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NMR NEWSLETTER

NO. 314

NOVEMBER 1984

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	WGH-...	...% Orthodichlorobenzene in Acetone-d_6	...olution	33.00
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TEXAS A&M UNIVERSITY

NMR NEWSLETTER

AUTHOR INDEX --- TAMU NMR NEWSLETTER, NO. 314, NOVEMBER 1984

Forthcoming NMR Meetings (Additional listings are solicited)

26th ENC - April 21-25, 1985, Asilomar, Pacific Grove, California; Chairman: P. Mark Henrichs, Research Laboratories, Eastman Kodak Company, Rochester, New York 14650

Seventh International Meeting on NMR Spectroscopy - July 8-12, 1985, University of Cambridge - see p. 47.

Book Reviews: Another in the series of book reviews ably edited by **William B. Smith** of Texas Christian University appears onpage 46 of this issue. Bill and I would be pleased to receive reviews of other books from any Newsletter readers who would like to write about new books of substantial NMR content. Such reviews should be cast in a format similar to that of the example on page 46, and must be confined to one single-spaced typed page.

Suggestions for other types of articles, news items, etc., to appear in the Newsletter would be welcomed - please make your wishes known.

All Newsletter Correspondence
Should be Addressed to:

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

DEADLINE DATES

No. 316 -------- 7 January 1985

No. 317 ------- 4 February 1985

McMASTER UNIVERSITY
Department of Chemistry
1280 Main Street West, Hamilton, Ontario, L8S 4M1
Telephone: 525-9140

September 25, 1984

Professor B. L. Shapiro
Texas A & M University
College of Science
College Station, Texas 77843
U.S.A.

Dear Dr. Shapiro:

^{45}Sc NMR Spectrum of $Sc(BH_4)_3$

The use of transition metal hydrides in organic and organometallic synthesis has prompted studies of their tetrahydroborate precursors. Scandium-45 is an excellent nucleus in this regard, due to its abundance and relative sensitivity, thus allowing its observation on relatively modest equipment. The figure shows the ^{45}Sc spectrum of $Sc(BH_4)_3$ under various decoupling conditions obtained at 21.9 MHz on a Bruker WH 90; spectrum C reveals a proton - scandium 45 coupling of 30 Hz which is not readily observable in the proton spectrum.

Sincerely,

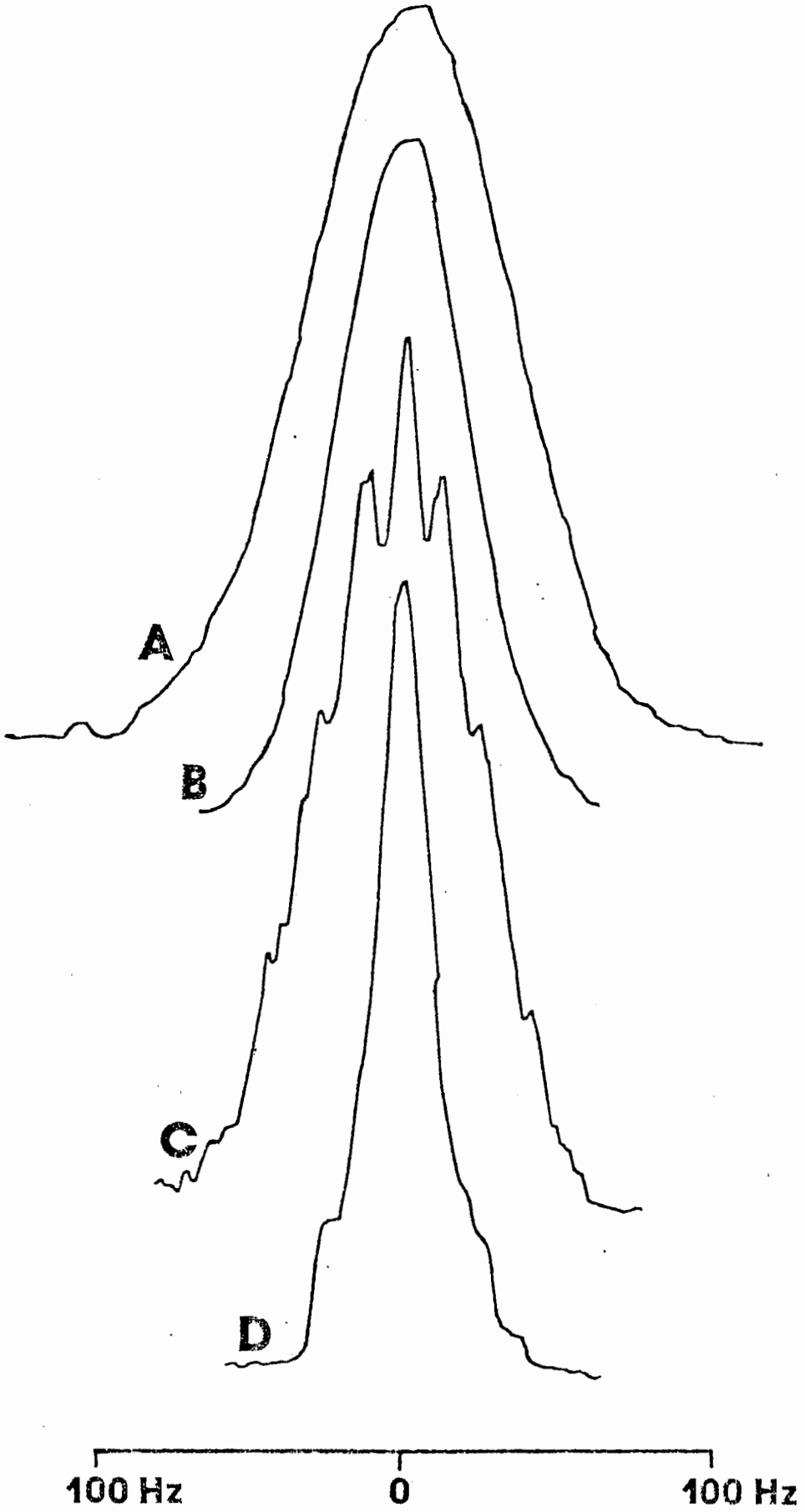
Brian Sayer Michael J. McGlinchey

Brian G. Sayer Michael J. McGlinchey

Please credit this contribution to the account of J.I.A. Thompson.

The Figure shows the ^{45}Sc NMR spectrum of $Sc(BH_4)_3$: (A) normal spectrum; (b) ^{1}H decoupled spectrum; (C) ^{11}B decoupled spectrum; (D) ^{1}H and ^{11}B decoupled spectrum.

BGS/MJMcG/cd


A
B
C
D
100 Hz
0
100 Hz

The Florida State University
Tallahassee, Florida 32306
Department of Chemistry

September 28, 1984

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

It's a dimer...No, it's the monomer

Dear Barry:

An unusual product, isolated as one of the components from the sodium borohydride reduction of 3-acetoxy-1-phenylbutanone was submitted recently for 270 MHz Proton NMR analyses and X-ray crystal structure. It proved to be the unexpected cyclic borate diester $C_{10}H_{13}O_3B$. (Fig. 1) Very few cyclic borate diesters are discussed in the literature.

The X-ray crystal structure[1] showed that the borate diester comprises three atoms of a six-membered ring. Intermolecular hydrogen bonding of the hydroxyl group of one ring with the ester oxygen atom of another ring leads to dimeric structure in the solid state.

^1H NMR studies[1] showed that the dimer structure persists in $CDCl_3$ solutions (Fig. 2A). The extensive chemical shift to higher field of the hydroxyl proton upon either dilution or increasing temperature implied the existence of association of the molecules.[2] Negative NOE enhancements for the hydroxyl proton also implied intermolecular exchange.[3,4] Addition of D_2O to saturate the $CDCl_3$ solution gave two sets of signals (Fig. 2B) corresponding to two different species in equilibrium. The new component had the same multiplet coupling patterns (determined from homonuclear decoupling experiments) but less rigidity was inferred from the more equivalent nature of protons H2a and H2b as seen in Fig. 2B.

It seems that the new component is a reasonably stable monomeric adduct with water, because step-wise addition of acetonitrile (to about 5%) increased the concentration of the adduct (Fig. 2C).

The crystallographic data and NMR chemical shift, coupling constants, and difference nuclear Overhauser enhancements are to be published in several months.

Sincerely,

Thomas Gedris
Virgil Goedken
Richard Rosanske

TG:sd

1. Submitted for publication.
2. J. A. Pople, N. G. Schneider and H. J. Bernstein, "High Resolution Nuclear Magnetic Resonance", Chap. 15, McGraw-Hill Book Company, Inc., New York, 1959.
3. J. Feeney and H. Heinrich, Chem. Commun., p. 265 (1966).
4. J. H. Noggle and R. E. Schermer, "Nuclear Overhauser Effect", Chap. 7, Academic Press, New York, 1971.

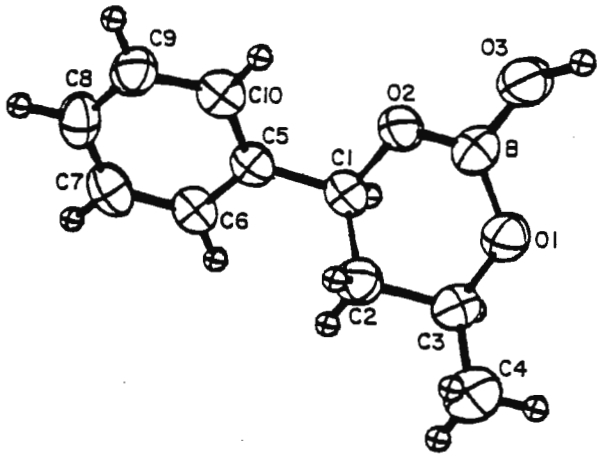


Figure 1. ORTEP plot of the cyclic borate ester, $C_{10}H_{13}O_3B$, with labeling scheme.

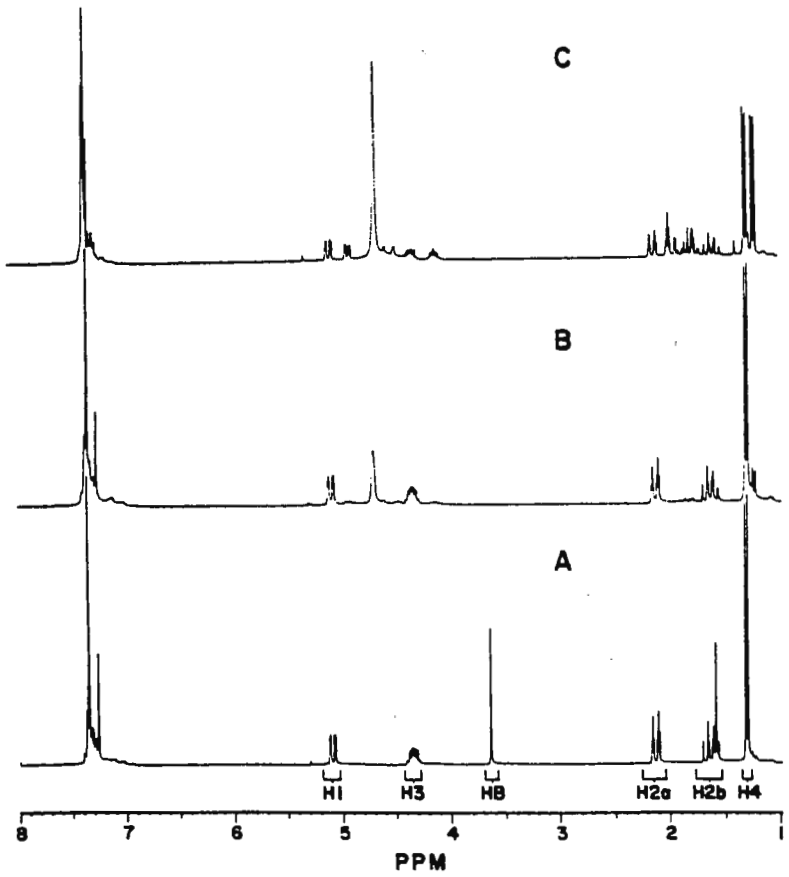


Figure 2. 270.13 MH_z ^1H NMR Spectra of $C_{10}H_{13}O_3B$ showing A) dimeric structure in $CDC\ell_3$ B) dimer/monomer equilibrium in D_2O saturated $CDC\ell_3$; and C) increased monomer concentration after addition of CD_3CN to about 5% total volume.

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PHYSICS DEPARTMENT
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(317) 923-1321

September 24, 1984

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

Title: Distinction Between Line Widths of Identical and Non-identical Dipolar Coupled Systems with and without Exchange

Dear Barry:

Recently we have found that Eq. (89) on p. 296 of Abragam's book, "The Principles of Nuclear Magnetism," is incomplete when the chemical shift between the two spins is comparable to the linewidths. The complete equation should be

$$\frac{d}{dt}\langle I_x\rangle^* = -\frac{\langle I_x\rangle^*}{T_2^I} - \frac{\langle S_x\rangle^*}{T_2^{IS}} \qquad [1]$$

where

$$\frac{1}{T_2^I} = \frac{3}{4}\gamma_I^2\gamma_S^2\hbar^2\left\{\frac{1}{6}J^0(0) + \frac{1}{24}J^0(\omega_I - \omega_S) + \frac{3}{4}J^1(\omega_I) + \frac{3}{2}J^1(\omega_S) + \frac{3}{8}J^2(\omega_I + \omega_S)\right\} \qquad [2]$$

and

$$\frac{1}{T_2^{IS}} = \frac{3}{4}\gamma_I^2\gamma_S^2\hbar^2\left\{\frac{1}{12}J^0(0) + \frac{1}{12}J^0(\omega_I - \omega_S) + \frac{3}{4}J^1(\omega_I) + \frac{3}{4}J^1(\omega_S)\right\} \qquad [3]$$

So for both the longitudinal and the transverse magnetization components one needs coupled equations. The complete solution allows for a continuous transition from the linewidths of the non-identical separate lines to the collapsed line for identical spins. Moreover when non-identical lines collapse under the influence of chemical exchange, the collapsed line has a width of $1/T_2^I + 1/T_2^{IS}$ which is contrary to the usual "dogma" that the exchange collapsed width is equal to that of the non-exchanging separated pair. Details are given in an article submitted to the Journal of Magnetic Resonance for publication.

Sincerely yours,

K. V. Vasavada and J. I. Kaplan

Please credit this contribution to the account of Dr. B. D. Nageswara Rao, Department of Physics, Indiana Univ.-Purdue Univ. at Indianapolis, Indpls., IN 46223

314-7

The University of Sydney

N.S.W. 2006
Department of Organic Chemistry

IN REPLY PLEASE QUOTE:

TELEPHONE: 692 1122.

Professor B.L. Shapiro
Department of Chemistry
Texas A and M University
College Station
Texas 77843, USA

October 2, 1984

Dear Barry,

High Field NMR for analysis of volatiles.
Measurement of Methane and Deuteromethane in Gas Mixtures.

In the course of some mechanistic studies involving organometallic compounds, we needed to analyse a gas mixture containing methane and deuteromethane in nitrogen.

A sample of the gas mixture (containing ca. 15-20 ug. of methane-deuteromethane) was injected into an empty (nitrogen-filled), 5 mm NMR tube fitted with a septum cap. The proton NMR spectrum of the gas was obtained on our Bruker WM400 spectrometer, operated in an unlocked mode. A spectrum (Fig. 1) constisting of a single broad resonance ($W_{\frac{1}{2}}$ ca. 20 Hz) was obtained in about 200 scans. Traces of benzene and other volatiles also gave broad peaks.

On addition of cyclohexane-d12 (0.5 ml) directly into the NMR tube through the septum cap, the gaseous hydrocarbons dissolved and the spectrum shown in Fig. 2 was obtained. Methane (at δ 0.223 ppm) and deuteromethane (at δ 0.208 ppm, J_{H-D} = 1.93 Hz, undoubtedly -ve) are conveniently separated by some 6 Hz due to the deuterium isotope effect.

