BRIAN SYKES

TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 354

March 1988

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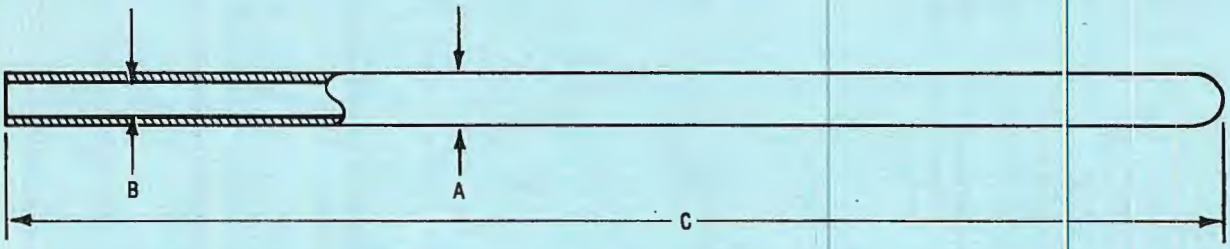

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FORTHCOMING NMR MEETINGS

29th ENC (Experimental NMR Conference), Apr. 17-21, 1988; Rochester, New York; Chairman: Professor Stanley J. Opella, Dept. of Chemistry, University of Pennsylvania, Philadelphia, Pennsylvania 19104, (215) 898-6459. For information, contact Professor Edward O. Stejskal, ENC Secretary, Dept. of Chemistry - Box 8204, North Carolina State University, Raleigh, North Carolina 27695-8204; telephone (919) 737-2998.

9th EENC (European Experimental NMR Conference), May 16-20, 1988; Bad Aussee, Austria; For further information, write Professor H. Sterk, Karl-Franzens-Universitaet Graz, Institut fuer Organische Chemie, Heinrichstrasse 28, A-8010 Graz, Austria. See Newsletter 348, 15.

2nd European Congress on NMR in Medicine and Biology, June 23-25, 1988; Berlin, West Germany; contact Prof. R. Felix, Dept. of Radiology, Charlottenburg University Hospital, Spandauer Damn 130, D-1000 Berlin 19, West Germany.

XIII Intl. Conference on Magnetic Resonance in Biological Systems, Aug. 14-19, 1988; Madison, Wisconsin. See Newsletter 349, 60.

NATO Summer School: "A Methodological Approach to Multinuclear Magnetic Resonance in Liquids and Solids: Chemical Applications", August 22 - September 2, 1988; Maratea, Italy; See Newsletter 353, 76.

★ 27th Annual Eastern Analytical Symposium and Exposition, October 2-7, 1988; New York Hilton Hotel, New York; General Chairman - Dr. Harvey S. Gold, Polymer Products Dept., E256/308, E. I. du Pont de Nemours & Co., Wilmington, DE 19898; (302) 695-3669.

★ New listing.

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, California 94303 U.S.A.

(415) 493-5971

DEADLINE DATES

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No. 358 (July) ---------- 17 June 1988
No. 359 (August) ---------- 22 July 1988

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OBJET :

GRENOBLE, le 15 Janvier 1988
(received 1/25/88)

Parity Non Conservation and NMR, why not ?

Dear Doctor Shapiro,

The first statement of a possible Parity Non Conservation (PNC) in weak interactions was made in 1956. Less than a year later, the first PNC experimental evidence came out in the study of the ^{60}Co β decay. Several experiments in nuclear physics then confirmed that PNC takes place in weak interactions. After several years of theoretical and experimental work PNC results came out from atomic physics. Nowadays there are no conclusive results in molecular physics. We propose that NMR could provide a good apportunity to observe PNC in molecular physics (1) the basic idea is the detection of

an NMR frequency difference, which would take place between the d and l forms of a chiral molecule in an achiral solution and environment. From our calculations a difference of about 5 mHz is expected. The observed nucleus must have a spin $\frac{1}{2}$ and a high Z value as the expected value increases as Z^2. Good candidates are : ^{195}Pt ; ^{199}Hg ; ^{203}Tl or ^{205}Tl ; ^{207}Pb.

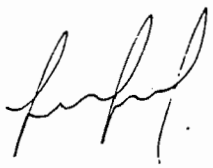
The main experimental problems we shall be facing are :

- existence of large ΔG which increases the natural linewidth
- temperature gradient in the NMR tube
- possible orientation of the molecule under study by the applied magnetic field.

The proposed experiment which is an ultrahigh resolution NMR experiment on a heavy nucleus is a kind of experimental challenge. Any suggestion, comment or critics will be welcomed.

A.L. BARRA

J.B. ROBERT



L. WIESENFELD

1) A.L. BARRA, J.B. ROBERT, L. WIESENFELD, Physics Letters A 115 (1986), 443 ; Europhysics Letters in the press.

Wageningen Agricultural University

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Enclosure(s)
Date January, 15, 1988 (received 1/26/88)
Re Ring current shift conformational analysis with CHEM-X.

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

Ring current shift conformational analysis with CHEM-X.

Dear Dr. Shapiro,

Proton-NMR can yield structural information on substituted porphyrins via ring current induced chemical shifts using Abrahams' double dipole model [1]. The conformation of the substituent with respect to the porphyrin plane is varied to yield optimum agreement between calculated and experimental ring current shifts.

In a previous method used at our department to determine the position of the substituent with respect to the porphyrin plane [2], the conformations were varied over all degrees of freedom without invoking the linking chain, and afterwards checked by molecular models. Although the results were satisfying, the method is now improved by generating conformations of the entire molecule by CHEM-X, a molecular modelling package released by Chemical Design Ltd. Oxford, England. The conformations are generated by rotating bonds of the connective chain. Subsequently the ring current shifts for each conformation are calculated until optimum agreement is found with the experimental data. We have applied this method to donor-porphyrin and porphyrin-acceptor complexes which are important models for biological photosynthetic processes.

As an example we present the data on zinc-5-(2(3-N-phtalimidyl(-1-propoxy)-phenyl)-10,15,20-triphenylporphyrin (fig. 1) in deuterated THF at 298 K, recorded on a Bruker CXP 300 spectrometer.

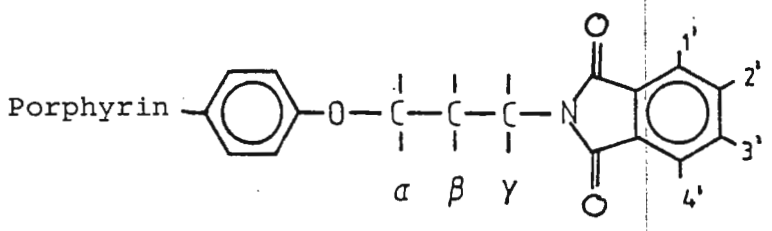

Fig. 1. The 3-N-phtalimidyl(-1-propoxy)-phenyl substituent

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The ring current induced chemical shifts with respect to the phtalimid reference spectrum and the results obtained with this method are listed below.

Proton	$\Delta\delta_{exp}$ (ppm)	$\Delta\delta_{calc}$ (ppm)
1'/4'	-0.40	-0.40
2'/3'	-0.30	-0.31
α	+0.06	+0.06
β	-0.85	-0.87
γ	-0.60	-0.62

The average conformation on NMR timescale which is calculated from the ring current induced chemical shifts is shown in fig. 2.

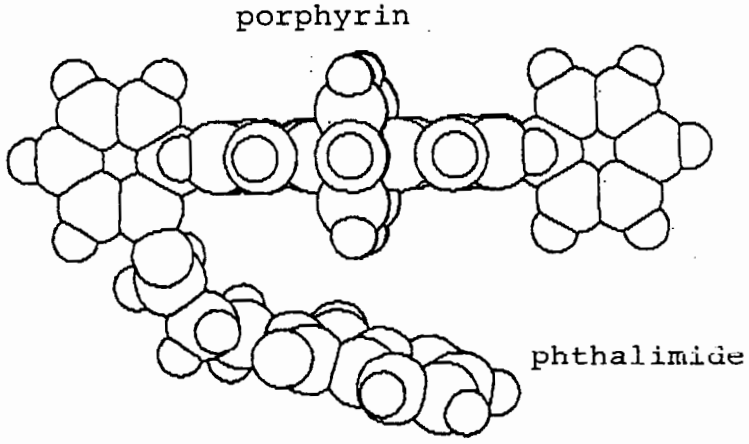


Fig. 2. The calculated average conformation of the porphyrin complex.

Yours sincerely,

D. Huckriede U. Hofstra T.J. Schaafsma

[1] R.J. Abraham, K.M. Smith, D.A. Goff, and Jan-Ji Lai, J. Am. Chem. Soc., Vol. 104, 1982, p. 4332.
[2] L. Benthem, R.B.M. Koehorst and T.J. Schaafsma, Magn. Reson. in Ch., Vol. 23, 1985, p. 732.

The University of Melbourne

DEPARTMENT OF ORGANIC CHEMISTRY

9th February, 1988
(received 2/11/88)

Dr B. L. Shapiro,
TAMU NMR Newsletter,
966 Elsinore Court,
PALO ALTO, CALIFORNIA, 94303.
U.S.A.

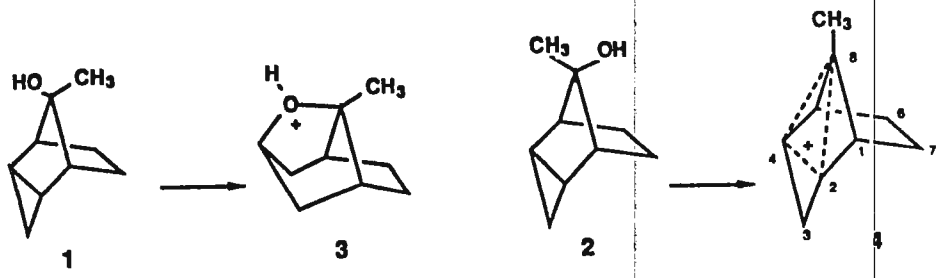
Tall Ships and Superacid Seas: ^{13}C Signals of a Trishomocyclopropenium cation

Dear Barry,

In keeping with the spirit of the Australian bicentennial celebrations our correspondence has taken several months to cross the oceans (indeed, the paper has changed colour, white to green to pink!). Apart from the visitors arriving in the tall ships we are pleased to have a number of scientific visitors arriving by more modern transport to attend the National NMR Conference next week at Thredbo, N.S.W. of which Dr Larry Brown wrote in TAMU 349-18.

For the last few years (but not 200) we have investigated $^{1}J_{C\alpha H}$ values of carbocations generated in superacids and found that in static tertiary classical systems the difference in $^{1}J_{C\alpha H}$ between the cation and the model ketone (ΔJ) is given by $\Delta J = A\text{-}B\cos^{2}\theta$, the values of A and B depending on whether the cation is methyl or phenyl substituted at the cationic centre.[1,2]

In our attempts to generate the trishomocyclopropenium cation 4, we treated the syn alcohol 1 with various superacids but the product exhibited ^{1}H and ^{13}C spectra inconsistent with 4. It was eventually identified as the protonated ether 3 (TAMU 331-21).[3] Subsequently we soaked the pure anti-alcohol 2 in a sea of FSO_3H/SO_2ClF and were pleased to find only highly shielded ^{13}C masts on the horizon at the coordinates shown on the illustration.



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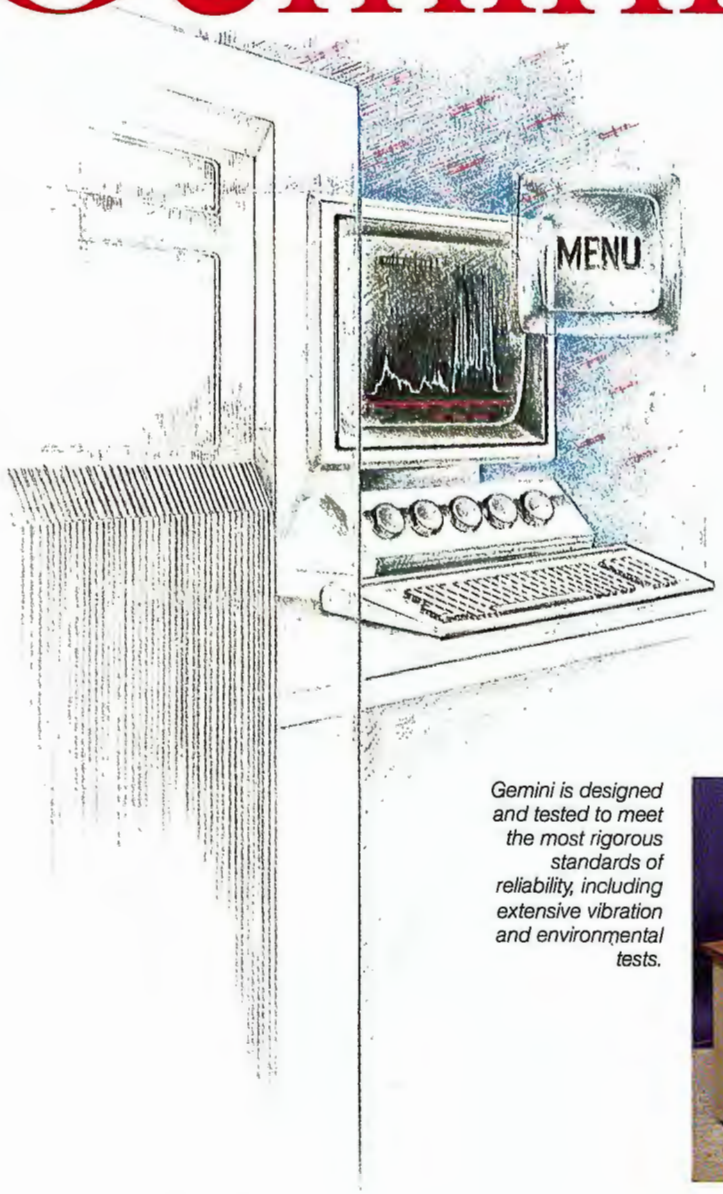

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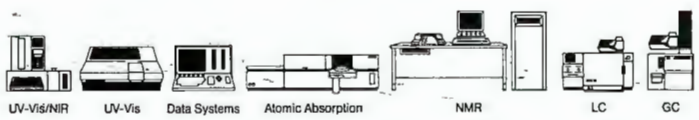

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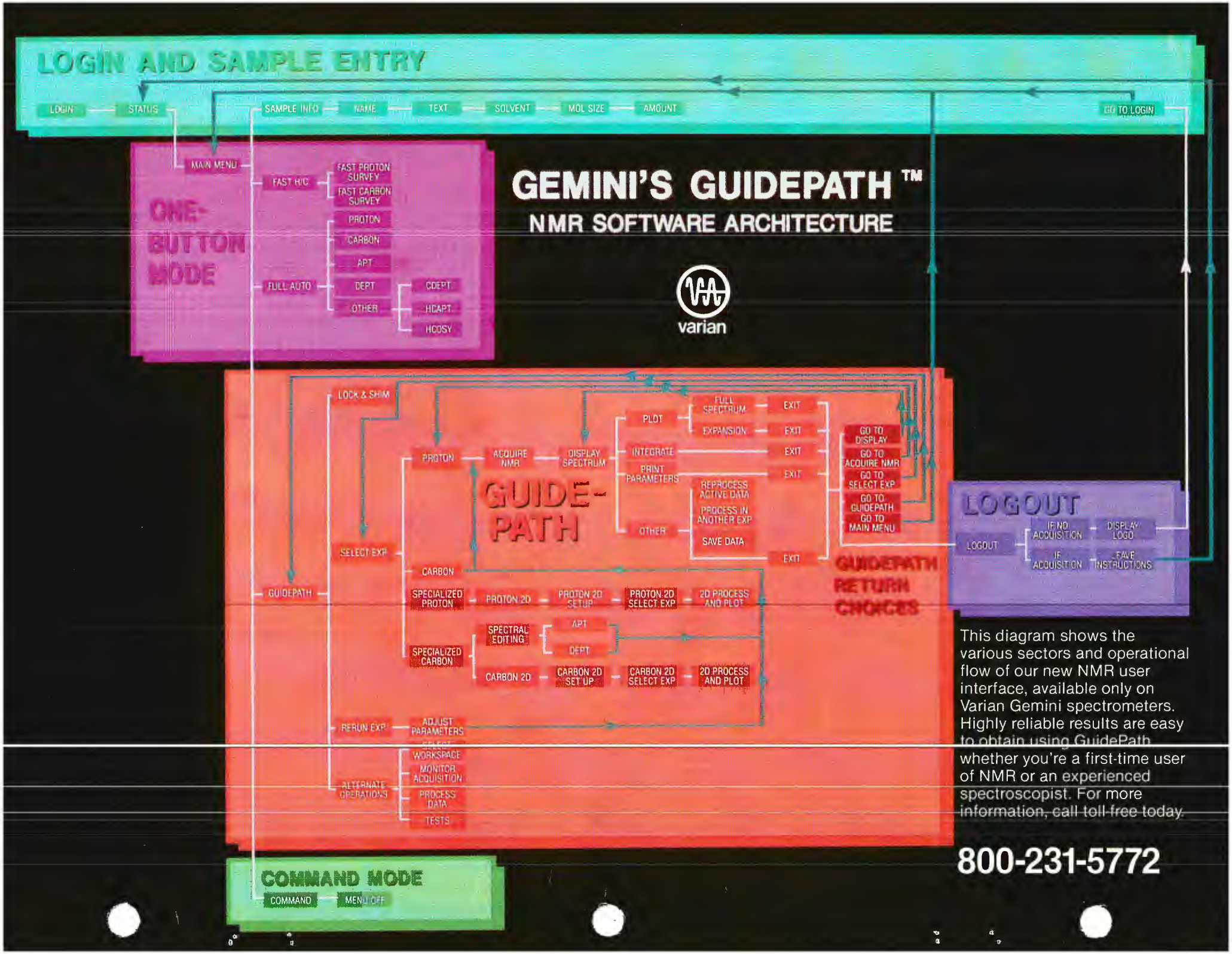

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When compared with the $^{1}J_{CH}$ values of the parent ketone, the $^{1}J_{CH}$ values give $\Delta J = 18$ Hz for the bridgehead carbons ($C_{1,5}$) and 21 Hz for the bridging carbons ($C_{2,4}$) which are larger than those anticipated for static or equilibrating cations. Together with the low ^{13}C shifts, these high ΔJ values are consistent with 4 having a nonclassical $\sigma\pi$-bridged structure.

This sighting completes the series of substituted trishomocyclopropenium cations first observed by Masamune.[4] A full report has been made to the Admiral of J.O.C.

Yours sincerely,

David

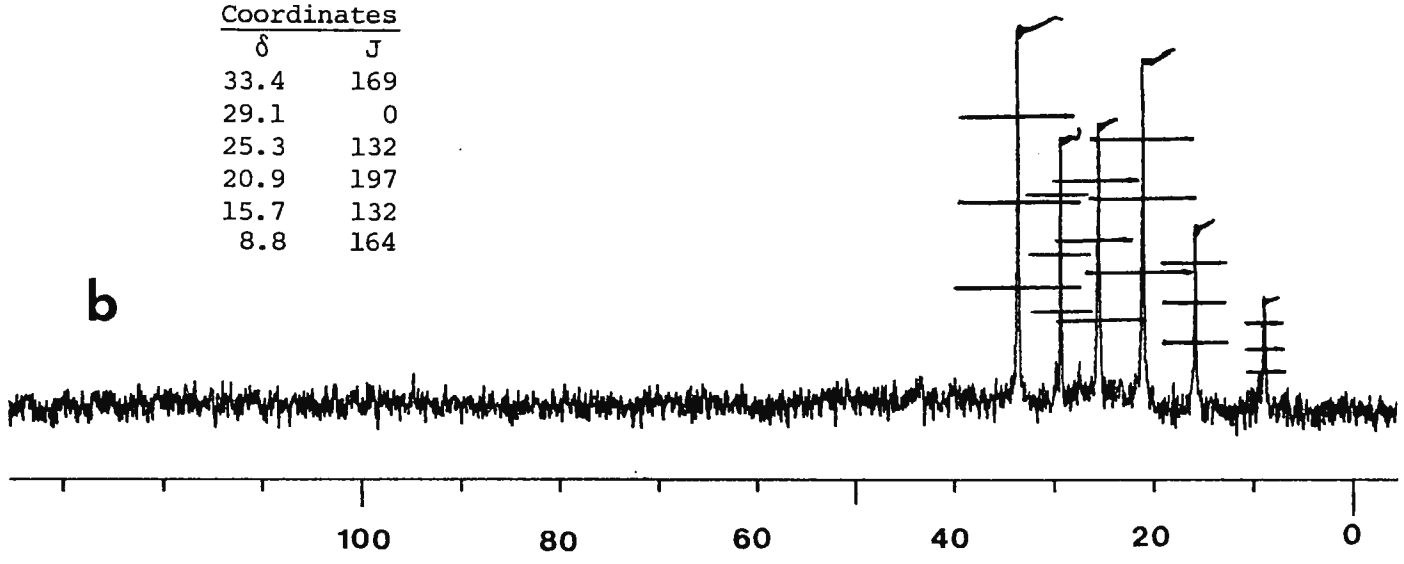
DPK:EC

D. P. Kelly.

1. Kelly, D. P., Underwood, G. R., Barron, P. F., J. Am. Chem. Soc., 1976, 98, 3106-3111.
2. Kelly, D. P., Farquharson, G. J., Giansiracusa, J. J., Jensen, W. A., Hügel, H. M., Porter, A. P., Rainbow, I. J., Timewell, P. H., J. Am. Chem. Soc., 1981, 103, 3539-3543.
3. Kelly, D. P., Giansiracusa, J. J., Leslie, D. R., McKern, I. D., Tetrahedron Lett., 1986, 27, 2311-2312.
4. Masumune, S., Sakai, M., Kemp-Jones, A. V., Can. J. Chem., 1974, 52, 858-860.

Coordinates

δ	J
33.4	169
29.1	0
25.3	132
20.9	197
15.7	132
8.8	164



First sighting of the 8-methyltricyclo[3.2.1.$0^{2,4}$]oct-8-yl cation at Port Phillip, Melbourne, February 1988 (Yards added under artistic licence).

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Telegrammi: ISTISAN-ROMA
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Rome, October 27th, 1987
(received 1/27/88)

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

19F NMR study of the interaction between 5-fluorocytosine and methylpartricin in Candida albicans

Dear Professor Shapiro,

The activity of 5-fluorocytosine (5-FC), a powerful antimycotic agent expecially active against Candida and Cryptococcus pathogenic fungi, is closely related to the metabolic transformations undergone inside the target cell.

Intracellular 5-FC is converted into 5-fluorouracil (5-FU) and then into a series of phosphorylated metabolites, the efficiency of these metabolic interconversions being closely related to the sensitivity or resistance of various strains to this antimycotic drug.

As already reported in one of our previous contributions, we are studying the 5-FC metabolism in intact cells of C. albicans, by utilizing 19F NMR approaches (1,2).

More recently we extended our analyses to study the synergism between 5-FC and methylpartricin (MeP), the methylated analog of amphothericin B, which exerts a well known activity on the cell membrane.

19F NMR analyses of perchloric acid extracts of cells incubated with 5-FC, either in the presence or in the absence of MeP, show that MeP induces a decrease in the levels of some of the 5-FC phosphorylated metabolites (Fig. 1) and suggest the interest of precisely identifying the individual signals comprised in the spectral region between 3.5 and 5.5 ppm. Such an assignment seems to be essential for possibly assessing the relationships between the specific alterations occurring in the pathways of 5-FC metabolic conversions and the structural effects of MeP on the cell membrane.

Yours sincerely,

Massimo Di Vito (1) — M.Di Vito
(2) — A.Torosantucci
Giulia Carpinelli (1) — G.Carpinelli
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Francesca Podo (1) — F. Podo

Laboratori di (1) Biologia Cellulare e (2) Batteriologia e Micologia Medica, Istituto Superiore di Sanita', Roma.
(Work partially supported by C.N.R. contracts TBMS n.86.01474.57 and CMI n.86.01563.52. We thank Mr. M. Giannini for technical assistance).

References: (1) M.Di Vito et al. Texas A&M University NMR Newsletter, 314, 25-26, 1984. (2) M.Di Vito et al. Antimicrobial Agents and Chemotherapy, 29, 303-308, 1986.

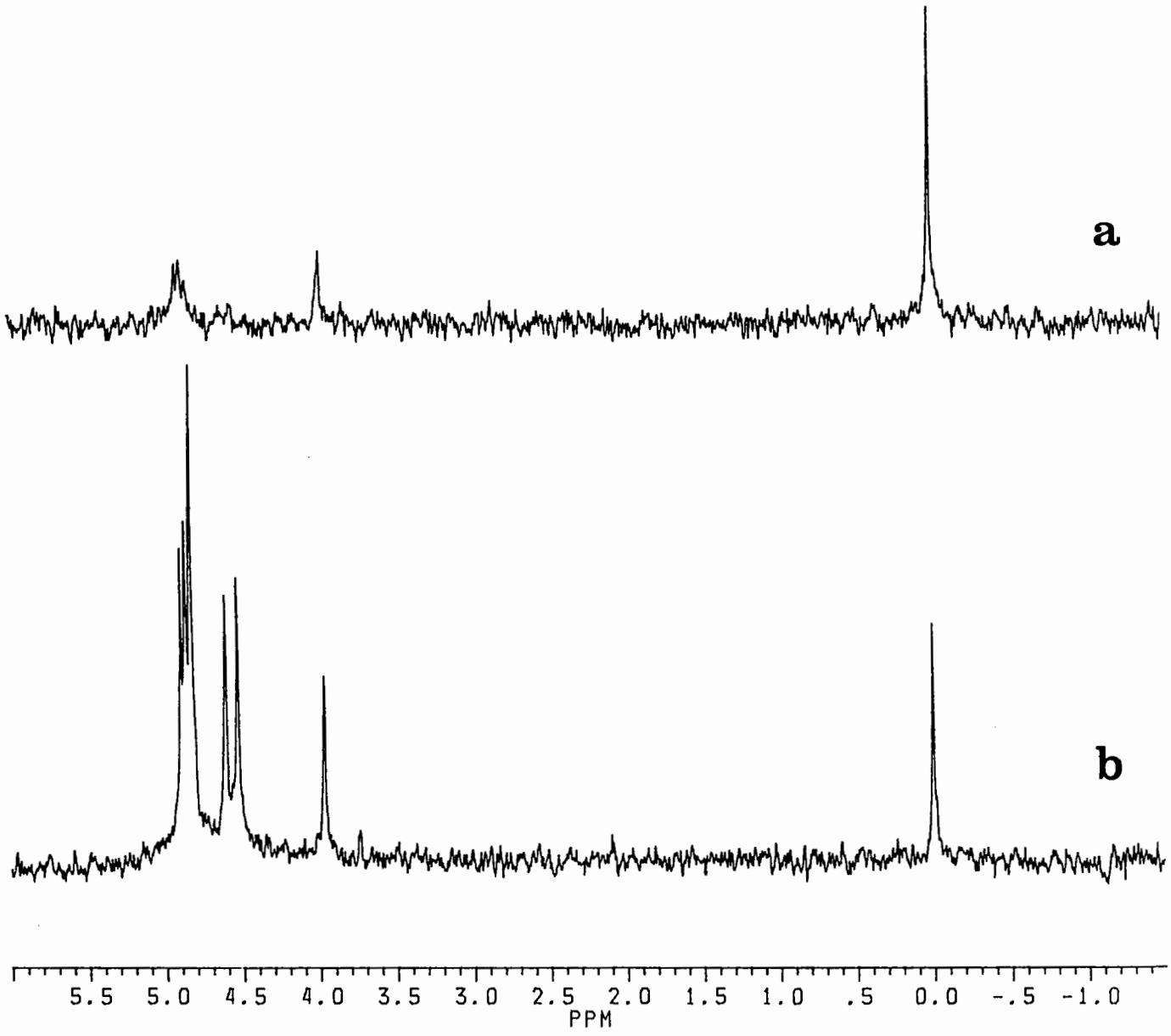

Figure 1. 19F NMR spectra (376.51 MHz) of perchloric acid extracts obtained from C.albicans cells (5-FC sensitive strain 72S) after 24 h incubation with 5-FC, either in the presence (a) or in the absence (b) of MeP. (5-FU signal, set at 0.00 ppm, is the internal reference. Amplification of spectrum (b) is 2x that of (a)).

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UCSF Radiologic Imaging Laboratory
400 Grandview Drive
South San Francisco, California 94080
(415) 952-1366

January 26, 1988
(received 1/28/88)

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

Dear Dr. Shapiro:

We have been working closely with some vascular surgeons on diagnosis and characterization of atherosclerosis with NMR zeugmatography. One question that comes up is whether a particular structure in the vessel is a fatty deposit or not. To address this and other applications we put together a spectroscopic imaging experiment [1] (sequence in fig. 1) that phase-encodes an inner volume at relatively high spatial resolution and reads the spin echoes in the absence of applied gradients to resolve (barely) the peaks of water and fat at 15 megahertz or 0.35 Tesla. The images are formed from peak integrals along the chemical shift frequency axis at the spectral locations of each major peak. The spectrum at any location in the images can also be plotted.

In Figures 2 and 3 are transaxial images of the fat and water signals respectively from a central region surrounding the abdominal aorta of a patient with vessel disease. Also shown are spectra from 2 individual volume elements 2 x 2 x 12 mm thick. One spectrum from the vessel wall suggests a fatty deposit. One more central in the aortic flow shows enhanced signal due to the replacement of saturated spins by the blood flow. There is also a crescent-shaped void in the water image of the aorta which might represent a flow disturbance.

An advantage of this sequence is the low receiver bandwidth that one can use by removing the imaging gradient during signal sampling. The disadvantages include a requirement of a large number of phase-encoding steps and the associated long minimum imaging time, in this case about 21 minutes using a 300 millisecond repetition time.

