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Forthcoming NMR Meetings (Additional listings are solicited)

New Directions in Chemical Analysis, the 3rd Annual Symposium of the Industry-University Cooperative Chemistry Program, Texas A&M University, College Station, Texas, April 1-3, 1985. Papers to be presented by T.B. Hirschfeld, R.D. Macfarlane, C.A. Fyfe, R.L. Swofford, G. Guiochon, A.G. Marshall, A.T. Hubbard, A.C. Parr, J. Ruzicka, J. Callis, N. Winograd, D.H. Williams, M.V. Novotny, W.P. Rothwell, J. Schaefer. For further information, write Prof. E. A. Schweikert, Department of Chemistry, Texas A&M University, College Station, Texas 77843 U.S.A.; (409) 845-2341.

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.

Effective January 1, 1985, "Organic Magnetic Resonance" (published by Wiley Heyden, Chichester, U.K.) will have a new name and expanded scope. The new name, "Magnetic Resonance in Chemistry," has been selected to indicate a broadened purview, including magnetic resonance papers primarily in the realms of inorganic chemistry, biochemistry, physical chemistry, and combinations of these. The traditional focus on organic chemical applications will, however, continue. The format and operation of the journal will remain unchanged.

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. 317 ----- 4 February 1985

No. 318 ----- 4 March 1985

November 26, 1984.

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Prof. Bernard SHAPIRO

TAMU NMR NEWSLETTER

Department of Chemistry

TEXAS A & M UNIVERSITY

College Station TX 77843

U.S.A.

Dear Professor Shapiro,

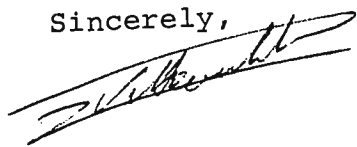
We showed recently that the analysis of deuterium quadrupolar relaxation rates is a convenient way for the study of pH induced motional changes and molecular volume variations of simple amino acids⁽¹⁾.

The same approach has been used to examine the behavior of amino acids with functional lateral groups : d,l-glutamic acid-2-[²H₁] and d,l-lysine-2-[²H₁].

It has been observed that the deuterium relaxation rates of these amino acids are minimum in the pH range near the neutrality (fig. 1 and 2).

The relaxation rates were used to calculate averaged molecular volumes of the different ionic forms of these amino acids. Molecular volume variation is actually related to the relative extent of inter and intramolecular ionic associations of the different functional groups, it gives therefore informations about the conformation of these molecules in their different ionic structures.

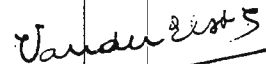
Sincerely,



Prof. Yves VAN HAVERBEKE



Dr. Robert MULLER



Dr. Luce VANDER ELST

(1) Y. VAN HAVERBEKE, R.N. MULLER, L. VANDER ELST, J. Phys. Chem., 88, 4978 (1984).