This simple experiment provides a very convenient method for observing (qualitatively) and probably measuring (quantitatively), the various deuteromethanes in a gas mixture. The scale on which these measurements were made clearly demonstrates that the sensitivity of modern NMR instruments makes NMR spectroscopy a practical analytical method even at the microgram level.

One does not normally consider NMR spectroscopy as an analytical method for volatile materials but this experiment was fair-dinkum because the detection of deuteromethanes by Mass Spectrometry is difficult due to its low molecular weight and overlap with the ever-present water peaks.

Yours sincerely

L.D. Field

S. Sternhell

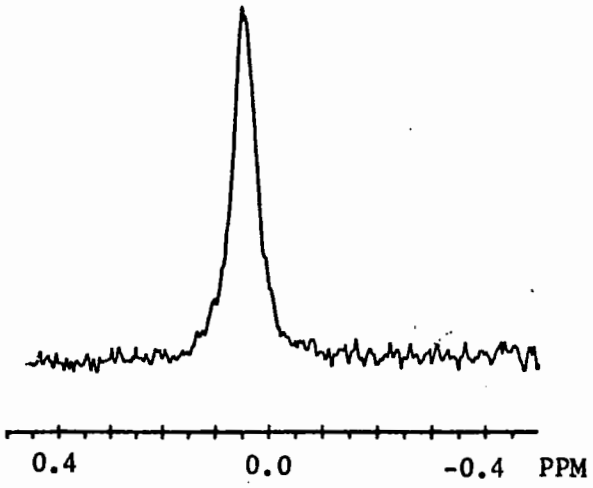

Fig 1. ^{1}H NMR Spectrum of a gas mixture containing methane and deuteromethane (400 MHz, shifts referenced to benzene vapour taken to be 7.15 PPM).

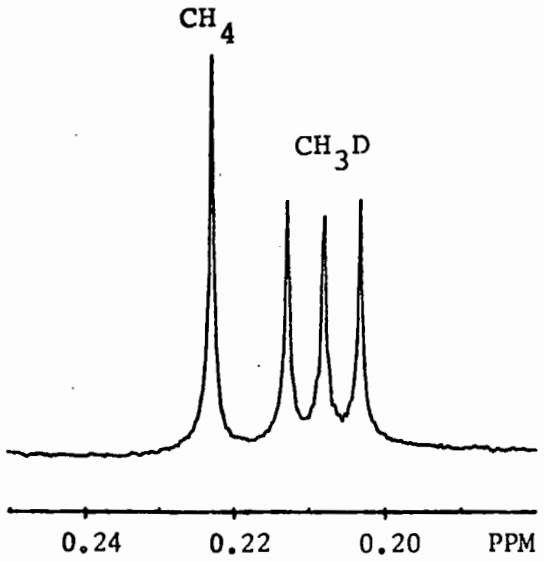

Fig 2. ^{1}H NMR spectrum recorded after adding C_6D_{12} (0.5ml) to the above sample (400 MHz, shifts are referenced to residual cyclohexane protons in the solvent, taken to be 1.42 PPM).

ES-3178 REV. 1/81

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CENTRAL RESEARCH & DEVELOPMENT DEPARTMENT
EXPERIMENTAL STATION

1984 October 4

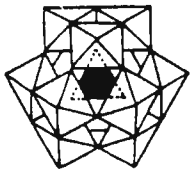
Dr. Bernard J. Shapiro
Department of Chemistry
Texas A&M University
College Station, TX 77843

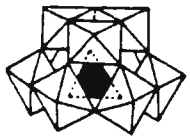
Dear Barry,

P-31 Chemical Shift Anisotropy of Phosphotungstates

Sites which exhibit very similar isotropic chemical shifts can have very different chemical shift anisotropies (CSAs). We are finding this to be a very useful feature of the P-31 NMR spectra of solid phosphotungstates.

Fragments of the complete dodecahedral Keggin structure from which a triad of tungstate groups have been removed are designated A- or B-type. They differ in that the central atom is bonded to either four bridging oxygens (A-type) or three bridging and one terminal oxygen (B-type). One phosphotungstate of this class, $W_9PO_{34}^{9-}$, has been reported and assigned a B-type stereochemistry with little or no evidence. The P-31 CSA clearly shows that this ion is actually A-$W_9PO_{34}^{9-}$. Thermolysis of solid A-W_9P (see (a)) at 140 to 180 C yields predominately B-W_9P (see (b)).

Keggin $W_{12}P$ 

A-type W_9P 

B-type W_9P 

Our interpretation is verified by examining the CSAs of $W_{18}P_2O_{62}^{6-}$, an anion of known crystal structure containing two A-W_9P groups. The spectrum (c) reflects a mixture of two isomers expected for this salt. Also, its fragment $W_{15}P_2$ has an A- and a B-type site (d), with CSAs comparable to those of the W_9P fragments.

An A-type site should have a small CSA, since it has four nearly equivalent tetrahedral bonds. The large, negative CSA of the B-type site is consistent with the CSAs of solid phosphates (Grimmer, Haubenreisser, Chem. Phys. Lett., 1983, 99, 487). The CSA of end groups (PO_3OP) are large and positive (126 ppm in $K_4P_2O_7$, 146 ppm in $K_5P_3O_{10}$), while the CSA of a branching group

$(PO(OP)_3)$ is large and negative (-295 ppm in h-P_4O_{10}). (Here, CSA is defined as $\delta_{\parallel} - \delta_{\perp}$, with positive δ to low field.)

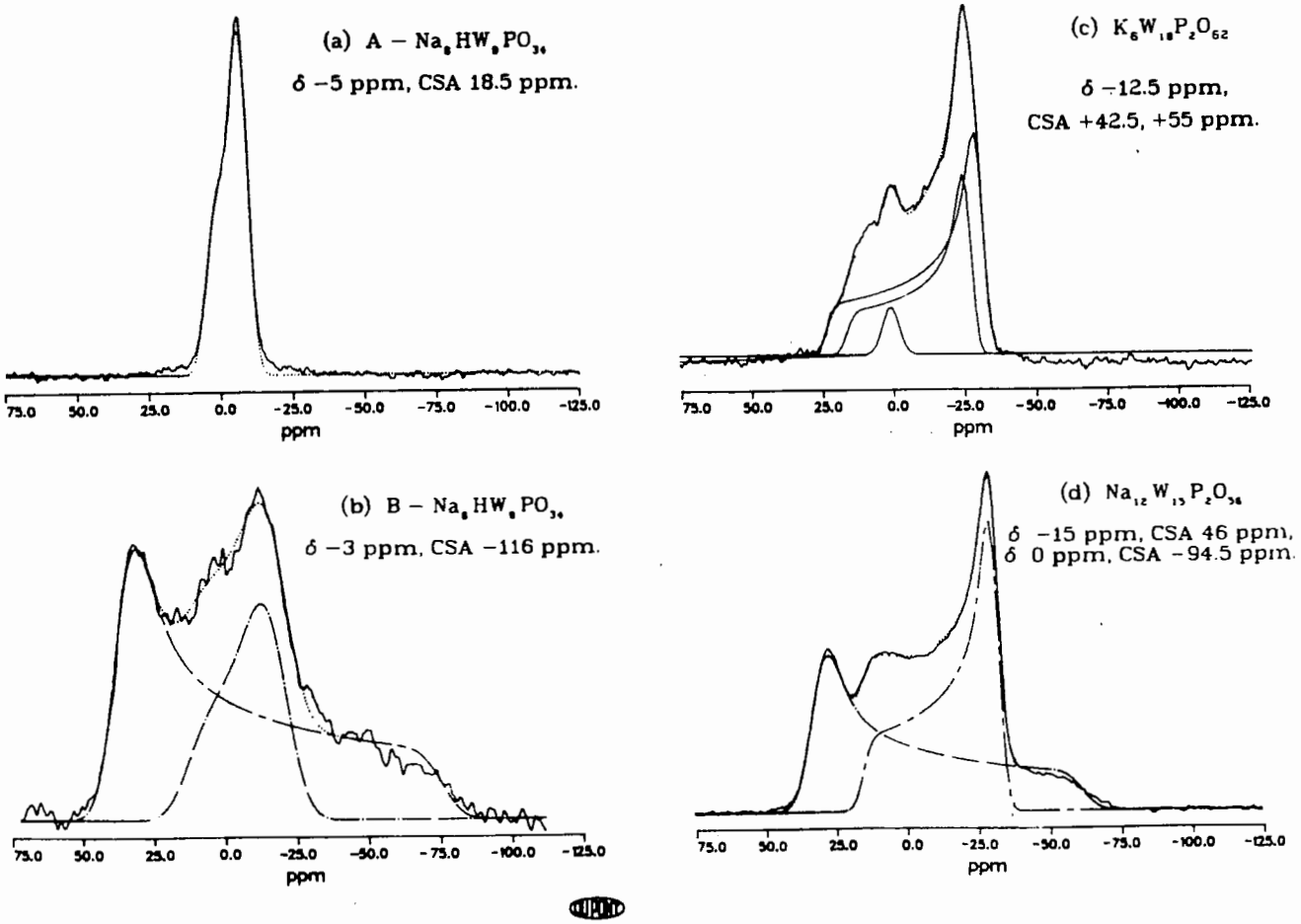
The CSAs are extracted by fitting nonspinning P-31 spectra (121.4 MHz on a CXP-300) obtained under quantitative conditions (typically, 30° pulse, 10 s recycle, 40 kHz decoupling). The fit (dashed lines below) is constrained to match the isotropic chemical shifts of the MAS spectrum, and is performed by software written by Mike Hanafey at Du Pont.

NMR of solids allows samples that are (like many heteropolyanions) insoluble, microcrystalline, or unstable in solution to be readily studied. We expect this to be a useful tool in understanding the structure of transition metal-substituted heteropolyanions.

Further details will be published in Organometallics. Please credit D. D. Bly's subscription.

Sincerely,

Rod Farlee Walt Knoth Peter Domaille



314-11

University of Nottingham

Department of Chemistry

UNIVERSITY PARK NOTTINGHAM NG7 2RD
TEL NOTTINGHAM 56101

LC/JL

1st October 1984

Professor B.L. Shapiro,
Department of Chemistry,
Texas A & M University,
College Station,
Texas 77843
U.S.A.

Dear Professor Shapiro,

Observation of $^{3}J_{HCNH}$

The observation of $^{3}J_{HCNH}$ coupling in amines requires relatively slow intermolecular exchange of NH protons. The required condition has usually been satisfied by lowering of temperature[1,2] and/or addition of basic alumina[3,4] to the n.m.r. sample tube. The state of both sample and solvent are of importance, and the need for very low temperature may be avoided by using thoroughly dried reagents. In the case of $CDCl_3$, the use of solvent from a freshly opened bottle is helpful, probably owing to the absence of HCl.[4] However, on one occasion we had to compromise by using 'aged' $CDCl_3$; in this way we avoided a situation in which broadening due to intermolecular N-H exchange occurred simultaneously with that due to a ring inversion.

Yours sincerely,

Harold Booth.

Dr. H. Booth
K.A. Khedhair.

1. H. Booth and R.U. Lemieux, Can.J.Chem., 1971, 49, 777.
2. P. Salvadori et.al., J.Chem.Soc., Perkin Trans.II, 1983, 1919.
3. F.A.L. Anet and I. Yavari, J.Am.Chem.Soc., 1977, 99, 2794.
4. cf. F.A.L. Anet and Xiuo-hui Ji, Tet.Letters, 1984, 1419.

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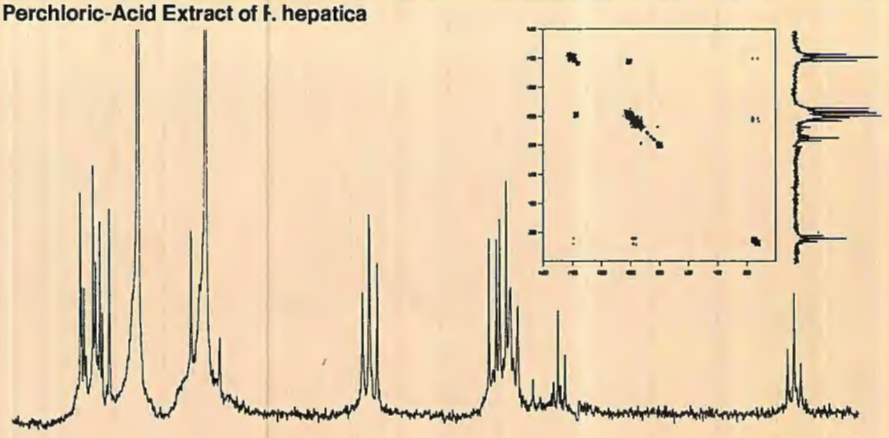
XL performance for demanding biological NMR studies

Recently, NMR has become an important tool for biochemists interested in studying metabolism *in vivo*. The applications shown on this page illustrate the broad range of capabilities required in an NMR spectrometer used in biological research. These capabilities demand superb sensitivity, flexibility in pulse programming, and software that permits taking advantage of available experiments and techniques.

Perchloric-Acid Extract of F. hepatica

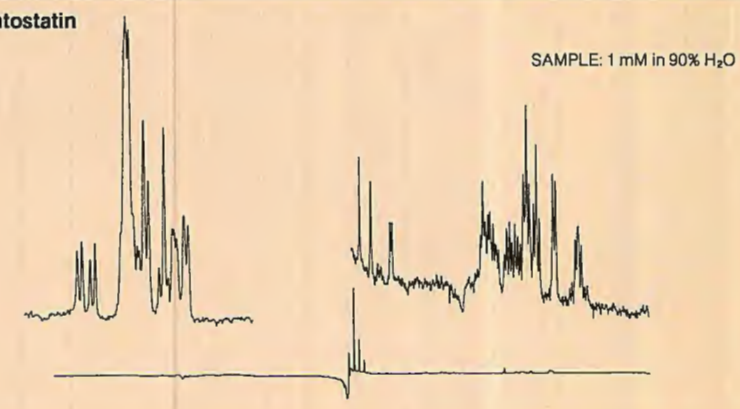


The high-sensitivity probes of the XL-200 allow spectra to be obtained from biologically relevant compounds at low concentration. The ^{31}P spectrum above is of a perchloric-acid extract of F. hepatica (bovine liver flukes) and was obtained in 4 hours (1500 transients) at 81 MHz, using a 10-mm probe. The spectrum has been resolution-enhanced to facilitate the identification of the ^{31}P-containing compounds present. The average concentrations of metabolites present in the sample are submillimolar.

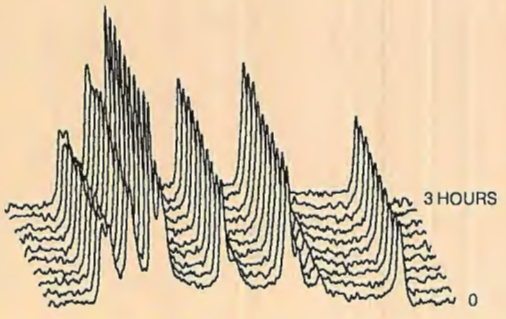
Two-dimensional NMR is a powerful method for analyzing complex mixtures. The contour plot shown above the spectrum is the result of a ^{31}P homonuclear shift correlation experiment carried out on a portion of the fluke extract using a 5-mm ^{1}H broadband switchable probe at 81 MHz. The total experiment time was approximately 12 hours. The experiment led to the discovery of a nucleotide pyrophosphate compound whose presence is indicated by the cross peaks between the P-α-nucleotide peaks and the peak at 800 Hz.

Biological NMR often requires the observation of protons in H_2O, particularly for observation of exchangeable protons. The strong signal from the solvent can be suppressed effectively by pulse sequences such as time-shared Redfield 2-1-4 or, as here, the Jump-and-Return pulse sequence. This XL-400 spectrum is the result of only 16 accumulations using a 1-millimolar solution. The aromatic expansion (rephased for upright presentation) shows the single-proton sensitivity that can be obtained in a half-minute period.

