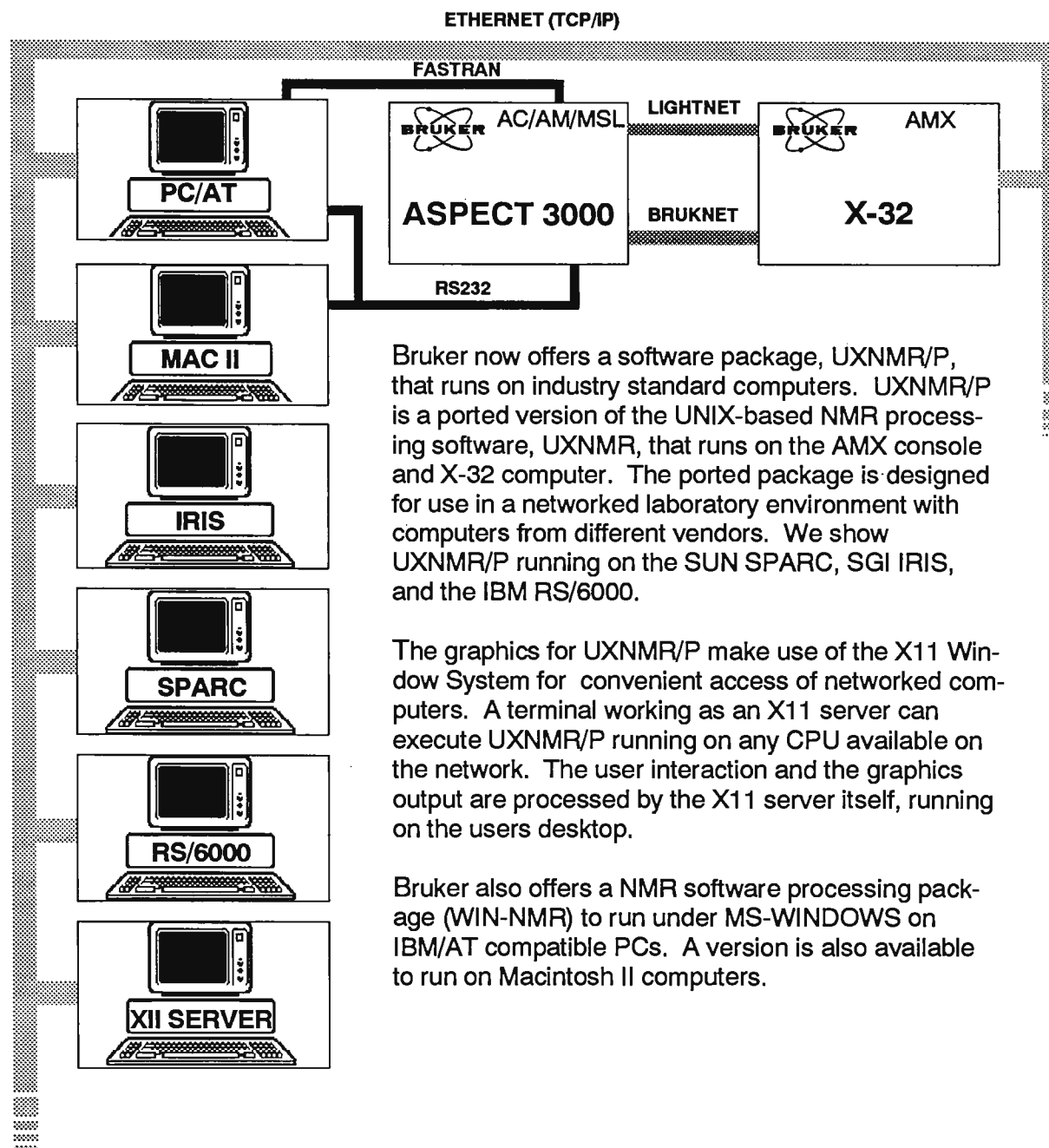


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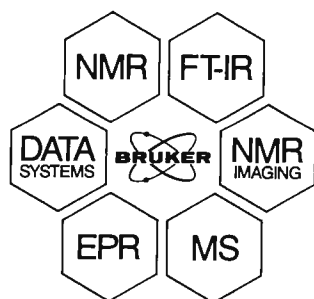
BRUKER SOFTWARE AND NMR NETWORKING



Bruker now offers a software package, UXNMR/P, that runs on industry standard computers. UXNMR/P is a ported version of the UNIX-based NMR processing software, UXNMR, that runs on the AMX console and X-32 computer. The ported package is designed for use in a networked laboratory environment with computers from different vendors. We show UXNMR/P running on the SUN SPARC, SGI IRIS, and the IBM RS/6000.

The graphics for UXNMR/P make use of the X11 Window System for convenient access of networked computers. A terminal working as an X11 server can execute UXNMR/P running on any CPU available on the network. The user interaction and the graphics output are processed by the X11 server itself, running on the users desktop.

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

U S A

N./Réf. 9011445/CB/RE
V./Réf.

Wissembourg, le November 16, 1990
(received 11/23/90)

Satellite talk: two dimensional chemical shift correlations between
 ^{119}Sn and ^{29}Si .

Dear Barry,

Heretenuclear two dimensional chemical shift correlation experiments are now widely used by chemists, either in direct or inverse mode. They are most commonly used to correlate ^1H chemical shifts with low abundance nuclei such as ^{13}C or ^{15}N .

They can be efficiently applied to correlate two low abundance, and low frequency nuclei and therefore be of interesting use for organometallic chemists. One has to notice that this correlation is done via respective satellites of each isotope¹.

Scalar coupling between ^{119}Sn and ^{29}Si was observed on a 90/10 solution of trimethylsilyltributylstannane in C_6D_6 . The standard $^{29}\text{Si}\{-^{119}\text{Si}\}$ heterocosity experiment² was done on an AC300 equipped with a BSV3 amplifier driven by a second PTS and a doubly tuned 10mm probehead. The pulse sequence given below was used, protons were broadband decoupled. The 2D was recorded using 1K datapoints, 576 scans and 16 experiments for a total experiment time of 13.3 hours.

The heterocosity experiment can be switched to a "reverse" approach: $^{119}\text{Sn}\{-^{29}\text{Si}\}$. The inverse bird sequence³ given below was used together with ^1H broad band decoupling. The 2D experiment was recorded using 4K datapoints, 200 scans and 16 experiments for a total experiment time of 3.4 hours.

Best wishes and Happy new year !

Barry
X'mas

C. BREVARD

Christian

A. BELGUISE

[Signature]

- 1: C. Brevard, R. Schimpf, G. Tourne, C.M. Tourne, J. Am. Chem. Soc. 1983, 105, 7059
- 2: G. Bodenhausen, R. Freeman, J. Magn. Reson. 1977, 61, 471
- 3: A. Bax, S. Subramanian, J. Magn. Reson. 1986, 67, 565



HETEROCOSY:

1	ZE	
2	D1	relaxation delay of ^{119}Sn
	SP	saturation pulse to remove natural ^{29}Si magnetization
	P1:D PH1	90 deg ^{119}Sn pulse
	D0	t1 evolution
	(P1 PH2):D P3 PH3	90 deg ^{119}Sn and ^{29}Si
	GO=2 PH4	acquisition of ^{29}Si FID
	WR #1	
	IF #1	
	IN=1	
	EXIT	

PH1=0

PH2=0 2 1 3

PH3=0 0 0 0 1 1 1 1 2 2 2 3 3 3 3

PH4=R0 R2 R1 R3 R1 R3 R2 R0 R2 R0 R3 R1 R3 R1 R0 R2

INVERSE WITH BIRD PREPARATION PULSE:

1	ZE	
2	D1	relaxation delay of ^{119}Sn
	P1 PH1	90 deg ^{119}Sn pulse
	D2	$1/(2J)$ ^{119}Sn - ^{29}Si
	(P2 PH1) (P4 PH7):D	180 deg ^{119}Sn and ^{29}Si
	D2	
	P1 PH9	
	D4	bird recovery delay
	P3:D PH10	90 deg ^{29}Si pulse
	P1 PH1	
	D2	
	P3:D PH3	
	D0	t1/2
	P2 PH2	180 deg ^{119}Sn decoupling pulse
	D0	
	P3:D PH4	
	D2	$1/(2J)$ ^{119}Sn - ^{29}Si refocussing delay
	GO=2 PH5	acquisition of ^{119}Sn FID
	WR #1	
	IF #1	
	IP3	
	IN=1	
	EXIT	