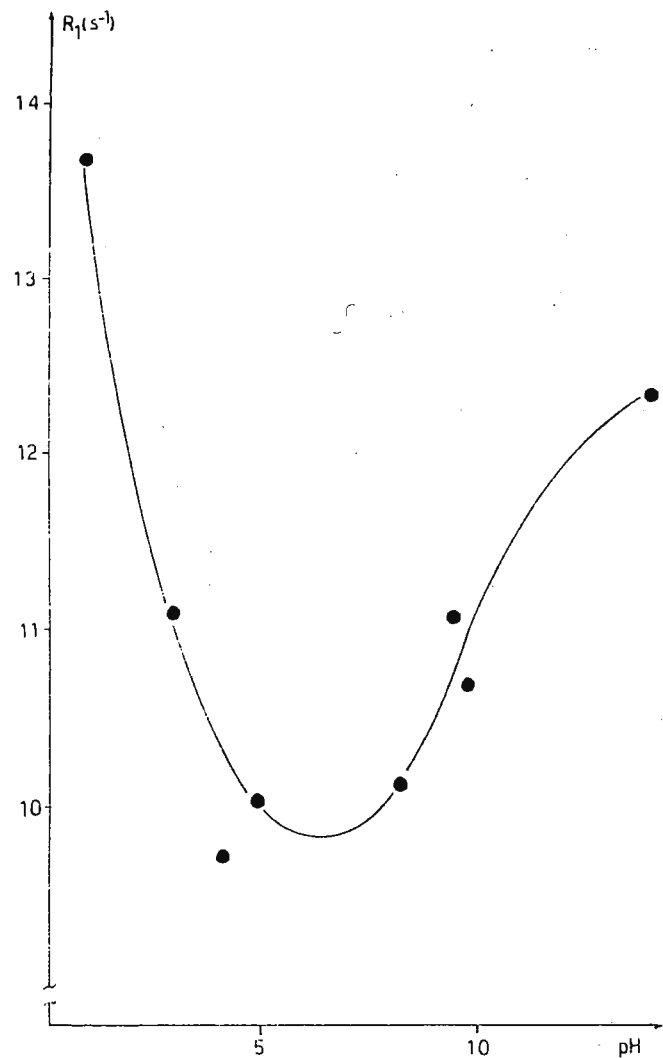


Fig. 1 : Deuterium longitudinal relaxation rates of d,l-glutamic acid-2- $[^2\text{H}_1]$ (0.2 M) vs pH at 9.2 MHz.

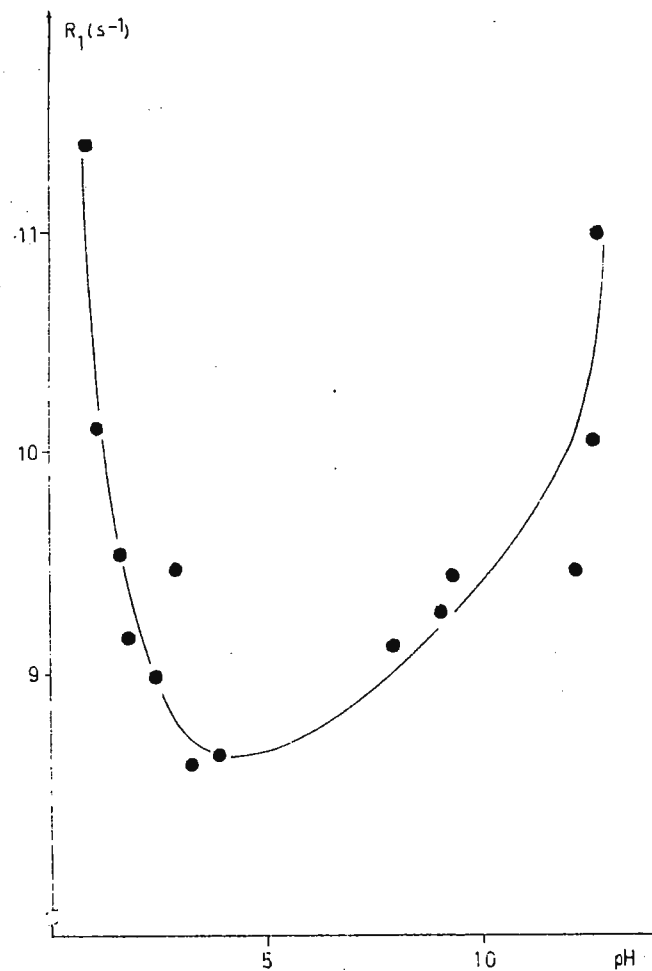


Fig. 2 : Deuterium longitudinal relaxation rates of d,l-lysine-2- $[^2\text{H}_1]$ (0.16 M) vs pH at 9.2 MHz.



November 19, 1984

Dr. Bernard L. Shapiro
Texas A&M University
College Station, TX 77843-3255

Dear Barry:

Subject: Magneto-Acoustic Probe Ringing

Recently we have been forced to deal with problems of spurious probe ringing following an initial pulse in two of our probes for solid NMR. In each case the ringing persisted for almost 100 μ sec when the probe was in the magnet but was absent when the probe was pulsed outside the magnet. The standard solutions to this problem (see Fukushima and Roeder, Experimental Pulse NMR, p. 463) were ineffective. Numerous coil designs and placement of lead shielding between the coil and the probe body did not significantly shorten the ringing time.

In each of the probes it turned out that the ringing was in subsidiary parts of the probe tuning circuitry. In one case the ringing was cured by a change of position and orientation of one of the chip tuning capacitors. In the other case, reworking of the proton trap solved the problem.