^{1}H Somatostatin

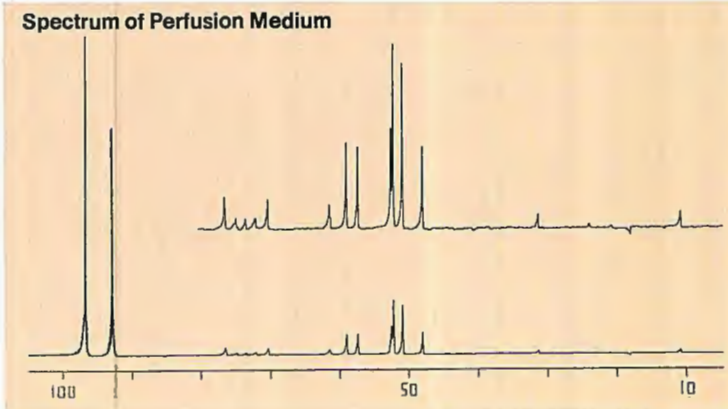


Perfusion of Tissue



Use of a modified 10-mm tube permits the NMR study of intact tissue while perfusing with temperature-controlled nutrient. No hardware modification of the spectrometer is necessary. The stacked plot shows the time course of ATP resonances during tissue perfusion with a nutrient medium and illustrates how tissue preparations can be maintained in a viable state during experiments. Insufficient or poor perfusion causes rapid degradation of the ATP and resultant cell or organism death. The ability to retain viability over many hours permits extensive study of metabolism in metabolic, nutrative, and cell research.

Spectrum of Perfusion Medium



NMR is a valuable tool for following metabolism in isotope labeling experiments. This spectrum is of the perfusion medium taken at the end of an experiment in which the bovine liver flukes (above) were perfused with (1-^{13}C) glucose. A large number of labeled species are formed as the (1-^{13}C) glucose is metabolized. Subsequent analysis of the sample using spectral editing pulse sequences and heteronuclear correlation experiments are essential for assignment of these resonances.

UNIVERSIDAD DE BUENOS AIRES
FACULTAD DE CIENCIAS EXACTAS Y NATURALES

Buenos Aires, October 1, 1984.

Professor B.L.Shapiro
DEpartment of Chemistry
Texas A & M University
College Station, Texas 77843.
U.S.A.

Title: Proton Deshieldingby Spatial Proximity.

Dear Prof. Shapiro:

The effect of the spatial proximityof molecular fragments on chemical shifts has been reported several times.However a quantitative evaluation of this effect is still lacking.Lately we have undertaken this study in ortho-substituted anisoles and so far we have concentrated on proton chemical shifts.Applying the aditivity rule of substituent effects we were able to quantitatively estimatethe influence of a cis-conformation of the methoxy group on the proton chemical shift.We have also studied this problemfrom a theoretical viewpoint.To this end a program was written in this laboratory which allows the calculation of the magnetic shielding tensorat the uncoupled Hartree-Fock approximation at the INDO level using GIAO's in the basis set.Good agreement between experimental estimate and the theoretical prediction of this deshielding effect was obtained.This result points towardsthe following conclusion:although it is very difficult to achieve a quantitative agreement between calculated and experimental chemical shifts,there are some contributions to the magnetic shielding tensorwhich canbe described using this simple approximation.

These results are in press in the Organic Magnetic Resonance.
Please credit this contribution to Prof.Dr. V.J.Kowalewski.
Yours sincerely,

Dr. Dora G. de Kowalewski

Prof. Dr. R.H.Contreras

UNIVERSITY OF STOCKHOLM
Physical Chemistry

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

Stockholm, October 12, 1984

VARIABLE TEMPERATURE MULTIPLE FIELD Cd-113 RELAXATION

Dear Dr Shapiro,

I apologize for the delay with my technical contribution to the TAMU Newsletters, caused by the hectic state of things here during the last few months.

Jim Dechter, at the University of Alabama, and myself have during last few years been cooperating on combined C-13 and metal nucleus relaxation studies for metal acetylacetonates (1,2). We have recently completed an investigation of Cd(acac)2 i DMSO-d6 solution. We have used three different magnetic fields: 2.35, 4.7 and 9.4 Tesla and covered temperature range of 20°C to 80°C. The results are collected in figure 1. The circles are experimental points and the solid lines are calculated assuming that all the field dependence comes from the CSA mechanism and that the field independent relaxation consists of the SR and DD mechanisms, the latter being separable by NOE experiments. The analysis of the various contributions at 4.7 Tesla is shown in Figure 2. The shielding anisotropy contribution is amenable for further analysis if the shielding tensor is assumed cylindrically symmetric, the reorientations is assumed isotropic and the correlation times are taken from C-13 relaxation times. This leads to the shielding anisotropy for Cd-113, $\Delta\sigma$, of about 150 ppm.

Yours sincerely
Jozef Kowalewski

1. J.J.Dechter, U.Henriksson, J.Kowalewski and A.C.Nilsson, J. Magn. Res., 48, 503 (1982).
2. J.J.Dechter and J.Kowalewski, J. Magn.Res., 59, 146 (1984).

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FIG. 1.

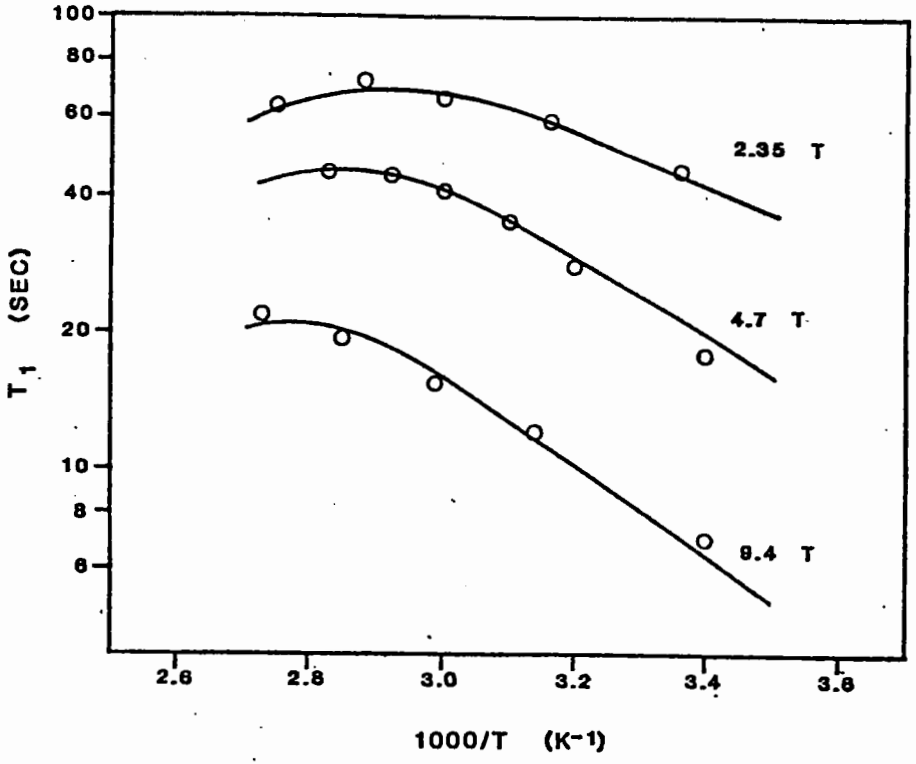
100
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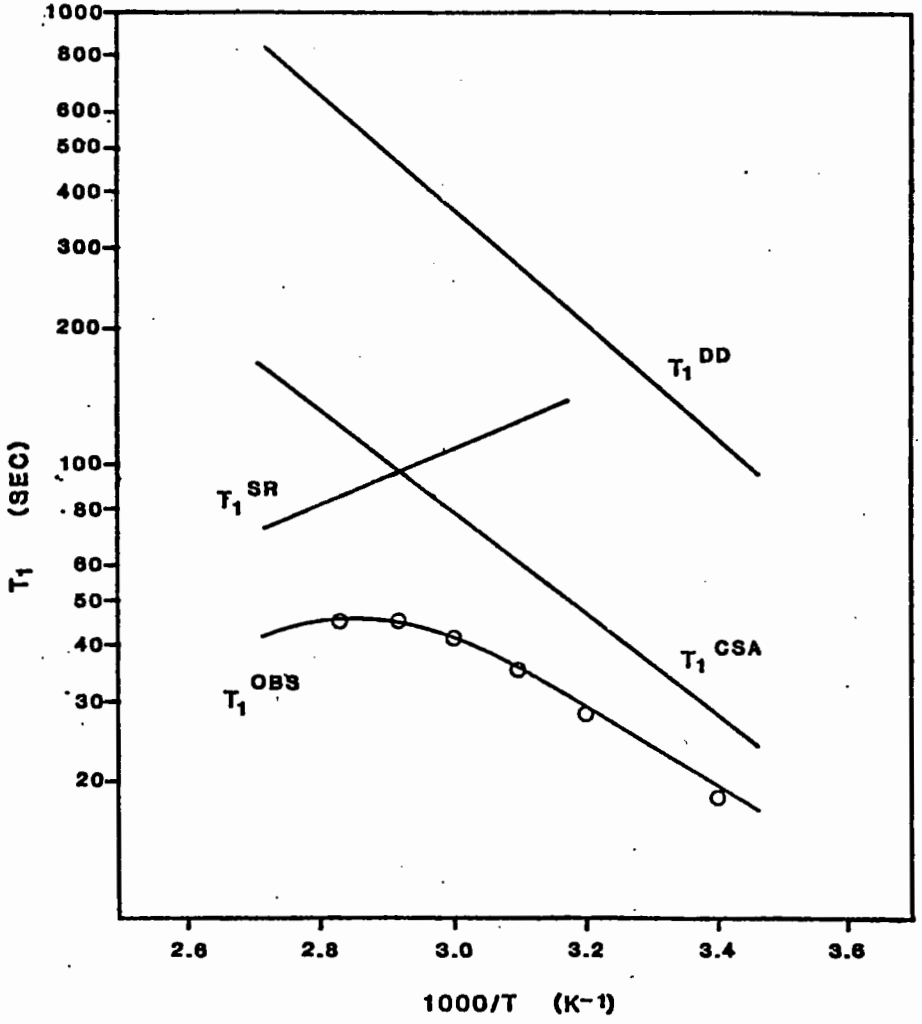
FIG. 2.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1085

Prof. B. L. Shapiro
Editor and Publisher
TAMU NMR Newsletter
Texas A&M University
Dept. of Chemistry

College Station, Texas 77843
U.S.A.

Zürich, October 1, 1984

Experimental Aspects of Relayed Coherence Transfer Experiments Applied to Small Proteins

Dear Dr. Shapiro,

Relayed Coherence Transfer (RCT) is a powerful method for identification of connectivities between spins within a spin system with zero or very small J couplings. This connectivity is established via a mutual coupling partner, thus, after a standard COSY-type magnetization transfer from spin i to spin j, magnetization is relayed to a third spin k:

$$H_i(\omega_1) \xrightarrow{\text{COSY}} H_j \xrightarrow{\text{RELAY}} H_k(\omega_2)$$

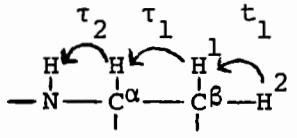
These experiments require that the relay period be adjusted (tuned) to a particular set of J couplings in the spin system of interest. By carrying out simulations, it is possible to maximize the relay for a variety of systems in a single experiment. The efficiency of the relay step is given by:

$$E = \sin \pi J_{ij}\tau \sin \pi J_{jk}\tau \prod_l \cos \pi J_{jl}\tau \qquad l \neq i,j,k$$

"Tuning" of the experiment involves selecting a τ which will optimize E.

One particularly useful application of RCT in small proteins is for the unambiguous identification of the amide, C^α, C^β, and C^γ protons of the amino acid residues in water solutions. Ordinarily, only the amide/$C^\alpha H$ connectivity is made in ^1H_2O; then, using the C^α proton chemical shift as a marker, the rest of the spin system is identified in ^2H_2O. Small differences in temperature or pH (p^1H versus p^2H), and crowding in the main $C^\alpha H$ region, leads to a certain degree of uncertainty in assignments. Using the RCT experiments in H_2O, it is possible to simultaneously establish $C^\alpha H$, $C^\beta H$, and $C^\gamma H$ connectivities to the more widely dispersed amide protons (figure 1).

In small globular proteins, the single relay experiment usually gives the connectivity between the amide and one of the C^{β} protons (as well as the COSY connectivity between amide and $C^{\alpha}H$). The second β proton is usually not observed, due to its small coupling constant to $C^{\alpha}H$ (magnetization transfer from β to α is inefficient via COSY). To avoid this problem, a double-relay experiment can be tuned for the following pathway:



$J_{\alpha\beta 1} > J_{\alpha\beta 2}$

These experiments have been used for assigning the ^1H spectrum of a chemically modified derivative of the small protein (58 amino acids) BPTI, circular BPTI. In a relay experiment connectivity between one β proton and the corresponding amide proton was observed for 39 of the 48 amino acid residues with observable amides (and at least one β proton). In an appropriately tuned double-relay spectrum, 60 out of the 84 possible amide-C^{β} proton connectivities were observed. In the accompanying figure, a few typical examples are given.

Sincerely yours,

W. Walter

Walter Chazin

References

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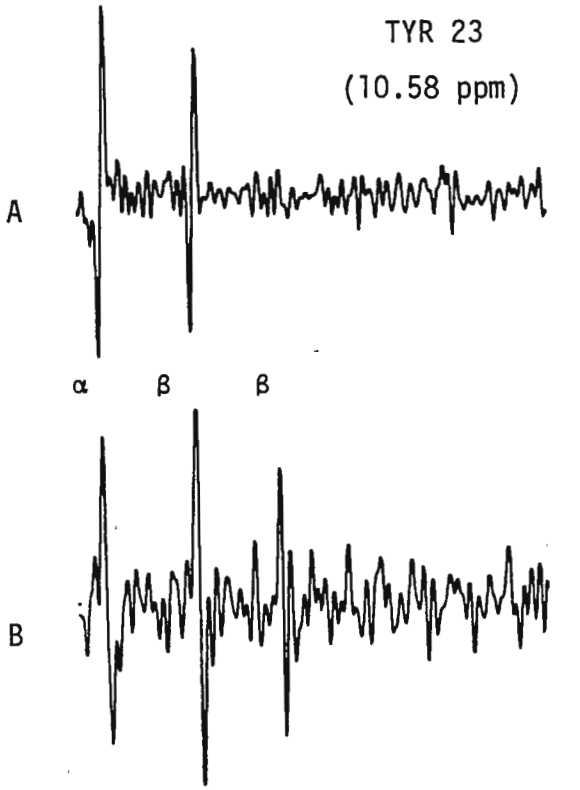
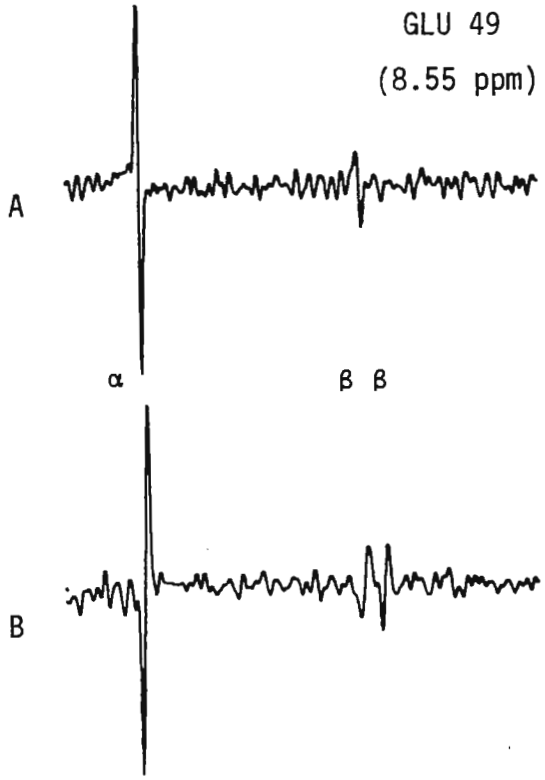
G. Wagner, J. Magn. Reson. 55, 151 (1983).