The results from a few different patients have been interesting but somewhat inconsistent, so it is much to early to decide how useful this is in clinical imaging. We are also using this type of sequence at 2.0 Tesla for imaging phosphorous. More on this in a future letter.

Sincerely,

David M. Kramer

[1] T.R. Brown, B.M. Kincaid, and K. Ugurbil, Proc. Natl. Acad. Sci. USA 79, 3523 (1982).

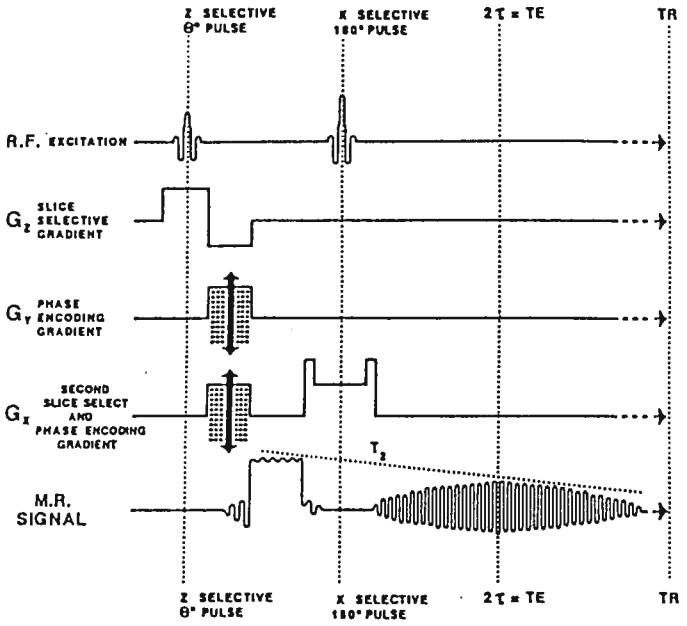


Figure 1. Pulse sequence diagram for the spectroscopic imaging technique. A slice is selected by the 90 degree pulse and an echo is rf refocussed from an inner volume within that slice using the 180 degree rf pulse in an orthogonal gradient to that used for the 90. Phase-encoding in both in-plane dimensions resolves into pixels the location of spectra obtained from the echoes, all of which are observed in the absence of applied gradients.

Figure 2: Region from fat image near abdominal aorta and a spectrum from indicated bright pixel in the vessel wall.

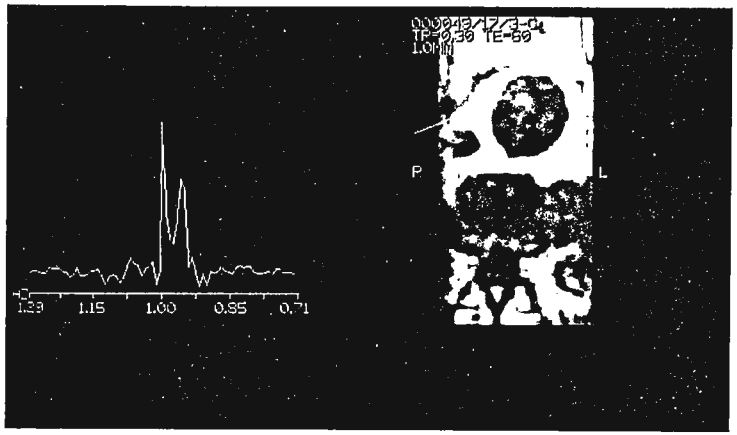
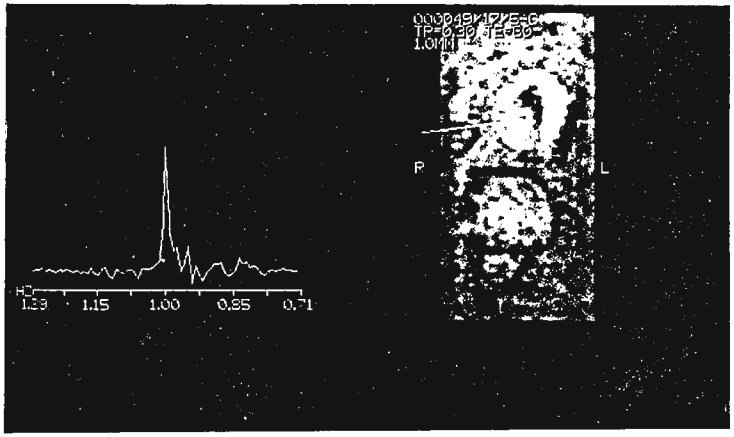


Figure 3: Region from water image and spectrum from pixel within the lumen.



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Professor Barry Shapiro
966 Elsinore Court
Palo Alto
California 94303
USA

1987-12-07
(received 1/28/88)

Subject: New BioNMR-laboratory, pattern recognition of SCS.

Dear Barry,

In response to your "ultimatum" we can report some planned improvement of the NMR facilities in the north of Sweden. In addition to our Bruker AC-80 and MSL-100 we will have a loaded AM-500 installed late next summer. We will also have an update of our WM-250 to a modern AC-P. Data handling will be performed on two SUN Specstations which will be communicating with several ASPECT-3000's via Ethernet (believe it or not). All these facilities are supposed to form a cornerstone to the new University Biocenter.

We are also continuing our work using pattern recognition methods to extract information from 1D and 2D NMR data. earlier we have shown that ^{13}C substituent chemical shifts (SCS) in monosubstituted, delocalized organic systems nicely could be predicted from the correspondingly substituted benzenes[1]. This analysis was accomplished by use of principal components and partial least squares data analysis. Together with prof Exner, Prague, we have now extended this study to include disubstituted systems and to our initial surprise we found , eliminating additivity SCS, a similar grouping behaviour (acceptor, donor, alkyl and halogen subgroups) as in the former analysis using monosubstituted benzenes. In other words, non-additivity effects are not continously distributed and could be predicted from the benzene pattern. From a statistical point of view, this finding also implies that the commonly used "electron-demand", three parameter models should not be used.

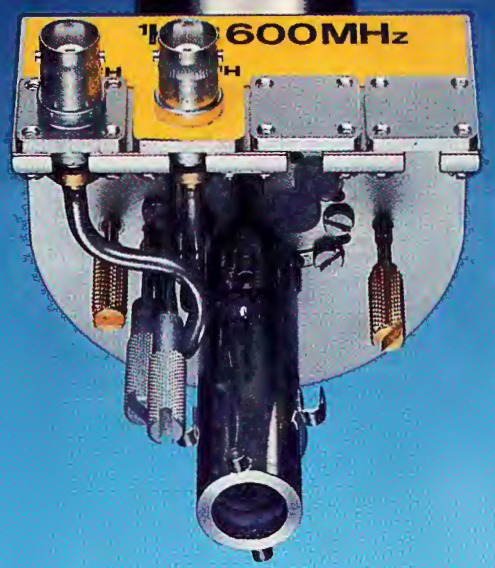
An important conclusion from this study is that **no other fundamental substituent effects could be demonstrated in the disubstituted systems other than those present in the monosubstituted benzenes.**

Best regards

Ulf Edlund

1. D. Johnels, U. Edlund and S. Wold, J. Chem. Soc. Perkin II, 1339 (1985) and earlier papers in this series.

Bruker delivers.



Again.

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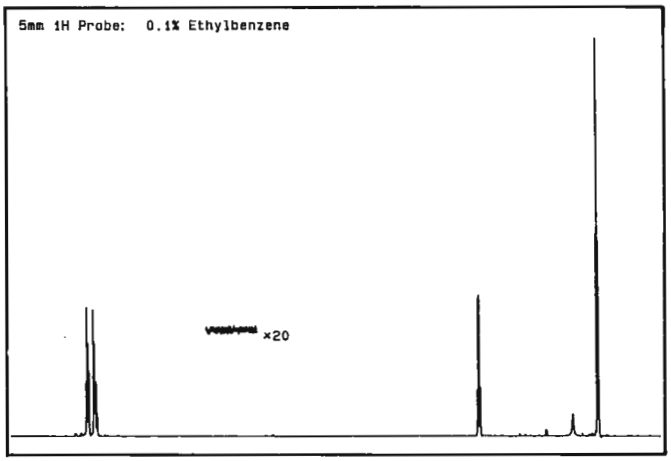

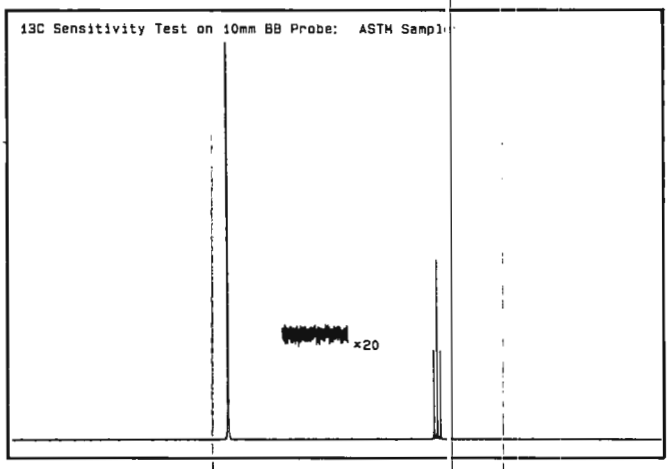

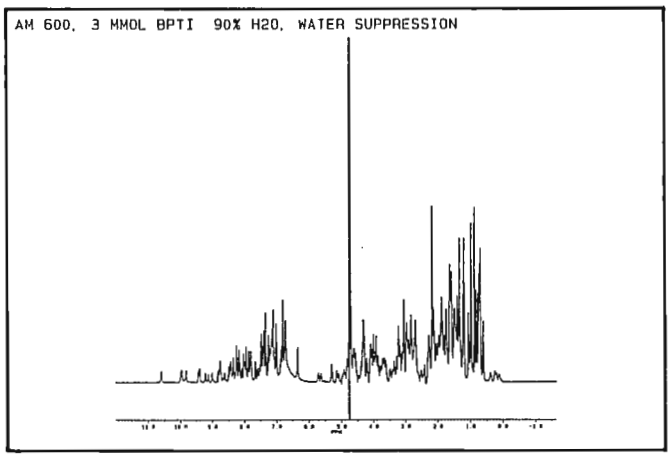

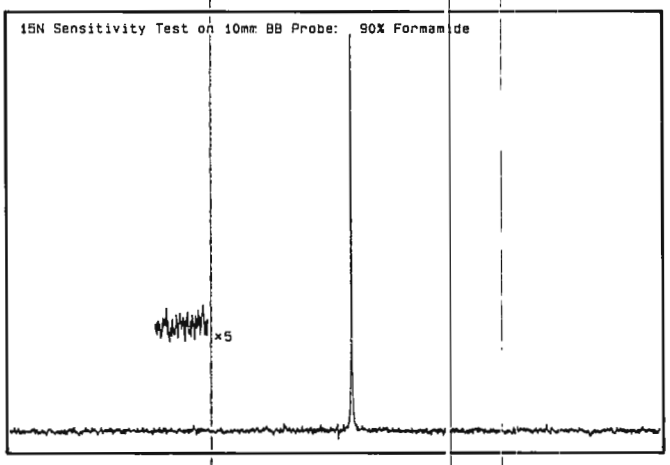

Current AM 600 specifications (subject to change):

Probehead	Nucleus	Test	Value
^{1}H selective (5mm)	^{1}H	lineshape	10/20
		S/N	600:1
Broadband (5mm)	^{13}C	S/N (ASTM)	220:1
		S/N (10% EB)	180:1
		lineshape	6/15
	^{15}N	S/N (FORM)	25:1
Broadband (10mm)	^{13}C	S/N (ASTM)	650:1
		S/N (10% EB)	375:1
		lineshape	6/15
	^{15}N	S/N (FORM)	80:1
Resolution (all probes)			0.25 Hz

Magnet drift: ca. <40 Hz/hr

EB = ethylbenzene with ^{1}H decoupling
ASTM = 60% C_6D_6 in dioxane
FORM = Formamide (^{1}H decoupling without NOE)

LINESHAPE: ^{1}H = $CHCl_3$ linewidth at height of ^{13}C satellite/at 20% this level
^{13}C = C_6H_6 linewidth at 0.55%/0.11% of peak height

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DEPARTMENT OF RADIOLOGY
AND
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Mailing Address:
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Baltimore, Md. 21205

January 25, 1988
(received 1/30/88)

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

"Detection of [4-^{13}C]-cyclophosphamide by ^{1}H{^{13}C} Spin-echo Difference Spectroscopy"

Dear Barry:

We have been interested in monitoring cyclophosphamide metabolism by NMR in perfused liver cells at clinically relevant concentrations. In order to detect such low concentrations, we will primarily be using ^{1}H or ^{19}F NMR with the latter requiring synthesis of a less active fluorinated derivative of cyclophosphamide. The ^{1}H NMR spectrum is complicated by the resonances from the cells, perfusate, and support medium (dextran beads). We have been evaluating the feasiblity of using ^{1}H-{^{13}C} spin-echo difference spectroscopy (1) with ^{13}C-labeled cyclophosphamide in these studies.

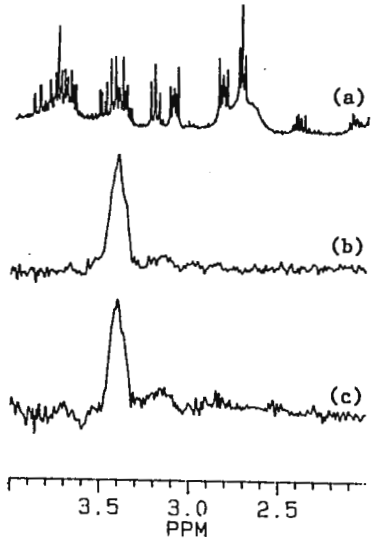

Figure 1

Figure 1(a) shows the ^{1}H NMR spectrum of 600 μM [4-^{13}C]-cyclophosphamide in Minimum Essential Medium Eagle (Sigma). Figures 1(b) and 1(c) show the edited spectra using a 1-3-3-1-τ-2-6-6-2 ^{1}H spin-echo pulse with carbon decoupling during the acquisition for the same mixture without and with Cytodex-1 beads, respectively. Total acquisition time is 8 minutes in the edited spectra using a 10 mm ^{1}H probe equipped with a heteronuclear decoupling coil from Cryomagnet Systems on our Bruker AM 360 WB. We estimate, that in our case, this method is approximately nine times more sensitive than ^{13}C-{^{1}H} spectroscopy.

We will soon be implementing experiments on perfused cell systems and evaluating some of the other ^{1}H detect ^{13}C editing methods.

Sincerely,

Michael P. Gamcsik
Instructor

Jerry D. Glickson
Professor

(1) Freeman, R; Mareci, TH; Morris, GA (1981) *J. Magn. Reson.* 42, 341.

INSTITUT FÜR PHYSIK DER UNIVERSITÄT BASEL
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Klingelbergstrasse 82, Telefon 061 - 44 22 80

CH-4056 Basel (Schweiz) January 21,88
(received 1/29/88)

Prof. Dr. P. Diehl

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

Title: Problems in the structure determination of linear molecules

Dear Barry,

In the structure determination by NMR of partially oriented linear molecules like e.g. diacetylene, it was always assumed, that a measurement of the 4 CH- and 1 HH-direct couplings definitely overdetermines the 2 unknown carbon coordinates with respect to the HH-basis and the unknown degreee of order.

Only lately, when we tried to determine not only the structure but also the interaction parameters (for the three different bond types), which induce the molecular order but simultaneously deform the molecule in a correlated way, it became clear, that such problems are in general exactly determined if the deformation is neglected but definitely underdetermined if the interaction parameters are also required.

The reason is, that for linear molecules with nuclei i,k,j at equilibrium positions the following additivity relation holds:

$$(k_{ij}/D_{ij}^{eq})^{1/3} = (k_{ik}/D_{ik}^{eq})^{1/3} + (k_{kj}/D_{kj}^{eq})^{1/3} \qquad (1)$$

If we add contributions due to anharmonic vibration to the D_{ij}^{eq}, this additivity is still correct. Also the contributions due to correlated deformation leave the relation unaltered. Only the addition of contributions due to harmonic vibrations which are quadratic in the displacements destroys equ. 1. On the other hand, experimental direct couplings corrected for harmonic vibration should fulfil equ. 1.

As a consequence, a linear molecule with n different bonds has only n independent direct couplings. Therefore its structure, corrected for correlated deformation (n unknown interaction parameters and n-1 unknown structure parameters) cannot be determined.

In the following tables results of equ. 1 for the molecule diacetylene illustrate the excellent additivity for the direct couplings corrected for harmonic vibration and simultaneously confirm, that the harmonic corrections have been performed correctly:

Tables: Addidivity relations for direct couplings of diacetylene ($H_1 - C_3 \equiv C_4 - C \equiv C - H_2$) solvent ZLI 1167, T = 302 K., direct couplings in Hz)

	D_{12}	D_{13}	D_{14}	D_{23}	D_{24}
D^{exp}	142.42(32)	5466.83(44)	601.61(27)	63.81(62)	149.77(47)
D^{vib}	143.31	6404.94	636.63	64.17	152.80

	basis(D_{13},D_{23})	basis(D_{14},D_{24})
D_{12}^{calc}	142.07 (1.12)	142.54(28)
$D_{12}^{vib} - D_{12}^{calc}$	1.24	0.77

D^{exp} = measured direct coupling

D^{vib} = " " " corrected for harmonic vibr.

D^{calc} = value calculated from equ. 1.

With best wishes,

Yours

R. Wasser P. Diehl

354-20

DEPARTMENT OF PHYSICS &
SOPHISTICATED INSTRUMENTS FACILITY
INDIAN INSTITUTE OF SCIENCE
BANGALORE-560 012 INDIA
Prof. Anil Kumar

Ref. :

Date : Jan. 13, 1988
(received 1/30/88)

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

Dear Professor Shapiro,

Degeneracy of Transitions and Names

(A) Transitions

One of our colleagues ran a coupled HETCOSY carbon-13 2D experiment of a peptide and found a central peak in the methylene carbon spectrum. This peak is always absent in decoupled experiments. We thought that the appearance of the peak may have something to do with strong coupling among the two inequivalent protons and calculated the X part of the ABX spectrum. It turned out that even under strong coupling, if the angle α of the proton coherence transfer pulse in the sequence $[90°(^1H)\text{-}t_1\text{-}\alpha(^1H)/\beta(^{13}C)\text{-}t_2(Aq\text{-}{}^{13}C)]$ is exactly 90°, the central peak is missing, if $J_{AX}=J_{BX}$. However, in the present case, unlike A_2X and decoupled cases, the central peak is retrievable by changing α, (Fig. 1). Look-out for JMR for more details.

(B) Names

Recently I encountered some problem due to degeneracy of names and wish to lift this degeneracy through your News Letter.

There are two Anil Kumar(s) in the NMR field. One (not me) has been working with Prof. George C. Levy, is a consultant in the company nmri [ref. TAMU NewsLetter No. 347, Aug. 1987 page 14] and is a coauthor of a book with him. I am the Anil Kumar who has at various periods of time worked with Professors R.R. Ernst and K. Wüthrich (E.T.H., Zürich), C.S. Johnson Jr. (UNC, Chapel Hill), S.L. Gordon (Ga. Tech., Atlanta) and B.D.N. Rao (IIT, Kanpur, now at IUPUI, Indianapolis). Since 1977 I am at Indian Institute of Science, Bangalore and have a modest group working on NMR.

With best regards,

Yours sincerely,

Anil Kumar

(ANIL KUMAR)

Telephone : 344411/343 Telegram : 'Science' Telex : 0845-8349 BG

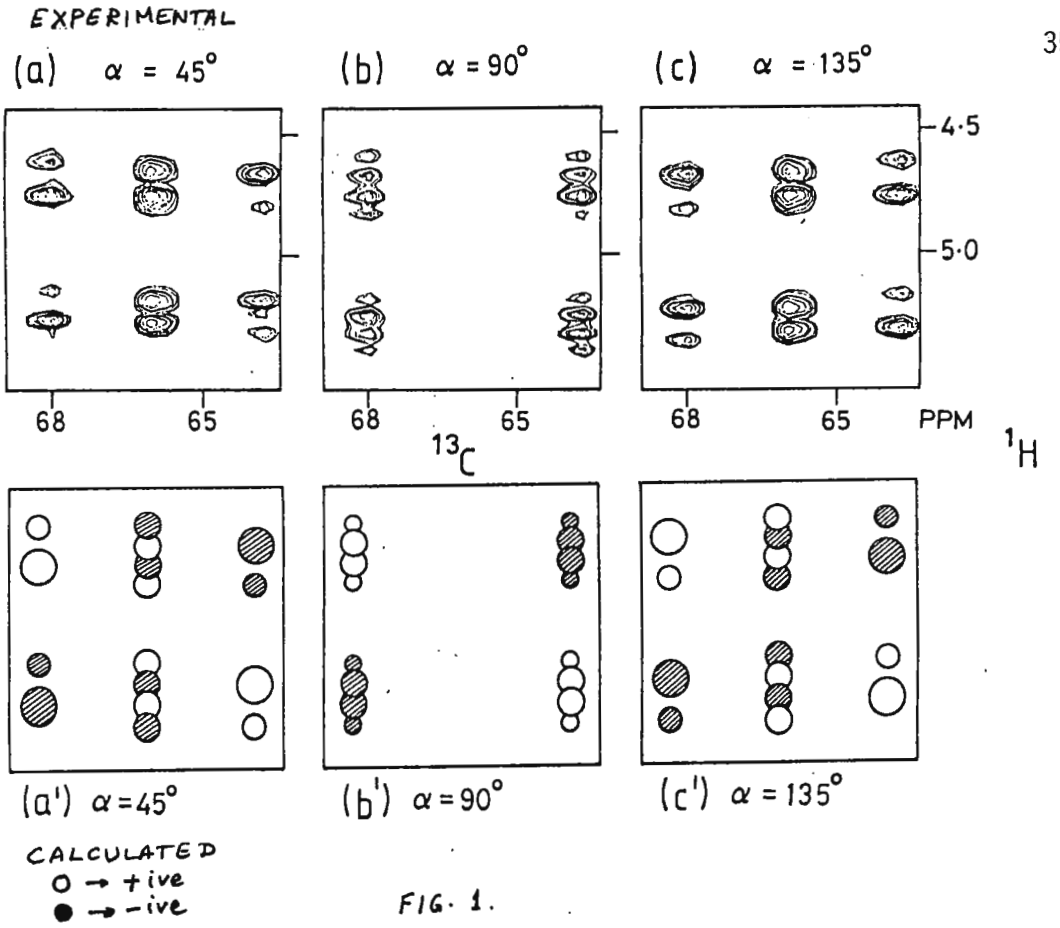

FIG. 1.

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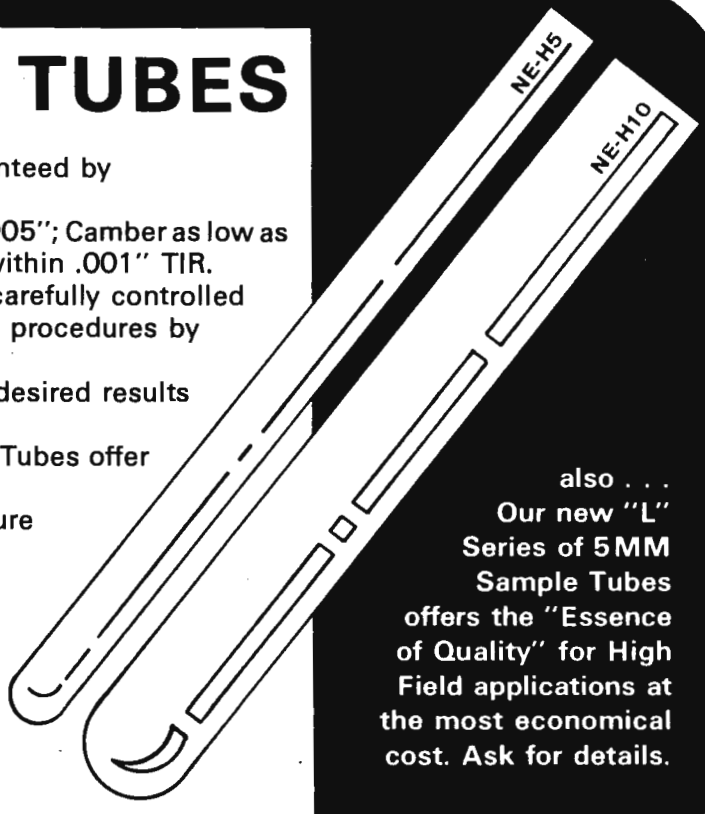

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Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

January 27, 1988
(received 1/30/88)

Re: ^{27}Al Cross Polarization of Aluminas and 87Rb NMR of Rubidium salts on γ-Alumina.

Dear Barry:

Pink and blue are attention grabbing colors....nice touch! I would like to relay to you some work that Doug Morris, John Cheng and I are doing in the area of nmr of surfaces and surface adsorbed species. We have demonstrated selective cross polarization of surface aluminum atoms and applied this methodology to γ- and α-Aluminas. With relatively high hydration levels of the γ-Alumina surface (out of the bottle) at least three surface species can be observed. Upon dehydration, only two somewhat broader resonances can be resolved. By comparison to Boehmite and α-Alumina, these resonances are assigned to surface octahedral sites (~0 ppm) and tetrahedral sites (~-72 ppm). All chemical shifts are reported relative to the bulk octahedral sites in γ-Alumina. This represents an overt shift of ~144 ppm to higher shielding for the tetrahedral sites relative to the bulk material. It is demonstrated that these resonances represent surface Brønsted sites and they are not contaminated by Lewis acid sites or sub-surface species. The Lewis acid sites can be selectively observed by subtraction of spectra of the surface resonances in the presence and absence of a Lewis base. For the α-Alumina used in this study (surface area of 1 m^2/g), there appears to be a significant number of strongly chemisorbed water molecules and/or Brønsted sites on the surface. The existence of these sites could have important impact on acid sensitive chemistry performed on such an "inert" surface. Overall, this approach appears to be an excellent way to nondestructively characterize these surfaces. More importantly, the success of surface selective cross polarization methodology illustrates that other nuclides are equally amenable to such experiments and that this approach could be very useful as a general surface chemistry method for the characterization of surfaces.

In another surface related project we have been examining alkali metals adsorbed to γ-Alumina. The solid-state ^{87}Rb nmr of Rubidium salts adsorbed to γ-Alumina has been utilized to characterize the Rb^+ species present at submonolayer coverages of Rubidium. Rubidium was chosen over other alkali metals because the moderate size of its quadrupole coupling constant meant that the $\pm\frac{1}{2}$-transition could easily be excited without the complications of contaminating the resulting lineshape by mixing in other transitions. By a combination of solid-echo and spikelet echo methods four species could be identified on the γ-Alumina surface at submonolayer coverages. There are two species of **surface salt**. By **surface salt** we mean a species whose nmr adsorption depends upon the identity of the counter ion used to impregnate the surface. The other complexes are identified as **surface species**. Here we mean the resonance position of this complex is only weakly dependent upon the identity of the counter ion. Within each category of species present there is a disordered phase. The two phases can be distinguished by their respective T_2's. It is hypothesized that the disordered phase has the shorter value of T_2 because of the possibility of cation motion. This is due to the reasonable assumption of the presence of interstitial vacancies that would be present within the disordered phase. The existence of such vacancies provide a mechanism for the Rb^+ cations to jump from site to site. The dynamical processes present on the surface are consistent with the notion of adsorbate islanding. These results will have significant impact on any models of the surface of γ-Alumina and less reactive phases such as α-Alumina.

As you can see we have been having a lot of fun with the application of solid state nmr methods to problems in surface chemistry. Both topics have been written up and have been submitted for publication. I hope this gets me off your pink stationary list. By the way have you seen any new books from our favorite author? *

Sincerely

John Cheng

John Cheng

Doug Morris

Paul

Paul D. Ellis

* Paul - My pink list is stationery, but not stationary. No new books by Tom Sharpe in the last 2-3 years, unfortunately.

Barry

University of Houston

College of Pharmacy
4800 Calhoun Road
Houston, Texas 77004
713.749.3476

Department of Medicinal Chemistry and Pharmacognosy

January 19, 1988
(received 1/30/88)

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

Does your amplifier have the droops?

Dear Barry,

Thanks for the gentle pink reminder. Despite my own interest in long range heteronuclear correlation experiments, we occasionally do run others. In particular, we had been having trouble getting good performance when we ran ^{13}C-^{13}C INADEQUATE 2D spectra on my NT-300 (vintage '84) spectrometer. In part, while checking over the system, we noted a severe case of front end "droops" in the ^{13}C pulse from the standard broadband power amplifier. In contrast, proton pulses were nicely square and proton double quantum spectra ran without difficulty.

We elected to remedy the problem by using the relays normally used to switch to ^{19}F, which my system didn't have (see block diagram in Figure 1), to switch instead to a dedicated ^{13}C amplifier shown in Figure 2. The new amplifier was mounted in the console in the space normally occupied by the ^{19}F power amplifier. The net result of this modification was a marked improvement in the shape of the ^{13}C pulse and a corresponding, dramatic improvement in the performance of the INADEQUATE experiment. About the only problem we've encountered with the new amplifier is the embarassment caused when we try to run other nuclei and forget to switch back to the standard broadband power amplifier.