PH1= 0

PH2= 0

PH3= 0 2

PH4= 0 0 2 2

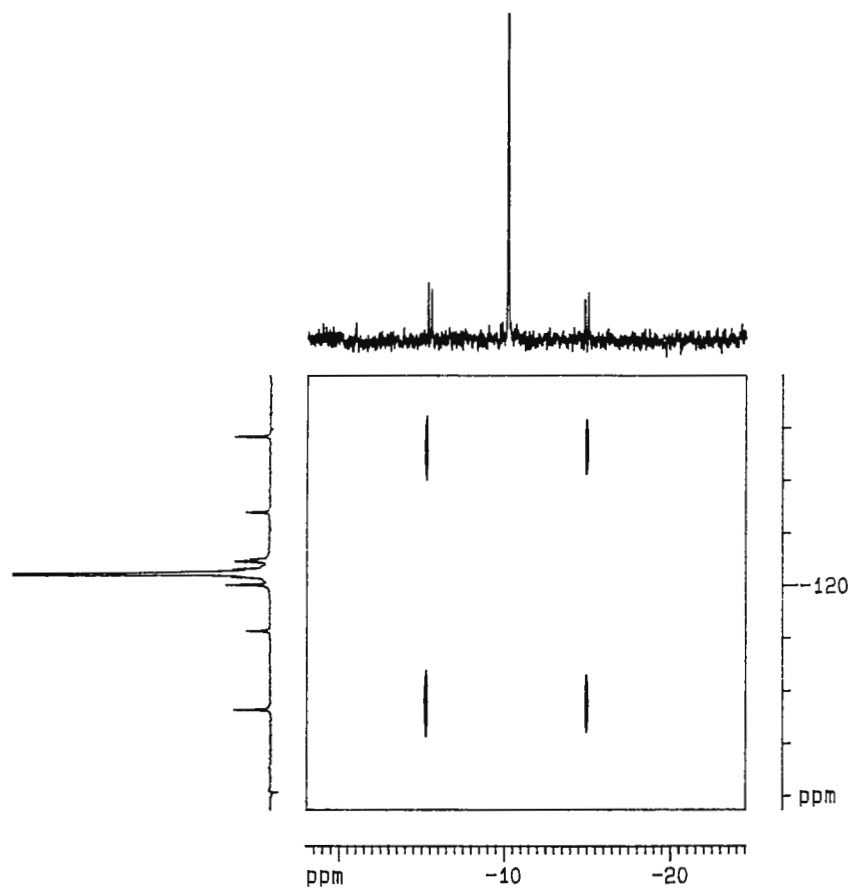
PH7= 0

PH9= 2

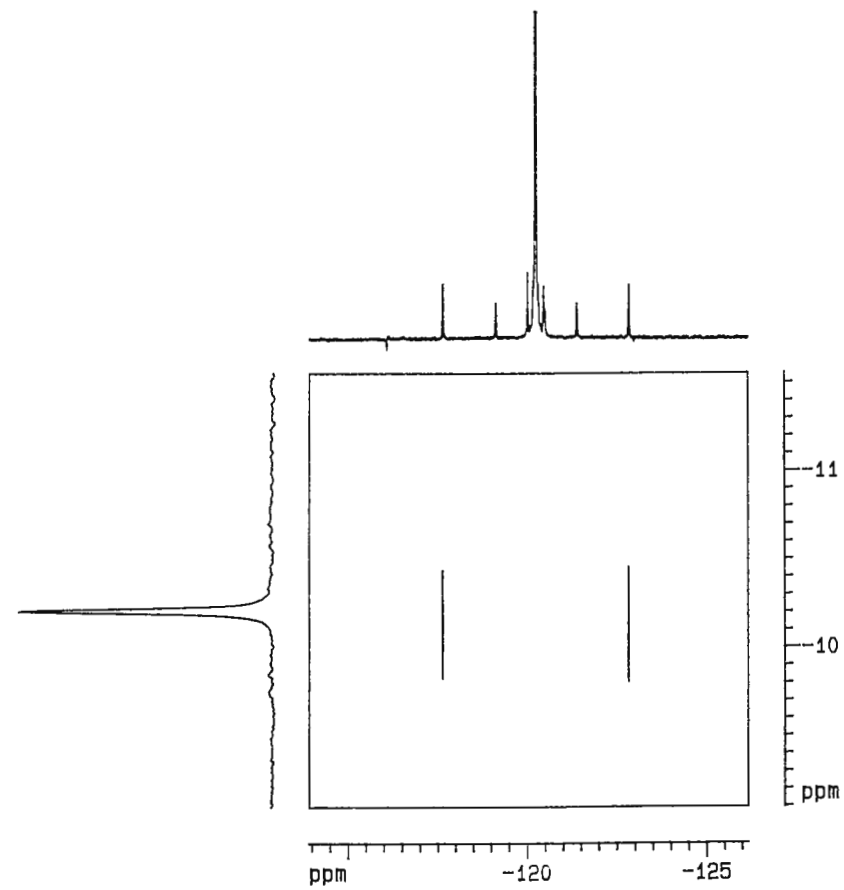
PH10= 0 0 0 0 2 2 2 2

PH5= R0 R2 R2 R0

heterocosity ^{29}Si - ^{119}Sn , TMSTBS, ac300



*"inverse" ^{119}Sn - ^{29}Si correlation
using bird sequence, TMSTBS, ac300*





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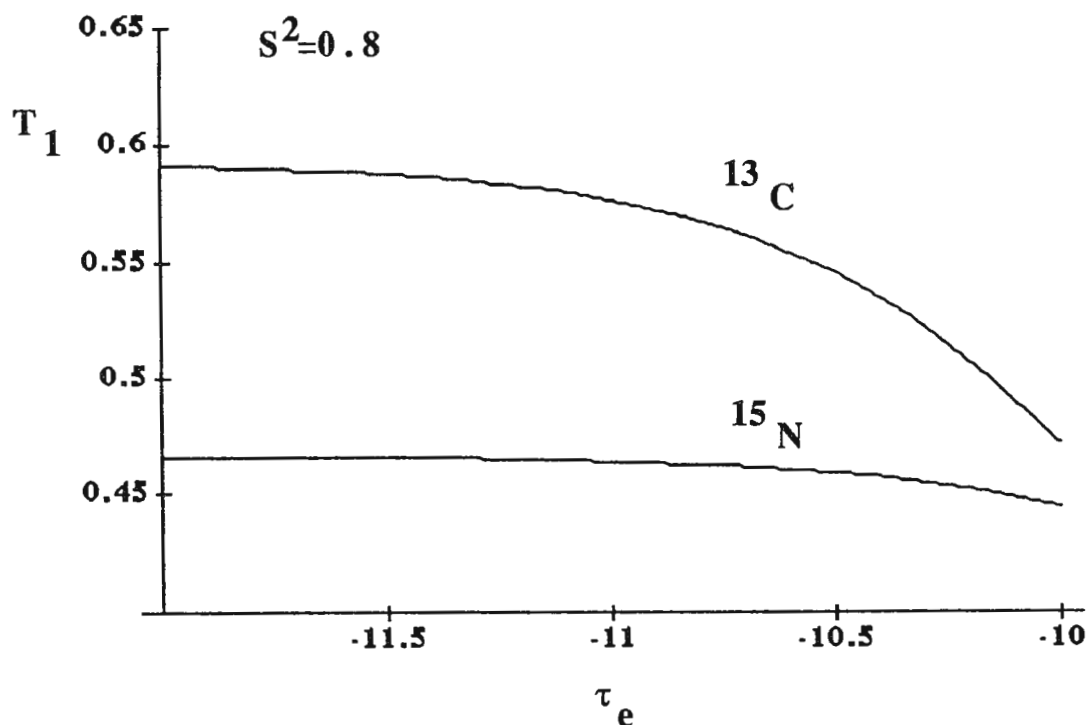
DEPARTMENT OF CHEMISTRY

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

Wednesday, December 5, 1990
(received 12/10/90)

Dear Barry:

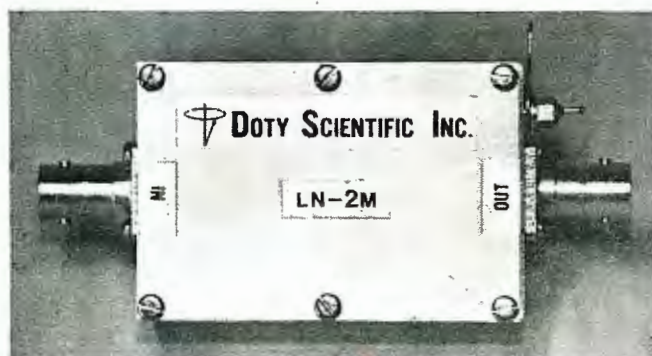
We have recently been examining the relaxation parameters of ^{13}C labeled Staphylococcal nuclease to investigate the dynamical effects of ligand binding, amino acid replacements and Ω loop deletion. When we compared our results to the previously reported results on the amide protons (L. E. Kay, D. A. Torchia and A. Bax, *Biochemistry*, **28**, 8972 (1989)) it was surprising how different the picture is when the $\text{C}\alpha$, $\text{C}\beta$, are examined rather than the amide. Calculations show that the ^{13}C relaxation is much more sensitive to internal motion (τ_e) than is ^{15}N . A typical plot is shown below for the case of 400 MHz and an order parameter of 0.8. We are in the process of writing this up in more detail.



Sincerely,
Philip Bolton
Philip Bolton

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Gain, ± 2 dB	31 dB
Output Power, 1 dB Gain Compression	17 dBm
Input Impedance, Small Signal	150 Ω + 10 pF
Input Impedance, for 100 W Pulse, approx.	0.2 Ω + 6 nH
Output VSWR, 50 Ω	2.0
Third Order Intercept	30 dBm
Power Supply Requirements	14-29 VDC, 100 mA
Input Protection from 50 Ω Source	1 kW, 5 μ s 5% duty cycle or 100 W CW

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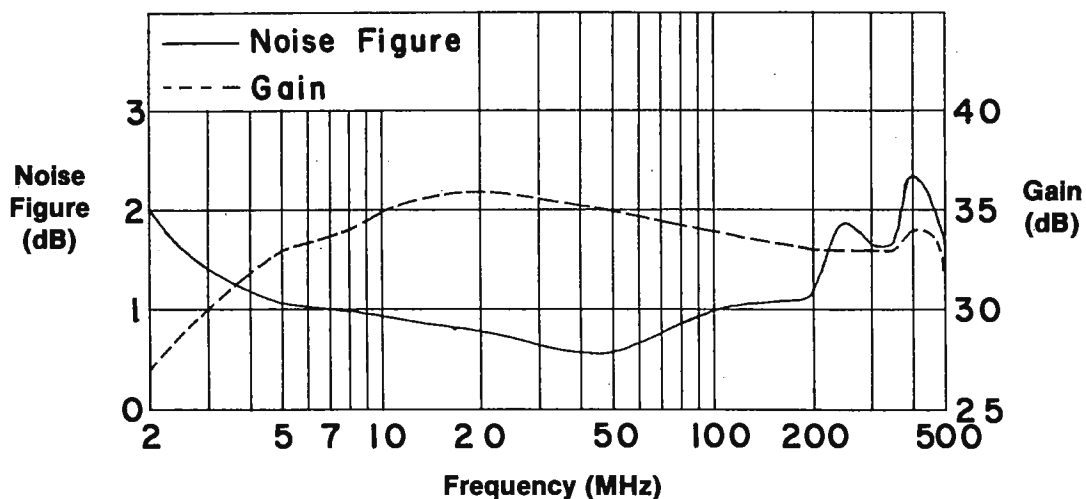
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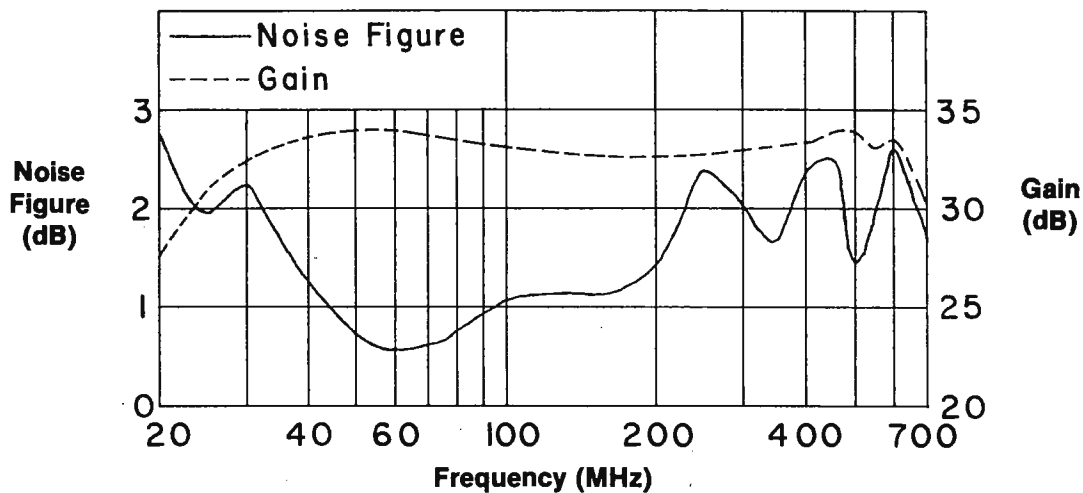
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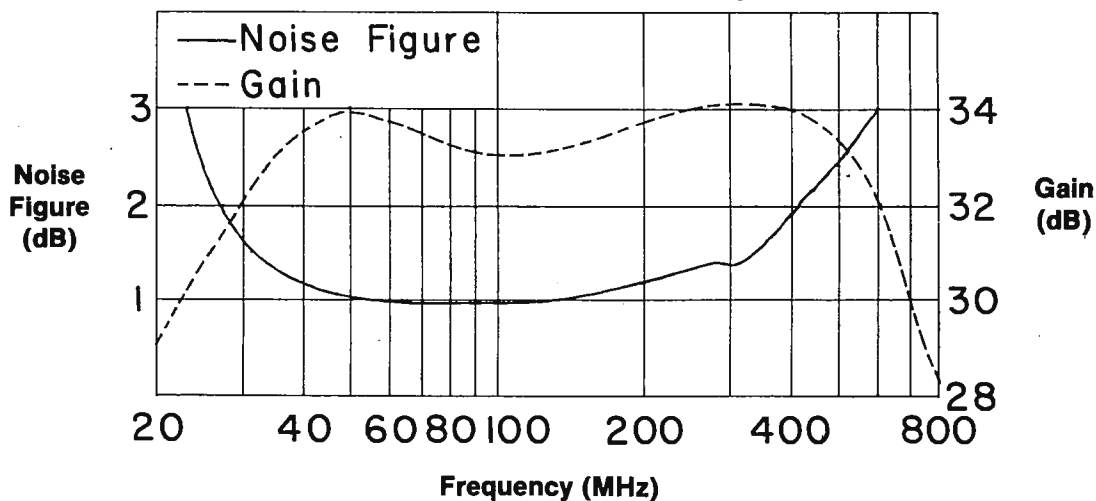
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LN-2H — .3 μ s recovery



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

(received 12/5/90)

Ihre Nachricht vom / Zeichen

Bei Beantwortung bitte angeben

Telefon-Durchwahl

JÜLICH

(02461) 61 3969

Oct. 31, 1990

A problem with an in vivo internal concentration standard.

Dear Prof. Shapiro,

It is a pleasure to present my first contribution to the Newsletter. After initial problems I hope that everything will run smoothly from now on.