"please credit this contribution to the subscription of Kurt Wüthrich"





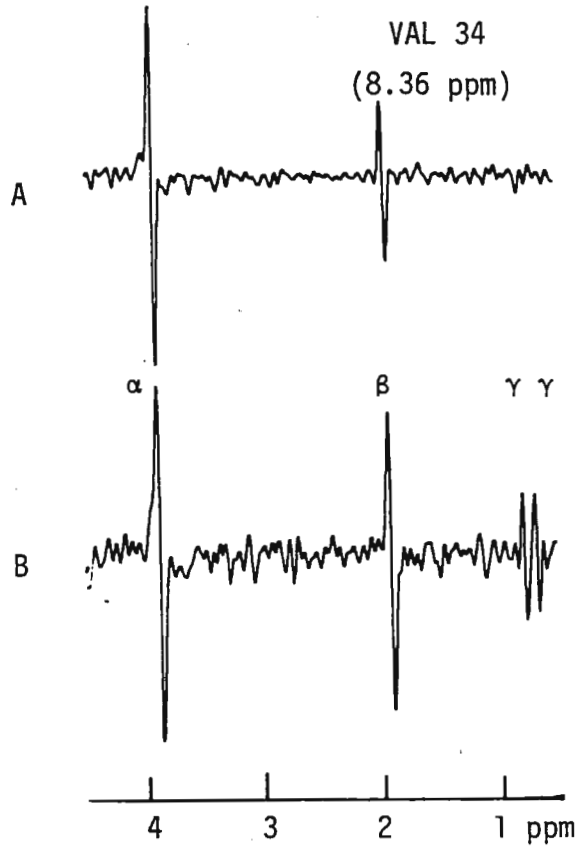
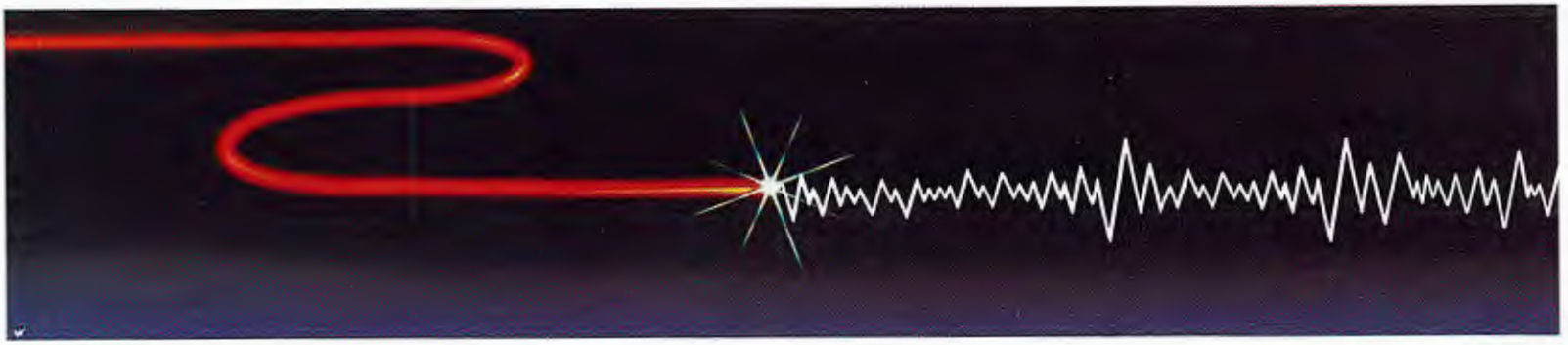


Figure 1

Relayed Coherence Transfer (A) and Double-Relayed Coherence Transfer (B) experiments on CBPTI (8 mM in H_2O/ 10% D_2O, pH = 4.6, 36°C, at 500 MHz). The pulse sequence and spectral parameters can be obtained upon request. Columns along ω_1 from the phase-sensitive spectra, corresponding to the amide (NH) proton chemical shift in ω_2 (given in parenthesis), have been plotted to show NH/H^{α}, NH/H^{β}, and NH/H^{γ} connectivities. The examples were chosen to demonstrate typical results for three different spin systems.

The NMR evolution continues:



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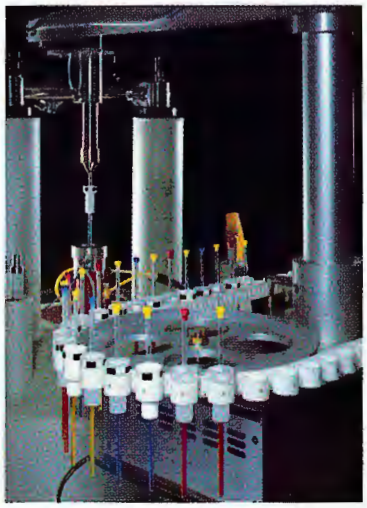

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Directeur : Professeur J. REISSE

September 22, 1984.

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

Re : J modulated spin echo accessory (2nd version)

Dear Professor Shapiro,

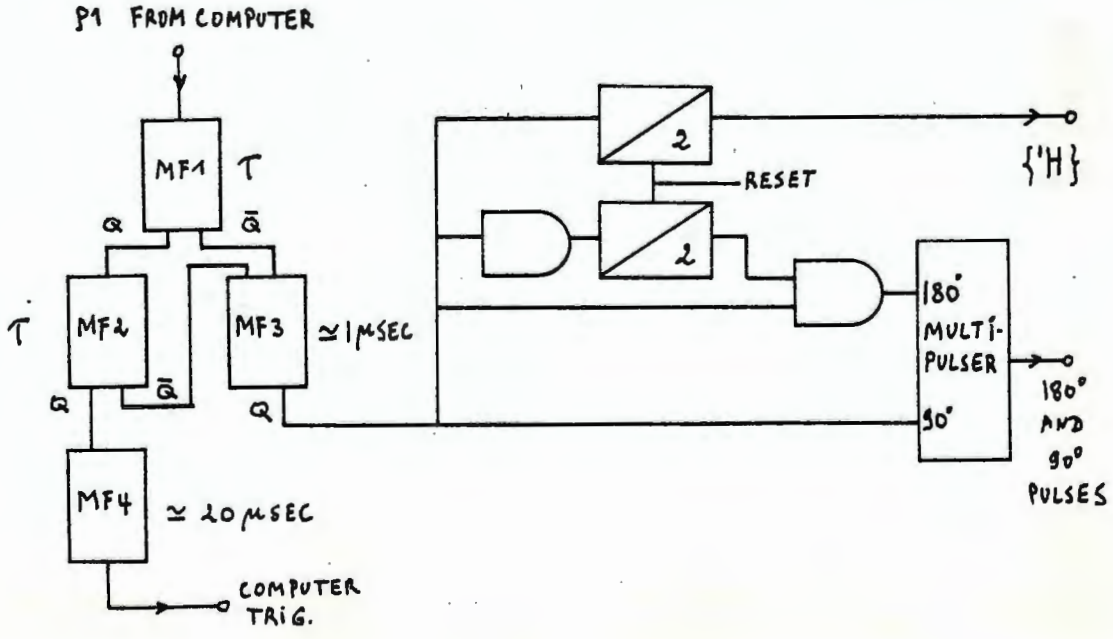
In a previous contribution(1) we described an accessory for a Bruker WP 60 spectrometer (equipped with a BNC 12 computer) which would enable it to perform J modulated spin echo (2). In order to avoid the unstable delay generated by the computer (Tau = L/5 ≃ 6.5 ms), we have modified our previous circuit as shown in the figure. The measurement is now set under way by a P1 pulse, and the acquisition begins under the control of the external trigger (PT).

Yours sincerely,

O. FABRE

D. ZIMMERMANN

(1) C. Lecocq, J. Lallemand, J.C.S. Chem. Comm. 1981, 150
(2) O. Fabre, D. Zimmermann, TAMU NMR Newsletter 291, 27 (1982).



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

October, I7, I984

Title: Molecular structure of Cs^+-gramicidin A complex

Dear Barry:

The double helical nature of gramicidin A (GA) dimers in organic solvents was originally proposed by Veatch, Fossel and Blout[1]. The molecular structures of two of these dimers in dioxane were evaluated by 2D NMR spectroscopy[2-4] - one as the left-handed antiparallel $\uparrow\downarrow\P\P^{5}_{LD}\cdot^{6}$ double helix, and another as the right-handed parallel $\uparrow\downarrow\P\P^{5}_{LD}\cdot^{6}$ double helix, both with 5.6 residues per turn.

Recently we were lucky to individualize the metal-GA complex in the I:I mixture of methanol and chloroform. With addition of caesium salt (CsCNS) the intensity and chemical shift of signals, arising from the complexed form are changing (see Figure) in comfirmity with a two-site binding reaction. Total signal assignment for the final I:I Cs^+-GA complex was achieved by analysis of COSY and NOESY spectra. By combined consideration of obtained NMR data (NOE commectivities, amide NH deuterium exchange rates and vicinal proton $H\text{-}NC^{\alpha}\text{-}H$ couplings) the molecular structure of the complex was evaluated as the right-handed antiparallel $\uparrow\downarrow\P\P\P^{7}_{LD}\cdot^{2}$ double helix with 7.2 residues per turn.

This cylindrical structure with 2.7 nm length and 0.4 nm diameter of the internal axial cavity is suited to accomodate caesium cations, which being 0.33 nm in diameter demonstrate greater than other alkali metal cations permeability via GA channels in bilayer phospholipid membrane.

The NMR study of GA conformations in organic solvents reveals stability of double helical structures, which have to be taken into account in further consideration of mechanism of GA induced membrane ion conductance and biological action of GA as endogenous inhibitor of RNA-polymerase of producing microorganism[5].

With best wishes,

Sincerely yours

V.F.Bystrov and A.S.Arseniev

References:

I. W.R.Veatch, E.T.Fossel, E.R.Blout, Biochemistry I3, 5249 (I974).
2. A.S.Arseniev, V.F.Bystrov, V.T.Ivanov, Yu.A.Ovchinnikov, FEBS Lett. I65, 5I (I984).
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5. Yu.A.Ovchinnikov, V.T.Ivanov, in: Conformation in Biology (Eds. R.Srinivasan, R.H.Sarma), Acad. Press, I982, p. I55.

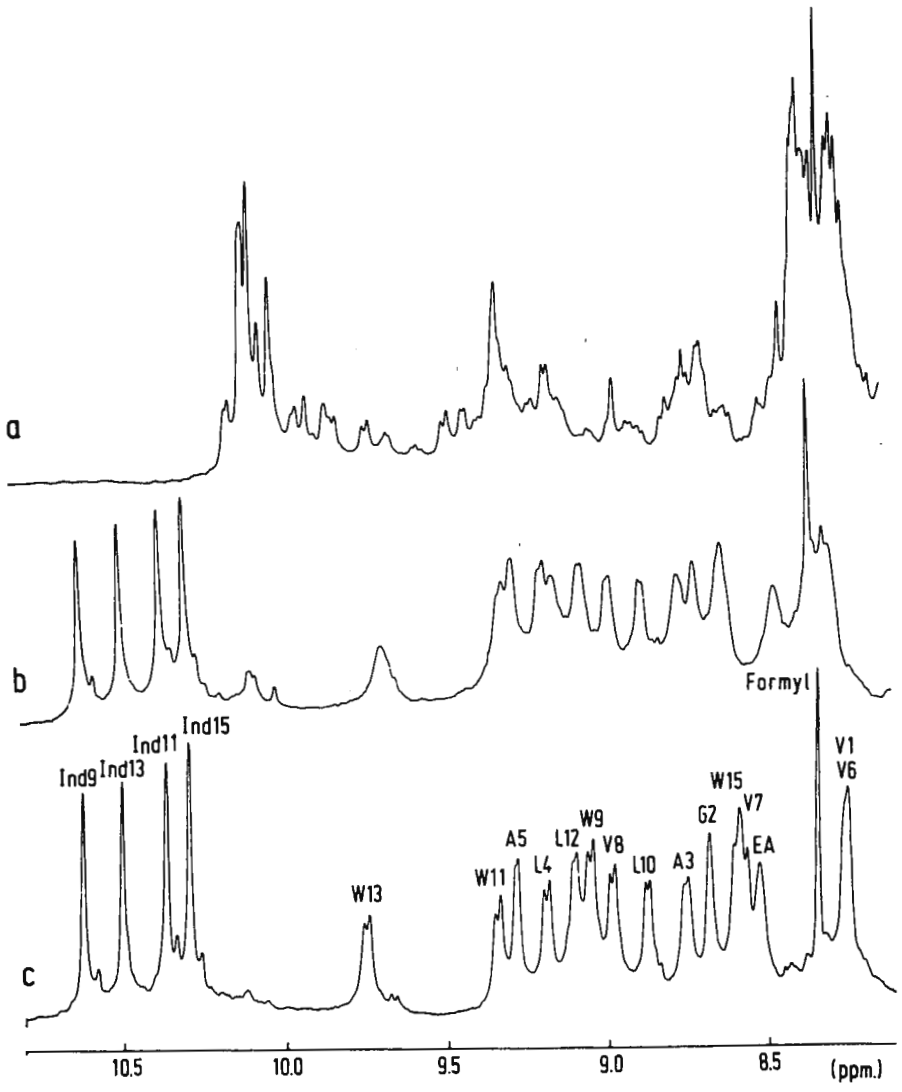

Figure: Low field region of the 500 MHz proton NMR spectra of a 30 mM solution of gramicidin A in $CD_3OH/CDCl_3$ (I:I), 30°C at different $|Cs^+|/|GA|$ molar ratios: a) 0, b) 0.6 and c) I.I. The backbone NH resonance assignments are indicated by one-letter IUPAC code for amino acid residues and the number according to the primary structure of gramicidin A. EA - amide proton of ethanolamine moiety, Ind - indole NH protons of tryptophan residues.

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Rome, October 22nd, 1984

Prof.B. L. Shapiro
Editor
TAMU NMR Newsletter
Texas A&M University
Department of Chemistry
College Station, Texas 77843-3255

Metabolism of 5-fluorocytosine in intact cells of Candida albicans, as studied by ^{19}F NMR.

Dear Professor Shapiro,

5-fluorocytosine (5-FC) is an important antimycotic agent especially active against pathogenic yeasts of the genera Candida and Cryptococcus.

Several biochemical studies on the metabolism of this compound have shown that 5-FC is taken up by the cells, deaminated to 5-fluorouracyl (5-FU) and,through phosphorylated intermediates, incorporated into RNA (1). However there is still some uncertainty about the exact mechanism of the antifungal action of 5-FC.

For this reason,as well as for the general interest of having more general information about the action of this drug in the intact microorganism, we undertook ^{19}F NMR studies on the metabolism of 5-FC in drug-sensitive and -resistant strains of C. albicans.
The kinetics of 5-FC uptake and transformation into 5-FU could easily be followed by examining ^{19}F NMR spectra taken at 28°C, 94.1 MHz,in packed yeast cells incubated in minimal medium containing glucose (50 mg/ml) and salts, in the presence of 5-FC (5 mg/10^{11} cells). Figure 1 shows the progressive formation of 5-FU in intact cells (strain BP C.albicans ISS). 5-FC and 5-FU (-45.6 and -46.8 ppm from external 25 mM NaF) were identified with standard compounds.

Preliminary data also suggest that this approach may be useful for the identification of intracellular phosphorylated compounds derived from 5-FU. In this line the use of 5-FC-resistant mutants is currently approached in our Laboratory.