Best regards,

Gary E. Martin Larry D. Sims Andrew S. Zektzer

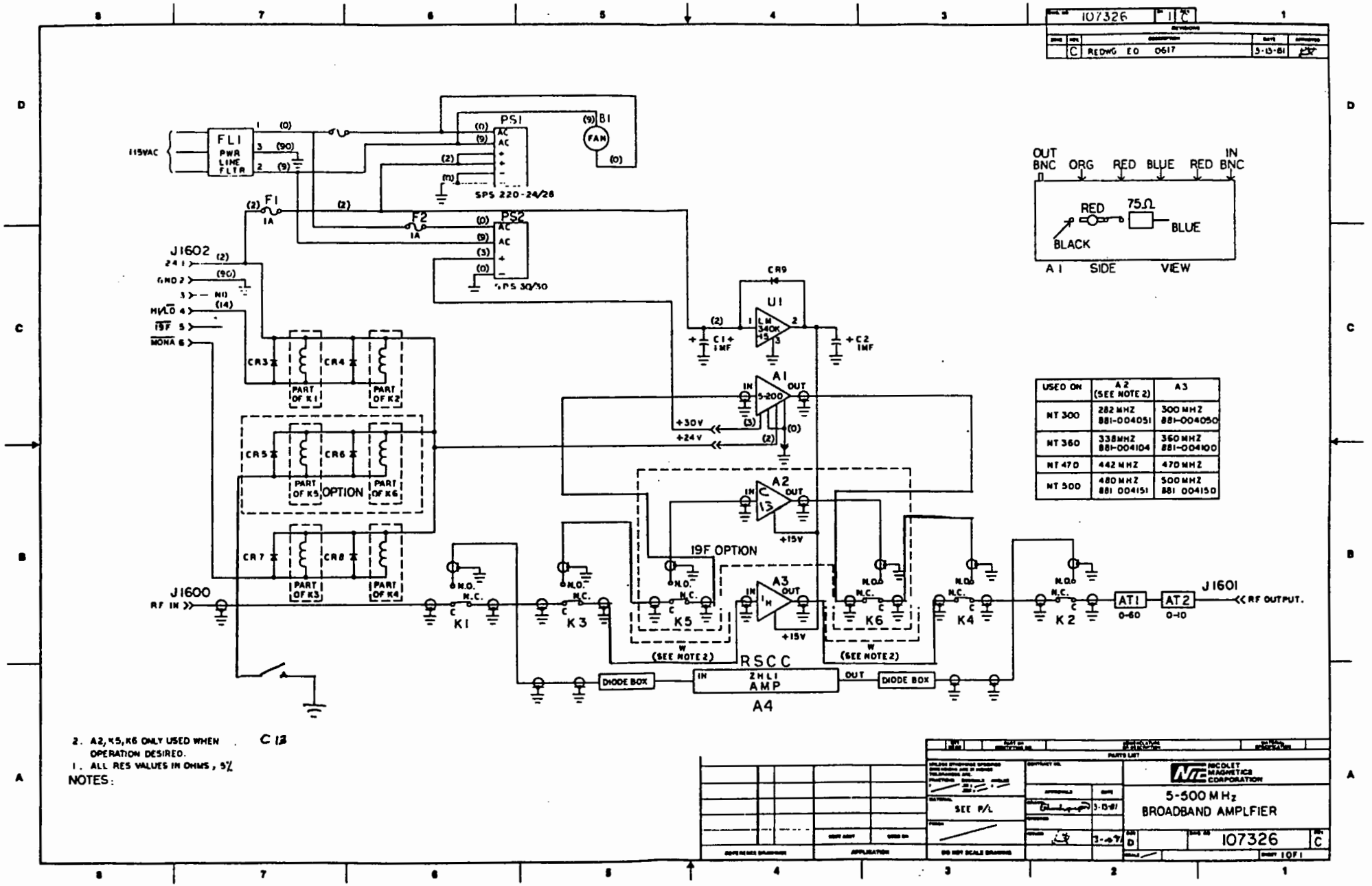

Figure 1. Block diagram of NT-300 showing location of modification (enclosed in dashed line).

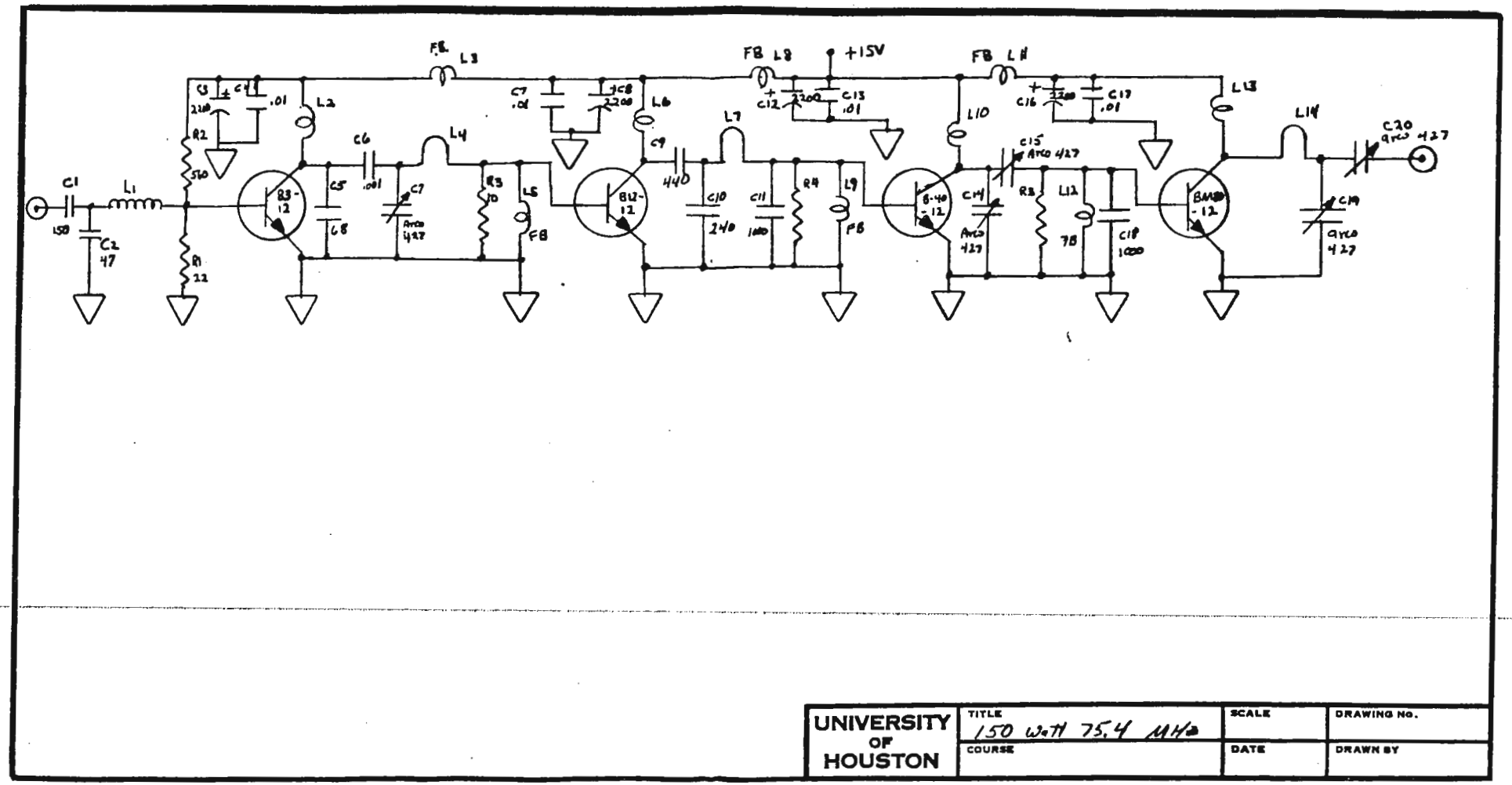

Figure 2. Schematic of dedicated ^{13}C amplifier used to replace the standard broadband power amplifier in generating carbon-13 pulses.

ABBOTT

Abbott Laboratories
Abbott Park, Illinois 60064

February 10, 1988
(received 2/13/88)

Professor Bernard L. Shapiro
Department of Chemistry
Texas A&M University
College Station, Texas 77843-3255

Re: Tweaking an AM500

Dear Professor Shapiro:

A year has already passed since our new Bruker AM500 arrived at Abbott Laboratories. In this brief letter, we summarize some of our experiences.

We are very pleased with the NOESY spectra of proteins acquired on our Bruker. Fig. 1 shows a 200 ms NOESY of a 0.2 mM (that is not a typo) H_2O solution of a 75 a.a. protein, acquired in 60 hours. This was not for testing purposes, but necessary since not more than 1 mg of this special mutant was available to us. Phase coherent decoupling for this experiment was obtained by feeding both frequency generating boards from a single PTS source using a power splitter (Minicircuits).

These good spectra did not come easily, however. In the beginning, we had much trouble with noise parallel to the diagonal (criss-cross noise) in our 2D spectra, which in some cases totally obliterated all cross peaks. After a long search, we finally traced the problem to phase instability of the 451 MHz IF receiver by feeding the observe frequency generating (TF1) board output signal into the receiver input and watching for changes in the obtained d.c. level. Large, random phase changes were observed in this way. Such instabilities result in different phasing for different slices of a 2D experiment and cause the criss-cross noise as we demonstrated in simulated spectra. Once a 451 receiver was installed that exhibited good phase stability, the criss-cross noise disappeared.

After a few years of G.E. experience, we had to (re)learn the tricks to obtain good spectra on Bruker systems. Especially for cosine modulated data, it is crucial that the baseline of the spectrum is flat to avoid t_2 type ridges. This is achieved in sequential quadrature when the spectra are acquired with

approximately orthogonal zero and 0° first order phase parameters (see Fig. 2). This is accomplished by setting the receiver reference phase to the appropriate value in the micro program itself (zero order) and by playing with the acquisition delay (first order). (For simultaneous quadrature only the first order needs to be close to zero).

We are generally happy with the flexibility of the process controller (freedom in number of pulses, loops, and delays), but are disappointed to find that overflow problems occur when pulse programs run more than approximately 100 steps of hard-timed excecutables. Even though this limitation can be circumvented in some cases (e.g. by acquiring hypercomplex data using WR #1, IF #1, IP1, LOOP TO 1 TIMES 2, IP1, IP1, IN=1 instead of programming the sequence twice) we are still looking for ways to run experiments requiring composite X decoupling such as GARP combined with other lengthy sequences such as TOCSY. Suggestions are most welcome.

Homospoil pulses are necessary for many jump-return type 2D experiments. However, our system required 50 ms for homospoil and 150 ms for homospoil recovery – characteristics which prevented us from running short τ_m JR NOESYs. A simple fix consisted of the replacement of the gradient capacitor in the shim unit with one of 30 fold less capacitance. With this modification, carried out in 10 minutes, we obtained good 5 ms homospoil and 25 ms recovery times.

We also have encouraging results for proton-detected heteronuclear correlation and isotope-edited proton NMR experiments. These experiments are set up by having the heteronuclear decoupler signal and X-transmitter both lead to the X-coil of the reverse probe through a power combiner (Werlatone). In this way, we have a versatile 3 channel spectrometer (Reverse mode ^{1}H decoupler, X-pulse BSV-7 and X-decouple BSV-3). The HMQC and isotope-filtered proton NMR experiments can be performed with short ^{1}H and X pulses; the X-decoupler power can be set freely and allows decoupling of the full ^{13}C spectrum without severe heating of the sample.

Sincerely Yours,

Stephen Fesik Robert Gampe David Nettesheim

Edward Olejniczak Erik Zuiderweg

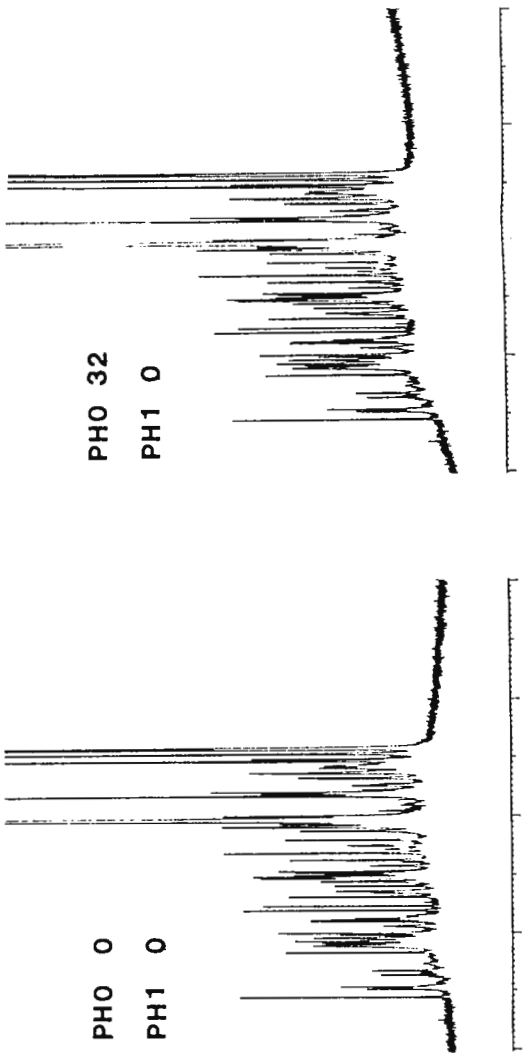

FIG 2

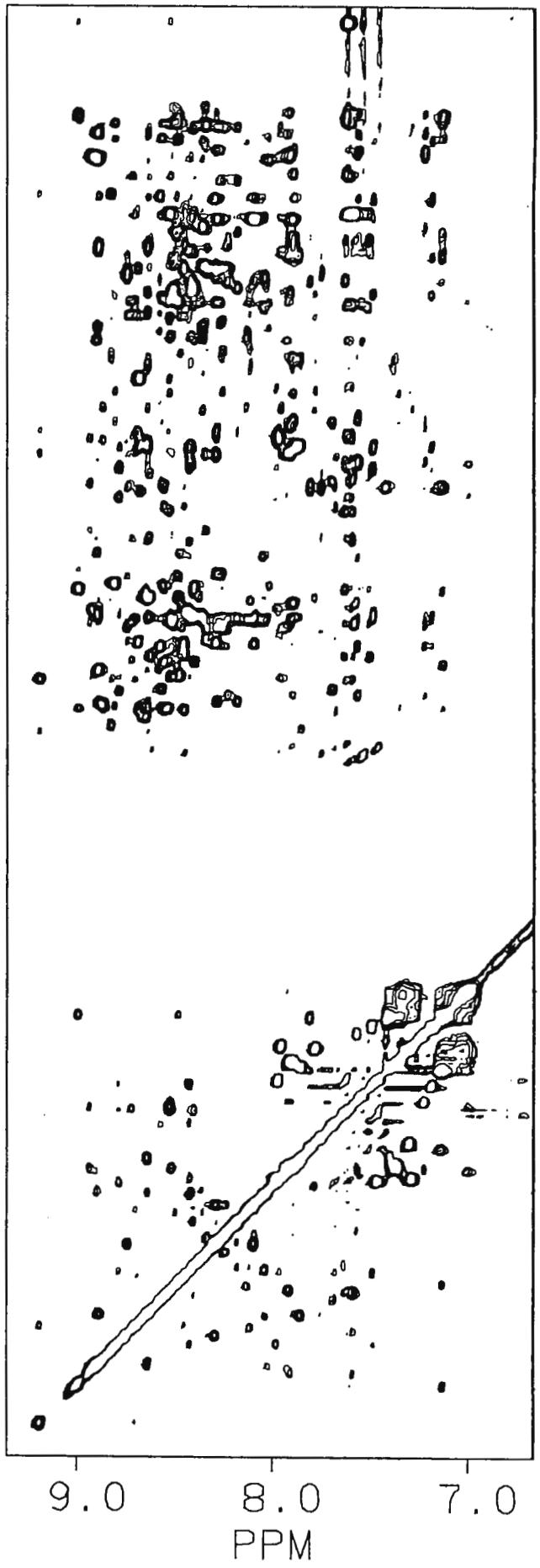

FIG 1

354-30 UNIVERSITY OF TURKU

Kalevi Pihlaja, Dr.
Professor
Physical Chemistry

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

Turku, February 8, 1988
(received 2/20/88)

Dear Professor Shapiro,

A handy stirring system for samples with cells in vivo

When studying living cells by means of ^{31}P NMR spectroscopy it is difficult to keep the sample homogeneous during the measurement. The cells tend to precipitate since the acquisition time has to be relatively long due to the low concentrations of substrates actually to be measured. There is also another problem with living systems: The sample becomes unaerobic during the measurement causing the death of cells and this ruins the experiment. Furthermore to avoid gathering of cells to the walls of the sample tube and even their possible breaking due to the centrifugal forces, a normal spinning cannot be used.

We did find a very simple means to overcome these problems: By bubling air through the sample during the measurement we do not even need to rotate the sample. In practice this can be easily achieved by leading a capillary GC column into the sample tube. There are two clear advantages of using this kind of approach: (i) the gas flow is easily controlled because of the small diameter of the column and (ii) the column is flexible enough to allow the lowering of the sample into the magnet. A disadvantage is, of course, the extra line broadening caused by the paramagnetic oxygen. Since the resonance lines for samples with e.g. living cells tend to be broad anyway this is not, however, too serious.

Sincerely,

Jorma Mattinen
Åbo Akademi

Kalevi Pihlaja

Karl Åkerman
Åbo Akademi

Department of Chemistry
SF-20500 Turku, Finland

Telephone 921-645705
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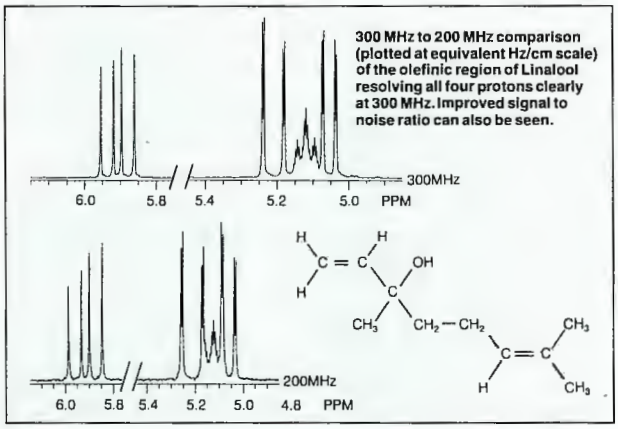

300 MHz to 200 MHz comparison (plotted at equivalent Hz/cm scale) of the olefinic region of Linalool resolving all four protons clearly at 300 MHz. Improved signal to noise ratio can also be seen.

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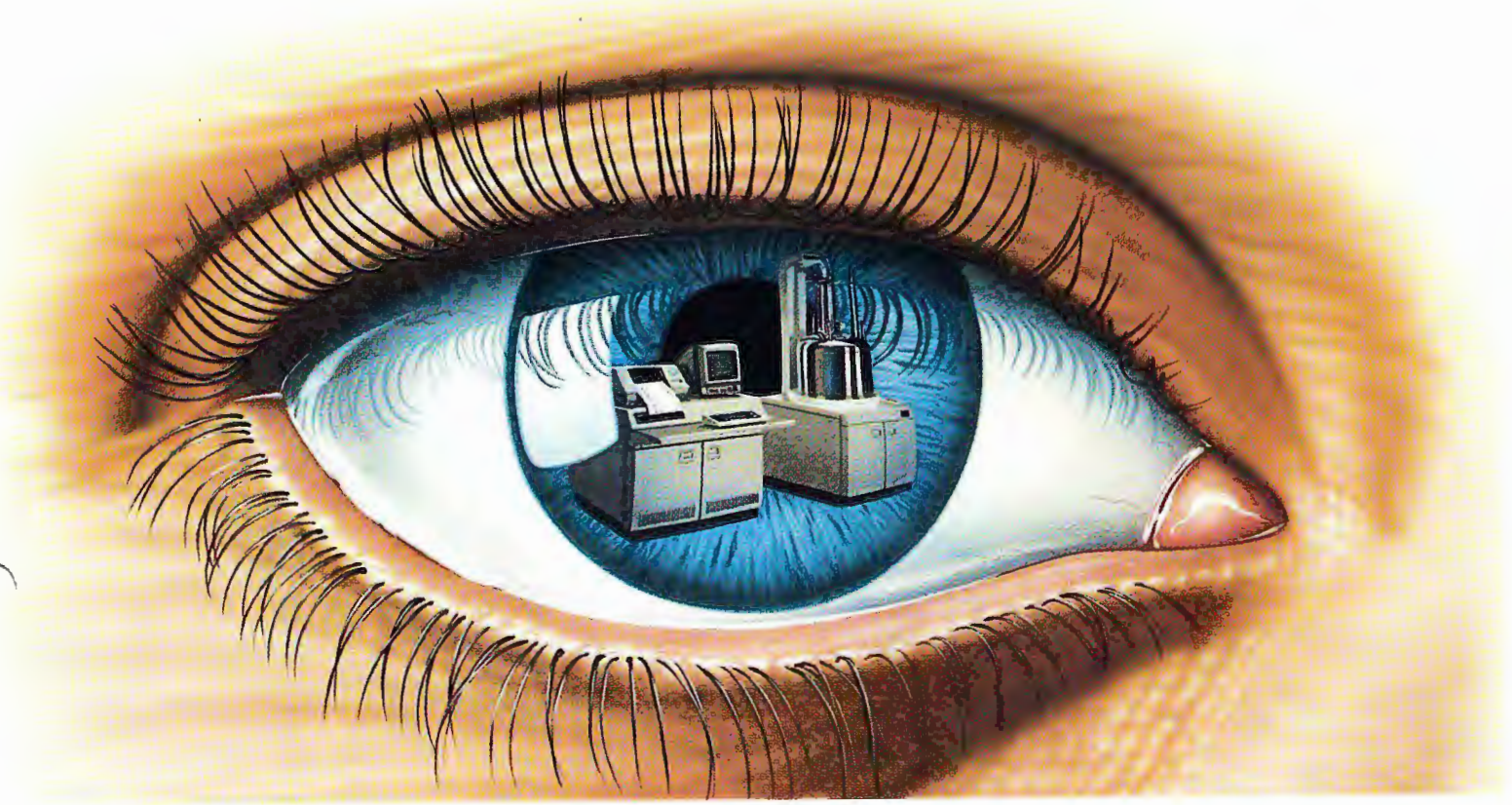

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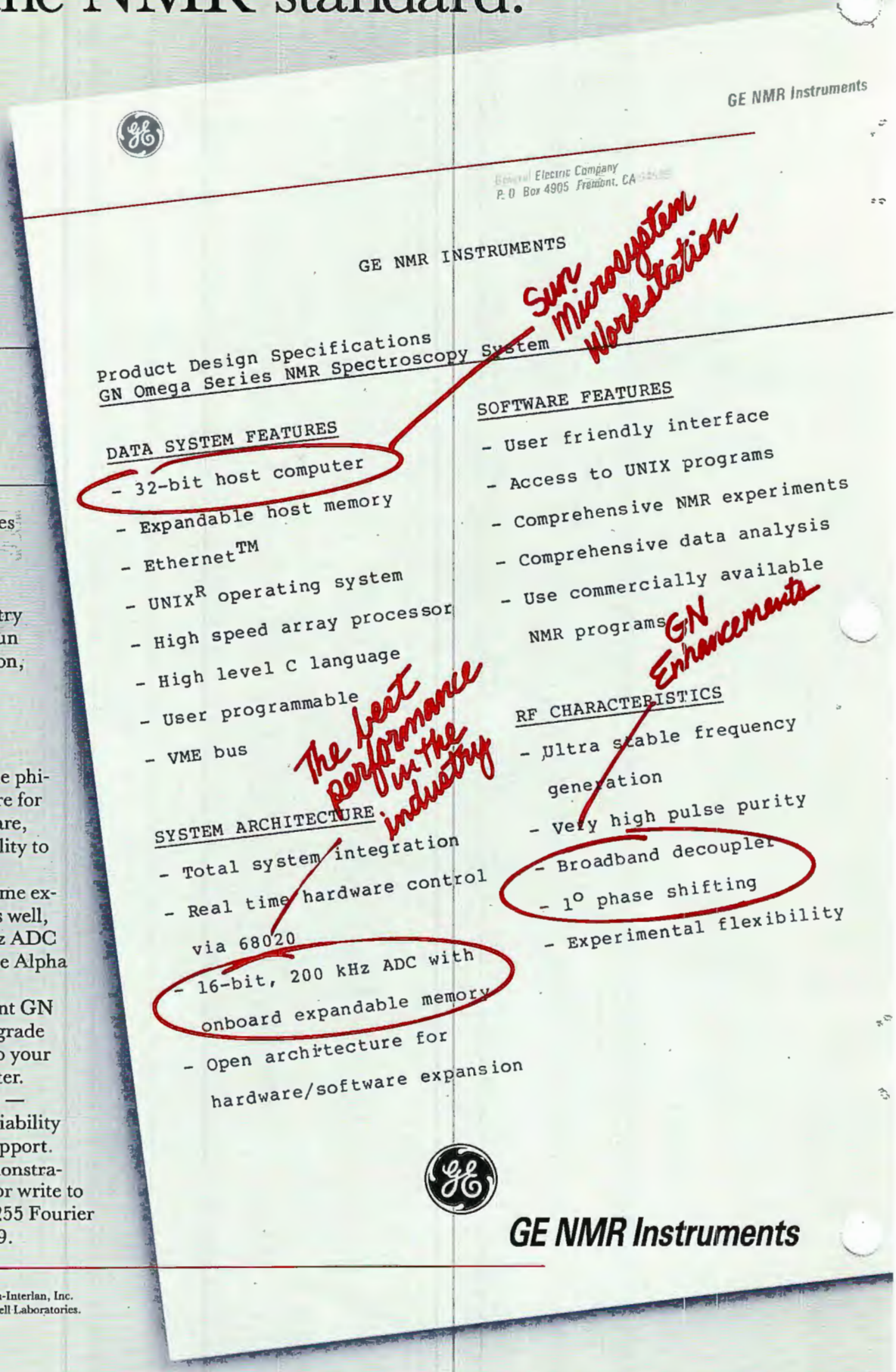

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Smith Kline &French Laboratories
A SMITHKLINE BECKMAN COMPANY

4. February 1988
(received 2/8/88)

Prof. Bernard Shapiro
966 Elsinore Court
Palo Alto CA 94303

Gray Scale Enabling of NDS Terminals: Benefits for Hare Programs

Dear Professor Shapiro,

Users of the programs **FTNMR** and **Dspace**[1], both written by the famous Dennis Hare, will appreciate getting the most out of their NDS GP-29 and GP-220 terminals[2]. One little-known bonus of the graphics on these terminals is their gray scaling abilities that, when enabled, allows one to get "colors" on a monochrome display. This contribution should make it clear how to set up the terminal to get gray scales, and some benefits when using **FTNMR** and **Dspace.**

On the GP-29, set the memory planes to *combined* and the GP-29 Tektronix extensions to *enabled* on the "General" set up page. For the GP-220 the same functions should be set on the "Operating Modes" and "Graphics" set up pages, respectively. These parameters can be saved using the "Home" set up page.

The changes will allow the user to draw different intensities on the screen; the intensities are controlled by the **FTNMR** *pen* and **Dspace** *color* commands. For example, one probably would like to draw DQF-COSY data such that the positive and negative contours are different intensities. To do this, set the following parameters: *pen 3, cyc 2, cpn 1.* Alternatively, one may want to draw contour plots with peak intensities "color-cued". Here, set *cyc* to 3 or 4. The benefits are obvious, but there are some drawbacks: text/graphics changes delete the memory (since they are now combined), and the cursor appears dim (though not to the point that it isn't useful). **Dspace** users can have atoms drawn with differing intensities by modifying the NDSxxx.DEV file; set the colors to pen numbers 1-4. The change is extraordinarily helpful for visualizing structures.

I am told by David Wemmer that proud owners of GP-220 terminals with the 8-plane memory extensions might also figure out how to set up their terminals to overlay different plots–very useful if you want to compare different 2D spectra or different sample spectra.

Please credit this to Luciano Mueller's account.

Sincerely Yours,

Paul L. Weber

[1] Hare Research Inc., 14810 216th NE, Woodinville WA 98072
[2] Northwest Digital Systems, PO Box 15288, Seattle WA 98115 (215)542-0014

354-34

Route 1 at College Farm Road
P.O. Box 191
New Brunswick, New Jersey 08903

The Squibb Institute
for Medical Research

January 29, 1988
(received 2/1/88)

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

Quantitative ^{31}P NMR

Dear Dr. Shapiro,

Recently, the Analytical R&D department of the Squibb Institute acquired a Bruker AM-250 spectrometer to be primarily used for quantitative and solid state NMR analysis. One of the first projects undertaken involved the use of ^{31}P NMR to support the method development of an HPLC assay to determine the purity of various batches of (4-phenylbutyl) phosphonic acid dichloride. Unfortunately the dichloride sample, synthesized by the action of oxalyl chloride upon (4-phenylbutyl) phosphonic acid, is very reactive and corrosive which prevented direct HPLC assay. Therefore, for HPLC assays, the sample had to be derivatized to the dimethyl ester by reacting the dichloride with anhydrous methanol. Obviously, this is unnecessary for the NMR experiment since the various phosphorus containing impurities and final product would display different chemical shifts.

Before utilization of the inverse gated decoupling pulse sequence for quantitative ^{31}P NMR measurements, the chemical shifts and spin-lattice relaxation times for all possible impurities and the final product were measured. The standard inversion recovery experiment was used to measure T_1 relaxation times. Deuterated chloroform was used as a lock solvent and each sample externally referenced to 85% H_3PO_4. All liquid samples were run neat by utilizing a 5 mm NMR insert tube with the lock solvent contained within the 10 mm tube. Any solid state samples were directly dissolved in the lock solvent and placed within the 10 mm NMR tube. Table I lists the chemical shifts for the various compounds/impurities. The dichloride sample displayed the longest T_1 time equalling 1.47 seconds. Consequently, a pulse delay time of 7.5 seconds was used for all data acquisition.