For probes having the tuning circuitry outside of the magnetic field, ringing of the components is no problem. If spin-temperature alternation is used in the cross-polarization experiment, the ringing may not be noticed even when it does exist. Thus even commercial probes may not be built for maximum suppression of residual ringing. When the problem does become important, reworking of the entire probe circuitry, not just the main coil, may be necessary.

Very truly yours,

A handwritten signature in cursive script, appearing to read 'Mark', written in dark ink.

PMH:slk

P. Mark Henrichs
Chemistry Division
Research Laboratories

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Research Institute of Scripps Clinic
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December 3, 1984

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

RE: A VERSATILE RING CURRENT SHIFT PROGRAM FOR
APPLICATION TO PROTEINS.

Dear Professor Shapiro:

A computer program has been developed to interactively calculate ring current shifts due to aromatic amino acids and heme ring systems using either X-ray or neutron diffraction structures of proteins. The program was developed on a VAX 11/750 computer running under the VMS operating system. The program is written in FORTRAN-77 and uses a number of VAX-11 FORTRAN extensions to the FORTRAN-77 standard.

The program accepts structural data in the Brookhaven Protein Data Bank format (Brookhaven National (USA) Laboratory Protein Data Bank), and can be readily modified to accept other data formats without disrupting the program structure. Proton coordinates may be calculated from the heteroatom locations using standard bond lengths and bond angle data, or read directly from the structure file in the case of neutron diffraction structural data.

The user may specify the proton for which the ring current shift is to be calculated in one of three ways. By specifying a type of residue, for example PHE, the program calculates the chemical shifts of all protons attached to phenylalanine residues. Including the residue index number, as in ALA 54, restricts the program to calculating the ring current shift for protons belonging to alanine 54. Finally, the user may specify the atom to which the proton is attached, MET 61 CE, and obtain the ring current shift for the CH_3 protons of methionine 61. The program calculates the average shift of methyl rotamers.

Ring current shifts are calculated using both the Johnson-Bovey model (C.E. Johnson and F.A. Bovey, J. Chem. Phys. **29**, 1012 (1958)) and the Haigh-Mallion model (C.W. Haigh and R.B. Mallion, Mol. Phys. **22**, 945 (1971)). Heme ring current shifts are calculated using

both an eight-loop and a five-loop model for the Johnson-Bovey model and a five-loop model for the Haigh-Mallion model.

Copies of the software and a guide to program usage are available from the above address.

Please credit this contribution to the Scripps's Clinic account in Dr. Peter E. Wright's name.

Sincerely,

Keith Cross

Keith J. Cross

KJC:st

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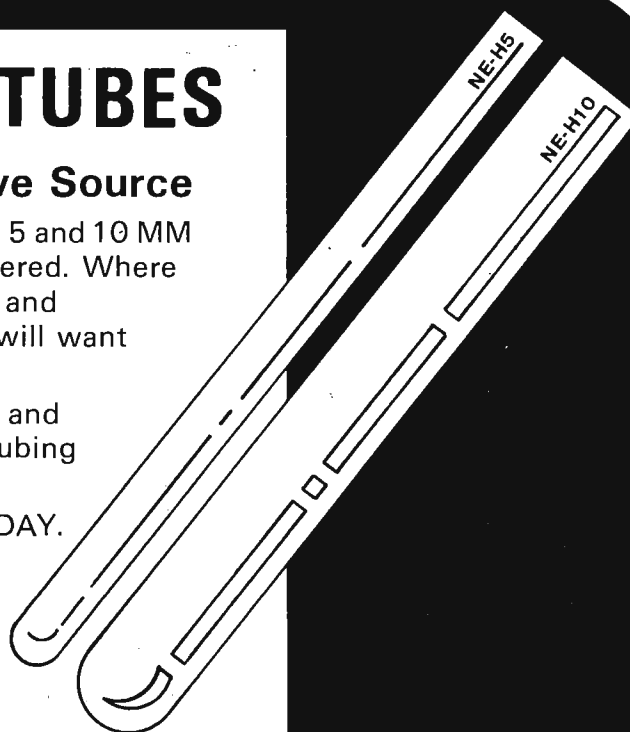
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Professor B.L. Shapiro
Department of Chemistry
Texas A & M University
COLLEGE STATION, TEXAS 77843, U.S.A.