Since the beginning of this year, I'm involved in In Vivo NMR spectroscopy studies on microorganisms in a small research group at the Institute of Biotechnology of the Jülich Research Center in Germany. Our instrument is an AMX400-WB spectrometer from Bruker. The general goal of our research is to develop methods for accurate determination of metabolic fluxes in bacteria, and apply these to monitor the effects of genetic manipulations. The latter are carried out in our institute with the aim of improving - from a biotechnological point of view - the characteristics of certain bacterial strains.

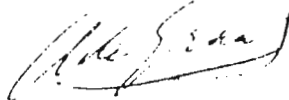
We are currently investigating the anaerobic organism *Zymomonas mobilis* by In Vivo ^{31}P NMR. This organism ferments glucose and/or fructose to ethanol in a very efficient way via the Entner-Doudoroff pathway [1]. In our experiments, we add 0.13 volume 50% glucose solution to a very dense cell suspension ($\sim 10^{11}$ cells/ml) and follow the resulting ^{31}P spectral changes in 10 consecutive 1 minute scans (60° pulses, 340 ms repetition time). The cultures immediately start heavy CO_2 gas formation. The spectra show a fast rise of sugar phosphates to a peak level at ~ 1 min, followed by a rapid decline to steady state levels that last from 3 to 6 minutes after glucose addition. During that period, high ATP levels are observed. Inorganic phosphate levels decrease simultaneously with the rise in sugar phosphates and recover only after ATP starts going down after ~ 6 minutes. A typical spectrum taken 5 min. after glucose addition is shown in Fig. 1.

We include triethylphosphate (TEP) as an internal concentration- and chemical shift reference. Upon proton decoupling, this non-titratable compound shows a sharp resonance 0.44 ppm downfield of 85% phosphoric acid [2]. Surprisingly, during the fermentation this peak behaves as depicted in Fig. 2. After an initial decrease, its area increases simultaneously with carbon dioxide gas production in the suspension, where at first sight one would expect a decrease due to sample dilution. The explanation for this phenomenon lies in the fact that the gas

formation induces stirring of the sample which acts to partially replace heavily saturated TEP magnetization inside the sensitive region of the RF coil by much less saturated magnetization coming from outside the coil region. Due to the relatively long T1 of TEP (we have measured a value of 8 sec) this results in a pronounced signal enhancement.

We plan to exploit the sensitivity increase and overcome this concentration referencing problem by using an air lift tubing system with adjustable forced flow as described in ref. [3].

Sincerely,



Albert A. de Graaf

- [1] Barrow et al., J. Biol. Chem. 259, 5711 (1984)
 [2] Kirk et al., J. Magn. Res. 70, 484 (1986)
 [3] Santos et al., J. Magn. Res. 68, 345 (1986)

Fig. 1

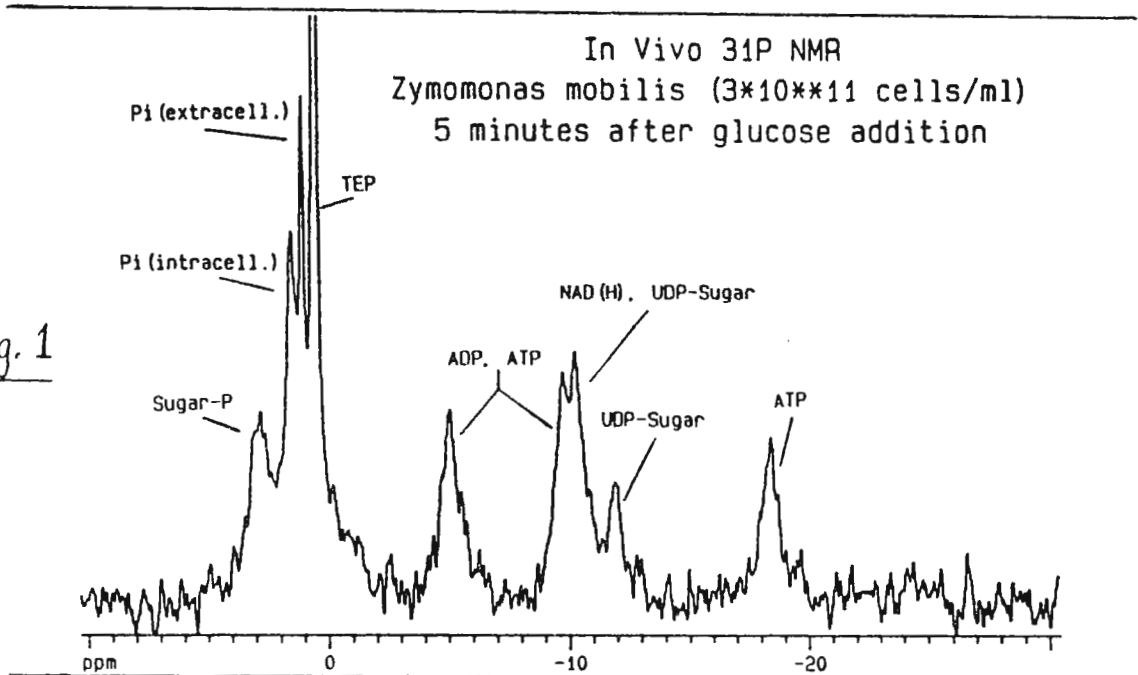
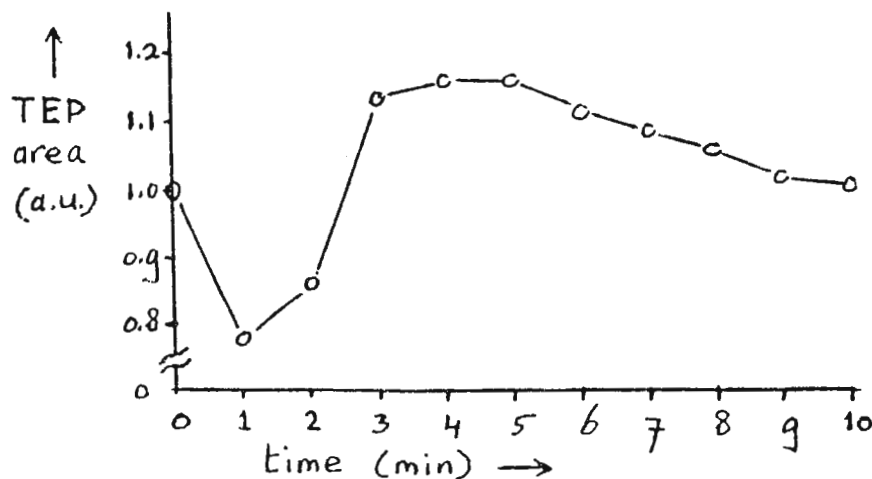


Fig. 2



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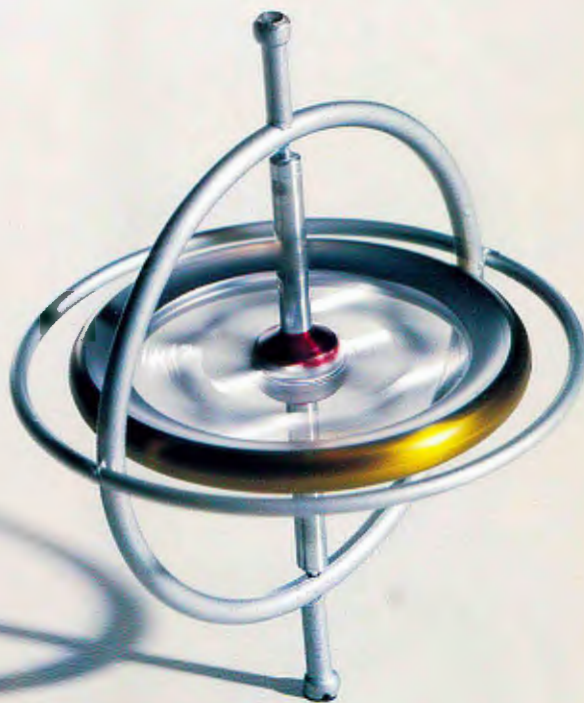


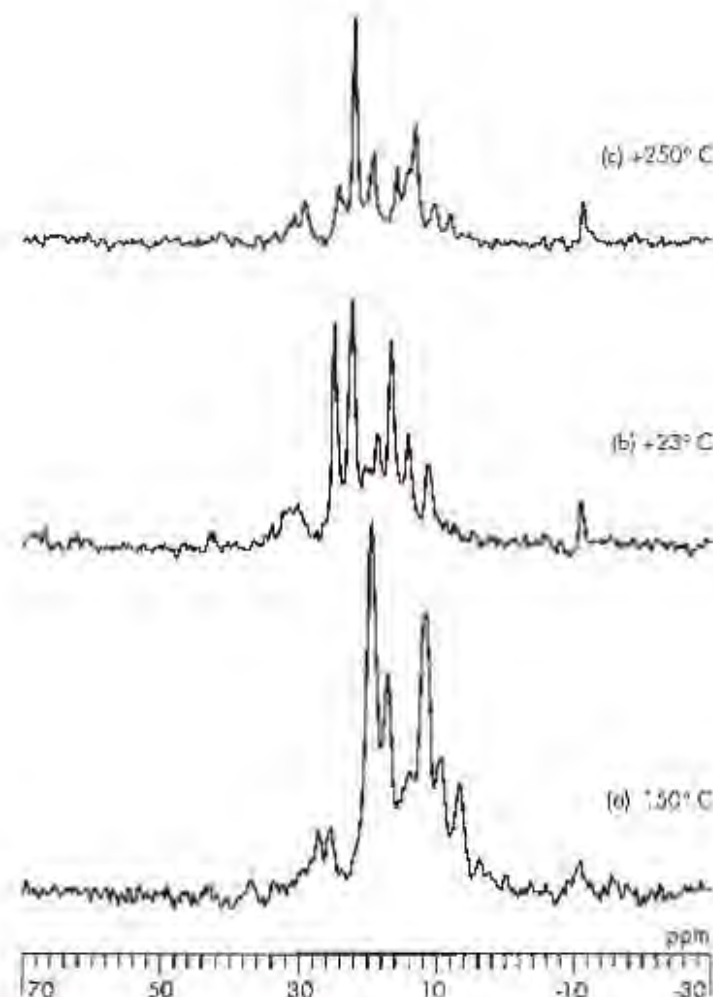
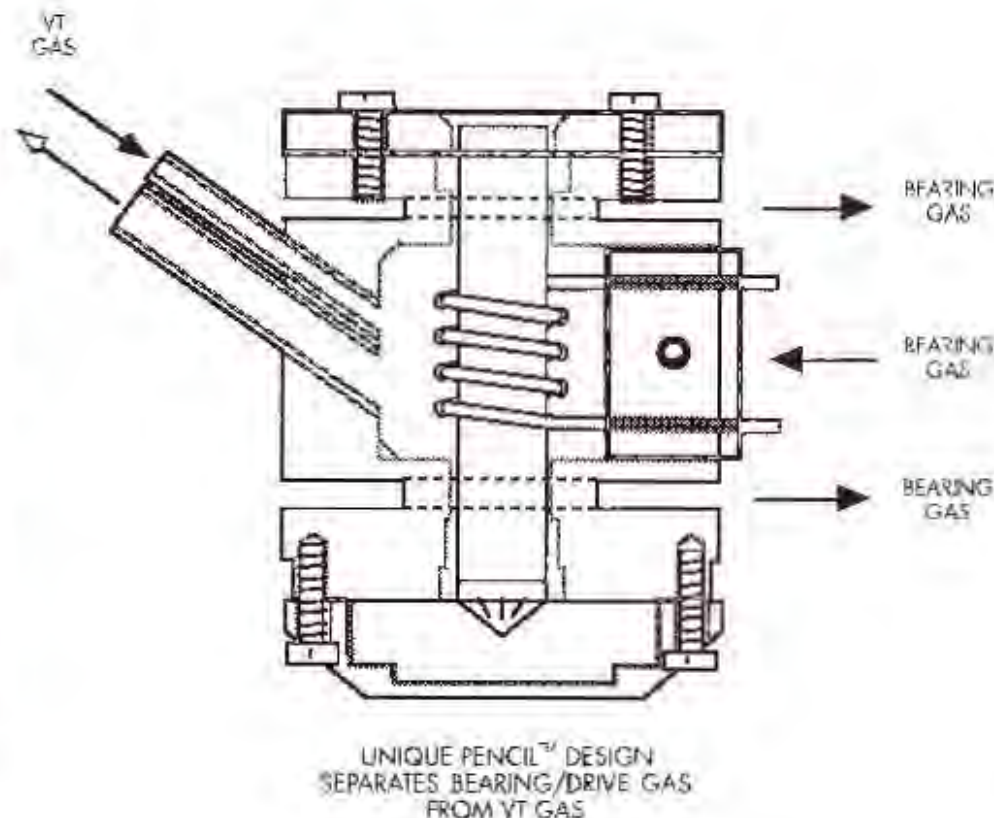
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Sealed-Sample ^{13}C MAS NMR of an Active Heterogeneous Catalyst

^{13}C MAS NMR is a very useful technique for characterizing the nature and structure of many solid organic systems. Anderson and Klinowski have applied MAS techniques to study the reactions of methanol in ZSM-5 [Natura, **339**, 200 (1989)]. Until recently, it has proved difficult to spin sealed samples at high and low temperatures rapidly and reliably with

the same probe. However, the design of the PENCIL™ CPMAS probe allows the spinning of a 7.5 mm rotor to 7 kHz over a temperature range of -150°C to $+250^\circ\text{C}$. This capability is illustrated here by spectra of the aliphatic region of a sample investigated by Anderson and Klinowski, spinning at 3300 Hz from -150°C to $+250^\circ\text{C}$.