In order to precisely and accurately measure the peak areas of the various resonances, Lorentzian curve fitting was utilized. After transferring data to the MicroVAX-II system, the curve fitting package contained in NMRi's NMR1 software package was used for peak area determination (1). Figure 1 displays the experimental spectrum along with the individual components used to make up the composite modeled spectrum. This particular batch of dichloride was determined to have a purity level of 98.0% with

regard to any phosphorus containing impurities. Unfortunately, these results did not correlate with the initial HPLC assay (92.0%). After repeated sampling by HPLC it was discovered that the anhydrous methanol contained a small amount of water which reacted with the dichloride to produce the mono-acid, mono-chloride intermediate which was not detected by HPLC techniques. After subsequent drying of the methanol, the HPLC results directly correlated with the NMR results.

Future correspondences to the TAMU NMR newsletter will include a number of NMR software programs which have been modified to be used with the GKS graphics system on our MicroVAX and also the use of solid state NMR for pharmaceutical analysis.

Sincerely,

David E. Bugay

(1) NMR1 is a trademark of New Methods Research, Inc., Syracuse, NY.

Table I

Compound	Sampling Condition	Chemical Shift
diacid	in $CDCl_3$	38.4 ppm
dichloride	neat	50.8
mono acid/mono chloride intermediate	in dichloride solution	46.0

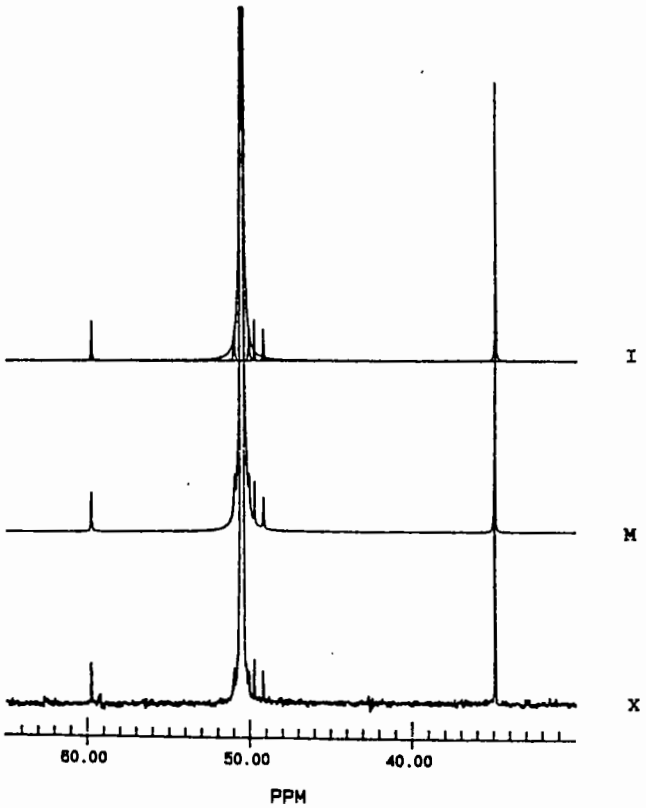

Figure 1. X - experimental spectrum, M - composite modeled spectrum, I - individual components. Dichloride resonance at 50.6 ppm (^{31}P-^{13}C coupling of 48 Hz), diacid resonance at 35.01 ppm (shift due to solvent effects), unknown impurities at 59.85, 49.82, and 49.28 ppm.

Research Centre
P.O. Box 5000
Kingston, Ontario
Canada, K7L 5A5

DU PONT CANADA
DU PONT CANADA INC.

1988 January 25 (received 2/1/88)

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

Dear Barry,

Polyethylene Packing Changes Induced By Sample Preparation

We recently had cause to look at samples of high density polyethylene highly filled with aluminum trihydrate. The samples had been extruded as sheets, rolled and drawn. The ^{13}C CP MAS spectra of the sheet (Fig. 1a) showed the expected crystalline peak at 32.9 ppm and an amorphous peak at 31.1 ppm. There was also a small resonance at 34.4 ppm which has been observed in highly oriented PE systems and attributed to chains with monoclinic packing[1] (the 33.9 peak arises from chains with orthorhombic packing). Static spectra of the sheet showed substantial orientation, so the presence of the 34.4 resonance did not seem unreasonable.

Samples were then scraped off the surfaces of the sheet and from the centres of the sheet to determine if the filler levels were constant. The ^{13}C CP MAS spectrum of samples scraped from the centre are shown in Figure 1b. There is clearly a much larger 34.4 resonance in this spectrum than occured in the whole sample, and this was found in all of the scraped samples, regardless of where they were scraped from in the sheet. It would appear that the resonance arises as a consequence of the sample preparation. The scraping/cutting which occurs may introduce new stresses and/or orientation which causes the chains to assume the new crystallographic form, much in the same way that packing changes occur during drawing.

This is my last contribution to TAMU for the time being. I will be working on reactive extrusion of polymers for the next while and Mike McKinnon will be making future TAMU contributions.

Eric

Eric Kelusky

Ref. 1. VanderHart, D.L. and Khopury, F. Polymer, 25, 1589 (1984).

P.S. Barry, it is -30C in Kingston today (and the windchill is worse). Any thoughts of coming back to this part of the world.?

Eric - Surely you jest!

BLS

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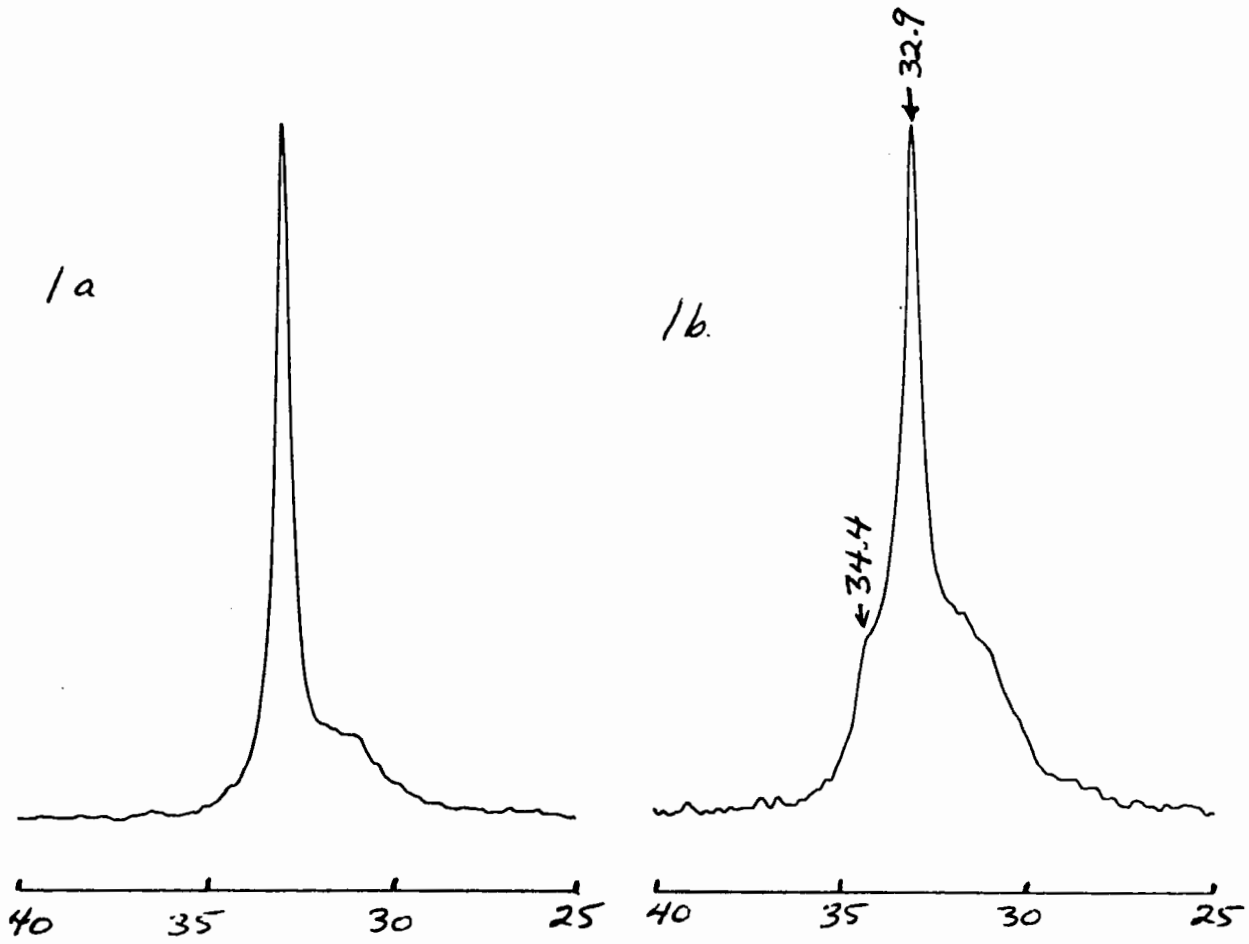

Figure 1) ^{13}C CP/MAS spectra of (a) ATH filled PE sheet and (b) sample scraped from the filled PE sheet. Linefitting indicates that 10% of the total intensity is in the 34.4 resonance in 1b while less than 1% of the total intensity occurs in that resonance in 1a.

POSTDOCTORAL FELLOWSHIP AT SYRACUSE UNIVERSITY - S.U.N.Y. HEALTH SCIENCES CENTER, SYRACUSE, NEW YORK

Biological applications of ^{31}P and ^{1}H NMR.

Currently active projects focus on NMR spectroscopy of living cells:
Steven J. Scheinman, M.D., Assistant Professor of Medicine – 31P NMR spectroscopy of cultured renal cells in suspension; metabolic changes in cell adaptation and with manipulations of phosphate transport. Robert A. Levine, M.D., Professor of Medicine – ^{1}H and ^{31}P NMR spectroscopy of isolated gastric epithelial and parietal cells, and red blood cells; metabolic changes and membrane effects of exposure to ethanol.

NMR facilities:

Under the direction of George C. Levy and Nik Szeverenyi, imaging and spectrosopic facilities include four high-field systems, 150-500 MHz, equipped for "all-nucleus" operation. In addition, there is a GE 2.0 Tesla 32 cm bore multi-nuclei chemical Shift Imager. Data processing facilities and software are uniquely powerful and collaborative research projects with radiologists and others are encouraged.

Interested candidates should write to Professor George C. Levy, Department of Chemistry, Bowne Hall, Syracuse University, Syracuse, New York 13244-1200 and have reference letters forwarded separately.

The University of Texas Medical Branch at Galveston

Medical School
Graduate School of Biomedical Sciences
School of Allied Health Sciences
School of Nursing

Marine Biomedical Institute
Institute for the Medical Humanities
UTMB Hospitals at Galveston

January 27, 1988
(received 2/5/88)

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

Dear Barry,

"IMPROVED PROBE TUNING"

When tuning the probes of our JEOL GX270 NMR spectrometer we have had difficulty at times because of the limitation of the LED. This contribution is targeted at spectroscopists who may have an older NMR spectrometer with an LED or VU meter read out for probe tuning. While this type display works fine for minimizing reflected power it does not allow for clear, concise optimization of tune and match capacitance when tuning over a broad frequency range with various solvents that have different RF characteristics.

Presented here is a suggested hardware set-up that can be used with the probe in the magnet or as a bench top apparatus.

The hardware needed consists of an x,y-oscilloscope; a frequency sweep generator and a VSWR bridge. Specifically, our lab uses a 50 MHz Tektronix model #2225 for the x,y display needed, but many older scopes that have fallen into disuse have the x,y display capability. The sweep generator of choice for us is the Wavetech model #1062 with harmonic type frequency markers of 1, 10 and 50 MHz up to the frequency limit of the instrument (400 MHz). A good commercially available VSWR bridge is the model #62BF50 (10MHz to 1000 MHz) from Wiltron company (be sure to specify 50 ohm impedance). Set-up is depicted in the block diagram.

The tuning network has given us good service in determining tune and match optimum settings for a wide range of sample volumes and constituents for all of our NMR probes.

Yours truly,

Robert Lipsey Felix Salinas III Edward Ezell

ELE/tw

Please credit to acount of Dr. Leland L. Smith.

AT THE 29th ENC

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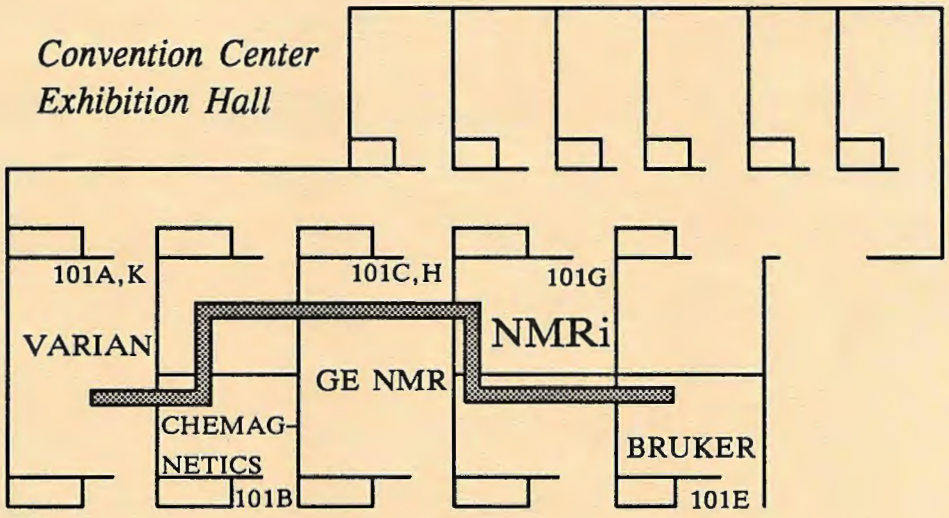

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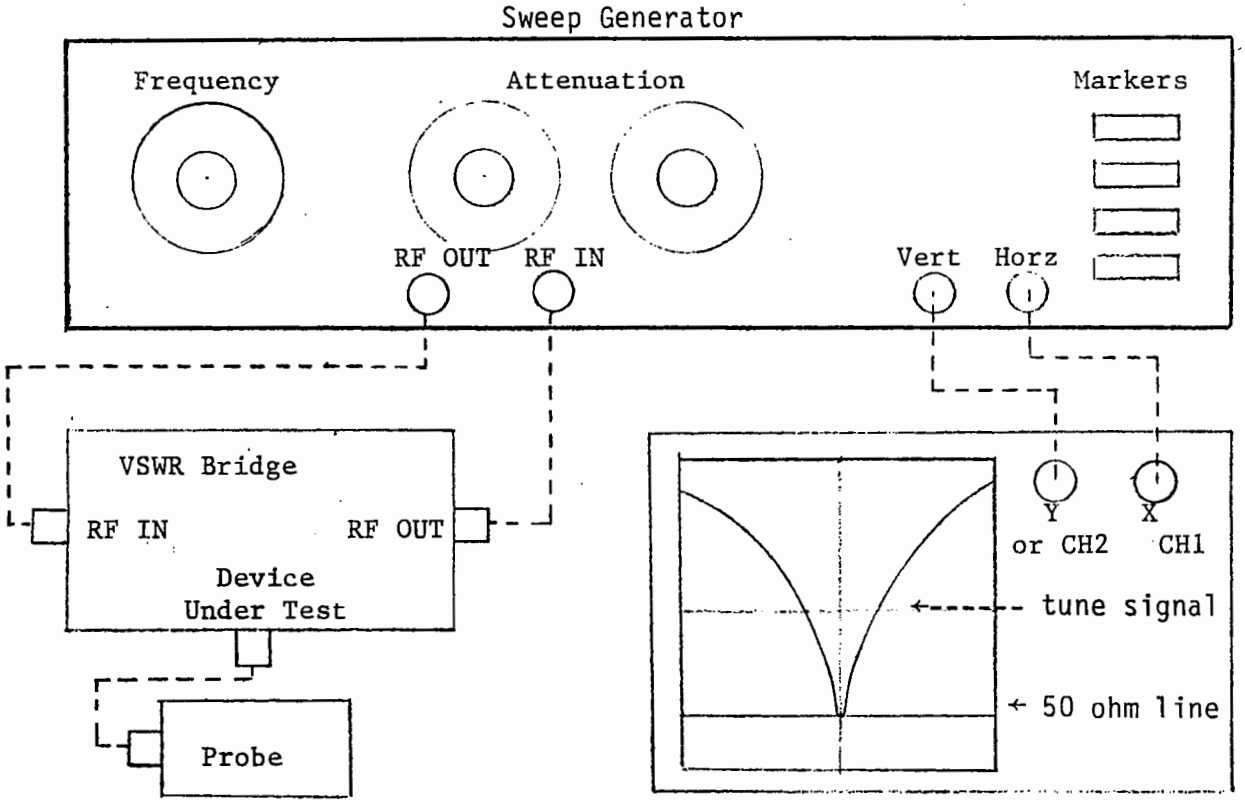

All Coax Belden RG 58 A/U

	Device	Approx. Cost
1)	Wavetech model #1062 with 1, 10 and 50 MHz Harmonic type markers; 50 ohm; 1-400 MHz	$2,065.00
2)	Wiltron VSWR Bridge model 62BF50 10 MHz to 1000 MHz; 50 ohm	$ 250.00
3)	Tektronix model #2225 oscilloscope	$ 900.00

DEPARTMENT OF BIOCHEMISTRY
UNIVERSITY OF CAMBRIDGE

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CAMBRIDGE CB2 1QW
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Dr. Bernard L. Shapiro,
Editor, TAMU NMR Newsletter,
966 Elsinore Court,
Palo Alto,
California 94303,
U.S.A.

26 January, 1988
(received 2/12/88)

Dear Barry,

Customized Microcomputer NMR Databases

Most NMR spectroscopists will be aware of the several large databases written on microfiche or stored in main frame computers. These suffer from a number of operational drawbacks, namely they (1) tend to be expensive; (2) are comprehensive and from an individual viewpoint therefore contain redundant information and (3) are not very portable and/or access to them may be limited.

The microcomputer is inexpensive, can easily be moved from room to room and a user can make a limited database targeted to the needs of an individual or group without having to justify a large capital investment. However, microcomputers do have the following limitations: (1) Memory and access speed are limited; data therefore has to be stored efficiently and floppy or hard disks used as virtual memory. Software is divided into several program segments (overlay files) which are moved on and off disk as required. System parameters are held in a separate area of memory or on disk; (2) There is no garbage disposal; certain areas of memory can be made reavailable only when memory is cleared as a block; (3) The most available high level language is "BASIC".

We describe here the way in which we have overcome these limitations. The computer chosen was the BBC microcomputer which is widely used in Cambridge and the U.K. generally. It has a version of BASIC which supports procedures and functions and allows the programmer to easily store system variables. We find that it is perfectly adequate to hold and search our *in vivo* ^{13}C and ^{31}P databases. We decided that a record should consist of:

- the name of the compound,
- a position label for the atom in question,
- the chemical shift value,
- a 2-letter code for the source e.g. "TI" tissue, "FR" free.
- the pH if known, "U" if not,
- the literature reference from which the data was taken.

The "data" record is written with 3 fields only: the "atom label" and "chemical shift" fields as described, and a 4 byte field, each byte of which is a pointer into the "name", "source", "pH" and "reference". This limits the size of any one entry to 255 characters saving approximately 60% of memory in comparison to a simple data storage system as well as speeding up most database search operations by up to a factor of 20. The list of compound names is too large either to search sequentially or to sort into alphabetical order within a reasonable time, so an internal hash table is used.

The following options are available:

A: Add data. New records are stored in internal data arrays. They are placed in the correct sequence by binary search and simple external sorting.
B: Backup up data - specific to the type of filing system.
C: Change a record - using a delete and add sequence.
D: Delete a record.
E: End.
F: Find a compound. Selecting a hash table entry results in a list of all records with that compound name, grouped into sets of unique "source", "pH" and "reference".
G: Global edit.
H: Help text.
I: Identify a peak. A single chemical shift identifies and displays the nearest 12 or 13 chemical shifts in the database. The user can repetitively choose any of these and the program will scan the database for full details and all associated chemical shifts.

J: Judge several peaks. A list of unknown chemical shifts will be matched against the database for possible compound identifications. The user needs to define the possible error (+/- ppm) and the minimum number of acceptable matches for a compound to be registered. The algorithm is simple and will err on the high side when there are closely spaced shifts.

K: Katalogue database. List all the database files.

L: List all details of each record.

M: Management of database structure. The user can change system parameters and remake the appropriate files.

Despite the limitations in hardware, we find such a system extremely useful and use it in conjunction with a card index. Although only a limited amount of data is stored in the computer, the user is rapidly directed towards more extensive references, saving a lot of time on searches. An example of an 'Identify' search is shown below.

^{13}C Database

Shift to identify (ppm)?57.2

Nearest results:-

Item Name	Shift (ppm)
256 glutathione (red)	56.60
257 L-tyrosine	56.60
258 HEPES	56.61
259 L-phenylalanine	56.65
260 L-serine	56.90
261 L-serine	56.90
262 L-tyrosine	56.93
263 L-serine	57.30
264 L-cysteine	57.50
265 L-tyrosine	57.60
266 L-phenylalanine	57.70
267 L-phenylalanine	57.90
268 L-serine	58.00

Item No - ?265

L-tyrosine

source = FR
pH = 7
ref = 12/13:Horsley et al '70

152 C3	37.80
265 C2	57.60
428 C6	118.30
457 C5	130.80
492 C7	157.30
552 C1	175.40

By request, copies of disks and/or programs are available on an experimental basis at cost price. We would be grateful for any comments by readers/users. We are grateful to Shell Research Limited for supporting this work.

Sincerely yours

Terry A. Moore Peter G. Morris

Sandia National Laboratories
Albuquerque, New Mexico 87185

February 3, 1988
(received 2/5/88)

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

Quantitative ^{29}Si NMR of Sol-Gel Solutions*

Dear Prof. Shapiro:

We have been using high resolution ^{29}Si NMR to study the reaction kinetics of sol-gel solutions. In addition to a reproducible signal response, our requirements included sensitivity, resolution and a flat baseline for accurate peak integrals of this rapidly reacting system. Our spectrometer consists of a Chemagnetics rf console interfaced to a G. E. 1280 data system and a Nalorac 4.7T wide bore magnet. The central feature of our capabilities is a silicon-free 20mm probe constructed by Cryomagnetics.

Figure 1 shows the spectrum of 2.24M solution of tetramethoxysilane (TMOS) in methanol after it has reacted at 25°C with 1 equivalent of H_2O for 5 minutes under acidic conditions. By using a relaxation agent, $CrAcAc_1$ the 8 FIDs could be accumulated in less than 2 1/2 minutes. Gated ^1H decoupling was used although CW decoupling resulted in an identical spectrum since the ^1H dipole-dipole contribution to relaxation is not significant. For longer reaction times, the number of resonances increase and more repetitions are required. Figure 2 shows the spectrum from 64 FIDs of the same solution after it had reacted for 1 hour (FIDs accumulated from 50 to 70 min). The magnet was used in an unlocked mode. This was usually not a problem since the field drift is only 0.2 to 0.3 Hz/hour. For some of the longer experiments, a "software lock"was used. An internal lock of deuterated water or alcohol was not satisfactory because the deuterium would be incorporated into the silicon structure by reaction or exchange and the resulting deuterium-silicon coupling would broaden the silicon line to several Hz.

Polymethylene oxide NMR tubes were used initially. Recently we have been using quartz tubes [1] from Wilmad which spin better and shim to less than 0.3 Hz with only a minimal amount of first order adjustment of z1 and z2. The integral of the total spectrum was usually reproducible to less than ± 5 % during the course of the reaction. Since the probe is silicon-free, the only correction applied to the baseline was to set the average baseline at each end of the spectrum window to zero. We are now using the RIDE [2] sequence to further flatten the baseline when looking at the latter stages of the reaction when many broad, low intensity peaks are present.

Combining the above techniques we have been able to accurately measure several of the reaction rates of the sol-gel system and have developed a model which predicts the concentration of the various

silicon species. High resolution and quantitative measurements were also necessary in order to identify the effects of substituents located four bonds from the observed silicon. The results will be reported in the Journal of Non-Crystalline Solids.

Please credit this to the account of Paul Cahill.

Sincerely,

Roger A. Assink
Physical Chemistry and Mechanical Properties of Polymers
Division 1812

*This work performed at Sandia National Laboratories supported by the U. S. Department of Energy under contract number DE-AC04-76DP00789.

1. B. L. Myers-Acosta, private communication. A very approximate measurement gave a T_1 of 25 to 30 min for the quartz tube.

2. P. S. Belton, I. J. Cox and R. K. Harris, J. Chem. Soc., Faraday Trans. 2, 1985, 81, 63-75.

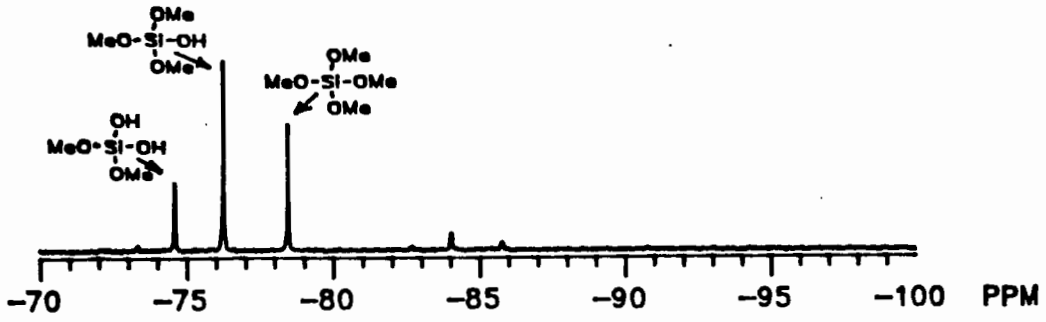


Fig. 1. ^{29}Si NMR spectrum of a 1:1 H_2O:TMOS reaction solution at 5 min.

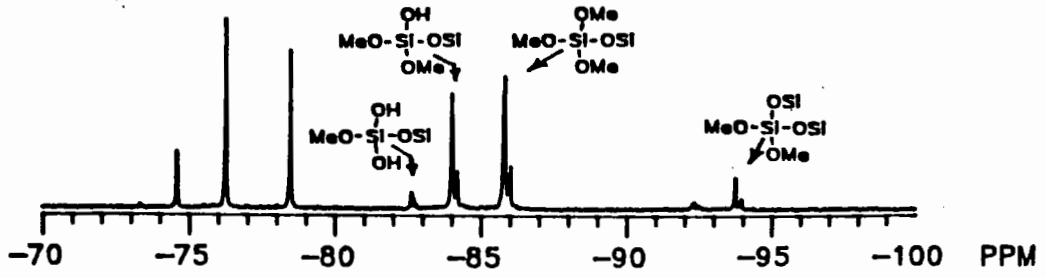


Fig. 2. ^{29}Si NMR spectrum of a 1:1 H_2O:TMOS reaction solution at 60 min.

PROFESSOR DAVID M. DODDRELL

Address: Computer Science Bldg.,
St. Lucia, Queensland,
Australia. 4067

Telephone: (07) 377 4501
Telex: UNIVQLD AA40315

DEPARTMENT OF CHEMISTRY

UNIVERSITY OF QUEENSLAND

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

- 3 FEB 1988
(received 2/6/88)

Water Suppression

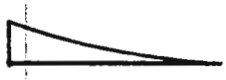
Dear Barry,

As you and your readers are aware, the S/N gain following water suppression is linked directly to the increase in efficiency of analog-to-digital conversion and the ability of the spectrometer's receiver to amplify low-level signals more effectively. The better the suppression, the better the S/N.

We have been looking at the combination of binomial water suppression sequences with gradient-induced destruction of water transverse magnetization as a means to yield the maximum amplifier gain. The pulse train is -

rf $\frac{\pi}{2}(+x)$ - τ - (binomial) - Acq.

Gradient (homo-spoil)

B_0 correction 

$\frac{\pi}{2}(+x)$ is a selective pulse (sinc), while in our initial experiments, a $\bar{1}\bar{3}31$ pulse train was used. The rationale behind this approach is as follows : The 'destruction" of the water signal means that there is less error signal read by the $\bar{1}\bar{3}31$, thus the overall suppression is increased - by typically an extra 30 - 40db. It is possible to achieve water suppression factors of 50,000 using this approach with the observation of 200μM concentrations in neat H_2O feasible. The B_0 correction is employed to maintain high-resolution conditons during data acquisitions. Some results are attached.

Yours sincerely,

D M DODDRELL I M BRERETON L LESTER

PS - is this the first time I have answered the reminder without the joys of an ultimatum ?

Probably.

BLS

NMR Sample Tubes

For evidence of **Norell's high performance quality,** we reproduce **CERTIFICATES** submitted by individuals **who actually tested and compared** our tubes vs those of our competition. SEE OTHER SIDE.

Don't let them fool you!

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

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Our top quality tube!

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

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100-299.....$4.25 ea
300 + ...$4.00 ea.

No. 507-HP HIGH PRECISION for high resolution NMR

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

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

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

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

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

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

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

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

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

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

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

No. 502 THROWAWAY TYPE

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

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

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i.d. 8.76 ± 0.025mm; (0.001 in.)
camber ± 0.007mm (0.0003 in.).
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6-25.....$13.75 ea.
26 +.....$12.95 ea.

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

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

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

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

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

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

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

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I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be: CERTIFICATE **107**

☐ Excellent ☒ Very Good ☐ Satisfactory ☐ Unsatisfactory

Norell's XR-55 NMR Sample Tube was compared with the following competitive tube or tubes: WILMAD CAT. No. 528 PP

(give catg. number and name of manufacturer)

Type of NMR equipment used in the above evaluation NR-80 - NMR

Date JULY 27 1982 Name [illegible] Title Res. Tech.