Yours sincerely,

Massimo Di Vito
M. Di Vito (1)

Fulvio Carpinelli
G.Carpinelli (1)

Franca Podo
F. Podo (1)

W.Whelan (2)

D.Kerridge (2)

A. Torosantucci (3)

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(1)Laboratorio di Biologia Cellulare, Istituto Superiore di Sanità; (2) Department of Biochemistry, University of Cambridge; (3) Laboratorio di Batteriologia e Micologia Medica, Istituto Superiore di Sanita', Roma.
(Granted by CNR contracts TBMS n. 83.00571.57 and CMM n. 83.02916.52)

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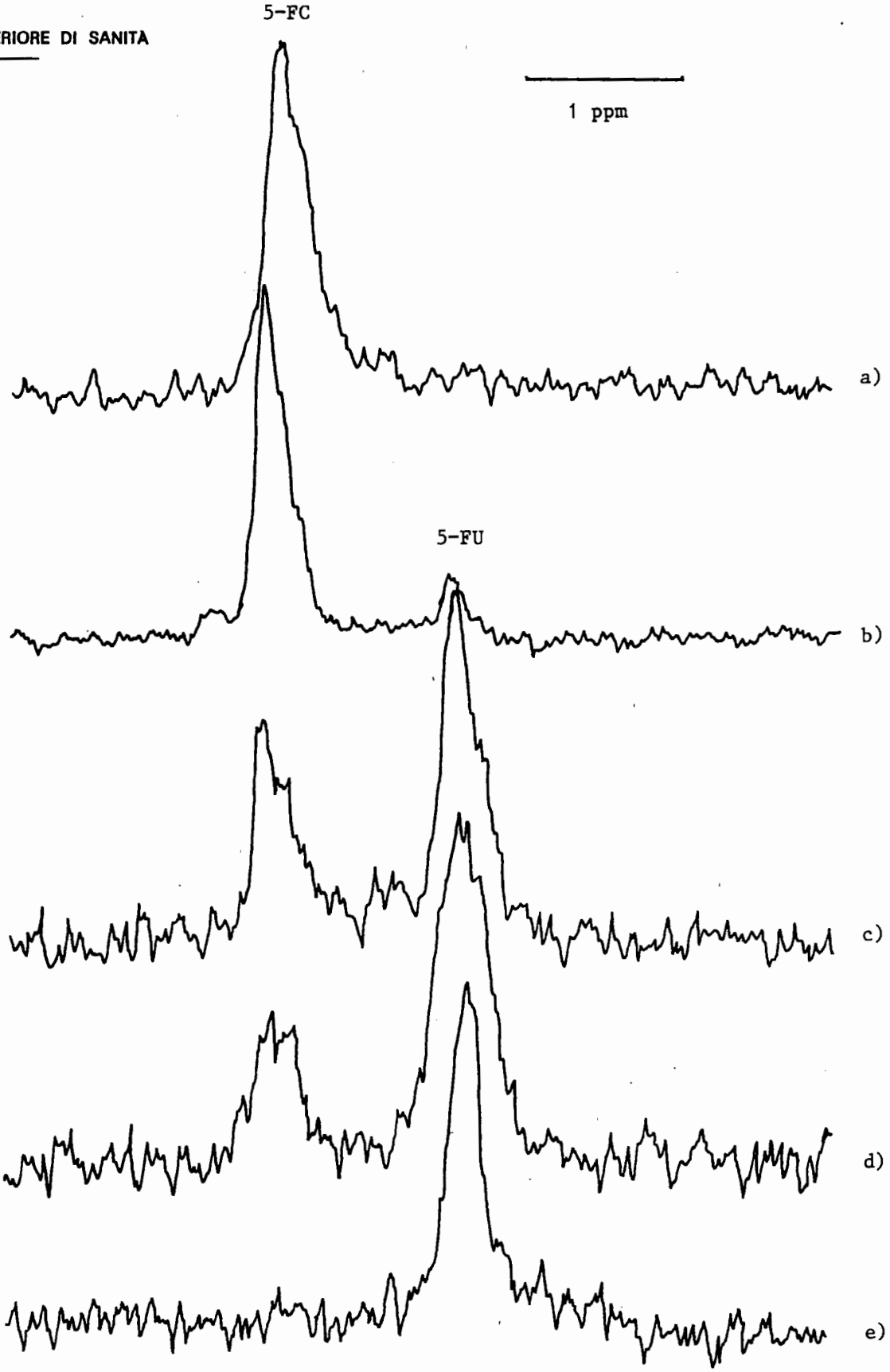

Figure 1. ^{19}F NMR spectra a)-d) were taken at various times after addition of 5-FC to the cells: a) 5', 287 scans (1 s each); b) 2^{h} 12', 700 scans; c) 19^{h} 15', 190 scans; d) 24^{h} 30', 190 scans. The cells were then washed and analysed (spectrum e), 657 scans).

University of Strathclyde

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Professor B. L. Shapiro,
Department of Chemistry,
Texas A and M University,
College Station,
Texas 77843-3255,
U.S.A.

26th October, 1984.

Dear Barry,

Dye Labelling of Melittin

We have had occasion recently to examine the 250 MHz proton spectra of Melittin (I) (for structure see ref. 1) and a derivative which has been labelled by reaction with the fluorescent probe erythrosin isothiocyanate (Molecular Probes Inc) with a view to determining the site of the label. The four possible sites are the ε-amino groups of the lysines (7,21,23) and the α-amino of glycine 1.

When the spectra are compared the greatest perturbation appears to involve either or both of valines 5 and 8 and this would suggest that lysine 7 is the point of attachment of the dye label (for the assignment of the spectra of melittin see ref. 2).

$^{*}H_2N$-Gly-Ile-Gly-Ala-Val-Leu-Lys*-Val-Leu-Thr-Thr-Gly-Leu-Pro-Ala-Leu-Ile-Ser-
1 5 10 15

Trp-Ile-Lys*-Arg-Lys*-Arg-Gln-Gln-CO.NH_2
20 25

Melittin (I) (free NH_2 sites marked *)

Yours sincerely,

Mark J. Dufton, Jackie Turner, Peter Bladon

1. T. C. Terwilliger and D. Eisenberg, J. Biol. Chem., 257, 6016 (1982).
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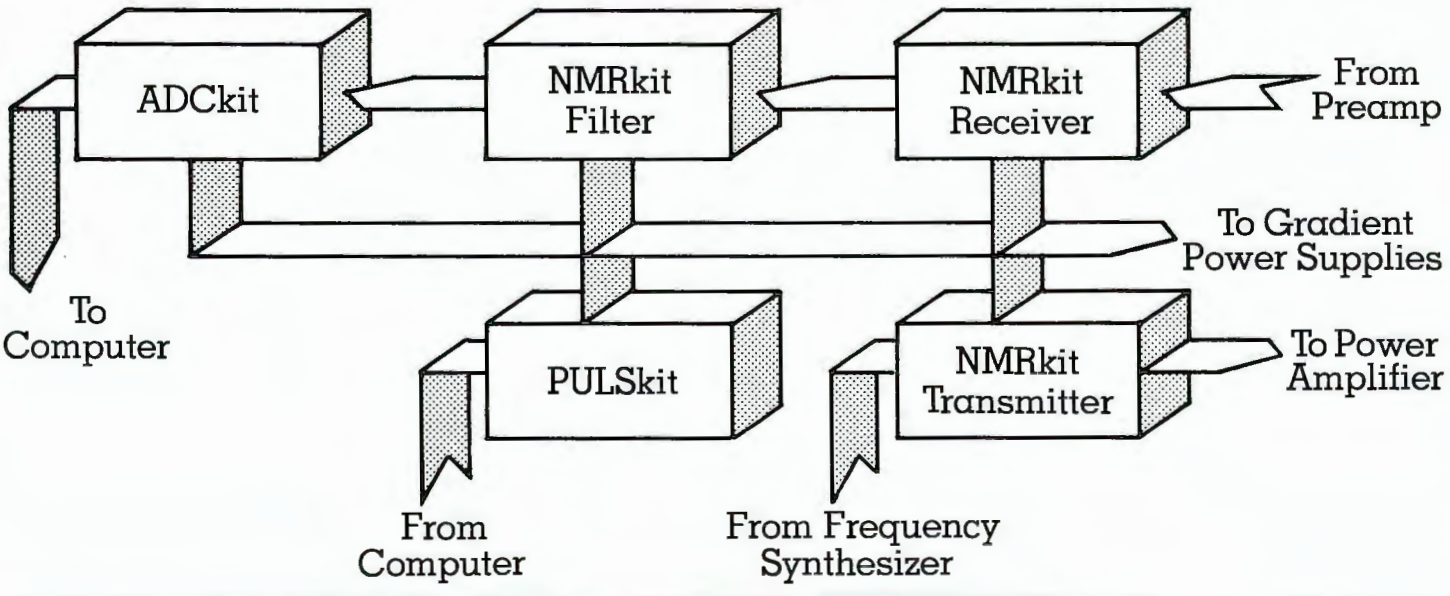

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—A 12-bit Analog to Digital Converter with a 3 μs conversion time
—A 16-bit adder/subtractor to control the sign of the output signal

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THE UNIVERSITY OF MANITOBA DEPARTMENT OF CHEMISTRY Winnipeg, Manitoba
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October 24, 1984

Dr. B.L. Shapiro
Department of Chemistry
Texas A & M University
College Station
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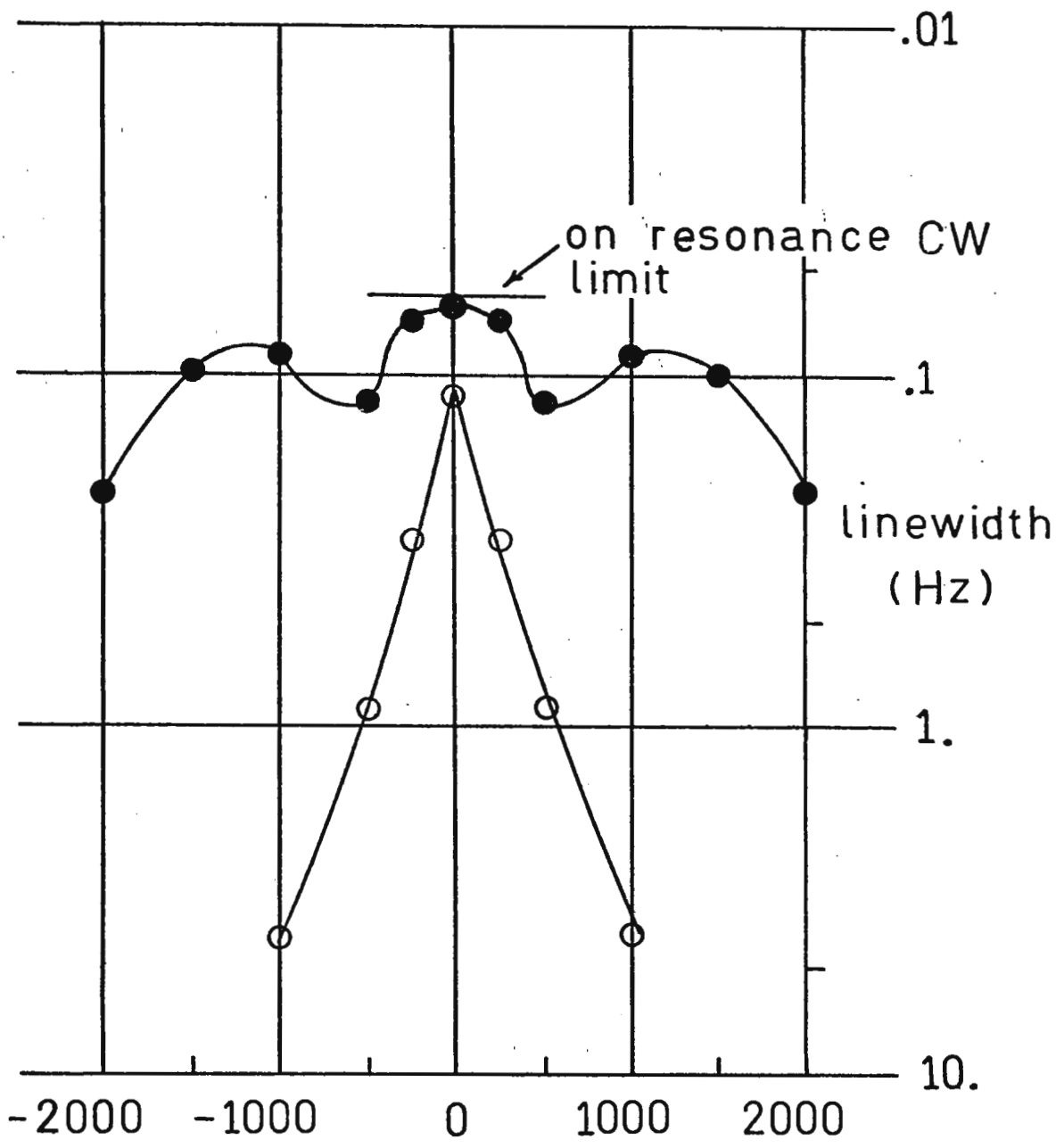
Dear Dr. Shapiro:

Re: Some observations on WALTZ16 decoupling.

Of the several new composite pulse decoupling schemes that have been developed over the last few years (1-6) one of the most effective is WALTZ16 (5,6). This method uses a series of phase shifted pulses to achieve a better decoupler bandwidth distribution than conventional broadband decoupling. On our AM300 this decoupling method may be used during any AU sequence by setting the MOD flag equal to 1 and a P9 pulse length equal to 90 degrees for the decoupler power being used. WALTZ16 gives very narrow ^{13}C lines over a wide range of proton decoupler offsets and has become our standard method of broadband decoupling. Fig. 1 compares the linewidth of benzene as a function of decoupler offset for WALTZ16 and conventional broadband decoupling. WALTZ16 almost equals the on-resonance CW limit of 0.065 Hz and produces lines narrower than 0.22 Hz over a range of 4 kHz. Linewidths using broadband decoupling at this power level are never less than 0.15 Hz and deteriorate rapidly with offset.

We have found this degree of resolution to be especially useful for measuring long range $^{19}F - ^{13}C$ coupling constants and small ^{13}C isotope shifts in protio-deuterio mixtures. We have noticed, however, that certain spin systems seem more difficult to decouple than others. Ethyl groups are especially bad: In 4-methylethylbenzene the ring carbons and 4-methyl carbon have linewidths of 0.1 Hz while the ethyl carbons have residual multiplet splittings of ca. 0.3 Hz. This effect does not appear to be due to mis-setting of the pulse length and is relatively independent of offset. Has anyone else noticed this effect? Does anyone have an explanation for it?

Fig 1.



DP = 14H = 1 W ; $\gamma B_2/2\pi$ = 1400Hz

o - BB

● - MOD = 1 (waltz16)

These residual splittings are probably less than the linewidths of most samples and WALTZ16 is still superior to broadband decoupling for most applications.

Please credit this letter to Ted Schaefer's account.

Sincerely,

Kirk Marat.

1. M. H. Levit and R. Freeman, J. Magn. Reson. 43, 502 (1981).

2. M. H. Levit, R. Freeman, and T. Frenkiel, J. Magn. Reson. 47, 328 (1982).

3. M. H. Levit, R. Freeman, and T. Frenkiel, J. Magn. Reson. 50, 157 (1982).

4. A. J. Shaka, T. Frenkiel, and R. Freeman, J. Magn. Reson. 52, 159 (1983).

5. A. J. Shaka, J. Keeler, T. Frenkiel, and R. Freeman, J. Magn. Reson. 52, 335 (1983).

6. A. J. Shaka, J. Keeler, and R. Freeman, J. Magn. Reson. 53, 313 (1983).

McMASTER UNIVERSITY
Department of Chemistry
1280 Main Street West, Hamilton, Ontario, L8S 4M1
Telephone: 525-9140

October 4, 1984

Professor B. L. Shapiro
Texas A & M University
College of Science
College Station, Texas 77843
U.S.A.

Dear Dr. Shapiro:

McMaster University has a fully operational Bruker WH90 for sale. In addition to standard ^{1}H and ^{19}F capabilities, multinuclear probes are available from 4 to 37 MHz. The instrument is fitted with a DIABLO (series 30) disc drive, NIC293 I/O controller and a Nicolet 1080 computer. For further information, please contact the undersigned.