These data were obtained under the following conditions:

Spectrometer: **CMX-300**
 Probe: 7.5 mm **PENCIL™ COAX™**
 Spinning Speed: 3300 Hz
 Temperature: (a) 150°C ; (b) $+23^\circ\text{C}$; (c) $+250^\circ\text{C}$
 Number of Acquisitions: (a) 200; (b) 300; (c) 1000
 Pulse Width (90°): 4 μsec
 Recycle Delay: (a) 10 sec; (b) 10 sec; (c) 2 sec
 Spectral Width: 45 kHz

Most critical parameter shown in **bold type**



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From: In Vivo NMR Research Center
National Institutes of Health

Date: 12/05/90 (received 12/7/90)

To: Dr. Bernard Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, Ca 94303

Proton spectroscopic imaging in humans at 1.5 T

Dear Dr. Shapiro,

Until recently, Positron Emission Tomography (PET) was the only technique that allows the clinician to obtain useful images with direct metabolic information. Of course, it was known that in vivo NMR spectroscopy is a promising alternative, because of its noninvasive character and its high information density. Single volume spectra have been obtained in vivo by many groups using ^{31}P , ^{13}C and recently, also ^1H NMR^{1,2,3}. However, since large variations in regional metabolism can be expected in many diseases, we prefer to use spectroscopic imaging with optimum resolution. Because of sensitivity reasons, we concentrate on ^1H spectroscopic imaging⁴.

In vivo proton NMR is hampered by the need to drastically suppress the water and the fat resonances. Another severe problem is that the RF coils for in vivo NMR are generally large, e.g. the regular quadrature head coils are cylindrically shaped with a length of at least 20 cm. Of course, it is impossible to obtain an excellent homogeneity over the whole sensitive volume of the coil, because of susceptibility effects and limited homogeneity of the magnet. For proton spectroscopic imaging, a large volume of interest is first selected in a single scan⁴. This is achieved using a double echo sequence:

$90 - \tau_1 - 180 - \tau_1 - \tau_2 - 180 - \tau_2$

where the three RF pulses are slice selective in the presence of mutually orthogonal gradients, thus selecting a volume of interest. A frequency selective pulse on the water resonance is used followed by a gradient dephasing pulse to suppress water⁵. Some other gradients are placed in the sequence to ensure that unwanted echoes or FIDs are completely suppressed⁵. RF phase cycling is not needed. Then phase encoding is added in two (or sometimes three) directions. The whole procedure of obtaining conventional images, choosing a region of interest in the brain (of the order of 120x120x20mm), shimming, water suppression, and data acquisition takes about 60-80 minutes, with the data acquisition part 34 minutes (32x32 phase encode steps, repetition time 2 s). The final data file (32x32x1024 data points) is processed using in house developed software. Voxel size is about 1- 1.5 ml.

The accuracy of these methods can be appreciated in the figure, showing on the left a conventional image of a patient with a recurrent glioma. On the right is a spectroscopic image obtained with a total echo time of 272 ms (when the lactate doublet is in-phase), representing the spectral intensity from 1.2-1.4 ppm representing lactate and/or fat. Since normal brain tissue does not show visible fat resonances at these echo-times, the tumor (or cystic part) is very well visualized in the spectroscopic image. Note the two moon shapes in the spectroscopic image

corresponding with the fat in the skull region. We found that it is not necessary to precisely select a region of interest well within the brain, especially in the left/right direction.

The pulse sequences were designed in collaboration with Joe Gillen from the Pittsburgh NMR Institute.

We hope that, with such techniques, the information of Magnetic Resonance Imaging can be enhanced from purely anatomical information to biochemical or functional imaging⁶.

Sincerely Yours,

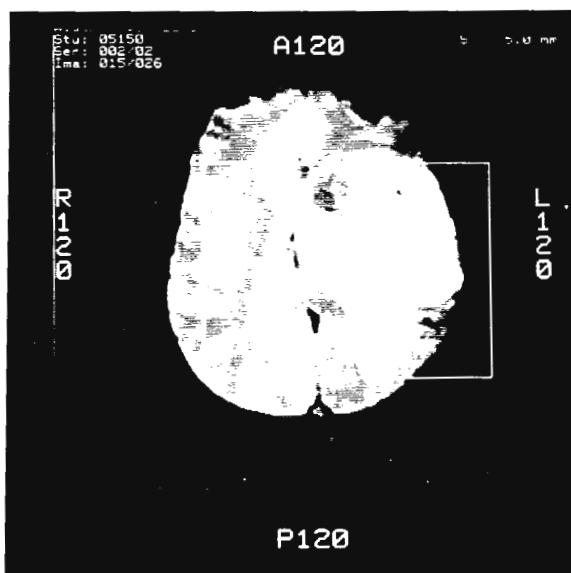
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Chrit Moonen Peter van Zijl

 Jeffrey Alger

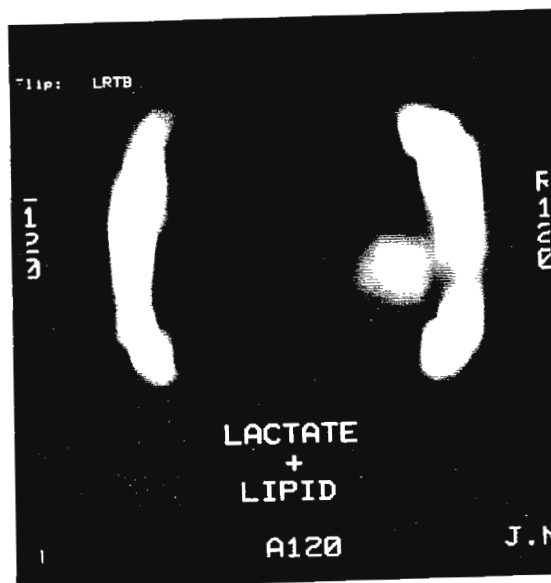
 Joseph Frank

 Geoffrey Sobering

- 1) G.K. Radda, Science 233, 640 (1986) 2) J.W. Prichard and R.G. Shulman, Ann. Rev. NeuroSci. 9, 61 (1986) 3) J. Frahm et al. Magn. Reson. Med. 9, 79 (1989); J.R. Alger et al. Radiology, 177, 633 (1990) 4) P.R. Luyten et al., Radiology, 176, 791 (1990) 5) C.T.W. Moonen and P.C.M. van Zijl, J. Magn. Reson. 88, 28 (1990) 6) C.T.W. Moonen et al. Science, 250, 53 (1990)



Conventional image with rectangle indicating region of interest



Proton spectroscopic image showing intensity between 1.2-1.4ppm

When faced with a tough analytical problem . . .

QUESTION

How do you find all the thiophenol derivatives matching YOUR CARBON SPECTRUM?

ANSWER

Sadtler Structure Assignment Library

STEP:1

Search For
Thiophenol Derivatives.

Structure Search Version 1.0
File Draw Search View Print Library Utility
CAS Registry Search
Exact Match Search
Sub-structure Search
Source Structure: Unknown Structure
Number of Hits: 128

Index	NQ1	Lib	Entry	Name
1	1	13C1	70	PHENYL DISULFIDE
2	1	13C1	98	BENZENESULFONYL CHLORIDE
3	1	13C1	207	BENZENESULFONYL FLUORIDE
4	1	13C1	208	BENZENESULFONYL FLUORIDE, -
5	1	13C1	227	P-TOLUENESULFONIC ACID, METHYL ESTER
6	1	13C1	355	P-TOLYL DISULFIDE
7	1	13C1	732	METHANOL, /P-TOLYLSULFONYL/-
8	1	13C1	982	BENZENESULFONIC ACID, HYDRATE
9	1	13C1	983	BENZENESULFONIC ACID, SODIUM SALT
10	1	13C1	987	P-TOLUENESULFONIC ACID, BUTYL ESTER
11	1	13C1	988	BENZENESULFONAMIDE
12	1	13C1	918	P-TOLUENESULFONAMIDE, N-BUTYL-
13	1	13C1	974	ACETIC ACID, /PHENYLTHIO/-
14	1	13C1	997	P-TOLUENESULFONYL CHLORIDE
15	1	13C1	1037	P-TOLUENESULFONAMIDE, N-METHYL-
16	1	13C1	1129	ANILINE, O-METHYLTHIO-
17	1	13C1	1178	SULFIDE, PHENYL 3-PHENYLPROPYL-
18	1	13C1	1296	SULFIDE, P-BROMOPHENYL METHYL-
19	1	13C1	1383	SULFIDE, METHYL N-NITROPHENYL-
20	1	13C1	1384	SULFIDE, METHYL N-NITROPHENYL-

STEP 2:

Match Those Derivatives
With Your Spectrum.

NMR Search 2.12
File Search View Library Print Utility Import Page
Unknown Entry
PEAK SEARCH PARAMETERS
Peak Tolerance: 0.3 ppm [value between 0.1 and 2.0]
Minimum No. of Peak Match: 4
Algorithm:
☐ Forward Search
☒ Reverse Search
Cancel
OK
READY

YOUR SEARCH IS OVER!