Address [illegible] STATION

WILMINGTON, DE 19898

Phone: 302-772-3311

We expect your findings from the supplied batch of 100 tubes to be unbbiased and we reserve the right to share your findings with others inquiring about our NMR sample tubes. Norell, Inc. 314 Arbor Ave. Landisville, NJ 08326 USA.

I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be: CERTIFICATE **167**

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Norell's XR-55 NMR Sample Tube was compared with the following competitive tube or tubes: 507 PP

Wilmad

(give catg. number and name of manufacturer)

Type of NMR equipment used in the above evaluation EM 390 90 MHz NMR

Date 3/22/83 Name L. WAYKOLE Title GRA. STUDENT

Address Box #208, Chem Dept

Emory University, Atlanta, GA 30322

Phone: 329-6500 (404)

We expect your findings from the supplied batch of 100 tubes to be unbbiased and we reserve the right to share your findings with others inquiring about our NMR sample tubes. Norell, Inc. 314 Arbor Ave. Landisville, NJ 08326 USA.

I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be: CERTIFICATE **125**

☐ Excellent ☒ Very Good ☐ Satisfactory ☐ Unsatisfactory

Norell's XR-55 NMR Sample Tube was compared with the following competitive tube or tubes: WILMAD 507-PP & 528-PP

(give catg. number and name of manufacturer)

Type of NMR equipment used in the above evaluation VARIAN EM-390

Date 11/3/82 Name J.C. HOWARD Title ASSIST PROF

Address BOX 40 MTSU

MURFREESBORO, TN 37132

Phone: (615) 898-2954

We expect your findings from the supplied batch of 100 tubes to be unbbiased and we reserve the right to share your findings with others inquiring about our NMR sample tubes. Norell, Inc. 314 Arbor Ave. Landisville, NJ 08326 USA.

I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be: CERTIFICATE **111**

☑ Excellent ☐ Very Good ☐ Satisfactory ☐ Unsatisfactory

Norell's XR-55 NMR Sample Tube was compared with the following competitive tube or tubes:

WILMAD 507 PP

(give catg. number and name of manufacturer) OCCI, direct research

Type of NMR equipment used in the above evaluation VARIAN EM 360

Date 4/7/82 Name STEVEN MASUO Title STAFF CHEMIST

Address 2100 S.E. MAIN ST.

IRVINE, CA 92714

Phone: (714) - 957-7350

We expect your findings from the supplied batch of 100 tubes to be unbbiased and we reserve the right to share your findings with others inquiring about our NMR sample tubes. Norell, Inc. 314 Arbor Ave. Landisville, NJ 08326 USA.

I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be: CERTIFICATE **105**

☐ Excellent ☒ Very Good ☐ Satisfactory ☐ Unsatisfactory

Norell's XR-55 NMR Sample Tube was compared with the following competitive tube or tubes: Wilmad 528-PP

(give catg. number and name of manufacturer)

Type of NMR equipment used in the above evaluation WH-250 Bruker

Date 2-8-82 Name L. Overman Title Professor

Address Department of Chemistry

University of California

Irvine, CA 92717 Phone: 714-833-7156

RECEIVED

We expect your findings from the supplied batch of 100 tubes to be unbiased and we reserve the right to share your findings with others inquiring about our NMR sample tubes. Norell, Inc. 314 Arbor Ave. Landisville, NJ 08326 USA.

I have evaluated Norell's XR-55 Precision NMR Sample Tubes and found them to be: CERTIFICATE **126**

☐ Excellent ☒ Very Good ☐ Satisfactory ☐ Unsatisfactory

Norell's XR-55 NMR Sample Tube was compared with the following competitive tube or tubes:

Wilmad 507 PP

(give catg. number and name of manufacturer)

Type of NMR equipment used in the above evaluation Varian EM 360 A

Date [illegible] Name [illegible] Title [illegible]

Address [illegible]

Phone: [illegible]

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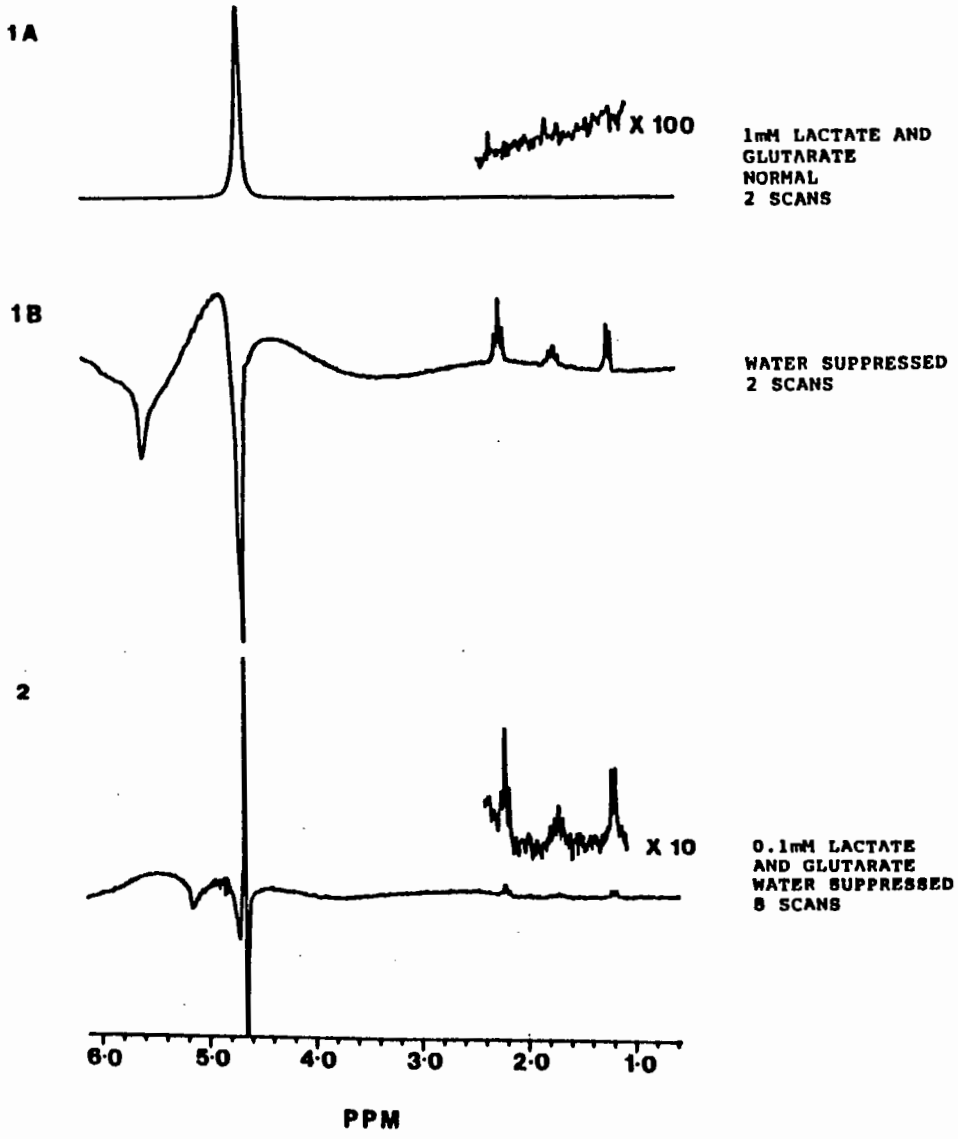
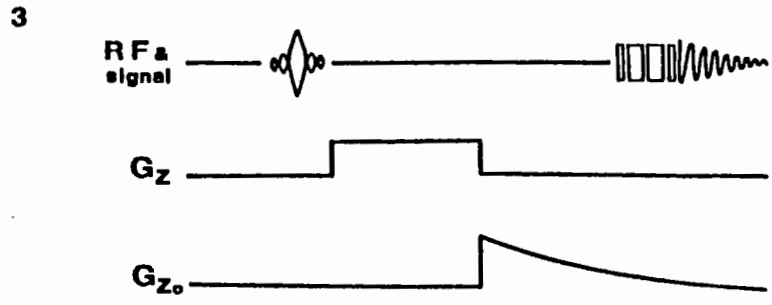
PERFORMANCE

While our competition makes many empty claims ..... we give you instead actual performance test results carried out by NMR Spectroscopists who signed their name to authenticate them. Norell, Inc. 314 Arbor Ave., Landisville, NJ 08326 USA

SUBMERGE – 1331 Water Suppression





SUBMERGE - 1331 PULSE SEQUENCE WITH Zo CORRECTION

OXFORD RESEARCH SYSTEMS

DATE: January 29, 1988
OUR REF: (received 2/8/88)
YOUR REF:

Oxford Research Systems Limited
Nuffield Way, Abingdon,
Oxon OX14 1RY,
England.
Telephone 0235 32421
Telex 83356

OSIRIS, an improved technique for localised spectroscopy

Dear professor Shapiro,

In our previous contribution (1), we illustrated the superior qualities for slice inversion of the complex hyperbolic secant pulse (2). In conjunction with the ISIS technique for localised spectroscopy (3), this pulse retrieves the maximum signal from the Volume of Interest (VOI).

The sensitivity of the ISIS experiment, however, also depends on the dynamic range between the signal from the whole sample and the signal from the VOI, and on the subtraction error. We now report the use of a noise modulated selective pulse (4) to suppress the signal from outside the VOI and thus improving both the dynamic range and the subtraction error.

We have called this technique OSIRIS (Outer volume Suppressed Image Related In-vivo Spectroscopy) (5). Because, as is the case with the hyperbolic secant pulse, the noise pulse is insensitive to the RF power, the OSIRIS experiment is perfectly suited for implementation with a surface coil.

In the figure profiles of a phantom are shown, measured with a surface coil, in which the excitation pulse amplitude is adjusted to cause approximately 270 degree nutation angle in the plane of the coil. (a) Normal profile, (b) Profile with inversion pulse only, (c) Profile with noise pulse only, (d) Profile with both inversion and noise pulses.

A. Connelly*, C. Counsell, J.A.B. Lohman+

*Address per 1-3-88 : Institute of Child Health,
University of London,
30 Guilford street, London WC1N 1EH, U.K.

+Address per 1-3-88 : Philips Medical Systems, PO box 10000,
NL-5680 DA Best, The Netherlands

References:

(1) TAMUN Newsletter, 345, 46 (1987)
(2) M.S. Silver, R.I. Joseph, and D.I. Hoult, Phys. Rev. A, 31, 2753 (1985)
(3) R.J. Ordidge, A. Connelly, and J.A.B. Lohman, J. Magn. Reson. 66, 283 (1986)
(4) R.J. Ordidge, Magn. Reson. Med., 5, 93 (1987)
(5) A. Connelly, C. Counsell, J.A.B. Lohman, and R.J. Ordidge, J. Magn. Reson., in press, (June 1988).

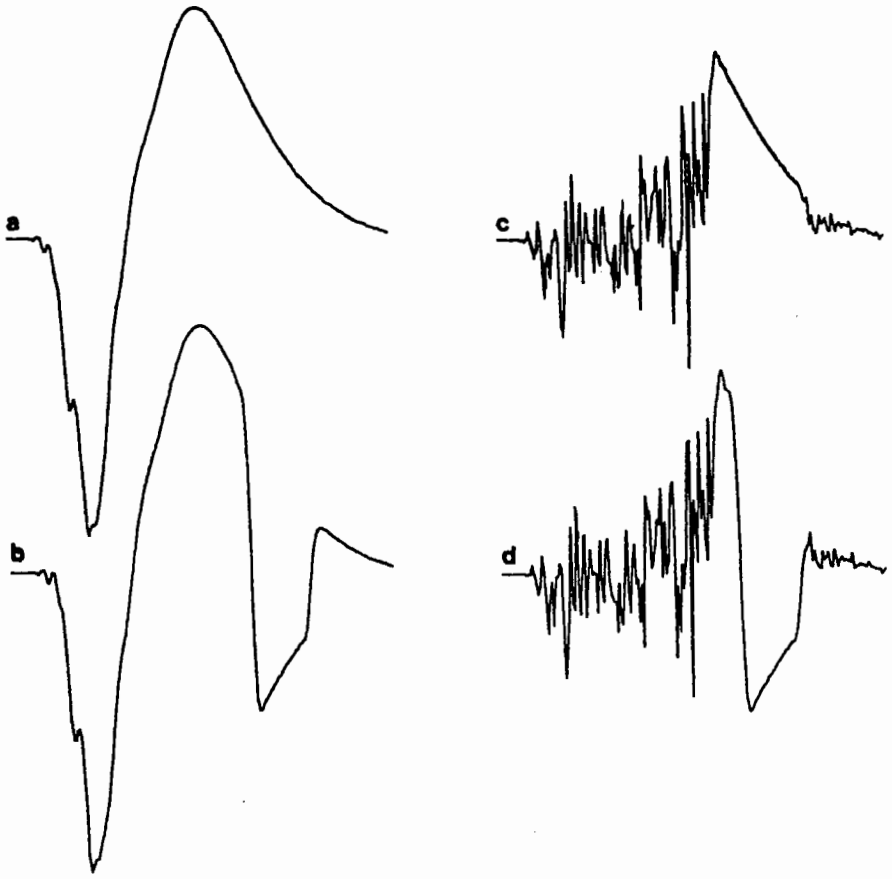


Figure 1 : Surface coil profiles of a phantom in a gradient (a) Normal profile with 270 degrees nutation in the plane of the coil; (b) profile after application of a slice inversion pulse; (c) profile after application of a slice selective noise pulse; (d) profile after application of both the inversion and the noise pulse.

NATIONAL TAIWAN UNIVERSITY *DEPARTMENT OF CHEMISTRY*

ROOSEVELT ROAD SECTION 4
TAIPEI, TAIWAN, REPUBLIC OF CHINA
PHONE: 3935357 · 3935359

Professor Bernard Shapiro
Editor/Publisher, TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, Califoirnia 94303
U. S. A.

February 2, 1988
(received 2/10/88)

Dear Dr. Shapiro:

The work reportd here have been done with Prof. T. C. Wong, at University of Missouri, Columbia, in our continuing collaboration on the study of molecular dynamics of an aggregate system. Besides the usual T_1 and T_2 measured, we have applied multiple quantum relaxation technique to re-examined methyl ^1H and ^{13}C relaxations in micelles of deoxycholate(DOC) at 7.05 T.(1)

In addition to the previous investigation(1) by proton two-quantum relaxation method, zero quantum relaxation techniques(2) are also employed to elucidate the dynamical parameters. In this system, the zero quantum relaxation contains the same information as T_2 process. The pulse sequence is shown in Fig. 1 with phase cycling as described elsewhere.(2)

Brainard and Szabo's cone model(1,3) has been used to calculate the auto and cross correlation functions. In the model it is assumed that the DOC micelle is spherical and have a overall reorientational correlation time $\tau_o=1/6D$, and that of C_3 axis of the methyl group rotates with a correlation time $\tau_i=1/4D_i$. The wobbling motion of the C_3-axis has a correlation time $\tau_w=1/6D_w$ with order parameter S. The diffusion coefficients D, D_i and D_w are defined for overall reorientational motion, the methyl rotation motion and the wobbling motion of C_3-axis, respectively. Isotropic random field model (with spectral density $j(\omega)$) has been used to account for the unspecified relaxation processes in addition to the dipolar relaxation among the methyl protons. The results are shown in Fig.(2). We have found three sets of parameters(Table I) that can fit the experimental data (proton multi-quantum and T_1 relaxations at 7 T) equally well. However, if we consider that the dipolar interactions between C-H is the dominant mechanism for C-13 relaxation we may neglect the contribution due to "isotropic random field". As shown in Table II, the calculated results of T_1 and NOE for C_{18}-13 of DOC do make some distinction between set 1 and sets 2, 3. The proton coupled ^{13}C spectrum can be fitted to a linewidth equation $1/T_2=a+bm+cm^2$ where m is the magnetic quantum number with total spin S=1/2, 1/2 and 3/2 for three protons in a methyl group.(4) The differential line broadening(DLB) effects may then be invoked to differentiate set 2 and set 3.

Sincerely yours,

Lian-Pin Hwang Pei-Lein Wang Der-Ming Duh

Lian-Pin Hwang
Professor of Chemistry

Pei-Lein Wang

Der-Ming Duh

References:

1. Kay, L. E.; Pretegard, J. H. J. Am. Chem. Soc. 1987, *109*, 3829.
2. Wokaun, A.; Ernst, R. R. Chem. Phys. Lett. 1977, *52*, 407.
3. Brainard, J. R.; Szabo, A. Biochemistry 1981, *20*, 4618.
4. Hwang, L. P.; Wang, P. L.; Wong, T. C. (submitted to J. Phys. Chem.).

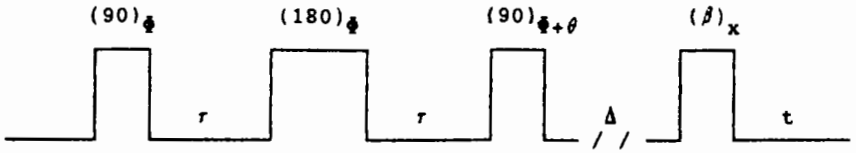


Fig.1 Pulse sequence for multiple quantum relaxation.

Table I	S	τ_o, ns	τ_i, ps	τ_w, ps	j(0)	j(ω)
Set 1	1	3.2	2.7	irrelevant	11.0	-1.40
Set 2	0.785	7.8	2.2	24.0	10.7	-1.07
Set 3	0.600	4.5	1.0	11.0	17.0	-0.94

Note: see Ref.(1) for definitions of j(0) and j(ω).

Table II	T_{1h}	T_{1c}	η_c	DLB C-Term
Experimental Data	0.41	0.71	0.96	3.5
Set 1	0.42	0.37	0.41	7.32
Set 2	0.42	0.71	0.97	9.60
Set 3	0.43	0.74	1.02	3.47

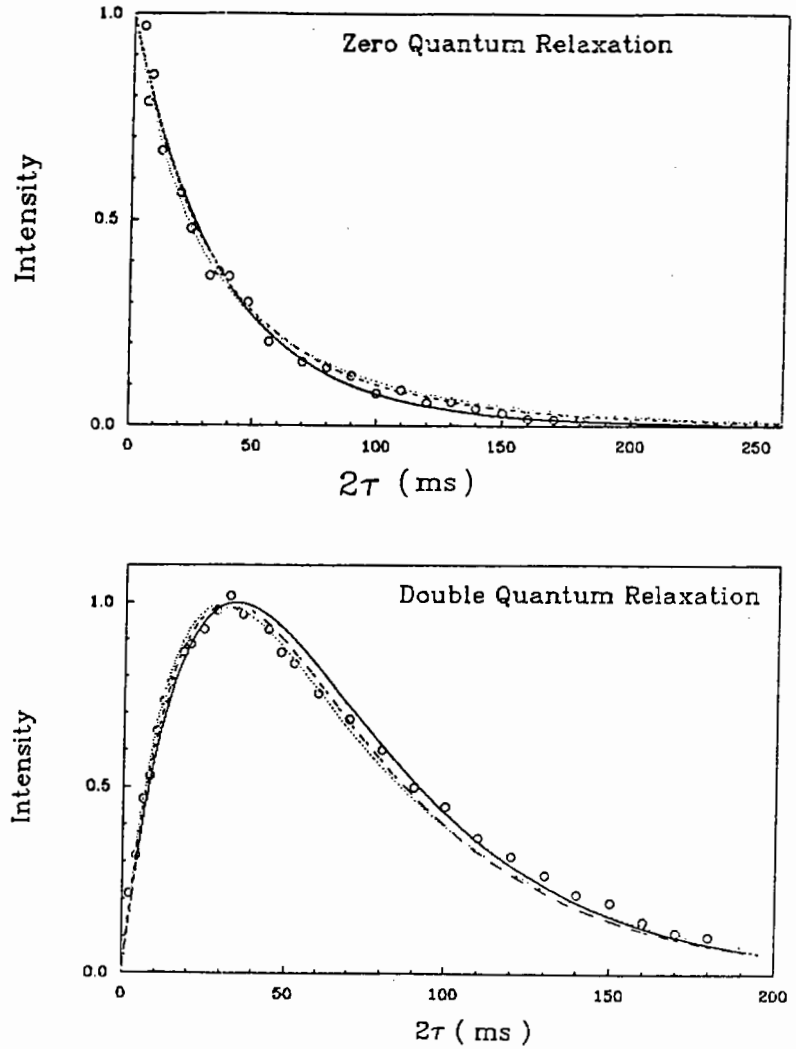


Fig.2 Proton multiple quantum relaxation as a function of evolution time 2τ for methyl #18 of 75mM DOC micelle in pH=9.0 aqueous solution. Dashed, dotted and solid curves are obtained according to set 1, set2, and set3 respectively.

AT&T Bell Laboratories

600 Mountain Avenue
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201 582-3000

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

NMR Microscope Images of a Mouse Brain; Positions Available

Dear Barry:

We have recently installed an NMR microscope accessory for our 360 MHz Bruker widebore spectrometer and want to share with you and your readers some of our initial results.

As you may recall, we are interested in understanding the changes that occur in neonatal rat brains during development. We have built surface coils to measure the amount of various phosphorus-containing metabolites in the rat brain during development, and have compared these levels to those found in cultured neuroblastoma cells [P. Glynn, T. M. Lee, and S. Ogawa, *Ann. N.Y. Acad. Sci.* 508, 474 (1987)]. We see fairly dramatic changes, especially in phosphoethanolamine levels, during critical developmental stages.

We are now attempting to determine whether various metabolites are compartmentalized during development. The initial step in these experiments is to obtain proton NMR images to determine the sort of resolution we can expect. One of our initial images is included in the Figure. It is a coronal slice, passing through the Bregma point, which is somewhat behind the eyes. This image was obtained using 4 repetitions with a 1 second delay, and 256 increments of the phase-encoding gradient. The pixel size is 90μ in the x and y dimensions, and 430μ in the z direction. Although the copy doesn't do it justice, I think you'll agree that the resolution is excellent.

In particular, the lateral ventricals (dark trianglular regions in drawing; dark triangular region near center of enlargement) are clearly visible. Since this scan was obtained in only 17 minutes, images such as these could be used to monitor the time course of drugs or other agents that affect vasoconstriction or vasodilation in the brain.

In addition to learning our new imaging hardware and installing our new SIS (Siemens/Varian) imaging system, we are currently trying to fill two positions; one permanent and the other postdoctoral.

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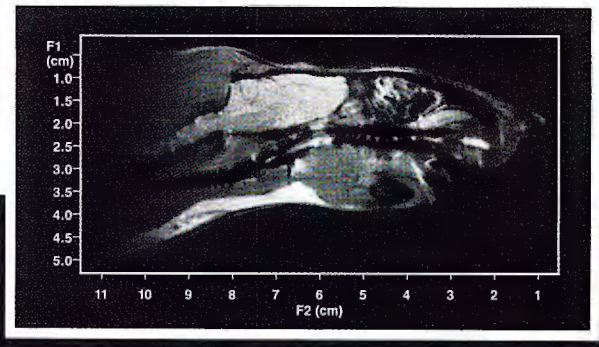

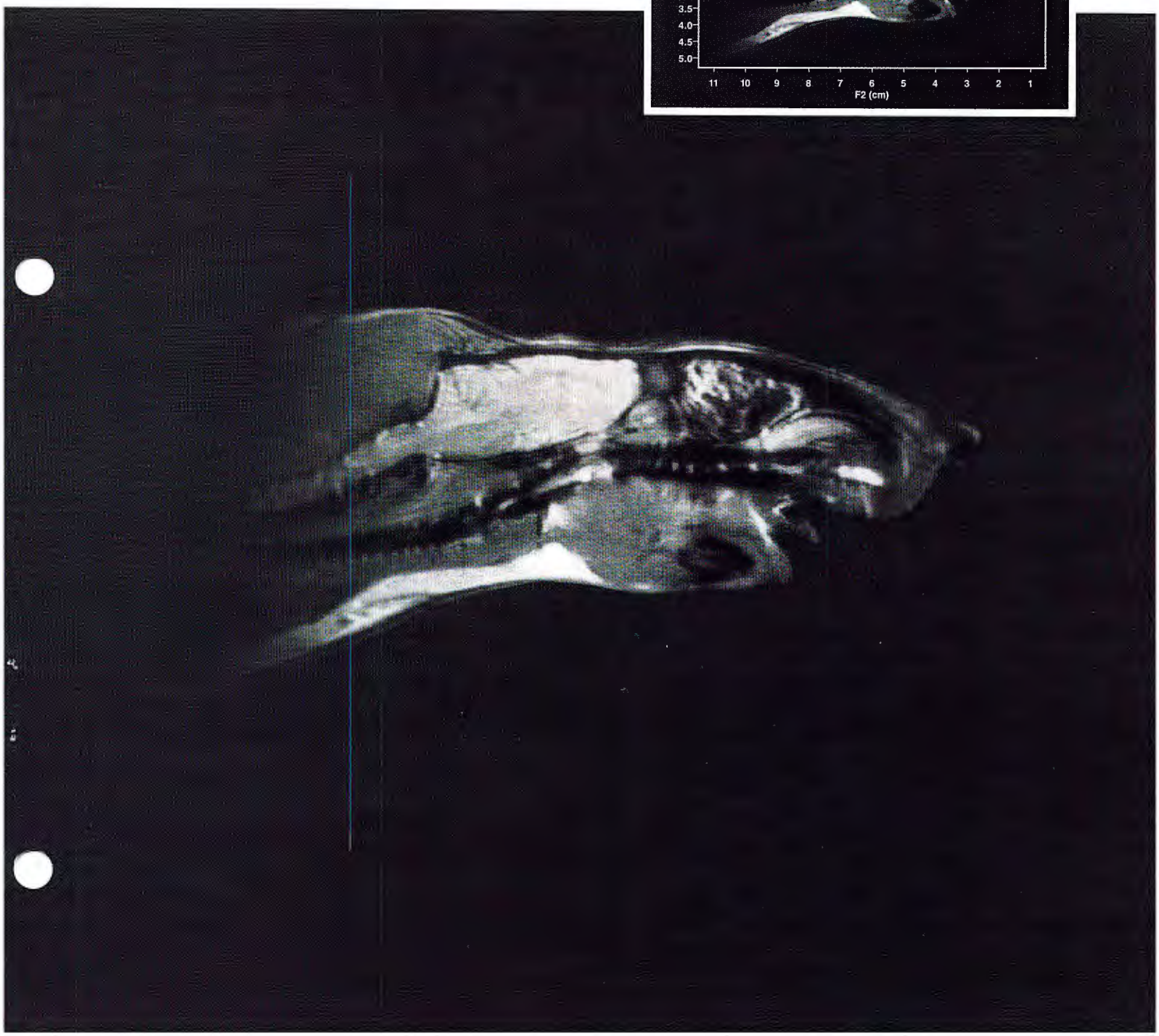

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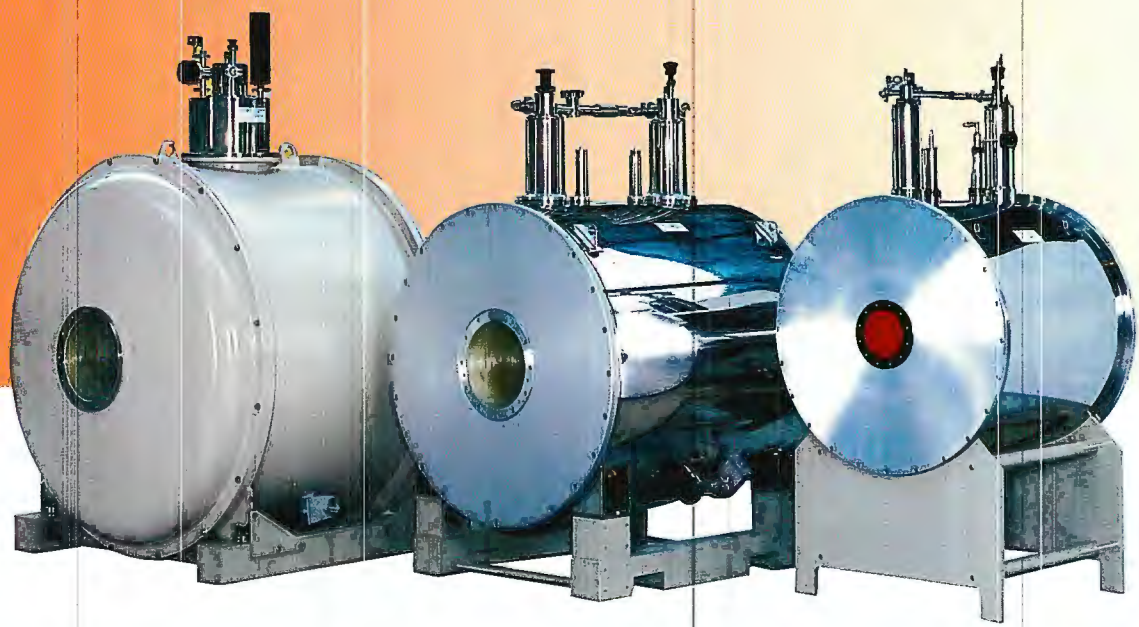
200/330	4.7T	330 mm	254 mm	2.3 G/cm	140 mm DSV ±5 ppm	70 mm DSV 0.1 ppm	6.95 m	5.60 m
200/400	4.7T	400 mm	324 mm	1.8 G/cm	140 mm DSV ±4 ppm	80 mm DSV 0.1 ppm	8.50 m	6.75 m
85/310	2.0T	310 mm	225 mm	3.0 G/cm	100 mm DSV ±5 ppm	70 mm DSV 0.1 ppm	4.50 m	3.63 m

DSV = Diameter Spherical Volume
HHLW = Half-Height Line Width
PPM = Parts Per Million



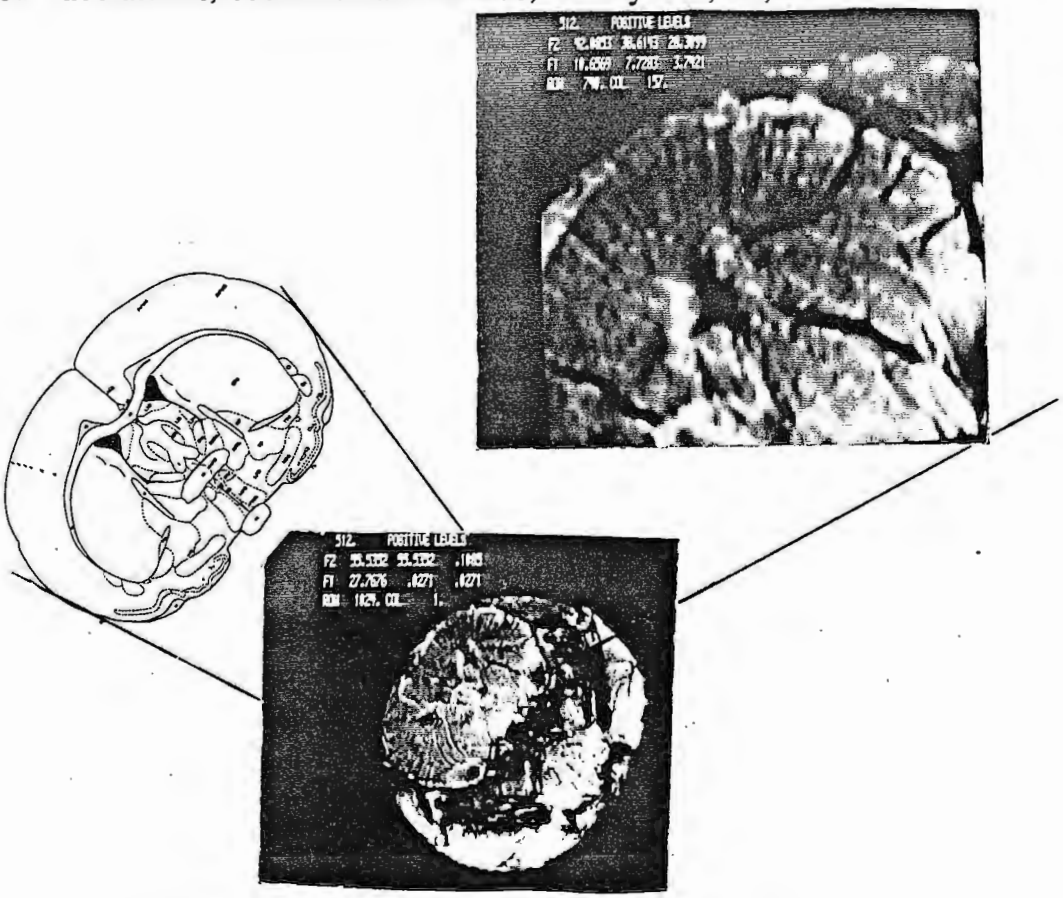
200/400, 200/330, and 300/180 magnets for the NMR Imaging Spectrometer System.
(Photos courtesy Oxford Instruments.)