16th November, 1984.

Dear Professor Shapiro,

Re: Improved Signal to Noise in Solid State NMR.

We wish you to advise your readers, particularly those interested in solid state NMR, of the significant advantages afforded by the use of MAS Spinners manufactured from PSZ ceramic material. The inherent strength of PSZ enables spinner wall thickness to be reduced as low as 0.6mm thus providing an approximate twofold increase in internal volume compared to spinners manufactured from boron nitride.

The consequences of increased internal volume and thus increased sample size, are obvious and are demonstrated by Figure 1. These silicon-29 spectra were recorded on a Bruker CXP200.

Our spinners were developed by Mr.G.W. West of the CSIRO Division of Materials Science, Melbourne, and are manufactured by Nilcra Ceramics Pty Limited from MgO stabilized zirconium oxide material. This material is virtually chemically inert, not easily damaged by abuse, and easily cleaned in an ultrasonic bath. Because of the spinners' hardness, a moderate reduction in the lifetime of the RF coil is expected, but the spinners should last for ages.

Nilcra Ceramics are holding stocks of these items and intend selling them through their worldwide sales outlets direct to users at a cost of US\$110-00 each. The current design has one end open with an internal thread (6.5 major diam, 0.6 pitch L.H. isometric form) but can be supplied unthreaded if required. Plastic end caps (kel-f) can be supplied at a cost of US\$45-00 each, or provided by the user.

Your readers will receive improved results if they use this new product.

Yours faithfully,

*David J. Cookson*Dr. David J. Cookson
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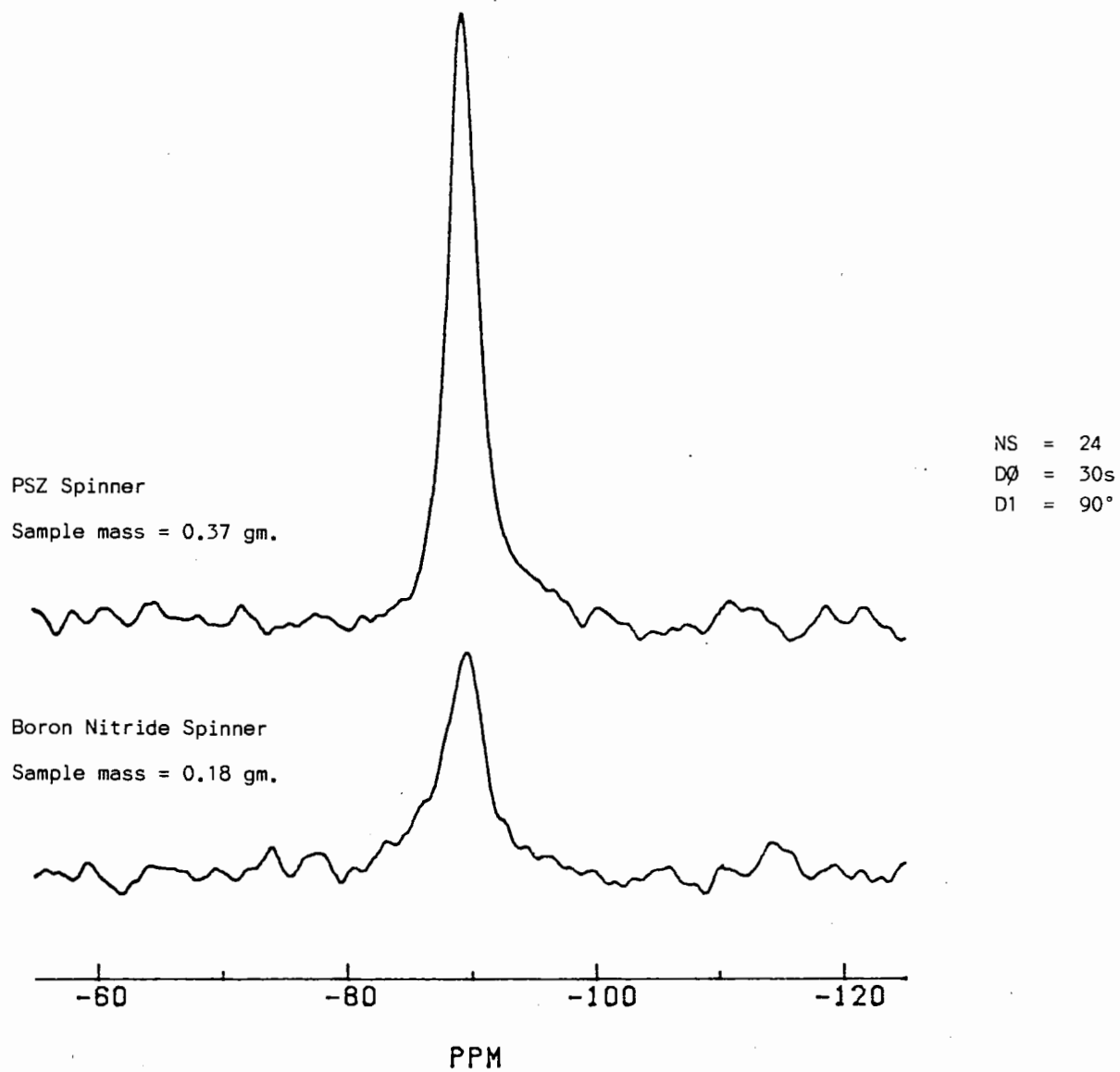