NMR Search 2.12
File Search View Library Print Utility Import Page
LIB PEAK LOCATION
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Sadtler ID: 8771
=== First Peak Table ===
(Total of peaks: 5)
No. Location Intensity
1 15.60 4
2 124.90 5
3 126.70 9
4 129.70 9
5 138.60 1
Source Library: 13C1 - C-13 NMR STANDARDS
<<Unknown Buffer>>
File Name: THIDEMO.NPF
Chem. Name: ODIFEROUS SPECIES
CAS Registry Number: unknown
Comment: process shutdown 12/9/89, green
READY

STRUCTURE
Print View
CS(=O)(=O)c1ccccc1

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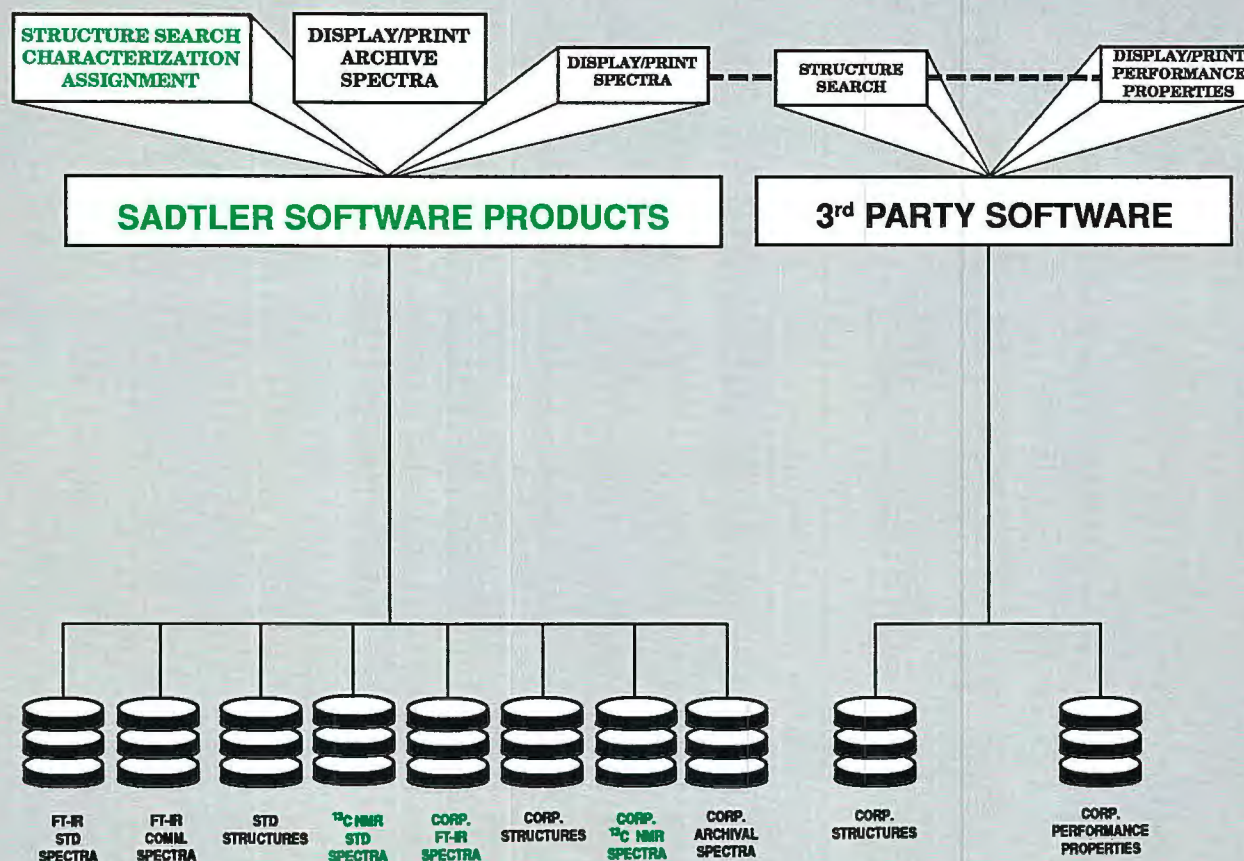
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Sadtler ^{13}C NMR Molecular Structure Library
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NMR AND DATA PROCESSING LABORATORY

.....

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

11/27/1990
(received 12/7/90)

Dear Dr. Shapiro,

Improved sensitivity for "twisted" DQ-COSY/INADEQUATE spectra

Processing DQ-COSY type homonuclear correlations 90° out of phase in F2, as it was recently proposed [Pelczer, I., Bishop, K. D., Levy, G. C., and Borer, P. N., *J. Magn. Reson.*, in press], leads to absorption-like cross peaks in many cases (Fig. 1). The cancellation of antiphase cross-peak contributions, well known for pure-phase or multiple-quantum filtered COSY spectra, is less severe for DQ correlation peaks, as those have in-phase character in one dimension (F1). If the above mentioned "twist" is applied during processing, signal-to-noise is improved when active coupling and the effective linewidth are comparable. This relative gain is even more expressed with decreasing ratio of the active coupling and the effective linewidth, as it is shown for calculated spectra in Table 1. Beside the improved sensitivity, this modification of processing results in not only an easier to handle spectrum, but also provides access to routine symmetrizations and application of reconstruction methods, such as MLM. A practical example is shown in Figure 2, where a subregion of the DQ-COSY spectrum of the small protein lysozyme is shown. Difference in results of conventional (Fig. 2a) and "twisted" processing (Fig. 2b) can be followed. Contours were set at the same levels for both spectra.

Sincerely yours,

(István Pelczer)

(George C. Levy)

Figure 1.



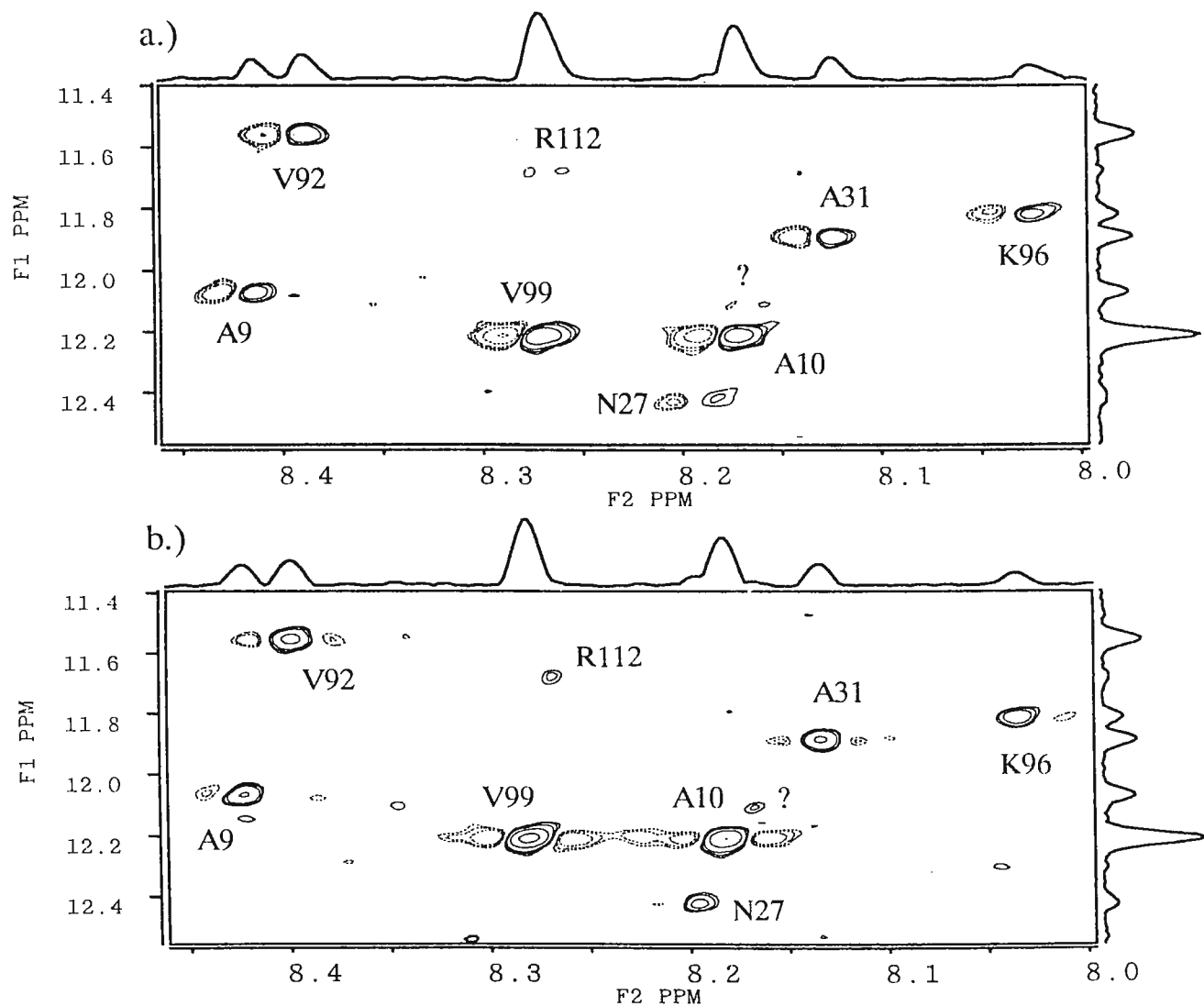
Phase the spectrum 90° out of phase in F2: a "twist" on DQ-COSY.

Table 1
COMPARISON OF RELATIVE INTENSITIES FOR SYNTHESIZED AX
DQ-COSY AND DQF-COSY CROSS-PEAKS

linewidth/J	DQ-COSY	DQF-COSY	DQ-COSY	DQF-COSY
1	100.00	70.83	143.70	146.26
3	6.49	2.51	10.00	5.96
5	1.53	0.38	2.33	0.89

Spectra were synthesized with 10 Hz line separation (coupling) and 3.9 Hz/point digital resolution. Maximum intensities were normalized to the height of the conventionally phased DQ-COSY cross peak.

Figure 2. Subregion of the DQ-COSY spectrum of lysozyme in D₂O, showing NH- $\{\alpha\text{CH}\}$ correlations. Assignments were done following Redfield and Dobson's data [*Biochemistry* 27(1988)122]. The subspectra shown were selectively processed using SDFT [TAMU N°382(1990)47, and Szalma, S., Pelczer, I., Borer, P. N., and Levy, G. C., *J. Magn. Reson.*, in press]. Negative contour levels are plotted with dashed lines.



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(To 220MHz)	0-600W	0-200W	
Gain (typ.)	65dB	60dB	
Gain Flatness	± 4 dB	± 3 dB	
Input/Output Impedance	50 ohms		
Input VSWR	< 2:1		
Pulse Width	20mS		
Duty Cycle	Up to 10%		
Amplitude Rise/Fall Time	200nS typ.	150nS typ.	
Amplitude Droop	5% to 10mS typ; 7% max		
Phase Change/Power Output	10 $^{\circ}$ to rated power typ.		
Phase Error Overpulse	4 $^{\circ}$ to 10mS duration typ.		
Noise Figure	11dB typ.	8dB typ.	
Output Noise (blanked)	< 20dB over thermal		
Blanking Delay	< 5uS on, 2uS off, TTL signal		
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	2. Input overdrive- up to +10dBm		
	3. Over duty cycle/pulse width		
	4. Over temperature		
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	3. Blanking- BNC (F)		
	4. Interface- 25pin D(F), EMI filtered		
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	2. Over temperature	6. Overdrive	
	3. Over duty cycle		
	4. Over pulse width		
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	2. DC power supply fault		
	3. Over duty cycle		
	4. Over pulse width		
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	2. Pulse width		
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Dr. B.L. Shapiro
TAMU Newsletter
950 Elsinore Court
Palo Alto, CA 94303
U.S.A.

Ha/66/90

(received 11/21/90)
31.10.90Splitting of Inorganic Phosphate Signal During Exercise.

Dear Prof. Shapiro,

the whole body solenoids enable the measurement of in vivo ^{31}P MR spectra of human skeletal muscle during exercise. During the exercise the most interesting changes in the spectrum are the increasing of inorganic phosphate signal intensities (Pi) and changes of its chemical shifts. Changes of chemical shift of Pi is connected with the change of intracellular pH.

In one case (healthy volunteer) we observed splitting of Pi signal during exercise and in rest immediately after exercise. Before exercise and 10 minutes after resp., we observed only one signal of Pi which of chemical shift corresponds to physiological pH value of about 7.1. Splitting of the signal is documented on figures enclosed. The difference of chemical shifts of Pi signals is in the range from 0.47 to 1.05 ppm and pH value calculated is from 6.2 to 7.24. The spectra were measured in one minute interval. The splitting of signals is time dependent. We have no definitive explanation of the effect, one hypothesis may be that signals are from different groups of muscles. But the protocol of the measurement was the same as in other cases, where we did not observed such splitting.

May be somebody has similar experience, if yes, we would welcome all comments.