Spectroscopy Imaging Systems
1120 Auburn Road
Fremont, California 94538
(415) 659-2600

Note: Equipment described is intended for investigational purposes, and is not approved by the FDA for clinical use.

possible. Facilities include a SIS 18.3 cm (room temperature) bore, 7 tesla multinuclear spectrometer and a Bruker NMR Microscope accessory for a widebore 360 MHz high resolution spectrometer. Please send a resume, publications list, outline of proposed research, and the names of three references to L. W. Jelinski, Department Head, Molecular Biophysics Research Department, AT&T Bell Laboratories, 600 Mountain Avenue, Murray Hill, NJ, 07974.

Postdoctoral: High resolution solution and solid state NMR studies of "tethered" biological systems. Some imaging. Send resume, publications list, and names of three references to L. W. Jelinski, Department Head, Molecular Biophysics Research Department, AT&T Bell Laboratories, 600 Mountain Avenue, Murray Hill, NJ, 07974.



Sincerely,

Seiji Ogawa Paul Glynn

Tso-Ming Lee Lynn W. Jelinski

INRA

THEIX
63122 CEYRAT
FRANCE

Professor B.L. Shapiro
966 Elsinore court
Palo Alto
California 94303
ETATS UNIS

INDIRECT DETECTION AND SEU ON BRUKER AM400 SPECTROMETER

Theix, 1988 February 4
(received 2/16/88)

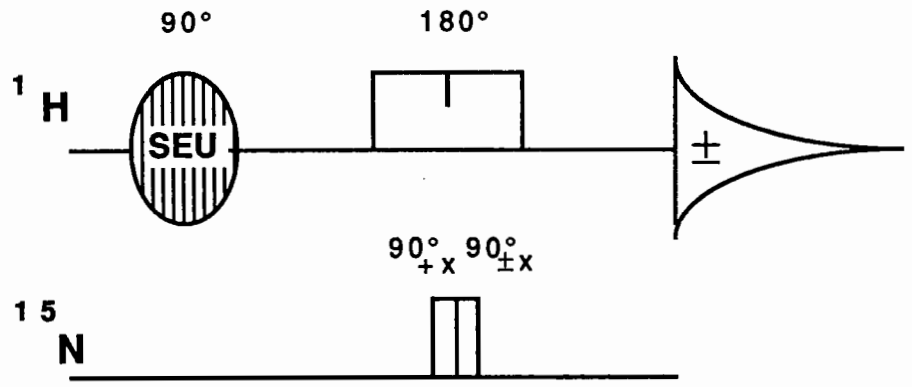
Dear Professor Shapiro,

We are interested in optimising the method for detecting the ^{15}N nucleus in metabolism of branched-chain amino acids. To follow the metabolic pathways, we recorded the ^{1}H spectrum through the effect of the ^{15}N -^{1}H coupling (pulse sheme I).
Last october we received our AM 400 Bruker and tested the Selective Exitation Unit to perform a 90° selective proton pulse and to overcome the strong water signal. At the same time, we can follow in a quantitative way the leucine ß protons and the valine ß proton coupled (3Hz) to ^{15}N nucleus. The labeled (95%) leucine released in the perfusing medium of muscle was thus detected in a less than 3mM concentration range.

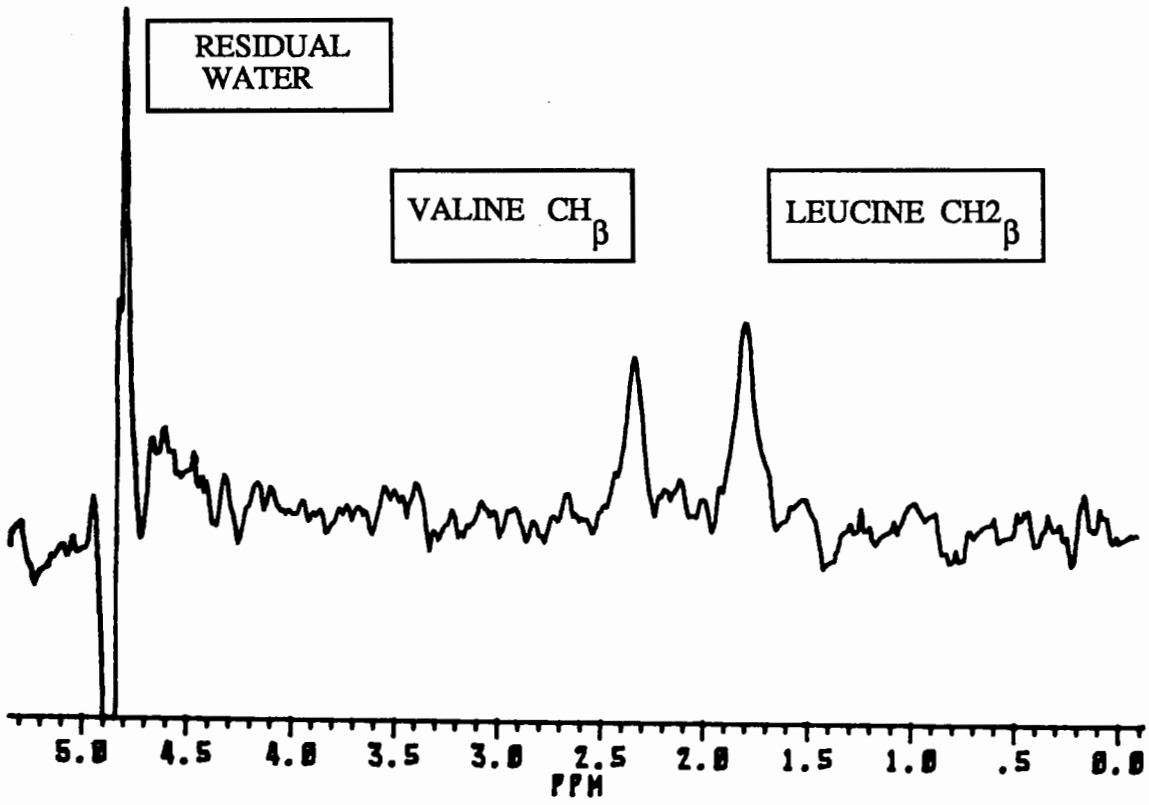
yours sincerely,

J.P. RENOU

D.MEYNIAL



pulse sheme I



Detection of leucine (2.5mM) ß protons and valine (2.7 mM) ß proton coupled to ^{15}N (95%) in muscle homogenate + 10% D_2O. Total experimental time: 100 minutes.

354-60

The University of Texas
Health Science Center
at Dallas

Department of Radiology
Biomedical Magnetic Resonance Center

15th February 1988
(received 2/22/88)

Southwestern Medical School
Southwestern Graduate School
of Biomedical Sciences
School of Allied Health Sciences

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

Dear Professor Shapiro,

Our group is actively engaged in assessing the application of magnetic resonance spectroscopy and imaging to study mouse tumor models during growth and therapy. This includes ^{31}P surface coil studies and $^{1}H/^{19}F$ imaging to examine the uptake and biodistribution of perfluorocarbons and gadolinium-containing contrast agents.

As we all know: "NMR is non-invasive"; but **Beware the shivering mouse!**

The graph indicates the variation in rectal and intra-tumoral temperatures measured using a Sensortek (18G) thermocouple following administration of anaesthetic to a MethA-tumor-bearing mouse. There is rapid decline in the body temperature to approach room temperature after about 80 minutes. This has serious implications for a number of <u>in vivo</u> NMR studies.

The spin-lattice relaxation times (T_1) of the ^{19}F resonances of perfluorocarbon emulsions are inversely proportional to the oxygen tension (pO_2). It has been proposed that this may be used to estimate tissue oxygen tension <u>in vivo</u>.[1]
However, we find that T_1 is also temperature sensitive e.g. in the pfc emulsion
FC-43 (perfluorotributylamine)

T_1	CF_3	(pO_2 = 160 T)	@ 25 C	= 578± 5 ms
	CF_2	(pO_2 = 160 T)	@ 25 C	= 405± 2 ms
but				
	CF_3	(pO_2 = 160 T)	@ 37 C	= 785± 1 ms
and	CF_2	(pO_2 = 160 T)	@ 37 C	= 558± 1 ms

When we exposed a mouse to 95% O_2 whilst in the NMR probe, we found that the T_1 obtained from the whole mouse spectrum decreased as expected. However as the mouse awoke from anesthesia and its temperature rose towards normal physiological the T_1 was found to lengthen, although the mouse was still breathing 95% O_2.

Temperature variation may also produce artifacts in the phosphorous spectra. It will present a problem in the use of the fluorinated amino acid analogues to measure pH in vivo, where a tolerance of ± 0.5 C is required.[2] We are currently designing methods of regulating mouse body temperature in the probe and evaluating devices to measure body temperature without introducing further artifacts. We wonder how other groups achieve temperature regulation?

Yours sincerely,

Ralph P. Mason, Ph.D.

Ray L. Nunnally, Ph.D.
Director Biomedical Resonance

P.P. Antich, Ph.D.
Director Radiation Physics

Reference 1. Fishman, J.E., et al Magn. Reson. Imag. (1987) 5, 279-285
2. Taylor, J.S., Deutsch, C., Biophys, J., (1983) 43, 261-267

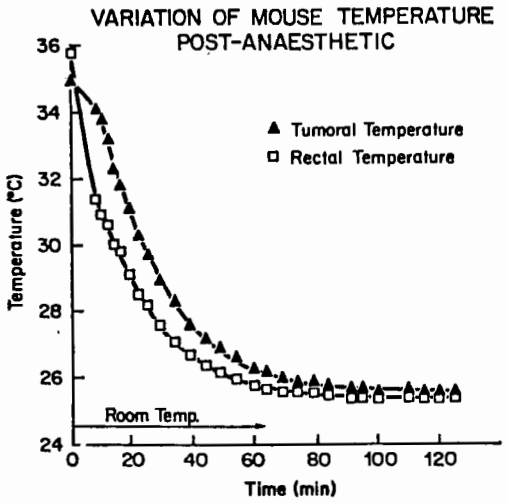


Figure 1

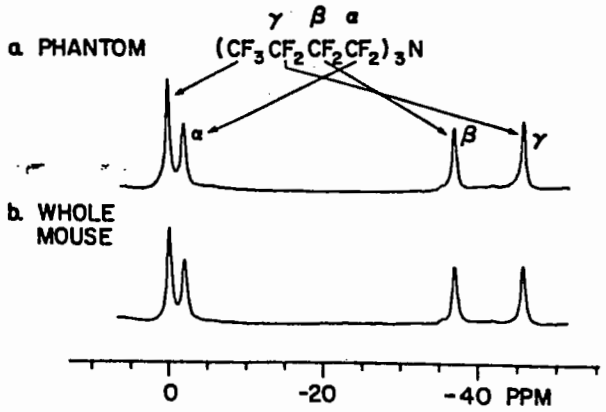


Figure 2

a. 167.5 MHz ^{19}F NMR spectrum of a phantom containing FC-43 emulsion.

b. ^{19}F spectrum of a mouse injected chronically with FC-43.

354-62

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service
National Institutes of Health

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

Professor Bernard Shapiro
966 Elsinore Court
Palo Alto, CA 94303

February 11, 1988
(received 2/19/99)

Temperature control on a Bruker

Dear Barry:

Upon testing our new AM-600 spectrometer we found the sample temperature to be quite unstable. Because the front panel indicates the desired and not the actual temperature, the user might be unaware of this instability unless (s)he uses a temperature sensitive chemical shift indicator. A first test, changing the air temperature going into the probe suddenly by 10 °C, had almost no effect (less than 0.3°C) on the sample temperature. Therefore, we concluded the regulation circuitry was working very well indeed. However, a subsequent test, changing the room temperature by 2°C, caused a sample temperature change by 1.4°C.

The reason for the correlation between room and probe temperatures is the absence of an ice point reference in the controller, which has the second thermocouple right on the printed circuit board. Adding an ice point reference still left a large temperature coefficient in the first amplifier.

Our temporary solution is to connect the sample thermocouple to a commercial digital thermometer (Newport Electronics 268). This gives an indication of the actual sample temperature as well as an analog output of 10 mV/°C. The latter is then injected into the control circuit in place of the original thermocouple signal (Fig.1).

At present, the calibration is slightly nonlinear since the linearisation for the thermocouple is sitting in a ROM. When we get the correct schematics for the unit, we will attempt to remove the ROM 's and make things linear.

Kindest regards,

Rolf Tschudin

Ad Bax

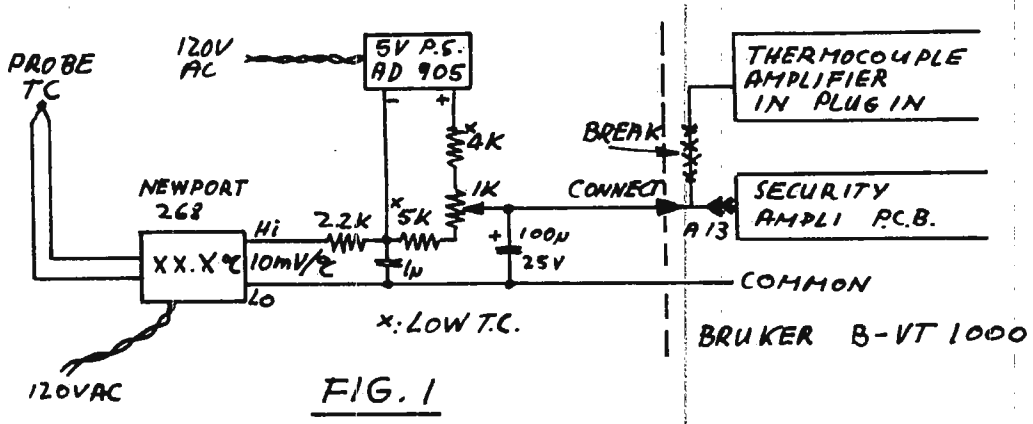


FIG. 1

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Department of Chemistry **Phone: 46-90-165000**
Umeå University S-901 87 Umeå Sweden **Fax: 46-90-136310**

POSTDOCTORAL POSITION AVAILABLE

At least one postdoctoral position will be available during 1988 in the Department of Chemistry, Umeå University, Sweden, applying high field NMR methods to the study of macromolecular structure and dynamics. Most of the work will be performed in collaboration with local medical chemists, biochemists, microbiologists, plant physiologists and molecular biologists. Modern 2D NMR techniques will be emphasized, applied to peptides, proteins and oligonucleotides. Other areas are: (a) reduction and automatic analysis of 2D NMR using pattern recognition methods, (b) protein, peptid-lipid interactions (c) structure and dynamics of lipid membranes, (d) membrane fusion, (e) lipid phase structure in particular cubic phases, (f) lipid regulation mechanisms in Acholeplasma laidlawii membranes. Modern Bruker instrumentation (AC, MSL and AM) ranging from 80-500 MHz will be available, including SUN workstations. Interested parties should communicate with prof Göran Lindblom or Ulf Edlund concerning salary, research areas etc. Please attach a short resume and names of a few references.

THE ROCKEFELLER UNIVERSITY

1230 YORK AVENUE • NEW YORK, NEW YORK 10021-6399

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Those interested should send me a biographical sketch, including the names of three references, and some indication of their expectations with regard to salary.

David Cowburn
The Rockefeller University
1230 York Avenue, Box 299
New York, New York, 10021-6399

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February 11, 1988
(received 2/16/88)

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

Dear Barry:

Title: Structure Proof by 2D NMR

We recently found that 2D NMR afforded the simplest method for determining which of two possible adducts had been obtained in a cycloaddition reaction of an o-quinone monoimide with diphenylfulvene (we received the compound from Professor Harold Heine of Bucknell University). The two possible isomers are shown in Figure (A and B) along with the H-H and C-H chemical shift correlated spectra. We suspected that compound A had been isolated from the reaction since we observed only one amide isomer in the carbon-13 NMR spectrum. Our prior experience with these N-p-nitrobenzoyl-substituted benzoxazines indicated that this was probably due to the steric effect of the exocyclic double bond and the phenyl substituents. While it was clear that the coupling patterns of protons a-d would indicate which isomer had been formed, we were not sure how to assign the proton chemical shifts. The carbon spectrum, however, was easy to assign, with the aliphatic carbon bearing nitrogen appearing at 57ppm and the aliphatic carbon attached to oxygen at 86ppm. Examination of the 2D spectra shows that the most shielded proton resonance (H_b in structure A) is attached to the carbon atom giving rise to the resonance at 86ppm. From the H-H correlated spectrum it is apparent that this proton is coupled to two other protons in the molecule (H_c and H_a). Since H_a is coupled only to H_b, this confirms the structure as A.

Sincerely,

Paul

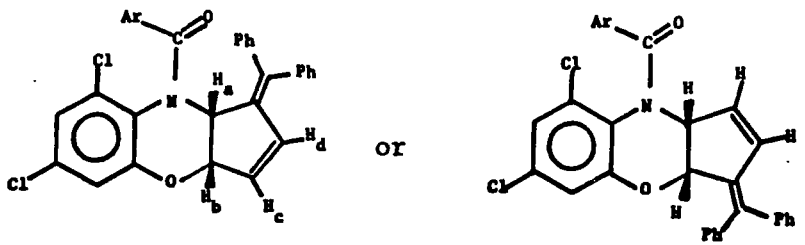
Paul Donahue

Joanne

Joanne Smith

Liz

Liz Williams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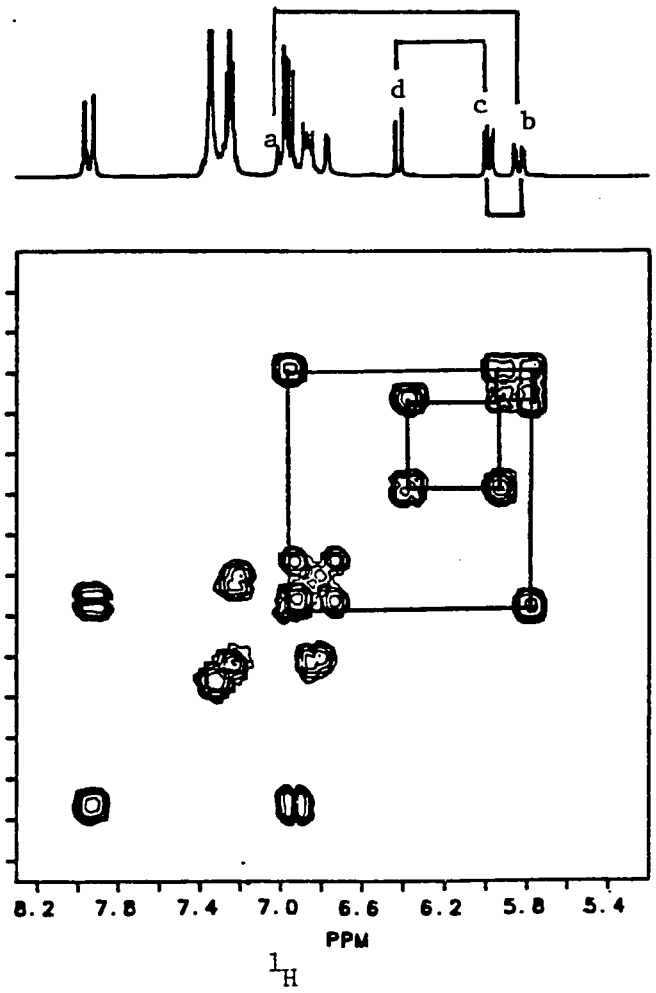
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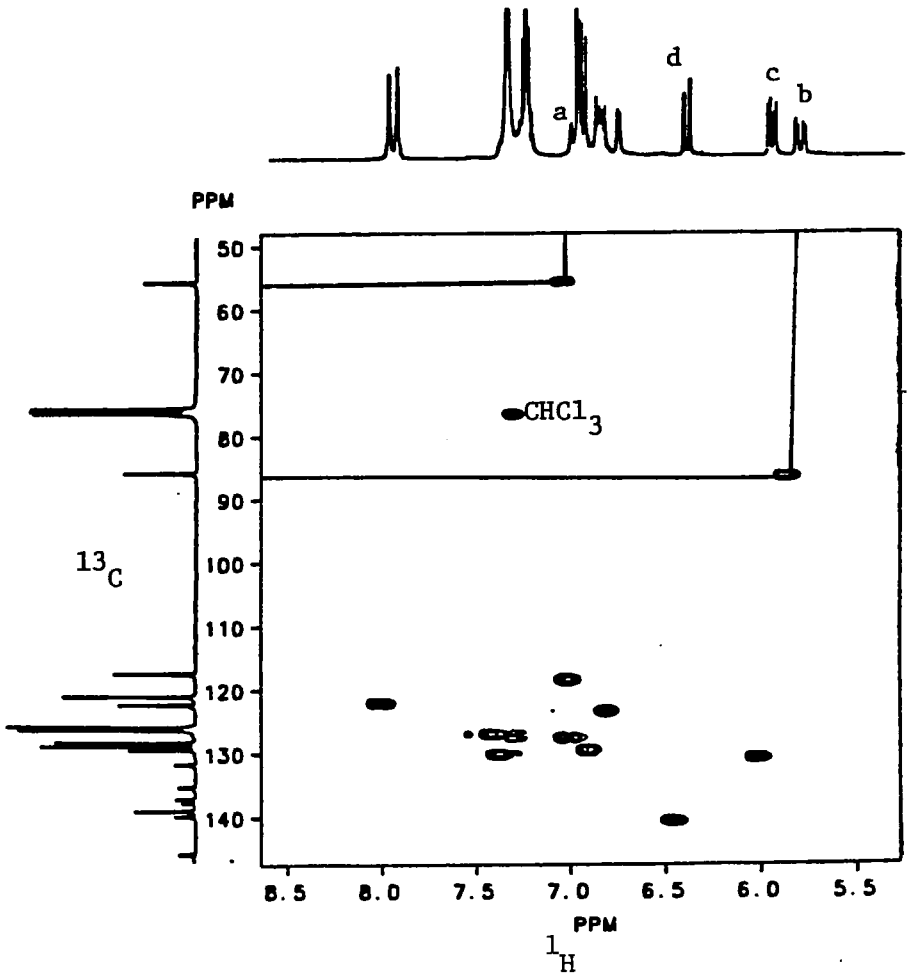
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The University of Alabama at Birmingham
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Feb. 10, 1988
(received 2/16/88)

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

Sensitivity-enhanced Indirect Detection 2D experiment on WH spectrometer

Dear Dr. Shapiro:

The sensitivity-enhanced indirect detection 2D NMR (HMQC) experiment has been demonstrated to be a superior method to perform the heteronuclear chemical shift correlation spectroscopy. Most spectrometers delivered by major manufactures within the past two years should be able to perform this experiment without any difficulty.

We have a Bruker WH-400 spectrometer which was installed in 1979. This spectrometer has been upgraded with an Aspect-3000 computer recently, but all the RF electronics, probes, preamps and transmitters are of 1979 design. With minor modification of the spectrometer, we have performed with partial success the {^{1}H - ^{13}C} indirect-detection experiment. The spectrometer hardware modification included the addition of a slide switch on one of the RF mixing units (PT-400). The purpose of this switch is to provide the transmitter and receiver with separate RF references. The ^{13}C pulse was generated through the BSV-3 transmitter power amplifier as in a routine ^{13}C experiment. The proton decoupler was used to generate the ^{1}H pulse. By properly setting the slide switch, the receiver was set-up to observe the ^{1}H signal. The indirect-detection spectrum of an oligosaccharide studied by us is shown in the enclosed figure. Since we could not do the ^{13}C decoupling during the data acquisition, the ^{13}C-^{1}H couplings were retained in the final spectrum. The data was obtained on a regular 10 mm ^{13}C probe, with the ^{1}H decoupler coil located outside the smaller ^{13}C coil. This configuration gives inherently low S/N ratio compared to those probes specially designed for the inverse experiment (with the ^{1}H coil inside). Nevertheless, the experiment was done within 6 hours (compared to 40 hours for the normal heteronuclear COSY experiment). The data was processed on a MicroVAX II computer using the FTNMR program kindly provided by Dr. Dennis Hare, and the result was very encouraging.

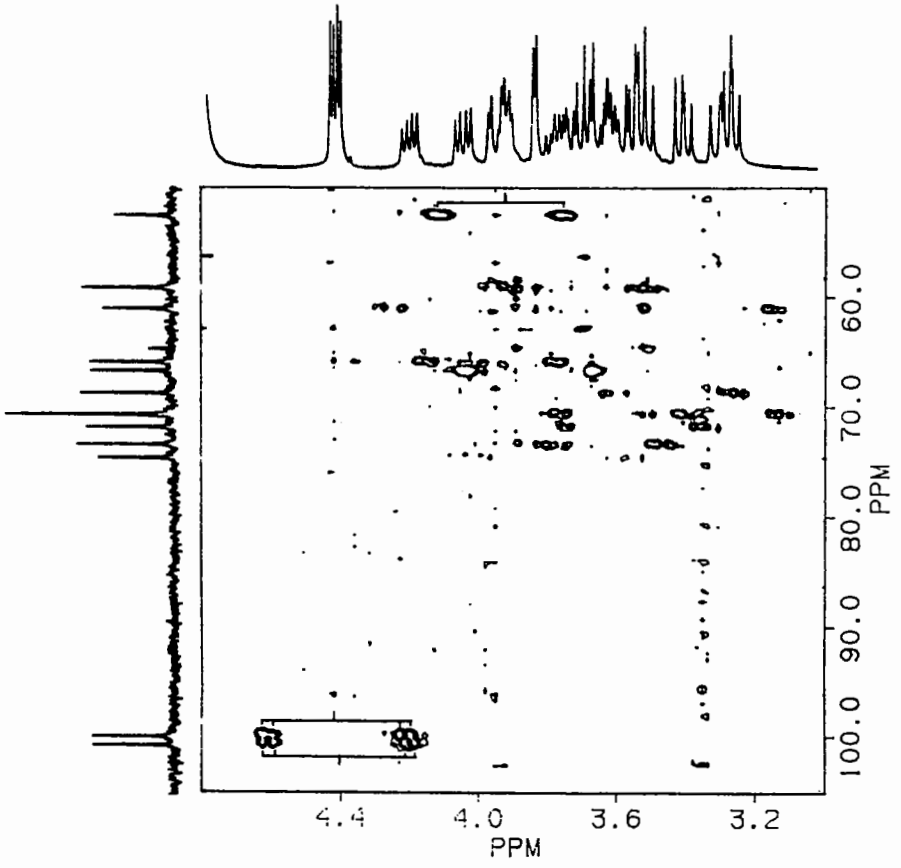
Because of the coil configuration on our regular 10mm ^{13}C probe, the extent of suppression of the center peak is critically depended upon the sample height. Best suppression was obtained by confining the sample volume to the ^{13}C coil. The ^{1}H - ^{13}C dual probe as well as the inverse probes now routinely supplied by the different manufactures should achieve a much better suppression of the center peak.