Sincerely,

Brian Sayer

Brian G. Sayer

(416) 525-9140 Ext 4443

BGS:cd

Department of Biochemistry

College of Agricultural & Life Sciences
University of Wisconsin-Madison

420 Henry Mall
Madison, Wisconsin 53706 USA

October 23, 1984

Professor B. L. Shapiro
Department of Chemistry
Texas A&M University
College Station, TX 77843

Subject: Implementation of a Heteronuclear decoupler on the Nicolet NT-200 NMR Spectrometer

Dear Professor Shapiro:

Decoupling of nuclei other than protons is often of interest but is limited by equipment availability. Shown in Fig. 1 is a relatively simple modification of the Nicolet NT-200 widebore spectrometer for decoupling of deuterium from carbon spectra. This strategy is similar to that of Griffey et al[1] for irradiation of ^{15}N in ^{1}H-^{15}N correlation spectroscopy. The system simply mixes the present proton decoupler frequency down to the frequency of interest, in this case to 30.7 MHz. A Bird Wattmeter is used to monitor decoupler power and filters are used to eliminate spurious signals from the decoupler and observe channels.

In Fig. 2 are shown spectra obtained for a mixture of 50% acetone/ 50% d_6-acetone. In part A the totally coupled ^{13}C NMR spectrum is shown, in B the deuterium decoupled spectrum is shown. Note the collapse of the seven-line pattern of d_6-acetone to give the single line pattern. In order to record these spectra the lock channel on a 5 mm ^{13}C probe was used for deuterium decoupling while the spectrometer lock channel was disconnected.

By using Texscan filters sufficient isolation between ^{13}C and ^{2}H channels at 50.3 MHz and 30.7 MHz respectively is obtained. (The ^{13}C probe has the ^{2}H signal riding on the outer coil which aids isolation of the observe channel.) With appropriate filters and probe this system may be used to decouple other nuclei such as ^{15}N and ^{31}P.

Sincerely,

Ed Mooberry

Ed Mooberry

EM:cap

Enclosure

[1] R.H. Griffey, C.D. Poulter, A. Box, B.L. Hawkins, Z. Yamaizum, and S. Nishimura, Proc. Natl. Acad. Sci., USA. Vol. 80, pp. 5895-5897, Oct. 1983, Biophysics.

Telephone: 608-262-3026, 262-3040
Telex: 26 54 52

Figure 1

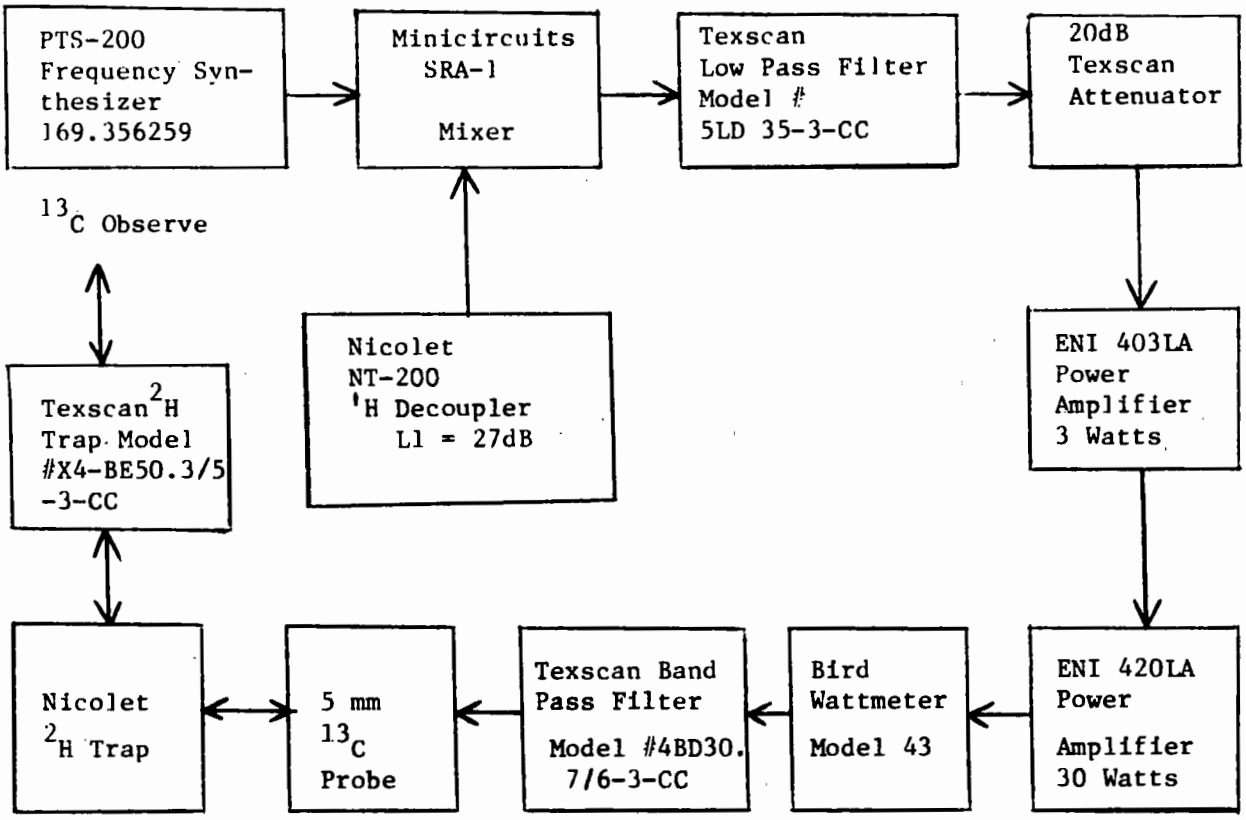
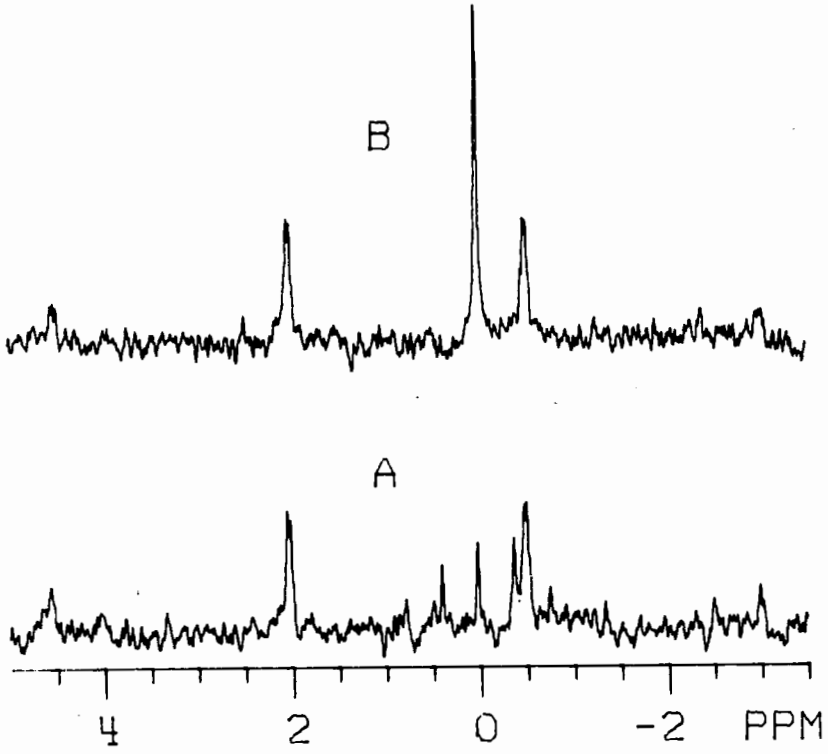


Figure 2



ONE-PULSE SEQUENCE

P2=	8.00 USEC
D5=	2.00 SEC
NA =	8
SIZE =	4096
AT =	4.10 SEC
QPD ON =	1
ABC ON	
BUTTERWORTH FILTER ON	
DB ATT.=	9
ADC =	12 BITS
AI =	-1
SW =	+/- 250.000
DW =	2000
RG =	10 USEC
DE=	2000 USEC
TL HIGH POWER ON	
OF=	52.24
SF=	50.908245
EM=	1.00
PA=	256.2
PB=	79.5
SCALE =	98.95 HZ/CM .7742 PPM/CM

StonyBrook

Department of Chemistry
State University of New York at Stony Brook
Stony Brook, New York 11794
telephone: (516) 246-5050

October 25, 1984

Dr. Bernard L. Shapiro
Department of Chemistry
Texas A&M University
College Station, TX 77843

Title: Views of pseudospace.

Dear Barry:

What the dreaded polychromatic barrage could not accomplish, the exponentially intensifying symptoms of a cold-turkey withdrawal from The Newsletter after all these years have done. I give in; here's a contribution. Is there any chance of catching up on the ones I missed?

Every time you deplore the appearance of an NMR spectrum with the natural linewidths concealed by broadening in a bad field you are complaining about a projection of a 4-dimensional object. The fourth dimension, of course, is just the high resolution spectrum, and it is the glimpse of the other three that is the problem. Can any use be made of this peculiar way of converting a commonplace observation into a confusing mystification? Again, of course. Think of it this way. In a perfectly homogeneous B_o field the spatial variables are absent because we are observing the projection of the 4-dimensional pseudo-object normal to the spectroscopic axis. In an infinitely large linear magnetic field gradient the spectrum is no longer relevant and we see only the real-space projection of the pseudo-object on a 4-dimensional hyperplane. At intermediate gradient amplitudes the projections are at other angles, giving views that contain mixed spatial and spectroscopic information. Quantitatively, the angle relative to the spectroscopic axis is given by $\theta = \arctan \gamma G D \Omega^{-1}$, where G is the gradient strength, D the spatial extent of the object, and Ω its spectroscopic extent. After scaling the projections by dividing by $\cos\theta$, and using enough different spatial directions of the gradient as well, any projection reconstruction method will give the 4D pseudo-image. Clear?

This exposition could be much clearer if you published 4-D figures, but I'm sure that you have excuses about mailing permits and the like for not doing so. Let me suppress one spatial dimension, then, and use an isometric view of a 3-D pseudo-object to make my point. For those who would prefer to start off easy, with a total of only two dimensions, see J. Magn. Res. 59, 536-541 (1984), but that paper has no experimental results in it.

Our experiment used two plastic containers, each 1 x 3 inches, shown projected on the X_1X_2 plane of Fig. 1. They actually had a third physical dimension, but that has now become almost irrelevant. Even at only 4 MHz, the water and acetic acid signals could be distinguished. After 1891 FIDs, in gradients ranging from about 3 μT/m to about 0.5 mT/m, a standard hybrid 3D reconstruction program was used to reconstruct an image. A single slice from that image is shown in Fig. 2, with the features of the spectroscopic zeugmatogram clearly resolved.

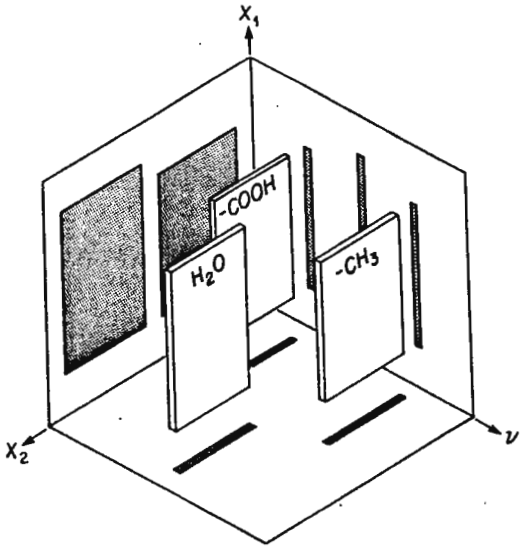


Fig. 1

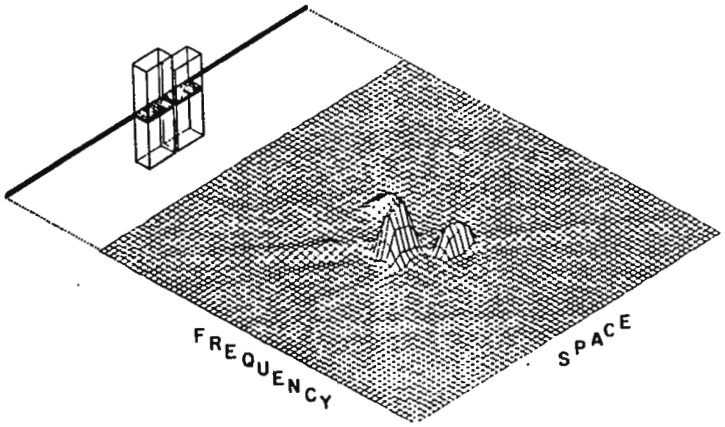


Fig. 2

The point to all this? It was fun, it showed that pure projection reconstruction can do what had previously required multidimensional FT methods, it works without spin echoes and can therefore handle very short T_2 values and homonuclear spin-spin couplings, and, because only static gradients are used, it makes it possible to do spectroscopic imaging on just about any NMR spectrometer, even an old A-60, if there are still any of those around. You can also use the same method to plot out magnetic fields.

Hopeful regards,

M. Bernardo, Jr. — Marcelino L. Bernardo, Jr.

Paul — Paul C. Lauterbur

L. Kyle Hedges/sg — L. Kyle Hedges

Eidgenössische
Technische Hochschule Zürich

Laboratorium
für Physikalische Chemie
Prof. Dr. R. R. Ernst
GEBO/mü

CH-8092 Zürich
ETH-Zentrum
Durchwahlnummer 01 256 43 68
Telefonzentrale 01 256 22 11

October 10/1984

0430

Professor B.L. SHAPIRO
Department of Chemistry
Texas A&M University
College Station
Texas 77843 U.S.A.

PATTERN RECOGNITION IN 2D NMR

Dear Barry,

We should like to inform you on recent progress made in the automatic analysis of two-dimensional correlation spectra (COSY). We have extended our work on pattern recognition[1] by feeding an IBM instrument CS 9000 computer with two complementary phase-sensitive double-quantum filtered COSY spectra[2,3,4] obtained with the sequence $\pi/2\text{-}t_1\text{-}\pi/4\text{-}\beta\text{-}t_2$ with $\beta=\pi/4$ in one experiment and $\beta=3\pi/4$ in the other. The program searches for multiplets with alternating positive and negative peaks. The 1D spectrum of a mixture of 2,3-dibromothiophen (AX), 1,1,2-trichloroethane (A_2X) and 1-bromo,3-nitrobenzol (AMKX) is shown in Fig.1. To improve the digitization, the spectral width has been reduced, and some signals are folded. One of the two complementary 2D spectra (obtained with $\beta=\pi/4$) is shown in Fig.2. The matrix consists of 512×512 points. If the program is instructed to search for a 4-spin system, the signals associated with 2- and 3-spin systems are disregarded, and the parameters of the 4-spin system are printed as shown in Table 1. The chemical shifts in this table have not been corrected for folding.

[1] B.U. Meier, G. Bodenhausen and R.R. Ernst, J. Magn. Reson., in press.

[2] U. Piantini, O.W. Sørensen and R.R. Ernst, J. Am. Chem. Soc. 104. 6800 (1982).

[3] A.J. Shaka and R. Freeman, J. Magn. Reson. 51, 169 (1983).

[4] M. Rance, O.W. Sørensen, G. Bodenhausen, G. Wagner, R.R. Ernst and K. Wüthrich, Biochem. Biophys. Res. Commun. 117, 479 (1983).