Sincerely

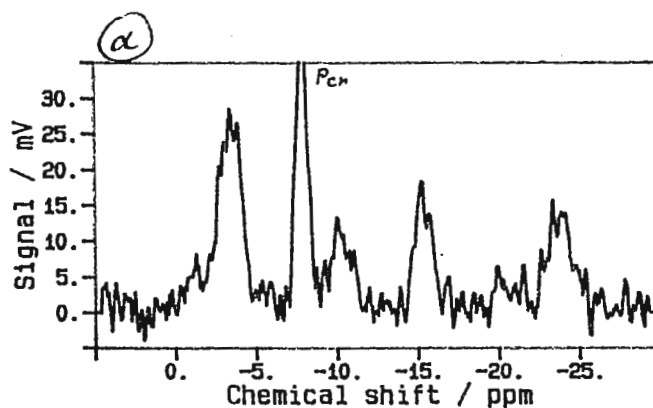
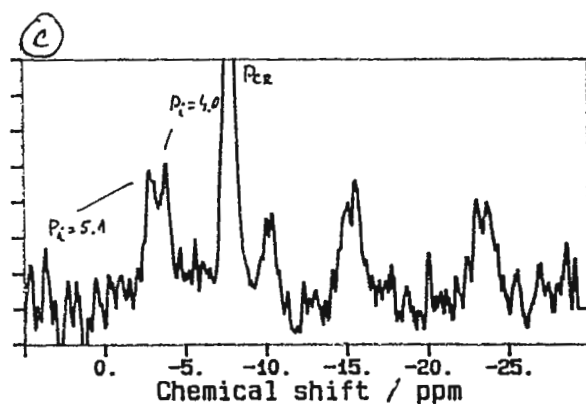
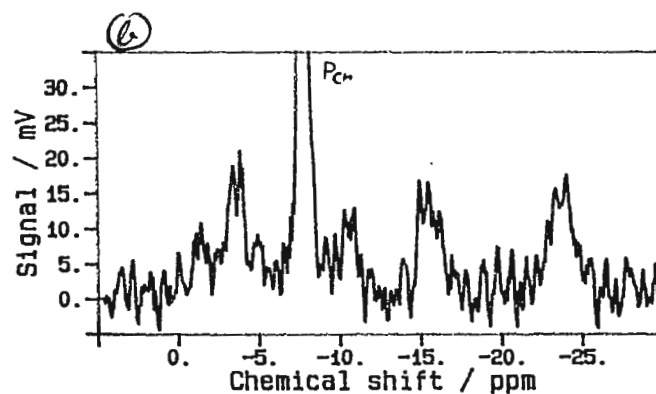
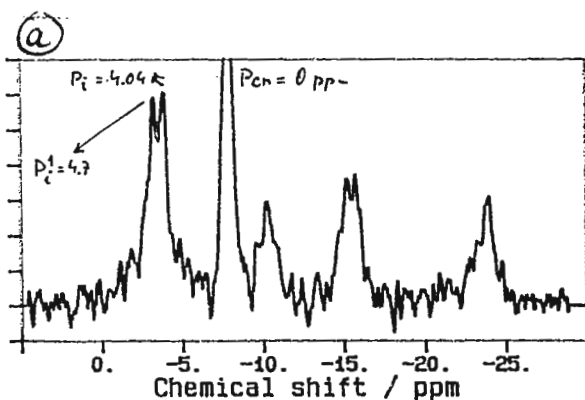
Milán Hájek
Milan Hájek
Alena Horská

Institute for Clinical and Experimental
Medicine, Prague, Czechoslovakia

Figure 1

The spectra were measured at 1.5 T Magnetom Imager with 8 cm surface coil positioned below the center of the calf. Home made ergometer was used. 32 acquisition was accumulated in one minute interval. The protocol of the experiment was: 5 minutes of exercises and 4 minutes of rest, cycle with increased workload repeated 3 times.

a) spectrum measured during 5th minute of 1st exercise, b) spectrum two minutes after 1st exercise, c) spectrum during 1st minute of 2nd exercise, d) spectrum measured during 5th minute of 2nd exercise.





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

November 26, 1990
(received 11/29/90)

Re: "Fun and Games with Surfaces"

Dear Barry:

The red dot is effective...I wondered what that code meant...now I know. In a few paragraphs I would like to summarize some of our recent solid-state nmr experiments on surface systems. First, is some work done by Yu-Hsing Chin concerning the carbon-carbon bond length in ethylene when it is bound to supported silver, i.e. in this case $\text{Ag}/\gamma\text{-Al}_2\text{O}_3$. He noted that at room temperature the ^{13}C CP/MAS nmr of the adsorbate gave, essentially, an isotropic spectrum. However, at low temperatures, $\sim -50^\circ\text{C}$ he obtained sidebands from what appeared to be an axially symmetric shielding tensor. When the temperature is lowered to approximately 100 K, one gets essentially a rigid lattice powder spectrum for the adsorbed ethylene. After a detailed line shape analysis of the mono-labelled ethylene (no homonuclear dipolar interaction) with the di-labelled material (with the homonuclear dipolar interaction) we arrived at a value for r_{cc} of $1.396 \pm 0.004 \text{ \AA}$. Further, the dipole and shielding principal axis systems are the same as in ethylene itself, i.e. oriented 90° with respect to one another. A paper describing this work has been submitted to JACS.

A second piece of work involves what I refer to as the O'Reilly paradox. O'Reilly¹ described some intriguing experiments in 1960 concerning the loss in ^{27}Al S/N as a function surface area for a series of transition aluminas. He concluded that as a result of such losses that the ^{27}Al nmr of the surface aluminum atoms would be impossible. This result (the experimental observation of which is absolutely correct) is at odds with some surface selective CP/MAS ^{27}Al experiments² we have reported on a few years ago. We have found that the origin of this loss is not due to a static electric field gradient at the surface, but due to dynamic processes at the surface. The dynamic processes are water motion (if the surface has not been dried) or proton motion. We have confirmed this by variable temperature experiments. Further we have calculated, via ab initio MO calculations, the electric field gradient tensor for a series of aluminum/oxygen cluster compounds. The calculations

support the idea that proton diffusion (or hopping) at the surface can modulate the aluminum electric field gradient by causing it to flip its sign in going from one state to another. Further, dynamic line shape theory for these systems demonstrates how such losses can occur and why the CP/MAS experiment still works. The experimental work for this project has been carried out by Mrs Beth Huggins. This work is being written up and will be submitted for publication shortly.

If any readers are interested in these projects they can write to me for preprints (when the papers have been accepted for publication). Let me take this opportunity to wish you and your wife the best during this up and coming holiday season and a most happy new year.

Sincerely,



Paul D. Ellis
George H. Bunch, Sr.
Professor of Chemistry
FAX: (803)777-7820
BITNET: N130006@UNIVSCVM

References

1. D.E. O'Reilly, *Advances in Catalysis*, 12, 31-116(1960).
2. H.D. Morris and P.D. Ellis, *J. Amer. Chem. Soc.*, 111, 6045-6049(1989).

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

P.O. Box 138
Medical Sciences Building
University Road
LEICESTER LE1 9HN

Telephone:
Direct Line: (0533) 52 3055
Switchboard: (0533) 522522

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

12th November 1990
(received 11/16/90)

Dear Prof. Shapiro,

Phase Sensitive Zero-Quantum Coherence Spectrum with Alternative TPPI Method

Zero quantum coherence (ZQC) spectrum has an attractive characteristic in that it has no evolution sensitivity to inhomogeneity of the static field, thereby giving rise to narrow linewidth along the F_1 dimension (that is the ZQC frequency dimension). Unfortunately, two problems have hampered the wide application of ZQC,s. Firstly, the ZQC cannot be separated from longitudinal magnetization, resulting in intense axial peaks although this problem has in part been solved by Cavanagh and Keeler [1] who suggested using a double-quantum filter. Secondly, it is very difficult to obtain a phase-sensitive absorption mode spectrum. We suggest in this letter an easy method to overcome the second problem.

The TPPI method is widely used to generate phase-sensitive 2D spectra. The phase of the coherence in t_1 is shifted by 90° for every t_1 increment; this is generally executed by shifting the phases of the pulses before the t_1 period. This method cannot, however, be applied in ZQC spectra since the ZQC is insensitive to the phase shift of pulses before ZQC evolution.

In reality, a phase shift of one pulse in a multiple pulse experiment simply changes the phase relationship between that particular pulse and the other pulses in the sequence. Therefore, phase sensitivity in ZQC experiments using the TPPI method can be achieved by shifting the phases of the mixing pulse(s) and receiver by 90° for every t_1 increment (instead of shifting the first two pulses). The result is a relative 90° shift of the ZQC. The figure shows a phase sensitive 2D ZQC spectrum of Gramicidin S (the N-H and alpha-H connectivities are as indicated).

The principles described here can also be applied to generate other phase-sensitive multiple quantum coherence data.

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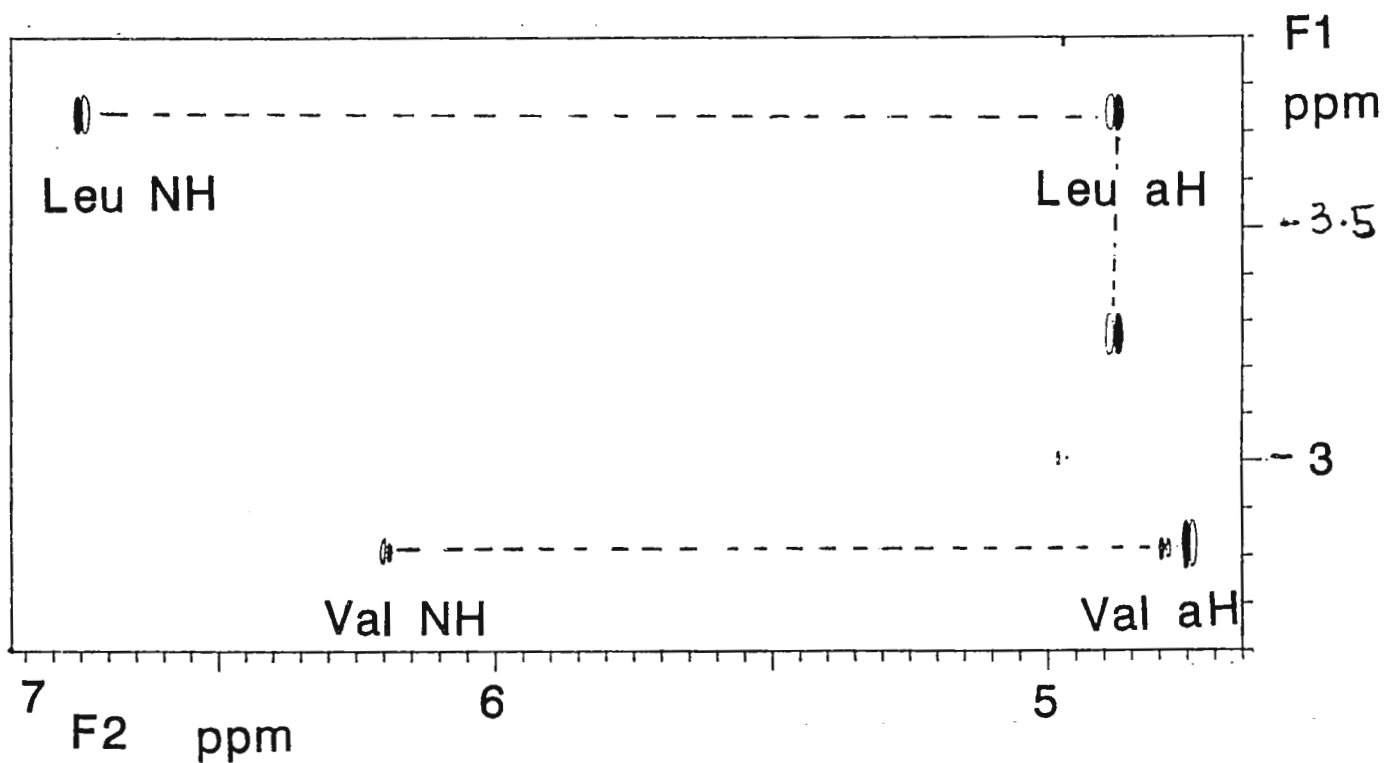
Yours sincerely,

Ji-Chun Yang

L.Y.Lian

[1] J.Cavanagh and J.Keeler, J. Magn. Reson., 1988, 77, 612-617.

Figure: Phase-sensitive ZQC 2D spectrum of Gramicidin S showing only the amide-alpha proton region



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Comparison and evaluation carried out at Harvard University by Scott Virgil on a Bruker AM-500 NMR unit, on November 30, 1988.