We would like to thank Drs. J.M. Geckle and Wolfgang Bermel of Bruker Instruments for their helpful suggestions and discussion on performing this experiment.

Please credit this to Dr. N. Rama Krishna's subscription.

Sincerely yours,

Dee-Hua Huang Mike E. Brown



THE UNIVERSITY OF UTAH

Department of Chemistry
Henry Eyring Building
Salt Lake City, Utah 84112

14 Feb. 1988
(received 2/20/88)

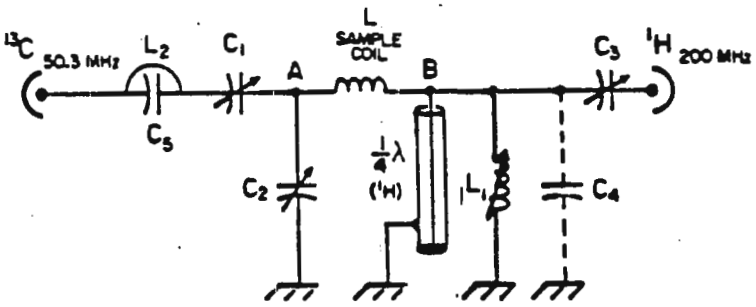
Dr. Bernard L. Shapiro
Editor/Publisher
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303
U.S.A.

A PROBE WITH HIGHER DECOUPLING EFFICIENCY AND SENSITIVITY FOR SOLID STATE NMR EXPERIMENTS

Dear Professor Shapiro:

Recently we have developed a more efficient double-tuned $^{13}C/^{1}H$ probe circuit especially for higher decoupling efficiency and improved sensitivity of the observation channel (^{13}C).

The basic circuit of the solid state probe for higher decoupling efficiency is as shown below.



Comparing this with the circuit in our previous paper(1), one notices that the C4 in the original circuit has been eliminated, and that the coil L1 can be adjusted smoothly by moving a piece of copper inside the coil. The capacitor C4 is now a stray capacitance, and the matching condition is met by adjusting the inductor L1 to satisfy the following formula (1):

$$R' = \frac{R_0}{\left(1-\omega^2 \frac{L_1 L}{L_1 + L} C_4\right)^2} = 50\,\Omega$$

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SPECIFICATIONS

MODEL	POWER BANDPASS (MHz)	GAIN (dB)	PULSE POWER (W)	CW POWER (W)	WEIGHT (lbs.)	DOMESTIC PRICE
200A	7-140	48	200	50	52	$8,000.
400A	8-130	50	400	100	57	9,800.
800A	8-120	54	800	200	62	13,700.

180dB minimum signal blanking.

Rise Time: less than 0.1μs to 90%.
Fall Time: less than 0.1μs to -80dB.

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

Flatness of response throughout power bandwidth:
±1.5dB at half rated pulse power.
±3dB at any power level.

THD is below 6% at any power level, any frequency.

Typical third order intercept (800W unit) 60dBm.

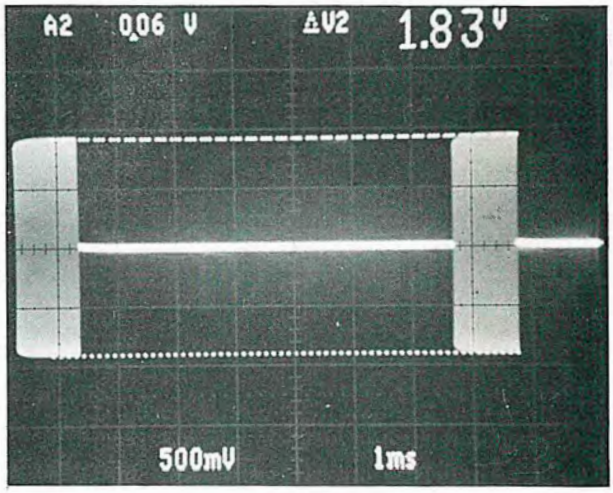
Phase shift change (10% to 80% of rated power): 8° max.

Adjustable average output power limiter from 0.1% to 25% of rated pulse power.

Control panel includes three rotory decade attenuators, directional pulse power meter with 0.3μs response time, and power limiter.

High frequency switching power supply operates off 105 to 125 VAC, 40 to 400Hz. Quiescent AC power demand is 8% of rated rf pulse power. Total efficiency at full power is 30%.

220 VAC, 50Hz models also available.

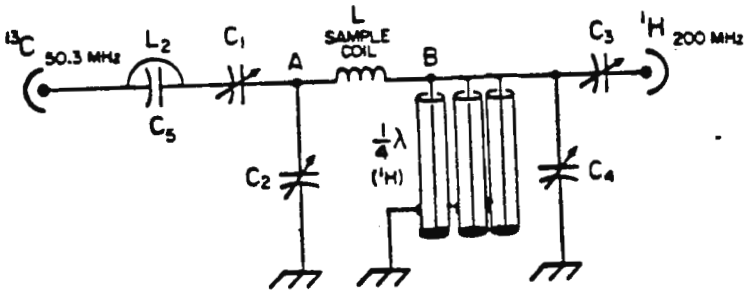


Typical 400 Watt pulse envelope.

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

This modification of the circuit not only eliminates an expensive capacitor, but also extends the space inside the probe, which lessens the arcing problem. Because the capacitor C4 has been replaced by a relatively small stray capacitance, the inductance L1 can be increased, resulting in more efficient decoupling power in the sample coil.

In a solid state NMR probe, for use at high frequencies (^{1}H at 200MHz or above) a small coil is often required, and hence the inductance of the coil is small. This decreases the sensitivity of the ^{13}C channel. This problem can be alleviated by replacing the single $\lambda/4$ coaxial cable in the original circuit (1) with three $\lambda/4$ cables in parallel, decreasing the inductance from point B to ground, and increasing the sensitivity of the ^{13}C channel, as shown below.



(1) Yi Jin Jiang, Ronald J. Pugmire and David M. Grant, J. Magn. Reson. 71 485 (1987).

Sincerely

Yi Jin Jiang — Warner R. Woolfenden — Mark H. Sherwood

Don W. Alderman — Ronald J. Pugmire — David M. Grant

Allied Signal

Allied-Signal Inc.
Corporate Technology
P.O. Box 1021R
Morristown, NJ 07960-1021

February 9, 1988
(received 2/16/88)

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

Peptides and Carbides at Allied.

Dear Dr. Shapiro:

The Corporate Technology NMR group at Allied-Signal currently has a Varian XL-400 and an aging XL-200 equipped to run solids. We are presently in the process of installing a Chemagnetics CMC-300 to be upgraded to a CMX by the end of the year. We are also taking advantage of the confusion caused by the renovation of the NMR laboratories to update our data handling capabilities with an NMRI data station.

NMR has been heavily involved in support of Allied's Biosciences group. We are obtaining high resolution carbon-13 spectra of peptides on solvent swollen PAM resins. As can be seen from Figure 1, high resolution spectra are easily obtainable. We are using this method for the study of side reactions that occur during solid phase peptide synthesis, and for the study of conformations of peptides bonded to the PAM resin. A communication describing these results has been submitted for publication.

We are also carrying out extensive silicon-29 (MAS) studies on a wide variety of ceramic and high performance materials. In many of these systems, we have noticed that the linewidth is sample dependent. This has been observed both in common ceramics and in more exotic materials, as well. For example, Figure 2 shows the silicon-29 spectra of two commercial silicon carbides. There appears to be no significant relationship between the concentration of paramagnetic impurities and linewidth, as might be expected. X-Ray Diffraction is often unable to distinguish such samples. We would be interested in hearing from anyone with similar experiences or insight into this observation.

Please credit this contribution to R. D. Sedgwick

Best wishes,

Raymond J. Brambilla
(201) 455-2984

Galen R. Hatfield
(201) 455-2794

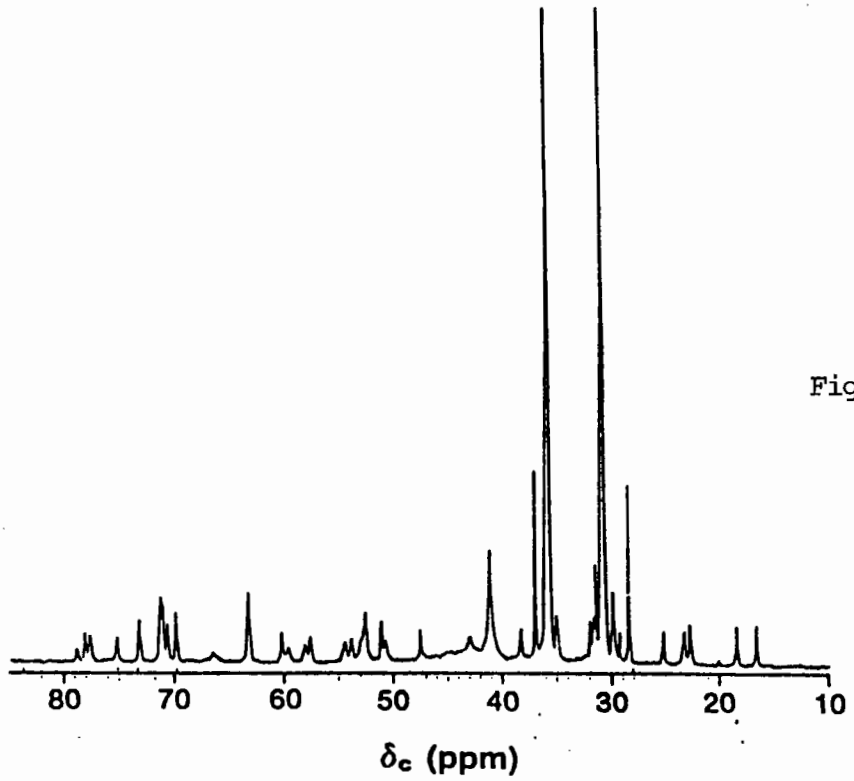


Figure 1

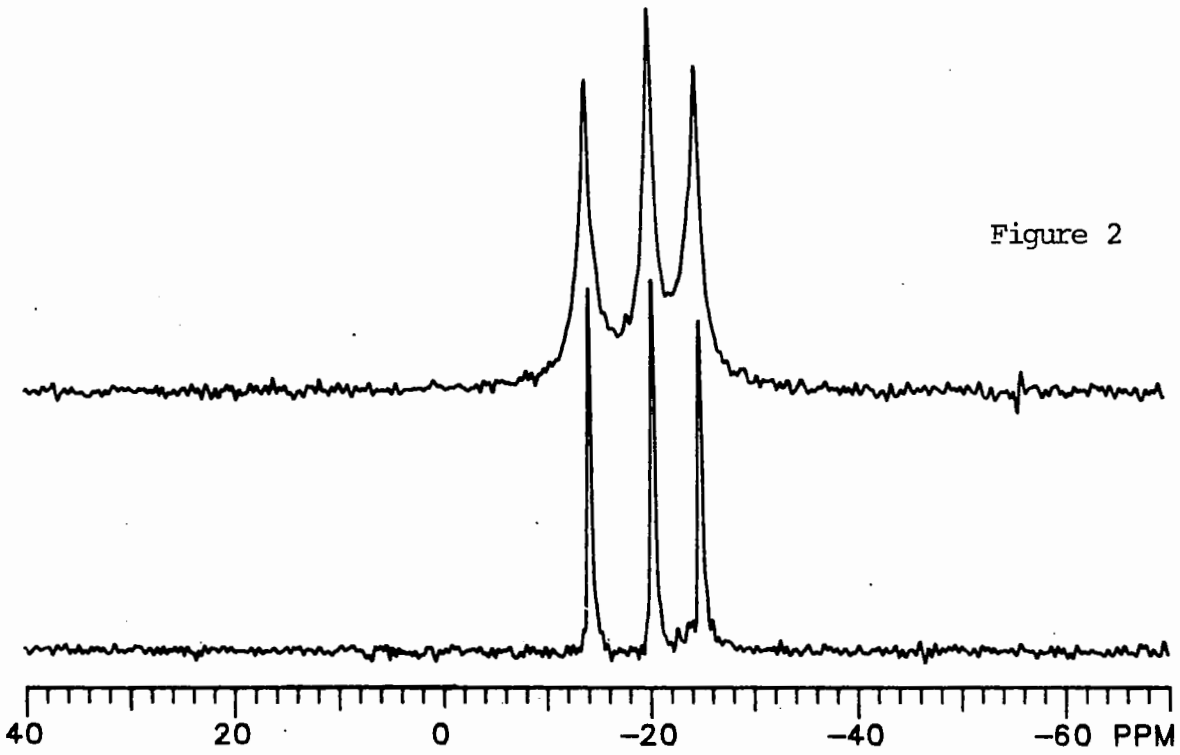


Figure 2

Catalytica

February 16, 1988
(received 2/18/88)

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

AN UNUSUALLY LARGE SUBSTITUENT EFFECT

Dear Barry,

In the course of investigating the ^{31}P chemical shift anisotropy of phosphonic acids, we discovered an unusual substituent effect. The effect is observed in the spectrum of m-sulfophenylphosphonic acid. The isotropic ^{31}P chemical shift for this molecule in the solid state is 18.8 ppm. The shift for the unsubstituted phenylphosphonic acid in the solid state is 16.1 ppm. The apparent effect on the isotropic chemical shift is 2.7 ppm, not an unexpected magnitude compared to other substituted phenylphosphonic acids. The unusual nature of the sulfonic acid substituent is revealed, however, when the ^{31}P chemical shift anisotropy and asymmetry parameter of the sulfonic acid substituted and the unsubstituted phenylphosphonic acid are compared. The anisotropy and asymmetry for the phenylphosphonic acid are -76 ppm and 0.93, while for the sulfonic acid sustituted system, the anisotropy and asymmetry are -145 ppm and 0.01. Thus, it appears that while the isotropic shift is only slightly perturbed by the sulfonic acid substituent, the chemical shift tensor is very drastically modified. The most probable explanation for this is shown in the attached figure. While the phenylphosphonic acid contains a phosphoryl oxygen, the sulfophenylphosphonic acid does not, the sulfonic acid group having transfered its strongly acidic proton to the basic phosphoryl oxygen site creating a sort of zwitter ion. Thus the chemical shift tensor becomes essentially axially symmetric in the sulfonic acid derivitive due to the formation of a third hydroxyl group equivalent to the other two. The axis of symmetry lies along the P-C bond axis. In the phenylphosphonic acid, no unique axis exists. The chemical shift tensor is strongly anisotropic.

The powder pattern spectra were determined on a Bruker MSL400. The spectra were acquired with cross-polarization and high powered decoupling during acquisition. The r.f. field strengths were matched at 56 kHz for the cross-polarization and the proton field was maintained at this level for the decoupling. The technique for eliminating baseline and phase distortion described by Henrichs, et al, in J. Mag. Reson. 69, 460-466(1986) was used in all cases. This technique is relatively straight-forward to implement on MSL-type spectrometers which allow one to control the reciever phase and I recommend it to those who have not tried it. The chemical shifts are referenced by sample exchange to 85% phosphoric acid with downfield shifts taken as positive. The principal components of the chemical shift tensor were determined by fitting the observed powder patterns using the program TENSOR (available through Abacus). The anisotropy and asymmetry as used here are defined as follows:

$\Delta\sigma$, the shielding anisotropy

$$= \sigma_{33} - 1/2(\sigma_{11} + \sigma_{22})$$

η, the asymmetry parameter

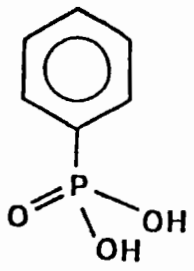
$$= (\sigma_{22} - \sigma_{11})/(\sigma_{33} - \sigma_i)$$

σ_{33} is taken as the unique axis.

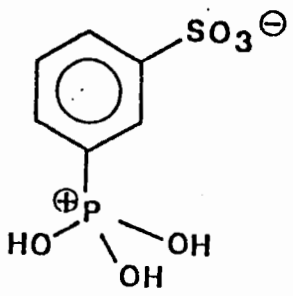
Sincerely yours,

Charlie

Charles M. Schramm



$\Delta\sigma = -76$
$\eta = 0.93$



$\Delta\sigma = -145$
$\eta = 0.01$

354-78

LUBRIZOL

THE LUBRIZOL CORPORATION
29400 LAKELAND BOULEVARD
WICKLIFFE, OHIO 44092-2298
TELEPHONE: 216/943-4200

February 11, 1988
(received 2/19/88)

Dear Professor Shapiro,

O,O'- Dialkyldithiophosphoric acid assignments by $^{31}P,^{1}H$ 2D Shift Correlation NMR

Dialkyldithiophosphoric acids (DPH) can be prepared from either a single alcohol or a combination of alcohols (scheme 1) to yield the corresponding single DPH or a mixture of DPHs. In the lubricant additives industry ^{31}P NMR is used extensively to characterize molecular functionality. The unique nature of observing a single ^{31}P peak for each phosphorus containing molecule provides a simple spectrum as compared with the corresponding proton or carbon NMR spectrum. This feature is valuable to characterize the functionality of a complex mixture of phosphorus containing compounds especially when dissolved in a complex organic medium such as a mineral oil. However, the simplicity of ^{31}P NMR can also be a problem. The lack of detail or information is problematic if one is interested in further characterizing the phosphorus molecules corresponding to specific peaks in a ^{31}P spectrum. In certain cases, we have been able to utilize the 2D heteronuclear correlation technique[1] ($^{31}P,^{1}H$) to circumvent this problem and unambiguously assign the phosphorus peaks of mixtures, e.g. a DPH mixture.

In this correspondence, we have used this technique to assign the ^{31}P NMR peaks of a DPH mixture (1-6) resulting from (+/-) 4-methyl-2-pentanol and isopropanol as the starting alcohols. Obviously, the two major requirements in successfully applying this approach are long-range P,H coupling and chemical shift assignment of the long range coupled protons. The $^{31}P,^{1}H$ long-range coupling provides three bond P,H correlations (7-13 Hz). In addition to three bond correlations, four bond correlations can also be observed in many cases when a secondary alkyl group is present. However, as was recently pointed out, when nonequivalent geminal protons are present, four bond coupling is observed only for one of the two available protons[2]. This is quite clear for H-3a of the meso/racemic mixture (1,2) as illustrated in Figure 1. The left contour plot (Figure 1a) is a 2D P,H HETCOR plot of a mixture of 1 and 2. The center spectrum (Figure 1b) is a column trace of the C-3 peak of 5 from a 2D C,H(decoupled) HETCOR[3-5] spectrum of the mixture 1-6. The doublet of doublets results from the H,H coupling of the C-3 nonequivalent geminal protons. The right 2D contour spectrum (Figure 1c) is a P,H HETCOR spectrum of (1-6). Peaks A-D are easily identified since the isopropyl methine protons (H-7) are downfield of the 4-methyl-2-pentyl methine protons (H-1). Clearly, peaks A and B are the racemic/meso diastereomers (1/2 and 3/4), peak C is the O-isopropyl,O-4-methyl-2-pentyldithiophosphoric acid (5) and peak D is the O,O-bis(isopropyl) dithiophosphoric acid (6).

In certain cases, we have used this technique successfully to unambiguously assign the ^{1}R and/or ^{2}R functionality (aromatic, aliphatic, primary, secondary, etc.) of phosphorus containing mixtures.

Please credit this contribution to Horton Dunn's account.

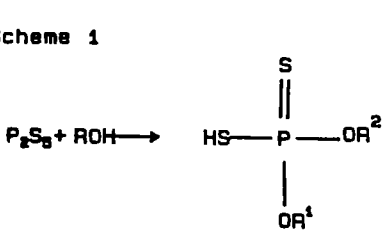
Sincerely,

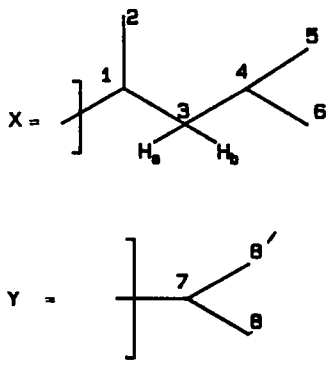
Kurt Wollenberg

Kurt Wollenberg

1) A. Bax, G. Morris J. Magn. Reson., 42, 501 (1981)
2) G.J. Ray, G. Caspari Magn. Reson. Chem., 25, 931 (1987)
3) A. Bax J. Magn. Reson., 53, 516 (1983)
4) V. Rutar J. Magn. Reson., 58, 306 (1984)
5) J.A. Wilde, P.H. Bolton J. Magn. Reson., 59, 343 (1984)

Scheme 1

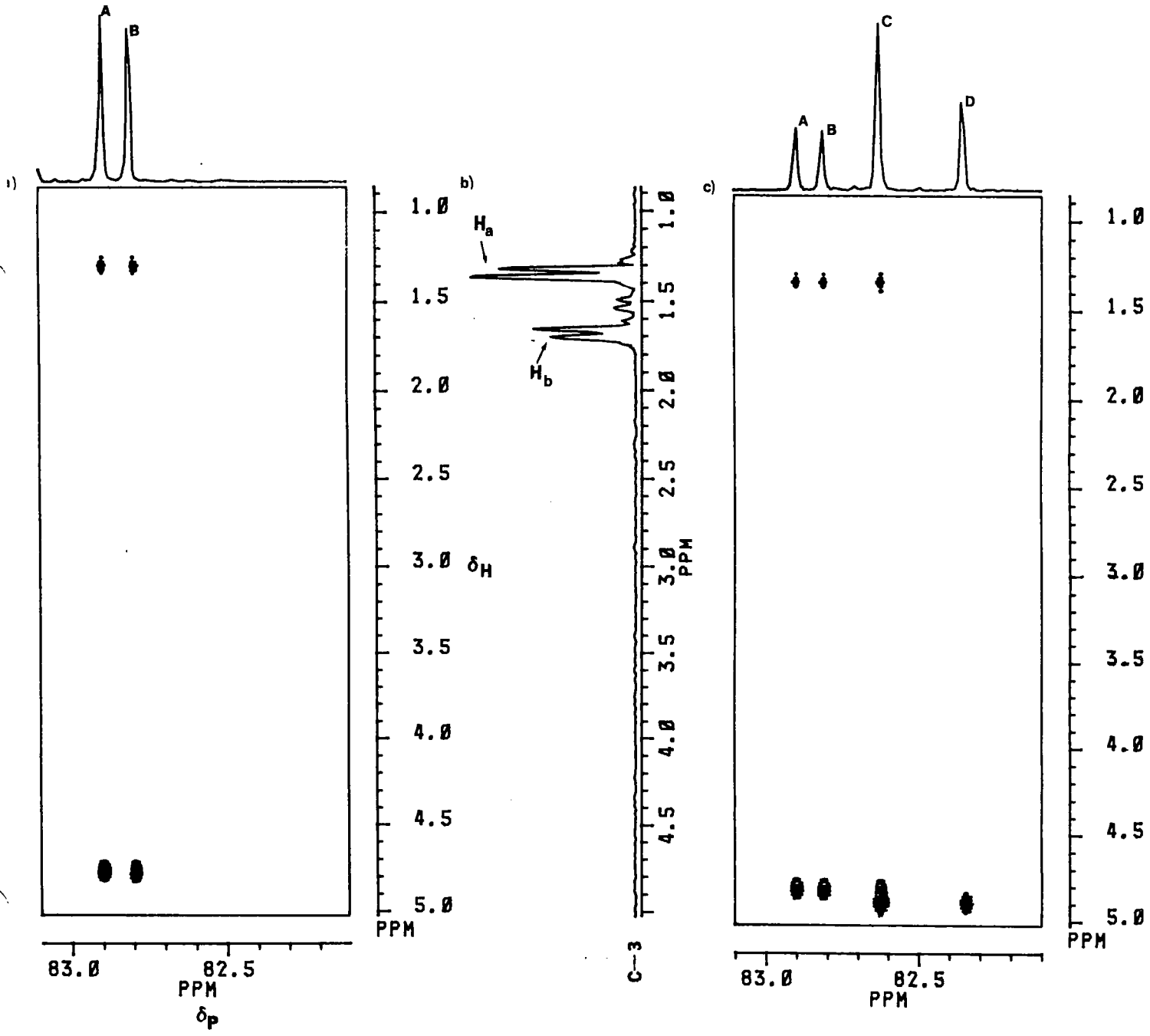




X=4-methyl-2-pentyl
Y=i-propyl
R,S refer to either R or S configuration

1) $R^1=R^2=R$ X
d,l pair 2) $R^1=R^2=S$ X
3) $R^1=R$ X, $R^2=S$ X
meso pair 4) $R^1=S$ X, $R^2=R$ X
5) $R^1=R$ or S X or Y, $R^2=R$ or S X or Y
where $R^1=X$ and $R^2=Y$ or $R^1=Y$ and $R^2=X$
6) $R^1=R^2=Y$

Figure 1 a) 121.5 MHz $^{31}P_1^1H$ HETCOR spectrum of a mixture of 1 and 2 b) 75.4 MHz $^{13}C,^1H$(decoupled) HETCOR C-3 column trace of 5. The doublet of doublets is due to the C-3 nonequivalent geminal protons. c) 121.5 MHz $^{31}P,^1H$ spectrum of a mixture of 1-6, peaks A-D are identified in the text.



Prague Institute of Chemical Technology

NMR Laboratory, Prague 6,Czechoslovakia

Dr. Bernard L.Shapiro

TAMU NMR Newsletter

966 Elsinore Ct.

Palo Alto, CA 94303

U.S.A.

(received 2/19/88)

Title: The NMR study of some 4H-pyran systems

Dear Prof. Shapiro,

recently we solved the problem of verification of the structure of 4-amino-5(2-furyl)-6-oxo-7-substituted-5,6,8-trihydro-pyrrolo [3,4´:5,6] pyrano [2,3-e] pyrimidines. For this purpose we employed the Bruker AM 400 spectrometer. At first we measured the ^{1}H NMR spectra, in which the most interesting role played the spin system of H5 and H8 protons of CH_2 group. Besides non-equivalency the H8 protons exhibit the long-range interaction with ^{5}J = 1.5 Hz classified as to be the homoallylic transoid coupling of the pseudoaxial H5 proton.But the key problem was to understand the carbon skeleton of the named compound, which bears 7 quarternary carbons. Because of that we measured the ^{13}C NMR data of the compound sequence beginning with the 2-amino-6-chlormethylen-3-cyano-5-ethoxycarbonyl-4 (2-furyl)-4H-pyran and have taken use of some NMR technics,

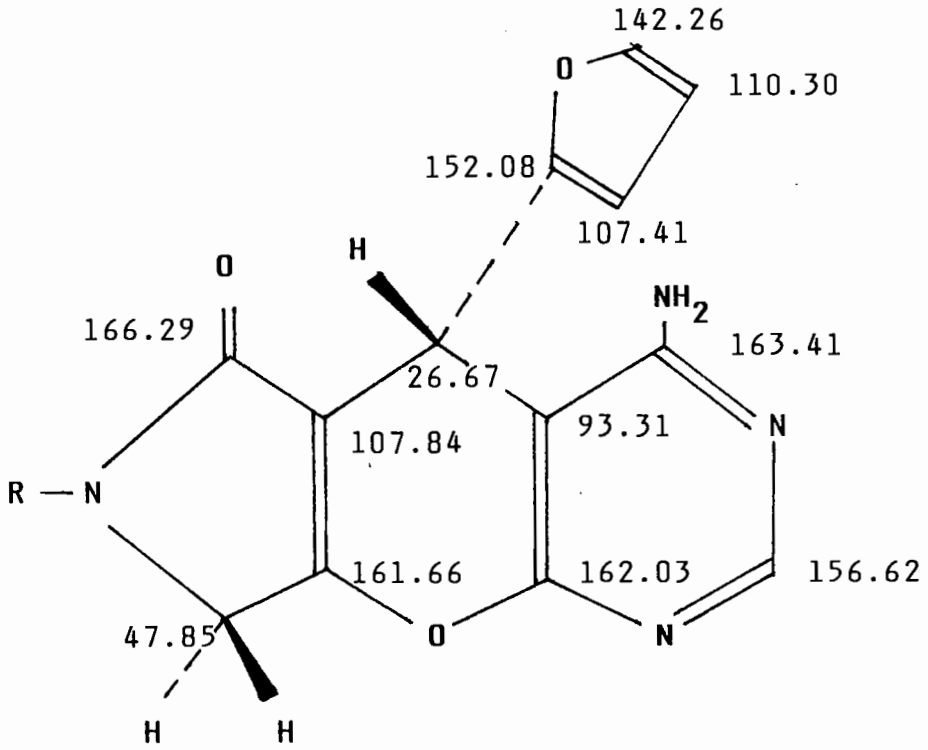


Fig.1. ^{13}C NMR data of one of the studied compounds ($R=CH_2Ph$).

above all the useful long-range INEPT. On the fig.1 the structure and ^{13}C NMR data of one of them are shown.

Sincerely

F.Pavlikova

F. Pavlík

NOTE: Please credit this communication to Dr.M.Hajek account.