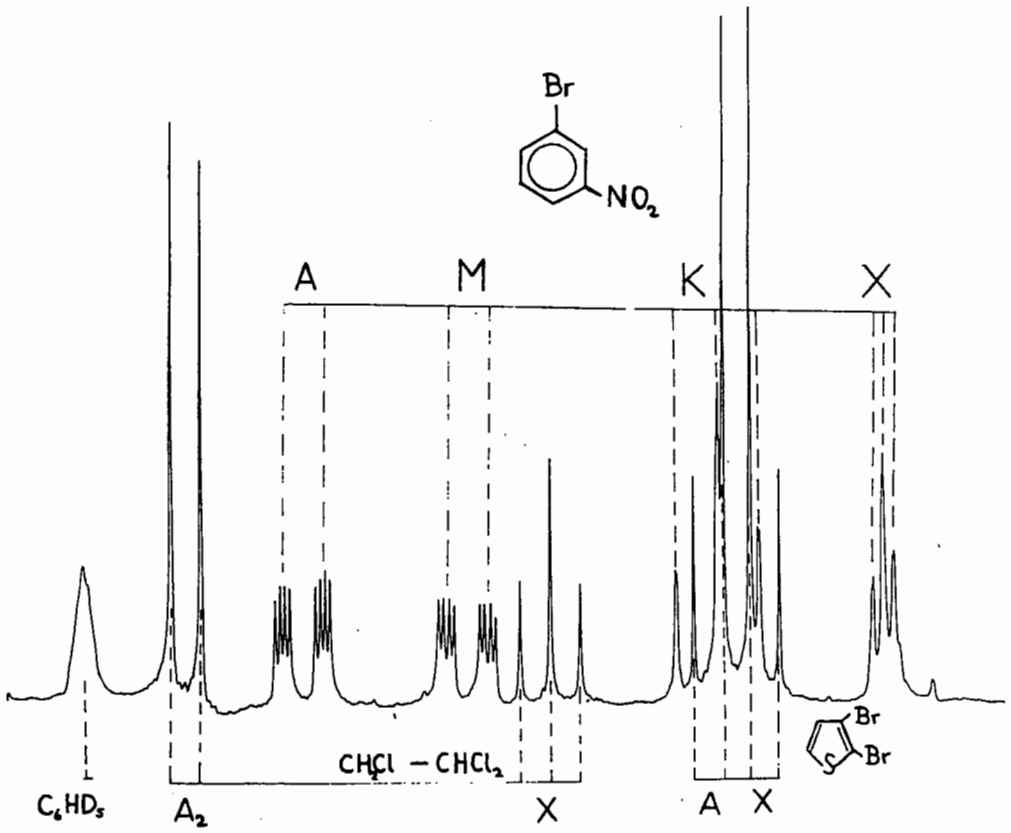


Fig.1

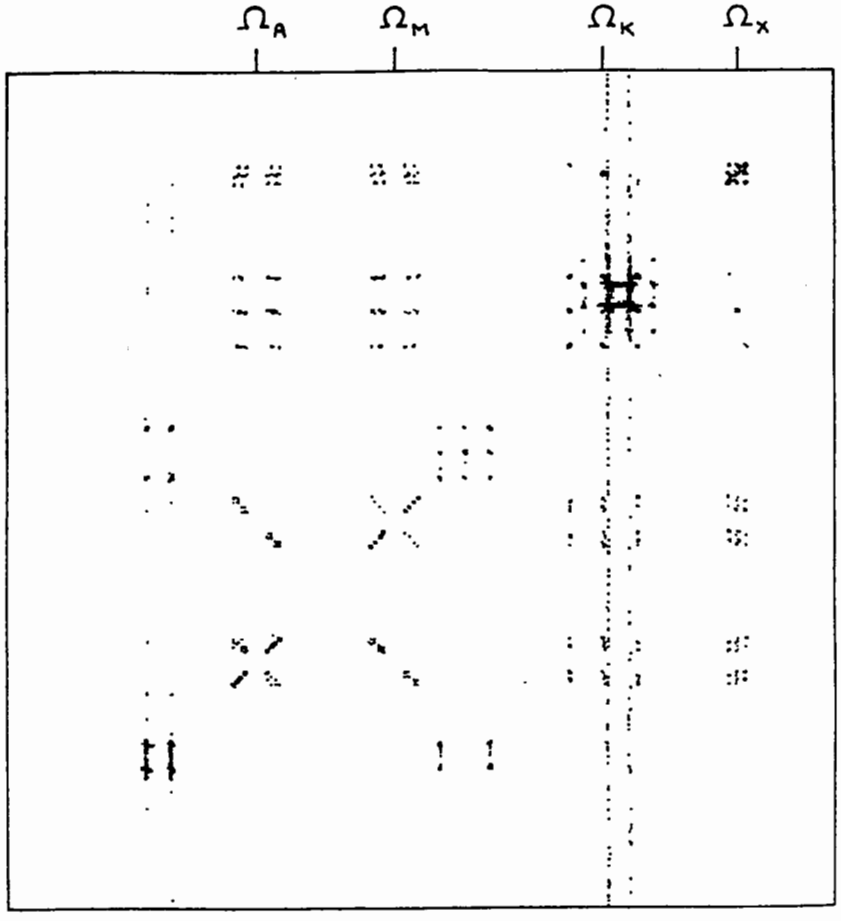


Fig.2

Pattern recognition has also been applied to 1,2-dibromopropane (AMK_3X). In this case, the program automatically identifies that there are three magnetically equivalent K protons, and correctly distinguishes the negative geminal coupling from the other couplings which are all positive.

Although these results are still preliminary, it appears that the laborious plotting and manual interpretation of COSY spectra may perhaps at some day be skipped, and the relevant information automatically be presented in a form similar to Table 1.

Yours sincerely,

Peter Pfändler Beat U. Meier Geoffrey Bodenhausen R.R. Ernst

Coupling Network of the AMKX-Spinsystem

Spin	Spin chem. Shift	A(1) 140.9	M(1)* 106.9	K(1)* 56.8	X(1) 24.3
A(1)	140.9	---	1.2	8.2	2.0
M(1)*	106.9	1.2	---	8.2	2.0
K(1)*	56.8	8.2	8.2	---	0.0
X(1)	24.3	2.0	2.0	0.0	---

* : folded Peaks all chemical Shifts and coupling Constants in [Hz]

Table 1

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IBM Instruments Inc.

314-41

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

National Institutes of Health
National Cancer Institute
Bethesda, Maryland 20205

October 31, 1984

Dr. B. Shapiro
NMR Newsletter Texas A&M University
College Station, TX 77843-3255

TITLE: 2D-NOE of Polydexoynucleotides

Dear Barry:

We have had our Varian XL-400 for ca 2 months now, but I haven't been able to enjoy it yet, having just returned from the Goa Conference.

Among the letters awaiting me was your reminder and acceptance by Biopolymers of our proton 2D-NOE study of sonicated polydeoxynucleotides. The figure shows the changes in the absorption mode 2D-NOE contour plots of double-stranded poly (dGm^5dC) upon the addition of $MgCl_2$ (3 mM), which induces the transition from the right handed B to the left handed Z-form. While there are changes in the 1D spectra, the 2D-NOE contour plot shows the changes much more clearly; for example, the presence of a strong (GH8-GH1') cross peak from the syn G base in the Z form. With this powerful method of conformational analysis we were able to show that there is no significant change in the overall conformation of poly (dAdT) as a function of salt concentration, i.e. it remains in a right handed B-form. Further details will be found in the paper by Borah, Cohen and Bax.

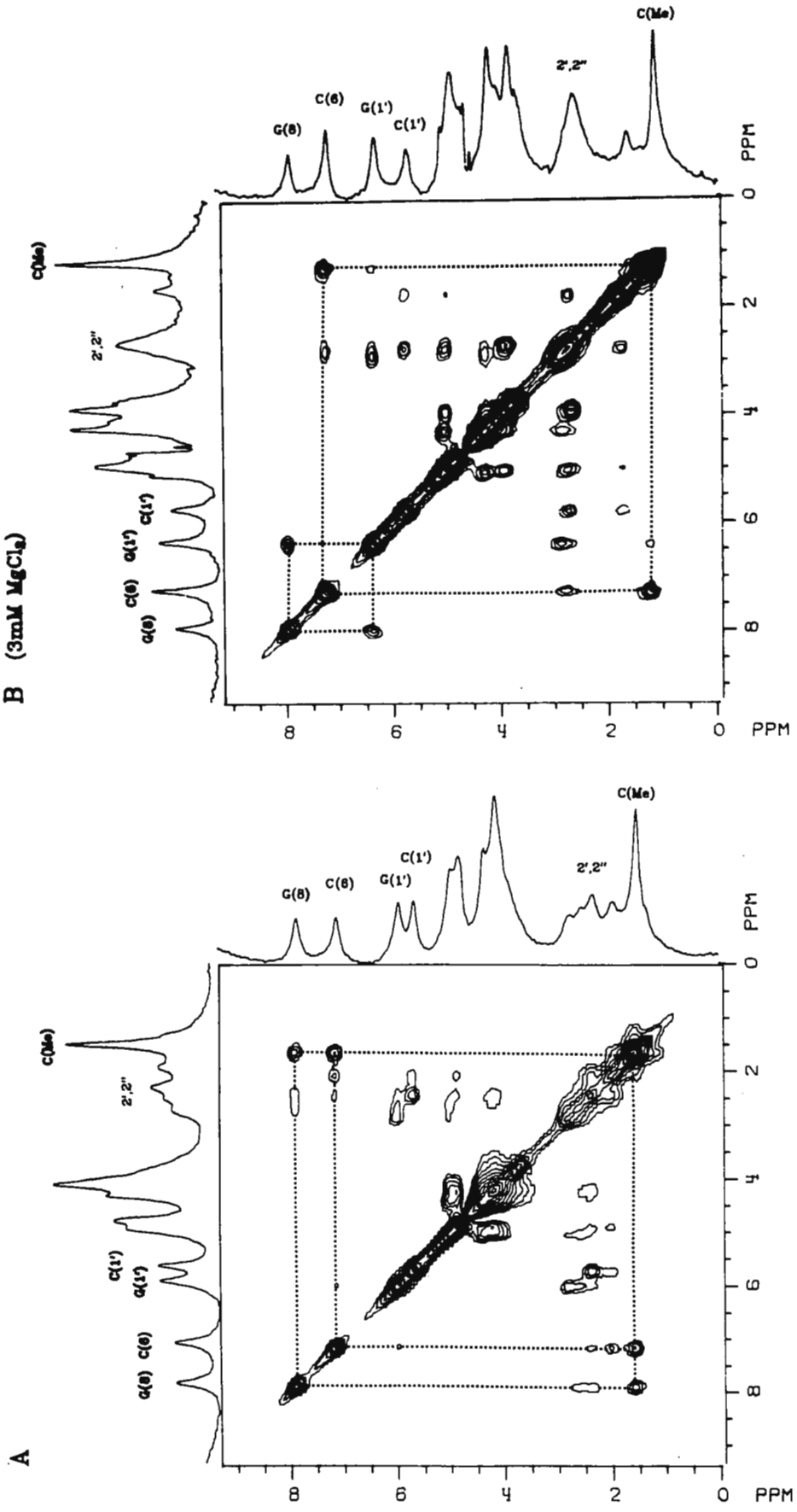
Yours sincerely,

Jack S. Cohen and Babul Borah
Clinical Pharmacology Branch
National Cancer Institute

JSC/cgr

Figure Legend - Contour plots of absorption mode 2D-NOE spectra of double-stranded poly($dG-m^5dC$) in (A) 50 mM NaCl and (B) 3 mM $MgCl_2$. Mixing time was 50 ms in both cases. Specific interactions are indicated by joining off-diagonal peaks for cross-relaxing proton.

Poly(dGm^{5}dC).Poly(dGm^{5}dC)



Carnegie-Mellon University

Department of Chemistry
4400 Fifth Avenue
Pittsburgh, Pennsylvania 15213
(412) 578-3149

November 1, 1984

Professor B.L. Shapiro
Texas A and M University
Department of Chemistry
College Station, TX 77843

Title: O Fickle $CDCl_3$!

Dear Barry:

Your green reminder has caught us in a state of mild shock, which I will be glad to share with TAMU readers. Like nearly everyone else, we have been routinely using $CDCl_3$ as a solvent and field-frequency lock for proton high resolution spectroscopy. We have noticed that our lock signals were not particularly good for such samples, but ascribed this to relatively low concentration, easy saturation, O_2, instrumentation, or other sources, depending on the day and our current prejudices. Recently, we had occasion to look closely at the $CDCl_3$ signal (but that, as Kipling said, is another story) and noted with surprise that it was a doublet, with a splitting of 0.22 Hz (Resolution enhanced signal, Fig. 1).

In retrospect, we see that we might even have anticipated this. Very high magnetic fields produce a slight alignment of molecules which are magnetically anisotropic, and C-Cl bonds have been demonstrated to be magnetically anisotropic by several means. Deuteron signals from aligned molecules are split (1). From the splitting we deduce $\Delta\chi$ for $CDCl_3$ to be about 0.2×10^{-28} cc/molecule, about one-fifth that of benzene. Adding benzene causes the splitting to decrease. If the complex of chloroform and benzene has the geometry of a vase of flowers on a plate, it means that the molecular diamagnetic susceptibility is least along the C-D axis.

For field-frequency locking at 10T and above, this could give trouble, depending on whether the resolution is better than the splitting. If so, some modulation of the lock signal due to saturation effects and lock jumping could be observed. If the lines were well resolved, the lock would probably jump back and forth. If someone saw the beat from the two lines, they would probably shim to make it go away.

Fortunately, most of the world does not have to worry about this problem, because the splitting is proportional to the square of H_o. At 300 MHz the splitting is only 0.05 Hz, and won't bother anybody.

Well, Cheerio.

Sincerely,

Aksel A. Bothner-By

Joe Dadok

P. K. Mishra

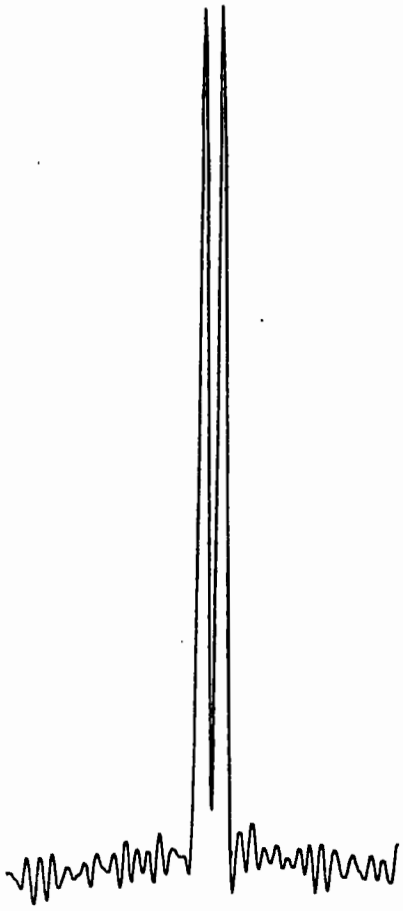


Figure 1 Deuterochloroform ^2H resonance, somewhat resolution enhanced. Splitting = 0.22 Hz at 14.09 T.

(1) P.C.M. van Zijl, B. H. Ruessink, J. Bulthuis, and C. MacLean, Acc. Chem. Res. 17, 172 (1984).

UNIVERSIDAD DE BUENOS AIRES
FACULTAD DE CIENCIAS EXACTAS Y NATURALES

Buenos Aires, october 1, 1984.

Prof. Bernard Shapiro.
Department of Chemistry.
Texas A. & M. University.
College Station, Texas 77843.
U.S.A.

Title: Looking for an old Nicolet.

Dear prof. Shapiro:

We have here a Nicolet Data Processor System, model BNC 812, similar to the 1080, which is still working (and working well) but without us having any chance of modernizing it.Therefore we are looking for a similar system to "canibalize" it and, if possible, to add some memoty to its formidable 30 K.

If anybody knows about a bargain, please let it be known to the underwriter at the below given adress.(I heard that some people even want to get rid of their old 1080 free of charge! Could that be true I would gladly take care of the shipping expenses!).

I would appreciate if you will publish this letter,besides the purely scientific contribution of my collegues from this lab.