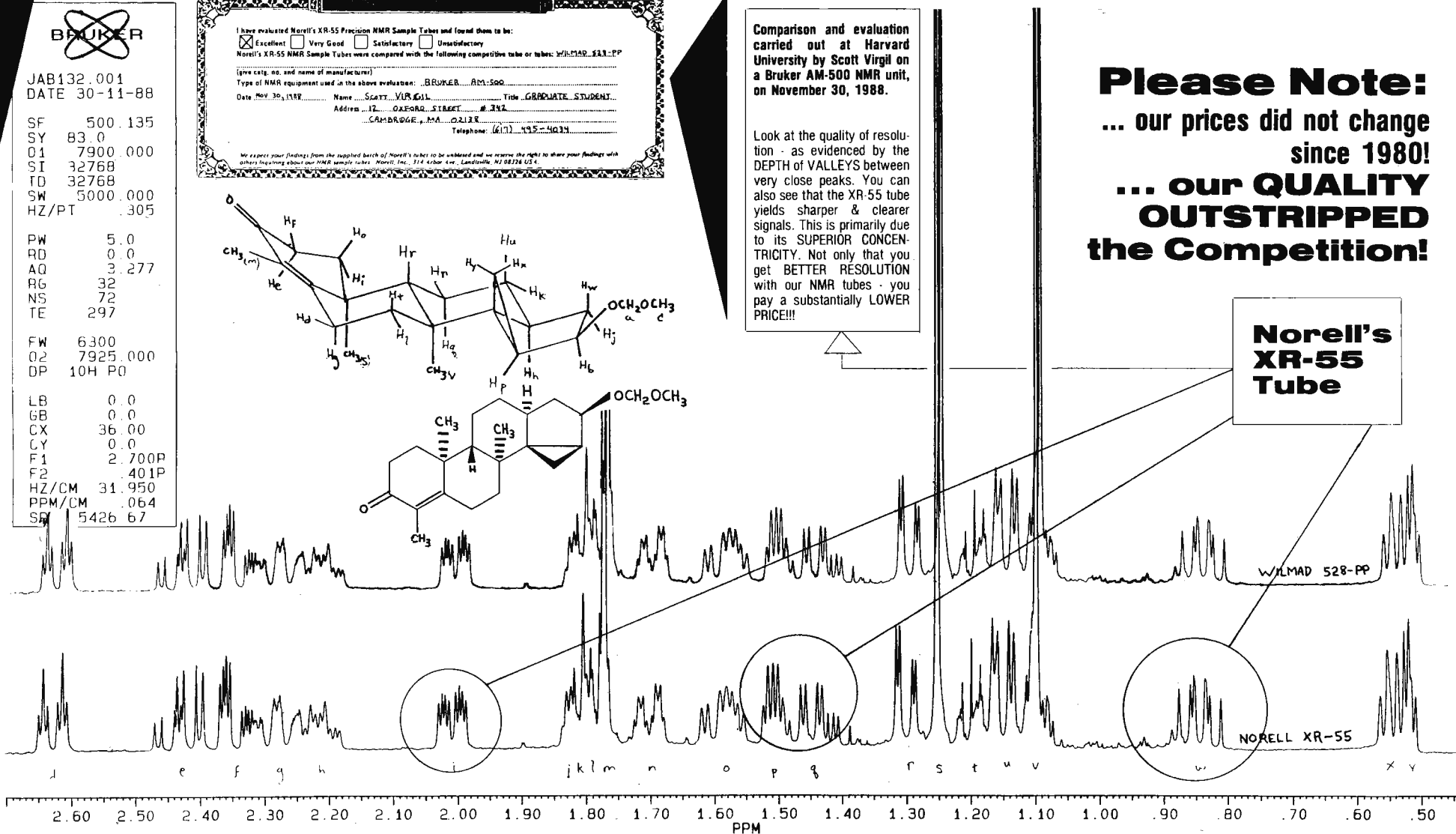
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LOVELACE MEDICAL FOUNDATION

MAGNETS AND PUMPS DON'T MIX: AN ADVENTURE TALE

(received 12/10/90)
6 December 1990

Dear Professor Shapiro,

An Oxford 1.9T/310mm horizontal superconducting magnet (#B26694) has been a workhorse for the past six years in our laboratory. On October 26th, the evening before the 18th (NMR)², New Mexico Regional NMR, meeting and shortly after receiving a "Reminder" from you, it vigorously ingested a 17-pound Cole-Parmer Masterflex peristaltic pump (Model # 7523-00). After glancing off the heavy end plate, the pump caught the shim/gradient coil and displaced it about 60 cm before coming to rest in the center of the magnet.

After the initial shock wore off, and after a sufficient number of people gathered to survey the situation, an exploratory effort was made to extract the pump by applying an axial force to the pump through a rope (yellow, polypropylene, 10 mm diameter). This effort was terminated after we moved the entire magnet 10 mm because we realized that there was a real danger of 1) pulling the magnet off the wooden pallet and 2) of displacing the solenoid with respect to the dewar.

At about this time, a serious thought was given to discharging the magnet in order to remove the offending pump but it was rejected on the grounds that 1) it was the sensible thing to do and 2) we never acquired the magnet power supply as we had hoped so there would be a significant delay before recharging the magnet. Therefore, we opted to take some risks, but not enough to cause any damage to the solenoid. At this stage, there were no overt damages to the magnet; coolant boiloff rates were normal and the magnet seemed to be working but probably with a less than optimal shim.

Another suggestion was to somehow separate the major components of the pump so they could be removed piece by piece. After some strenuous efforts at wielding a magnetic hacksaw blade and a magnetic screwdriver in the bore, we abandoned this effort.

Reasoning that a lot of harm could result from exerting forces between the solenoid and the dewar, we decided to try a scheme to let the solenoid push out the pump by pulling in lots of smaller ferromagnetic items that could be removed from the magnet individually after the pump was removed. After protecting the fiberglass bore tube with a 25.4 cm diameter PVC water pipe that enclosed the pump, we built a 16 cm long wooden piston that fit tightly in the PVC pipe and located it adjacent to the pump. We then confined a bundle of 16 120 cm long and 19 mm dia PVC water pipes to the center of the large PVC pipe and placed it next to the piston. Finally, in a scene similar to that of the operation of some early nuclear reactors, we inserted 5-foot long, 12 mm diameter steel "rebars" one-by-one into the 19 mm PVC pipes, starting at the center of the bore.

Initially, the process seemed to be working. However, after the pump had been displaced about 30 cm, it became impossible to keep the pump centered in the bore and we finally appreciated that the radial force on the pump was formidable when the pump was displaced from the uniform field region. As we were contemplating our next move, we noticed an alarming increase in the liquid helium boiloff rate. The steel rebars were extracted immediately to reposition the pump in the center of the magnet but it was decided to quench the magnet because we could not afford the wait to see if the boiloff would calm down before we ran low on liquid helium without any supply on hand. During the gentle quench, we used up less than 20 liters of liquid helium to end up with about 17 liters in the helium can. The pump was removed without much fanfare.

We blew out the coolants, warmed the magnet, and disassembled the dewar to discover that the various cans were reasonably centered so that just removing the pump and possibly also warming the dewar was probably sufficient to correct the enhanced boiloff. However, we were glad to make certain that the cans were centered and also discovered that the fiberglass rods that kept the helium can and the radiation shield centered in the nitrogen can were looking more like brooms than rods. We replaced those spacers and recentered all cans on reassembly. In addition, we inserted 15 cm by 100 cm multilayer pads of aluminized mylar superinsulation and applied them under the joints between the superinsulation in the vacuum space around the girth of the nitrogen can and the

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superinsulation pads applied on the annular end plates bolted to the nitrogen can.

We pumped out the vacuum space, precooled the magnet with liquid nitrogen, blew it out into the nitrogen can after two days, waited overnight to boiloff residual nitrogen (with appropriate pumping of the helium space), leak checked one last time, and finally cooled with liquid helium before powering back up. The only other fix we had to perform was to work on the output transistors for the z^2 and z^3 shims which blew when the shim/gradient coil was displaced by the pump.

Besides reporting that we have extricated ourselves from a brink of disaster, we wish to pass on some other good news. First is that for the second time in six years, we allowed a cleaning crew to clean and wax the floor while the magnet was down. The second is the fact that our helium consumption is back to where it was (we buy a 100 liter dewar of it every 6.5 to 7 weeks) and the nitrogen consumption has been reduced from 8% to to less than 6% per day, presumably because of the extra superinsulation.

Somehow this adventure reminds us of a quote attributed to G. L. Mallory "...because it is there."

We acknowledge the help of Jim Carolan of Nalorac, Dick Marsh of Oxford, and Neal Munro of Magnex. We also thank Clif Unkefer and Scott Ekberg of Los Alamos National Laboratory for the loan of an Alcatel leak detector and James Abbott of MIT and Los Alamos for overseeing its transportation. Finally, we thank Leon Axel for not only lecturing at the (NMR)² meeting but also for suggesting the title for the would-be picture (not available for this letter) of the magnet containing the pump.

Sincerely yours,

Steve Altobelli

Steve Altobelli,

A. Caprihan

Arvind Caprihan,

Eiichi Fukushima

Eiichi Fukushima,

PDMjor

Milt Icenogle, and Paul Majors.



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November 23, 1990
(received 11/29/90)

Comparison of solvent
suppression techniques

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

Dear Professor Shapiro:

We recently carried out pH titration studies on the peptidyl nucleoside antibiotic nikkomycin X, a potent chitin synthetase inhibitor produced by *Streptomyces tendae*. These studies yielded information on the conformation of the antibiotic in aqueous solution (1).

As a preliminary experiment for this work, ^1H -nmr spectra of dilute samples of nikkomycin X (≈ 2.5 mg/mL) were run in water and different techniques for suppressing the immense solvent peak were tried. All spectra were run at 25°C , and a closed capillary with D_2O was placed inside the nmr tube for signal lock. The top spectrum in Figure 1 was obtained without suppression. Obviously, the presence of the enormous water resonance peak introduced serious problems associated with the high dynamic range of the spectrum. The solvent resonance, with its first and higher order spinning sidebands, obscures most of the spectrum and severely distorts the baseline.

The bottom spectrum (Figure 1d) was obtained by irradiating the sample with the ^{133}I sequence proposed by Hore, which was designed to selectively excite all the protons but those of the solvent (2). In this way it was possible to obtain a higher than 250-fold suppression of the solvent signal and observe exchangeable protons, such as the amide NH (≈ 8.95 ppm) and the imidazolinone NH-3 (≈ 11 ppm). These resonances cannot be observed in D_2O solution (Figure 1b).

Attempts were also made to suppress the water peak by irradiating the sample with a selective low power saturating pulse prior to pulsing and observation (Figure 1c). This procedure is straightforward to implement, but runs the risk of saturation transfer by chemical exchange from the solvent to the solute spins. Indeed, the imidazolinone NH-3 resonance is not seen in spectrum c. Furthermore, in a similar run at pH 5.65 (data not shown) the amide NH was also saturated. In contrast, by using the ^{133}I sequence, this latter resonance could be observed at a pH of almost 6.0. The ^{133}I sequence thus proved superior to the saturating pulse technique, and was used for carrying out the titration studies.