354-82

University of Arkansas for Medical Sciences

4301 W. Markham
Little Rock, Arkansas
72205-7199

February 17, 1988
(received 2/22/88)

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

SUBJECT: NMR Imaging of Rubber

Dear Barry:

We have recently been investigating the utility of NMR imaging for certain materials-science applications, particularly those involving elastomeric systems. Below are two NMR images of a block (80 x 40 x 15 mm) of bulk, uncured cis-polybutadiene, a rubber of high molecular mobility (Tg = -102°C). The top image is the 2D FT, spin-echo image of a 2-mm axial slice (40 x 15 mm) (TE = 12 ms, NA = 24, FOV = 120 x 123 mm, resolution 256 x 256). The bottom image is a gradient-echo image of a 1-mm axial slice at the same location (TE = 6.4 ms, NA = 48, the rest as above). The images were acquired at ambient temperature at 200.1 MHz on a GE CSI-4.7/33 system. The maximum gradient strength was 2 G/cm. The slices were taken through a section of the block that was riddled with trapped voids or air bubbles of average diameter of about 1 mm. We attribute the mottled appearance in the spin-echo image to the voids. The appearance of the spin-echo image is not changed significantly for a 1-mm slice. The gradient-echo image, which is sensitive to T_2^*, is considerably different than the spin-echo image. Apparently the voids, which have a range of sizes and are unevenly distributed in the block, cause a significant shortening of T_2^* relative to T_2. This is probably due to differences of magnetic susceptibility at the polymer-void interface. Similar behavior has been observed in medical imaging when a gradient-echo sequence is used. This and other results from our laboratory suggest that gradient-echo imaging may be the method of

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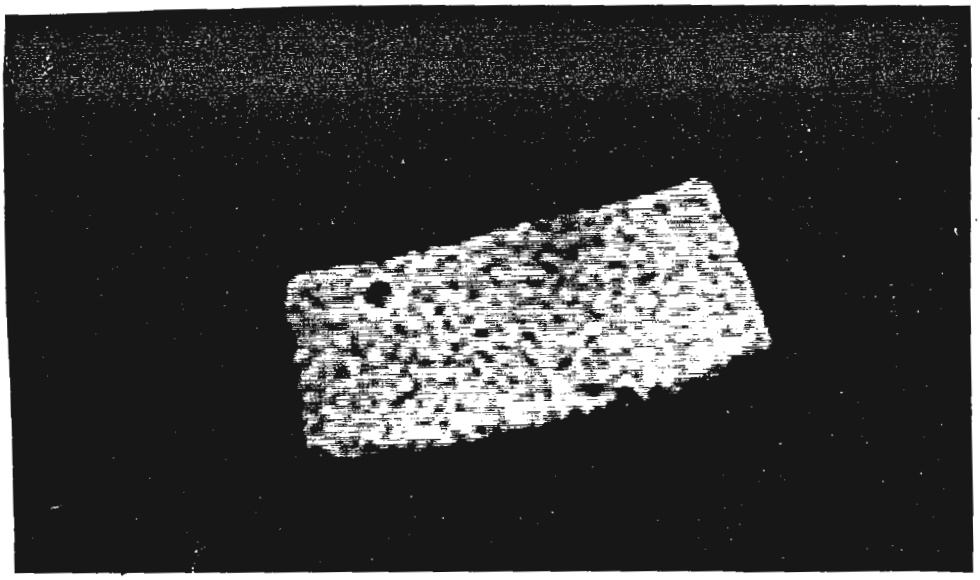
choice for detecting small physical defects such as voids, tears, or gaps in manufactured elastomer products.

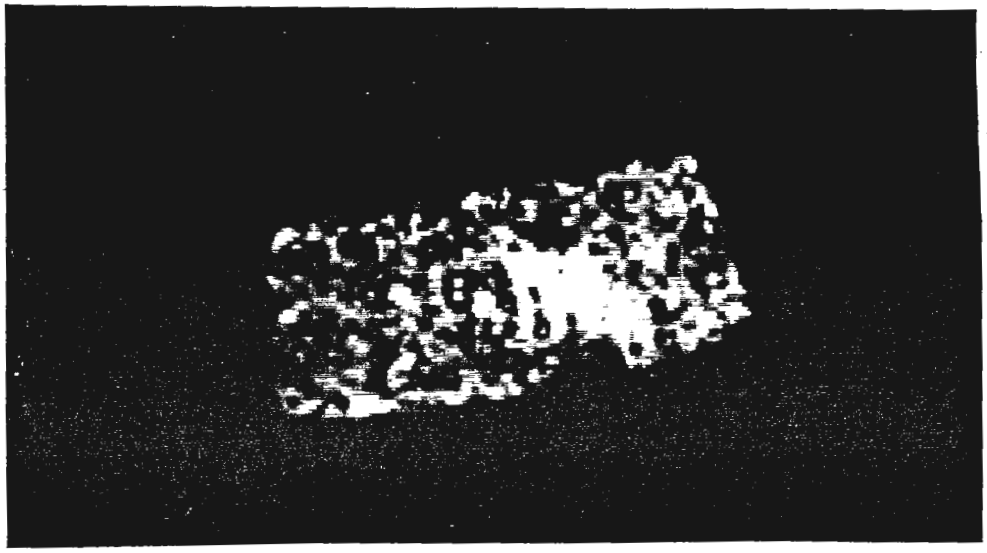
With best regards,

Chen Chang

Chen Chang

Richard A. Komoroski





Université de Nancy I

Laboratoire de Méthodologie RMN

D. CANET
Professeur

dc8811

Professor B.L. SHAPIRO
TAMU NMR Newsletters
966 Elsinore Court
PALO ALTO, California 94303
U.S.A.

Self diffusion measurements, spatial localization and signal suppression by use of rf field inhomogeneity

February 17, 1988
(received 2/22/88)

Dear Professor Shapiro:

I would like to mention some simple experiments currently performed in this laboratory and which resort to B_1 inhomogeneity:

Self diffusion measurements can be carried out by means of the axial gradient of the rf field produced by a single turn coil, positionned so that the sample experiences a linear gradient. The sequence also includes pulses of a homogeneous rf field delivered by a conventionnal saddle shaped coil orthogonal to the single turn coil. Denoting periods of application of the rf field gradient by braces, this experiment is schematized as:

$\{\delta\}_x - \Delta/2 - (180°)_y - \Delta/2 - \{\delta\}_x - (90°)_x$ - Acquisition

The two rf channels corresponding to the two coils must be made phase cohertent at the spin level. The resulting magnetization, as obtained after the last $(90°)_x$ observing pulse, is given by:

$$M(\Delta,\delta) = -M_0 \exp[-D\gamma^2 g_1^2 \delta^2 (\Delta + 2\delta/3)] \exp(-\Delta/T_1) [2 - \exp(-\Delta/2T_1)]$$

D is the self diffusion coefficient and g_1 the rf gradient; other symbols have their usual meaning. Measurements are best performed by varying δ while keeping Δ constant.

Another interesting application of this experimental arrangement concerns the spatial localization of a slice perpendicular to the single turn coil axis. Again, it makes use of both rf fields (homogeneous and

inhomogeneous) and of a DANTE-like sequence which is the replica of the conventionnal DANTE sequence in the rotating frame:

$(\pi/2)_x[(\theta)_{-x}(\tau)_y]_n(\pi/2)_{ax}(\text{Acquisition})_{ax}$

The angle θ is such that $n\theta=\pi/2$ and τ is chosen as a function of the abcissa a of the slice to be selected: $\gamma B_1(a)\tau=2\pi$. The repeated cycles $(\theta)_{-x}(\tau)_y$ bring the magnetization of interest towards the z axis while magnetization corresponding to other regions is essentially kept along the y axis. The capabilities of the conventionnal DANTE sequence may be transposed to the present experiment, especially the adjustment of selectivity (here spatial selectivity) as a function of n. This enables to obtain the spectrum pertaining to a slice whose both abcissa and thickness can be defined with great accuracy.

Finally, it can be pointed out that the inherent inhomogeneity of B_1 produced by a saddle shaped coil may be employed to suppress the longitudinal and one of the tranverse components of nuclear magnetization. It suffices to apply for a time sufficiently long (in practice, of the order of several milliseconds) an rf field along, say the x axis of the rotating frame, for achieving a complete scatter of the magnetization in the yz plane, whereas the x component remains locked along the B_1 field. This feature can profit a lot of situations, especially when there is a need of suppressing a strong solvent peak; magnetization of the latter has simply to be taken along y while leaving other magnetizations (or part of them) along the x axis.

I hope that this letter will reach you before the deadline date for the March issue.

Yours sincerely,

D. Canet

POSTDOCTORAL ASSOCIATE
BIO-NMR

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UER DES SCIENCES PHARMACEUTIQUES ET BIOLOGIQUES

UNITÉ DE PHARMACOLOGIE MOLÉCULAIRE (INSERM U 266)
LABORATOIRE DES INTERACTIONS BIOLOGIQUES (CNRS UA 498)

LABORATOIRE DE CHIMIE ORGANIQUE

DIRECTEUR : PR BERNARD P. ROQUES

Paris, February 16th, 1988
(received 2/22/88)

Doctor SHAPIRO
Department of Chemistry
Texas A & M University
College Station
TEXAS 77843 -3255.

Dear Doctor Shapiro,

Still involved on elucidating drug nucleotide complexes, in which broad lines are often obtained and the sensitivity poor, we have decided to set up the 2D homonuclear Hartmann-Hahn (HOHAHA) (1) technique on our Bruker AM 400 MHz spectrometer. However reading the litterature published on the subject since 1985 it was not clear for us, what was the power necessary and how to get it. Therefore we have measured the dispatching of the power available from the decoupler to the probehead.

As shown on scheme 1, they are two possibilities to drive of the decoupling power to the probehead :

A) proton receiver selected (PR = 1,1). In this case the loss in the directional coupler are very high (~17 dB) and the highest power available at the probehead is very weak around 0.6 W leading to a 180° pulse width of 150 µs.

B) X nucleus receiver selected (PR= X,2) there, the power lost through the RF coaxial relays C_1/C_2 is much weaker and the power available at the probehead is 26 W.

As the experiment needs high power the coaxial cable from the decoupler was connected to the BNC Jack specified "F1 transmitter in" on the preamplifier cabinet. A 6 dB attenuator was inserted to avoid ^1H preamplifier damage ; in this case the 180 pulse width is 52 µs. This low cost modification, allows to cover a substantial bandwidth (~10 KHz)

1. BAX, A., DAVIS, D.G. (1985) J. Magn. Res. 65, 355-360.

J. BELLENEY M. DELEPIERRE

Please credit this contribution to the subscription of Professor B.P. ROQUES.

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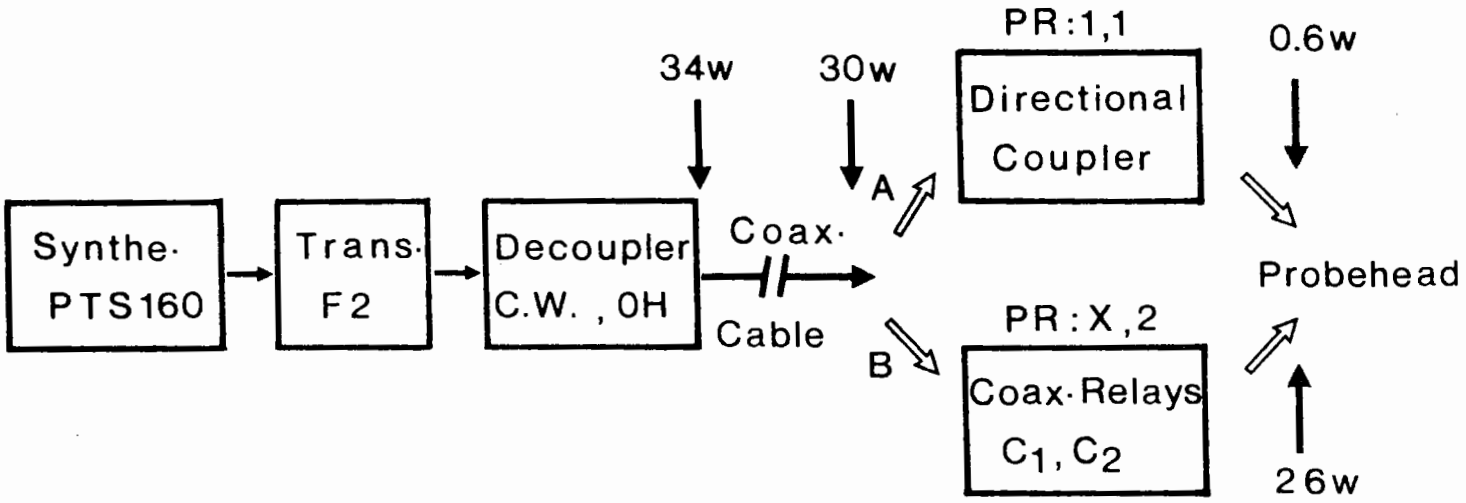

Scheme 1 : Power loss measured in the C.W. mode with a BIRD Wattmeter.

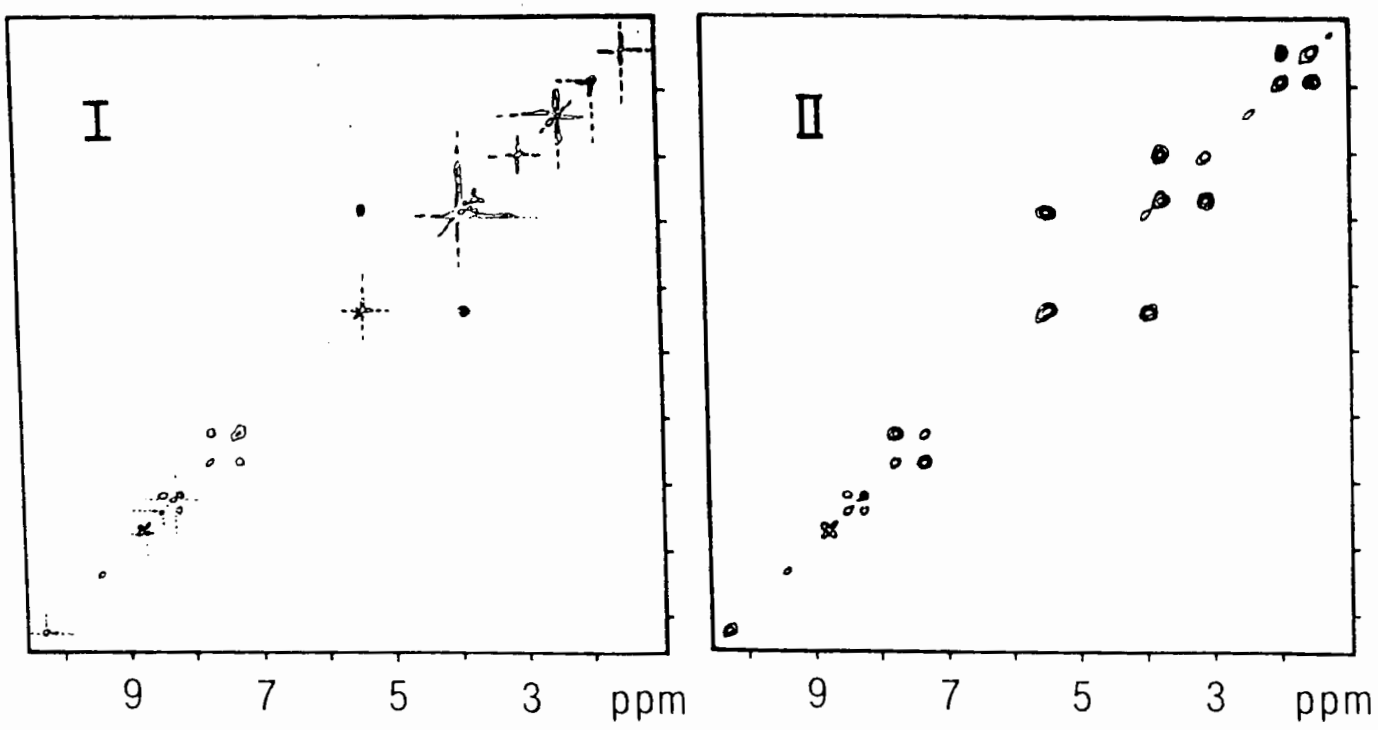

Figure 1 : HOHAHA spectra on Ditercalinium recorded with the MLEV-17 pulse sequence (1) with a trim pulse of 2.5 msec (512 x 2048 data matrix, exponential line broadening of 2 Hz, mixing period 25 ms).
(I) Proton receiver selected (without modification); (II) Decoupler cable connected to the BNC "F_1 trans." with 6 dB attenuator.

Texas A&M University NMR Newsletter - Book Reviews

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

" Recent Advances in Organic NMR Spectroscopy. "

Edited by

Joseph B. Lambert and Roberto Rittner.

Norell Press, 314 Arbor Avenue, Landisville, NJ 08326, U.S.A.; 1-800-222-0036, in NJ 609-697-0020; 1987; 185 pages; softcover $15.00, hardcover $25.00.

In the summer of 1986, an NMR workshop was held in Brazil to review the status of the field, both world-wide and specifically in Brazil. This volume is a compendium of the papers presented at that workshop, and has been edited by Joe Lambert and Roberto Rittner, both of whom also presented papers.

The chapters (in order of appearance) and authors are as follows: *The Production of Very High Fields for High Resolution NMR* (Bothner-By and Dadok); *Chemical Shift Theory. Orbital Symmetry and Charge Effects on Chemical Shifts* (Grutzner); *The IPP Method as a Complementary Tool for High Resolution Spectroscopy* (Contreras, Giribet, Ruiz de Azua, and Diz); *Spin-Spin Coupling in Peptides* (Barfield); *Spin-Lattice Relaxation as a Structural and Kinetic Tool* (Lambert); *Carbon-13 NMR Assignment, Structure, and Dynamics of Deoxyoligonucleotides* (Zanatta, Borer, and Levy); *Two Dimensional NMR Studies of Biomolecular Structure* (Wemmer); *Substituent Effects of alpha-Heteroatoms. Stereochemical Consequences* (Rittner); *Ambient Effects in Comformational Analysis as Detected by NMR Spectroscopy* (Eliel); *Carbon-13 NMR Spectroscopy of Oilseeds* (Seidl and Colnago); *Structure and Characterization of Trichothecenes* (Mazzola); *Molecular Structure Determinations by NMR Using Liquid Crystals* (Fujiwara); *NMR Imaging* (Panepucci, Beckmann, Tannus, and Bonagamba).

Since these articles are packed into a total of 185 pages (including a brief but adequate index), they are not in-depth studies. They are, however, uniformly good, brief reviews of the start of the art with respect to the subjects covered. This was, of course, what the workshop was intended to provide. The quality of the printing, illustrations, and production are all very satisfactory. All are appropriate for this useful 'proceedings' volume, which has been produced on a reasonable, useful time schedule after the workshop itself. Given the price, this volume is a very good value for its intended audience. The work should also prove useful in a variety of teaching situations.

W. B. S.

Continued from page 89:

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

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

All of the above fussbudgetness (Sorry, but I don't know corresponding term in other languages!) notwithstanding, we are suffering from an embarrassment of riches other than the fiscal kind. Your helpful attention to these practical matters will be appreciated. To those already doing a sterling job of saving space, etc., many thanks - your efforts are noticed and most gratefully received. Suggestions for improvement of the Newsletter or making the operation more efficient and cost-effective are always welcome.

B.L.S. 2/25/88

TAMU NMR Newsletter

Editor/Publisher: Bernard L. Shapiro
966 Elsinore Court, Palo Alto, CA 94303, U.S.A. (415) 493-5971

* * * Help ! Help ! Help ! **Help ! Help !** * * *

For the kind attention of all Newsletter recipients:

As evidenced by this record-size issue and the few immediately preceding it, the Newsletter has become rather large (Perhaps 'Newsletter' is no longer an accurate term, but we'll retain it for now.) While I am pleased that the number of subscriber/participants, sponsors and advertisers continues to grow, and interest in this basically self-regulating Newsletter shows no signs of atrophy, certain practical considerations have to be faced.

One factor is, of course, the financial viability of the Newsletter - I remind you that it is a completely self-supporting enterprise, which exists totally on funds raised from the three sources indicated above. Increases in funding have been gratifying in absolute terms, but have not kept pace with increased costs of labor, printing, supplies, and (especially) postage. While I believe the Newsletter will survive for the current subscription year which ends with the September 1988 issue, continuation beyond that month is predicated on success in the following:

1. Raising additional funding. Advertising rates were raised this year, and will not be raised again until at least September 1989. More advertising by present and new advertisers would help greatly. Our rates are modest and our readership is highly targetted - please enquire if you can help.

Subscription rates, on the other hand, have not been increased since October 1983, which is probably unprecedented in this age for anything you purchase but computer hardware. Costs have grown to the point where the academic/personal subscription rate is not fiscally viable. There is only so far one can push the economies of scale, for some of the Newsletter expense *rates*, most notably postage, are independent of the size of the issues (*v.i.*) or the number of copies mailed.

Accordingly, a modest increase in the basic subscription rate for current subscribers will go into effect with the summer invoicing for the October 1988 - September 1989 subscription year. The increased rate, to be set soon, will start now for any *new* subscriptions initiated after today's date. The Sponsorship rate, in effect for many years, will also be reviewed.

2. Decreasing the size of each Newsletter issue. The increasing numbers of pages in each Newsletter issue which are devoted to technical and other non-advertising material is a matter of concern, and this is an area in which significant savings can, I believe, be effected now. [To anticipate a suggestion certain to be made both facetiously and seriously, let me indicate my extreme reluctance to lower the frequency with which each subscriber must send something technical in to the Newsletter. I find it difficult to believe that one technical letter every 10 months or so is excessive, or needs to be a burden with a little planning ahead.]

Where significant savings of Newsletter pages and total space *can* be made however, is by exercising much better control over the formatting and type sizes of the contributions. **Please consider the following:**

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

ii) **PLEASE** avoid excessive margins. *Instruct your secretaries to avoid normal correspondence esthetics or practices, however time-honored or 'standard' !* This page has margins at the top and both sides of 0.6" (*ca.* 1.55 cm), which is very adequate. The same is enough for the bottom also, but don't worry if the bottom has more space at the end of your document, for I can often use such spaces for notices, etc. You are reminded that regardless of the standard paper length you use, *all material* - letterhead, text, figures, addresses printed at the page bottom, *everything - must not exceed 10" (ca. 25.3 cm) from top to bottom.*

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

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

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TEXAS A&M UNIVERSITY
NMR
NEWSLETTER

No. 354
March 1988

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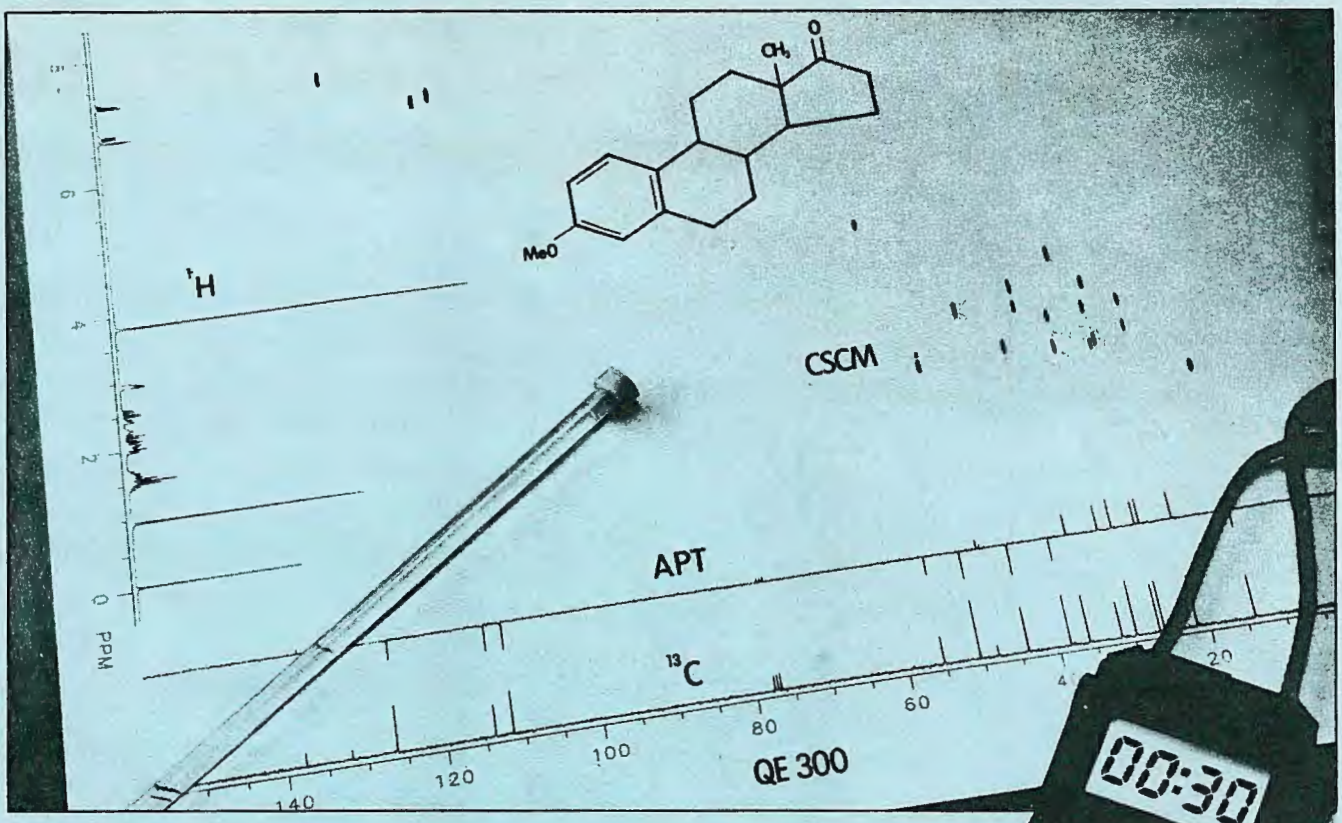
★ *New record!!*



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Subject: Automation

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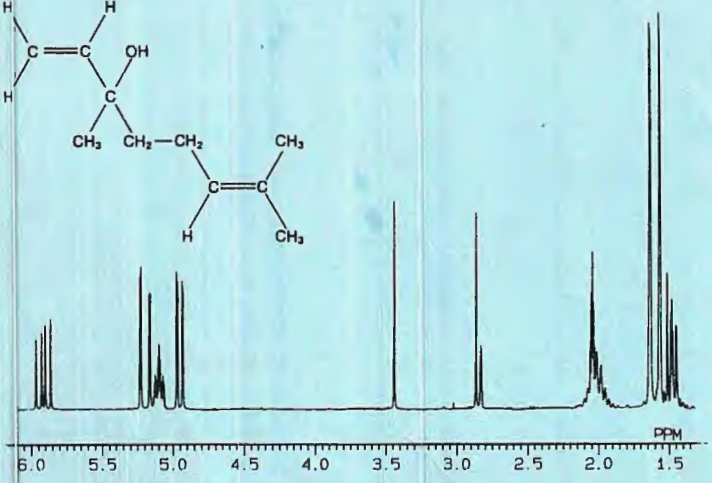
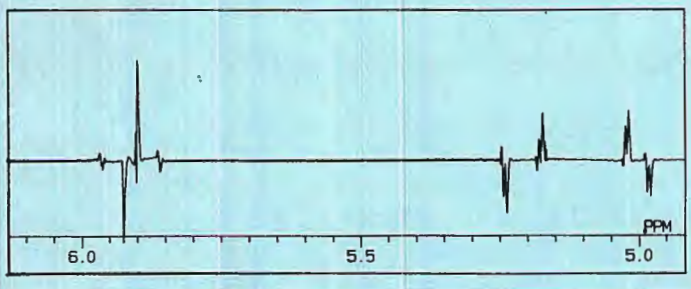
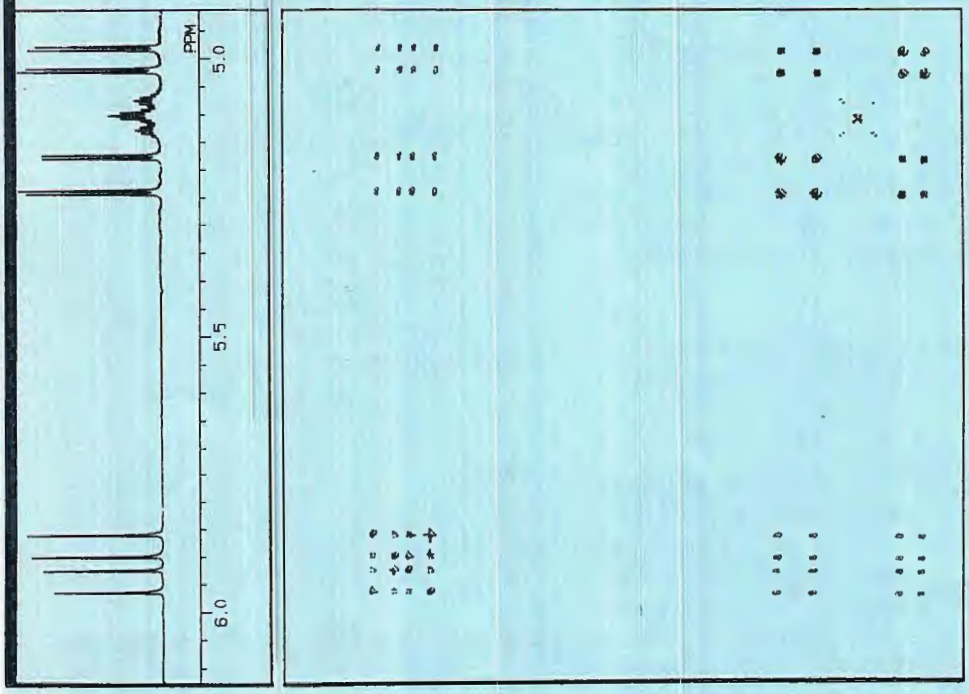
Linalool







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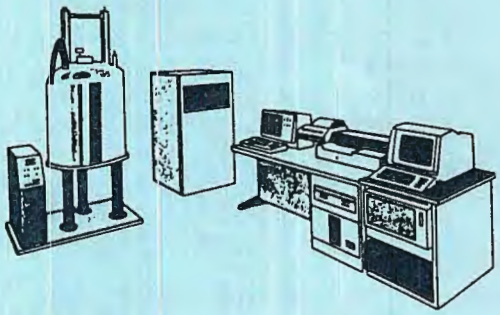

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