Yours, sincerily

DR. V.J.Kowalewski.

my adress: Dr. V.J.Kowalewski.
Facultad de Ciencias Exactas
(1428) Ciudad Universitaria.
Buenos Aires, Argentina.

Book Review

Editor: **W. B. Smith**
Texas Christian University
Fort Worth, Texas

Theory of NMR Parameters

by **I. Ando** (Tokyo Institute of Technology, Tokyo, Japan) and
G. A. Webb (The University of Surrey, Guildford, Surrey, U.K.)

Academic Press
New York, 1983, 217 pages, $52.

This small volume deals primarily with the quantum mechanical calculation of chemical shifts and spin-spin coupling constants. These two parameters are briefly introduced in the first chapter. Also introduced here are the definitions of T_1 and T_2, along with a discussion of the factors contributing to their values. Relaxation is not considered further because this quantum mechanical approach does not deal with the subject.

Chapter 2 is a brief introduction to quantum chemistry. The flavor of the presentation can best be gained by pointing out that the Schrödinger equation, the variation method, first- and second-order perturbation theory, MO theory with LCAO SCF equations, unrestricted SCF methods, empirical MO methods, semi-empirical and non-empirical MO methods, valence bond theory, and applications of quantum chemistry to second-order molecular properties are all covered in 24 pages. From this you may infer that this book is not for those who are looking for an introduction to quantum mechanics.

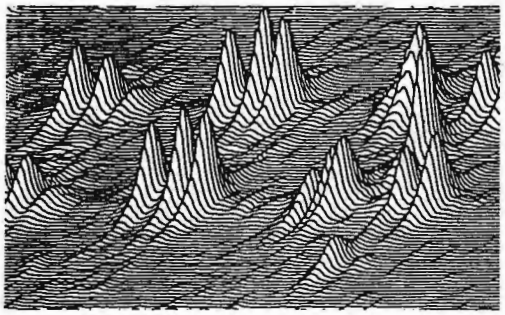
In Chapter 3 these methods of approximating molecular wave functions are applied to the calculation of chemical shifts, using the shielding equation of Ramsey as the point of departure. Both the sum-over-states (SOS) and the finite perturbation theory (FPT) methods are discussed and exemplified. This chapter is designed to lead one to the ultimate presentation of two computer programs which allow calculations of chemical shifts to be carried out. The best part of this chapter for those not planning to attempt such calculations is the perspective given on available methods and the limitations of the various programs. In the early days of NMR it seemed to some of us that there were lots of theoretical calculations and not much data to test them. It becomes more apparent on reading Chapter 3 why today there are a great many data but not so many calculations, which do have their limitations.

Coupling constants are given a similar treatment in Chapter 4. Since there is an extant program available from the Quantum Chemistry Program Exchange at the University of Indiana, the treatment here is rather brief. Chapters 1-4 total 113 pages.

The final 97 pages are devoted to the program listings and documentation for the FPT and SOS methods of calculating chemical shifts. Apparently not readily available hitherto, these programs would have been of more use to the unitiated if samples of input and output for a typical case had been included. I note in looking at the last list available from QCPE that a semi-empirical program to calculate chemical shifts up through third row elements now exists. The magnitude of the task can best be indicated by the fact that it requires 10,000 symbolic cards. To the best of my knowledge, no data based on this program have been published. I'm not in a position to judge if this program duplicates those given in the text.

10/31/84 W.B.S.

NMR DISCUSSION GROUP



Chairman: Dr. D. Shaw, I.G.E. (NY) Limited, 260 Bath Road, Slough, Berks, SL1 4ER. Telephone: (0753) 874495

Secretary: Dr. I. P. Jones, Bruker Spectrospin Limited, Unit 3, 209 Torrington Avenue, Coventry CV4 9HN. Telephone: (0203) 463770

Treasurer: Dr. P. Beynon, Kodak Limited, Research Division, Harrow, Middlesex HA1 4TY. Telephone: 01-427-4380

24th October, 1984.

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

Dear Barry,

May I take this opportunity to inform your readers of the 7th International Meeting on NMR Spectroscopy to be organised jointly by the Royal Society of Chemistry and the NMR discussion Group.

The meeting will take place at Cambridge University from Monday 8 July to Friday 12 July 1985, with participants foregathering on the afternoon and evening of Sunday 7 July 1985. The Scientific sessions will be held in the University Chemical Laboratory, Lensfiedl Road, whilst participants will be accommodated in Queen's College.

The meeting will deal with selected topics in NMR Spectroscopy, and will comprise the following seven symposia:-

1. NMR in Bio-Organic Chemistry
   Chairman: Dr. D. H. Williams
   Invited Speakers:
   Professor D. Cane
   Professor H. Kessler

2. Solid State NMR
   Chairman: Dr. D. G. Gillies
   Invited Speakers:
   Dr. D. Vanderhart
   Dr. C. M. Dobson

3. New Experimental Techniques
   Chairman: Dr. R. Freeman
   Invited Speakers:
   Professor A. Pines
   Professor R. R. Ernst

4. In Vivo NMR
   Chairman: Dr. D. G. Gadian
   Invited Speakers:
   Professor R. Andrew
   Professor R. Shulman

5. NMR in Industry
   Chairman: Dr. A. M. Chippendale
   Invited Speakers:
   Dr. W. Bremser
   Dr. A. Thomas
   Dr. J. C. Waterton

6. NMR in Inorganic Chemistry
   Chairman: Dr. O. W. Howarth
   Invited Speakers:
   Dr. B. T. Heaton
   Professor J. M. Thomas

7. Large Molecule NMR
Chairman: Dr. I. D. Campbell
Invited Speakers:
Professor R. J. P. Williams
Professor K. Wüthrich

Two poster sessions are planned for the programme. There will also be an opportunity for a limited number of short contributions to be presented orally in the symposia. Anyone wishing to contribute a paper to one of the poster sessions, or to one of the symposia, should submit, NOT LATER THAN 31 JANUARY 1985, a title and synopsia (up to 250 words) to the Chairman, at the above address.

A full programme of evening social events is planned, to include:-

Receptions

Organ Recital

Conference Dinner

The fees for attendance at the meeting have not been finalised, but it seems likely that the conference fee will be of the order of £65 (Students £30), and the accommodation for the full fvie day period will be of the order of £110.

Further details may be obtained from:-

Dr. John F. Gibson,
Secretary (Scientific),
The Royal Society of Chemistry,
Burlington House,
London. W1V OBN.

Very best wishes,

Yours sincerely,

DEREK SHAW,
CHAIRMAN

314-49

UNIVERSITY OF SOUTH CAROLINA

COLUMBIA, S.C. 29208

SOUTH CAROLINA MAGNETIC
RESONANCE LABORATORY

(803) 777-7341

The NSF Regional Instrumentation Facility in NMR Spectroscopy at the University of South Carolina is seeking qualified applicants for the position of Facility Manager. Applicants should have a Ph.D. in Chemistry with extensive hands-on experience in the applications of high field, high resolution FT nmr. Successful candidates should be knowledgeable with a variety of two-dimensional nmr experiments. The principle instrument of the facility is a WH-400. Salary is commensurate with experience - usually in the range of $19-23K. In exceptional cases the upper end can be increased. The position is available immediately.

Research opportunities for the successful candidate are impressive. In addition to access to the WH-400 my own group will take delivery of a 300 and 400 MHz nmr spectrometer in late February. These instruments are capable of both solid state (single crystal, MAS, ^1H powder) and multinuclear liquids research projects. This individual will have good access to both of these new instruments and a 200 MHz wide bore system.

If you have any potential candidates, please ask them to apply and to provide two or three letters of reference. Thank you very much for your time, and if we can help you at the facility, please give me a call.

Sincerely,

Paul

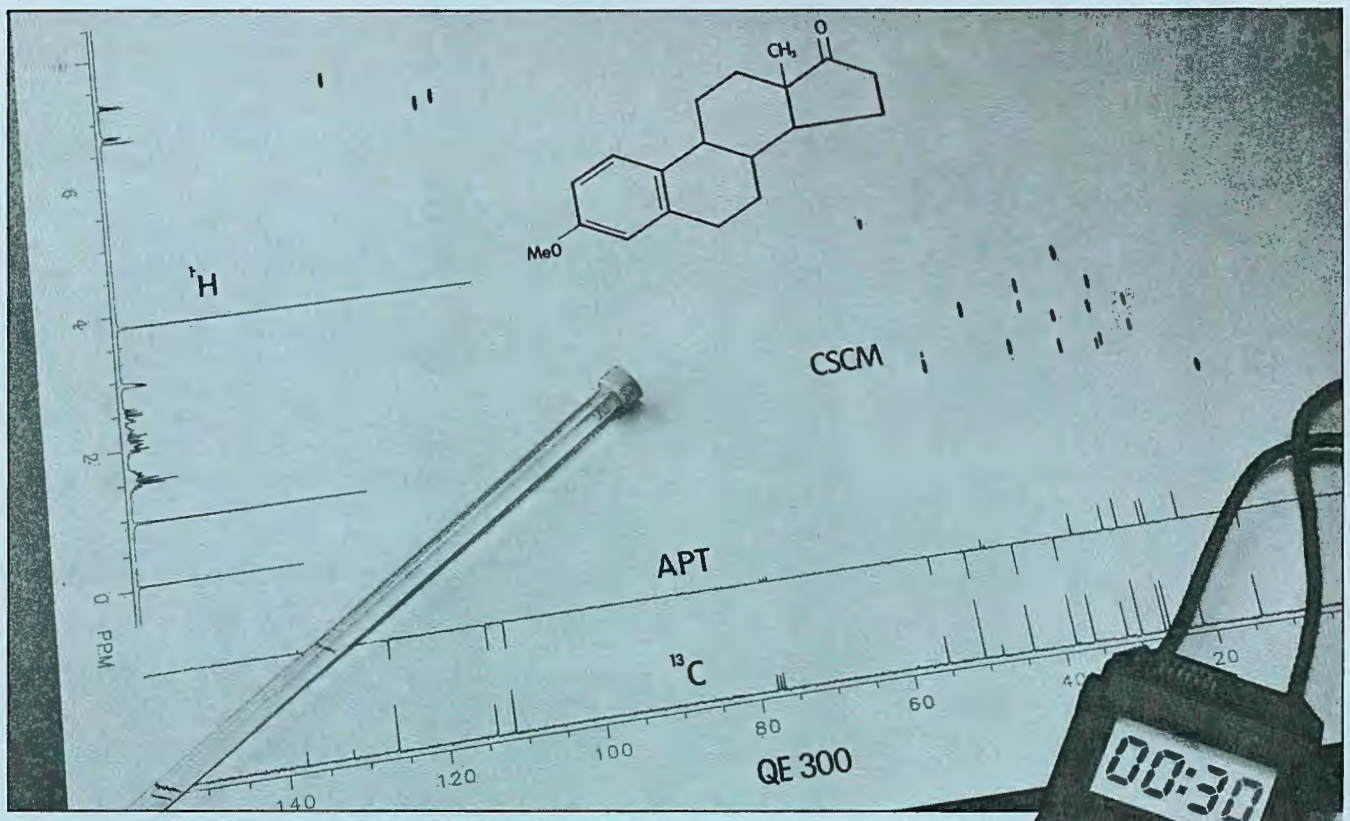
Paul D. Ellis
Professor of Chemistry
Facility Director

We would like to announce that we have a position open for an NMR spectroscopist in our Analytical Research and Services Division. We are looking for a Ph.D. with experience in high-field multinuclear NMR of solids. The applicant should have a good chemical physics or physical chemistry background. Experience in electronics with an ability to maintain and modify commercial spectrometers is desirable. A good understanding of computer interfacing and software development is also sought.

Interested applicants should send their resume to Dr. M. J. Deveney, Supervisor, Spectroscopy Group, Standard Oil Company (Indiana), Amoco Research Center, P. O. Box 400, Naperville, Illinois, 60566.

G. J. Ray

G. J. Ray, B-5



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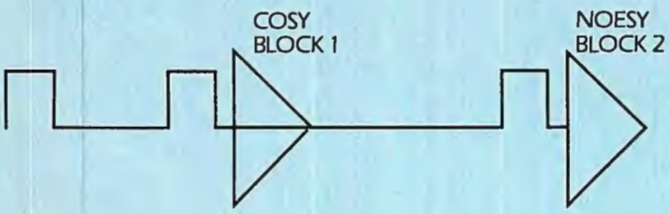

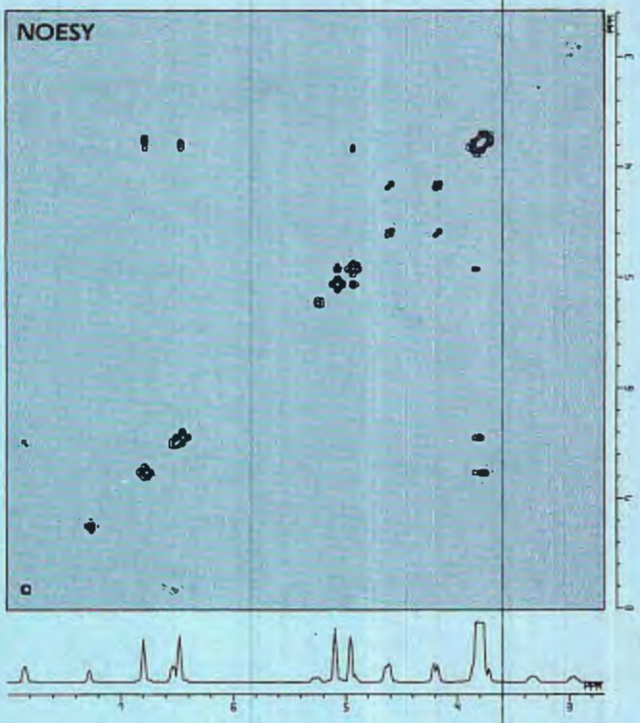

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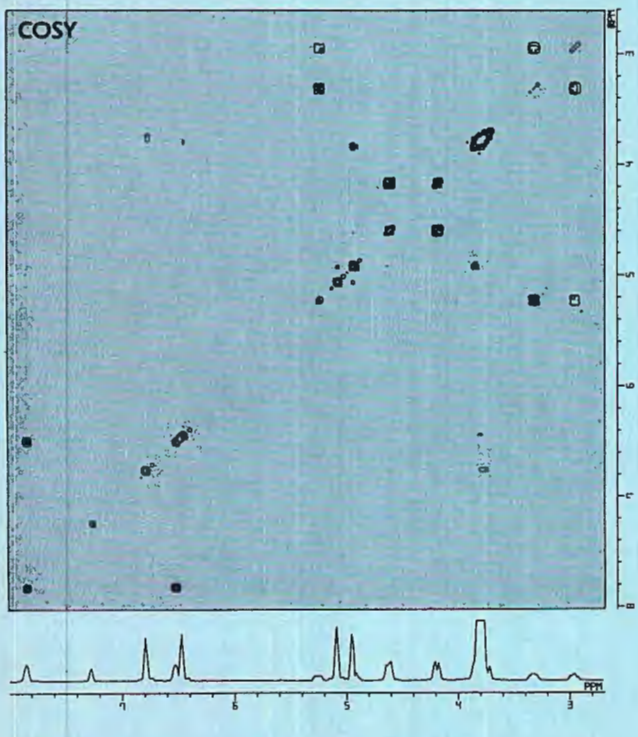

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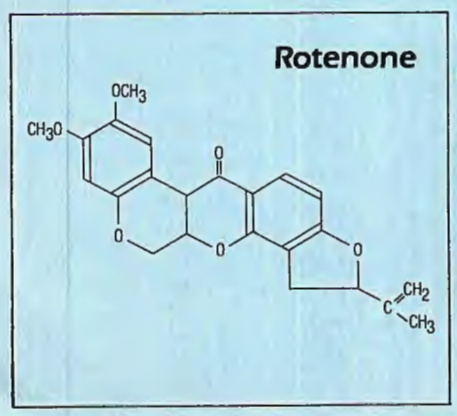
*COCONOSY (Haasnoot, et. al., J. Magn. Reson., 56,343 [1984])



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