The problems associated with the ^{133}I pulse technique were the following. First, the resonances within ≈ 0.6 ppm from the water peak could not be detected. This region includes both α -CHs and most of the sugar ring protons. Secondly, due to the limited spectral region that is excited, three independent runs were necessary in order to obtain a complete spectrum. Specifically, spectrum 1d is a

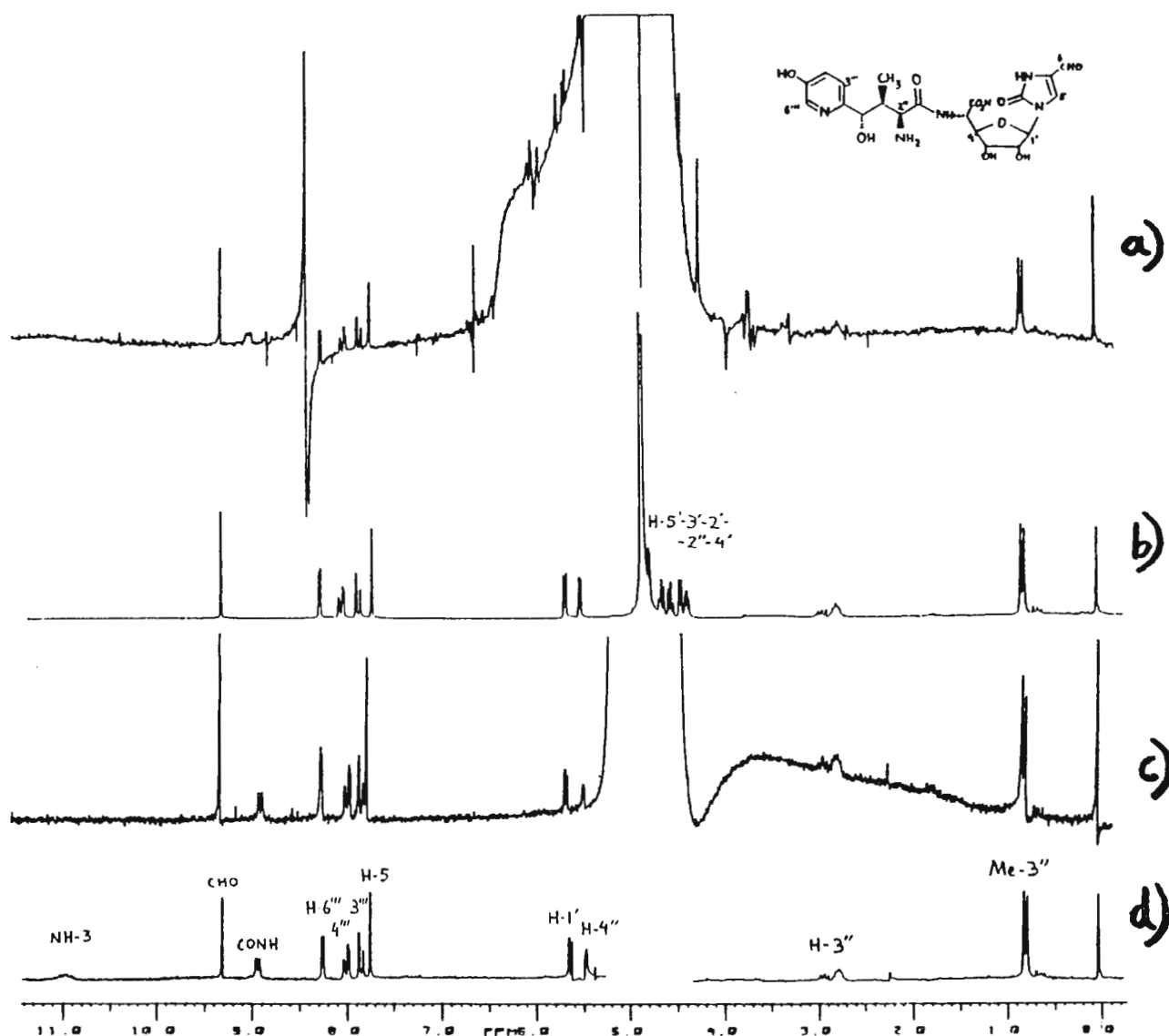


Figure 1 200 MHz ^1H -nmr spectrum of nikkomycin X, obtained
 a) in H_2O ; b) in D_2O ; c) in H_2O , using a presaturating
 low power 6 sec pulse. pH=3.48; d) in H_2O with the
 ^{133}I pulse sequence. pH=3.14. Reference: internal DSS.

composite of three separate spectra where the regions of interest were selectively excited by using three different sets of delays between pulses (0.7, 1.2 and 2.5 msec).

References:

1. E. Krainer, F. Naider, J. M. Becker. *Biopolymers*, **29**, 1297 (1990).
2. P. J. Hore. *J. Magn. Reson.* **55**, 283 (1983).

Sincerely,


 Eduardo Krainer


 Fred Naider

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Direct Dial

November 21, 1990
(received 12/3/90)

500 to FTNMR Revisited

Dear Barry:

In the April 1989 Newsletter we described the indirect process necessary to transfer data from our VXR-500S, through an XL-400, to our VAX-8800 for use with the FTNMR program. With the recent ability to implement DNI on the Sun 3/160 which controls our spectrometer, this pathway has become much less tortuous, and much faster. However, the conversion program we described, which puts the data in a format compatible with FTNMR, has been modified.

The fid portion of a data set (ex.- data.fid/fid) is transferred directly to the VAX over Ethernet using DNI. At this point, the file has a record length of 512 bytes, unlike the 133 byte length obtained using LimNet. Thus, there is no longer a need for the "PACKFID" portion of the program we described earlier.

The "VARIAN1D" portion of the program (which is obtained from the Sun version of FTNMR) still needs to be run. However, some initial byte swapping is required. The incoming data has successive bytes in the order: $a b c d$. They must be rearranged to the order: $b a d c$. Then VARIAN1D, in addition to other things, rearranges the order to: $d c b a$.

We have simplified things by mutating VARIAN1D so that it creates the correct ordering in a single step.

We thank our colleague Dr. Charles Cook, who did the programming.

Sincerely yours,

David Fry, David Greeley, Ross Pitcher, and Sudha Narasimhan
Department of Physical Chemistry



Department of Chemistry

First Announcement

Fifth Washington University- ENI/Emerson Electric Company Symposium on Nuclear Magnetic Resonance

Monday, May 20, 1991

A symposium on modern techniques in nuclear magnetic resonance will be held from 8:45 a.m. to 5:00 p.m., Monday, May 20, 1991 in Louderman Hall, Room 458, Department of Chemistry, Washington University, St. Louis, Missouri.

The symposium will feature invited papers by:

- * **Robert S. Balaban**
*National Heart, Lung and Blood Institute
Bethesda, MD USA*
- * **Ray Freeman**
*University of Cambridge
Cambridge, UK*
- * **Chien Ho**
*Carnegie-Mellon University
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Jeffrey A. Reimer, Associate Professor
DEPARTMENT OF CHEMICAL ENGINEERING
BERKELEY, CALIFORNIA 94720-9989
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"Be Careful with those Catalysts..."

Dear Barry:

Thu Dec 6 1990
(received 12/10/90)

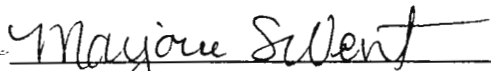
We would like to report some observations and make some suggestions regarding sample preparation for those NMR researchers that are studying adsorption on catalysts. It occurred to us that some of the TAMU readers may benefit from our observations.

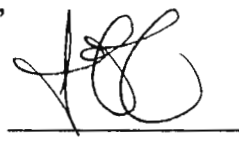
We have an *in-situ* probe containing a microreactor for studies of adsorbates on surfaces. The catalyst sample is loaded into the microreactor which is then attached to the probe with ultra-torr fittings. We take the probe to a vacuum rack where we can flow gases over the sample, or adsorb gases statically from a dosing volume, as the sample is being heated by an external heater. When attached to the vacuum rack the probe acts as a microreactor under conditions of plug flow, with the pelletized sample packed against a quartz frit. When we are ready for NMR we typically evacuate the microreactor with a diffusion pump, back-fill with helium, and seal the probe from the atmosphere.

We are currently studying ammonia adsorption and decomposition on supported vanadium oxide catalysts. In these systems the oxidation state of the vanadium determines the color of the sample: it is yellow in its most oxidized state (+5), and grey-bluish in its reduced state (+4/+3). Therefore when we adsorb ammonia, a reducing agent, we see a color change in the sample. This has allowed us to observe, in our preparation procedures, the presence of both (radial) diffusion gradients and temperature gradients. Useful dimensionless numbers which help in characterizing these systems are the Peclet number (the ratio of convective to diffusive forces) and the particle Reynolds number. In our systems we have also noticed channelling effects. In order to eliminate diffusion effects we have reduced our gas flow rates when exposing the sample. Channelling is reduced by paying careful attention to how we fill and pack catalyst into our reactors. The thermal gradients arise from the way the reactor fits into the heater (horizontal reactor length is about 1/2 the heater diameter). By doing the adsorptions with flowing gases in a carrier such as helium, we have reduced our thermal gradients.

We surmise that other groups may be preparing samples in an analogous fashion and may be operating under unnoticed conditions of diffusion and/or temperature gradients. Based upon our observations, we stress the importance of reactor design and suggest that interested researchers consult textbooks such as Smith's "Chemical Engineering Kinetics".

Best regards,


Marjorie S. Went


Jeffrey A. Reimer

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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. 390 (March)-----15 February 1991
No. 391 (April) -----15 March 1991
No. 392 (May) -----19 April 1991
No. 393 (June)-----17 May 1991

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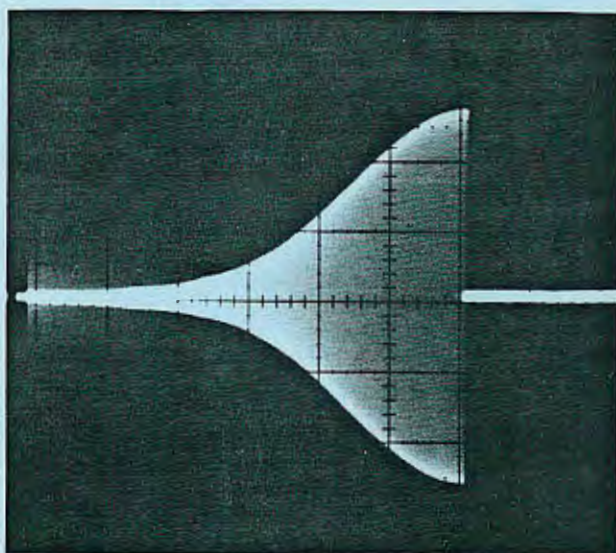
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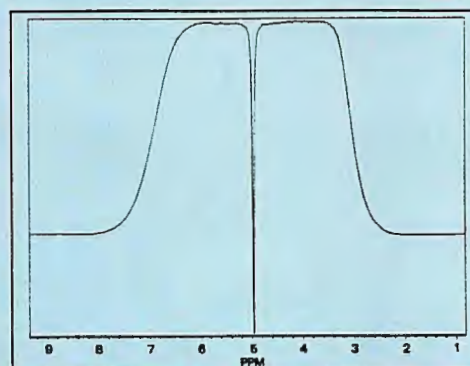
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easily created by the user. Normalized waveforms can be recalled and modified in amplitude or duration by a single instruction resulting in very efficient pulse programs.

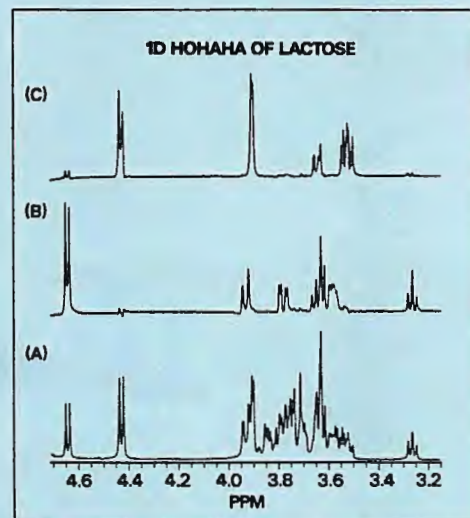
Wave Shaping on the Omega 500 PSG



An oscilloscope trace of a half-Gaussian pulse. The pulse is defined by 250 points and the duration is 10 ms.



The result of applying a 180° half-Gaussian pulse to a sample of doped water. The water resonance has been broadened by introducing a large Z1 current in the room temperature shims. The half-Gaussian pulse width is 200 ms and the width of the "burned hole" is 12 Hz.



TOCSY of Lactose (10mM in D_2O).

Bottom spectrum (A) is a simple one pulse spectrum. Middle spectrum (B) is a 1D-TOCSY spectrum, where anomeric proton at 4.43 ppm has been selectively irradiated with a half-Gaussian pulse. Top spectrum (C) is a 1D-TOCSY spectrum when the anomeric proton at 4.65 ppm has been selectively irradiated with a half-Gaussian pulse.



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