Fig.1.

Los Alamos

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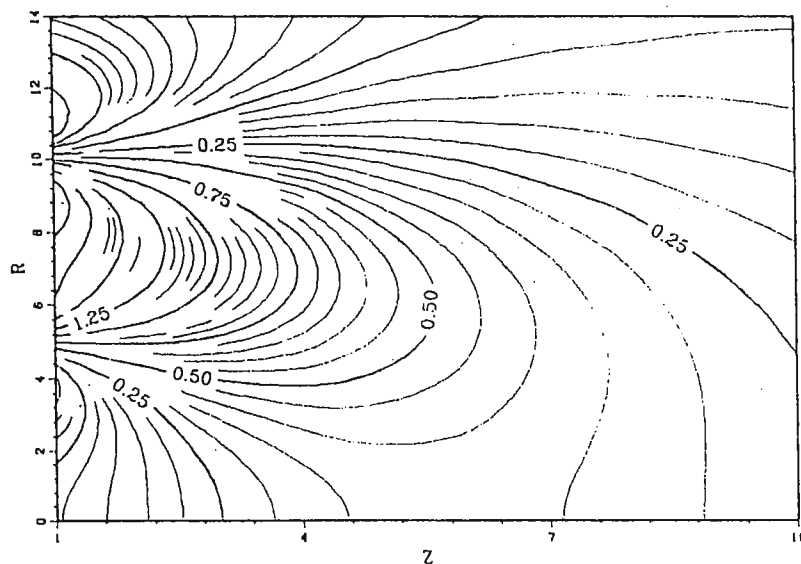
Dr. B. L. Shapiro
Department of Chemistry
Texas A & M University
College Station, TX 77843

The Opposed Loop: An Improved Surface Coil?

Dear Professor Shapiro:

With the realization of whole body NMR as a viable technique, remote sensing methods (obtaining signals from samples not contained in the sample coil) have assumed a renewed importance. [Remote sensing NMR had been used primarily for industrial and geological applications.] Furthermore, with the emergence of topical NMR as an important biomedical tool, the spatial selectivity of sensitive probe regions has become very important.

We describe a coil (the opposed loop) which is potentially useful for spatial localization of the signal. It is a surface coil consisting of a pair of unequal loops, coaxial and coplanar, with currents 180° out of phase to cancel the field on the cylindrical axis near the plane of the two loops.



The sketch shows the Z-component of the field for a pair of loops having radii 5 and 10 with currents adjusted so the field on axis is zero in the plane of the loops. [Note that the Z-scale is expanded compared to the R-scale.] The uniform field region is a saddle point and, for this geometry, occurs on the axis at a distance approximately equal to the radius of the smaller loop. The field intensity at this point is about 60% of the field intensity from the larger loop alone.

A way to take advantage of the field localization near the axis may be to use both loops to transmit and only the smaller loop to receive. In addition, this coil might be used to "sharpen up" the effects of depth pulses.

Please credit this note to Bill Earl's account. With best wishes.

Alan

Alan Rath
Univ. of New Mexico

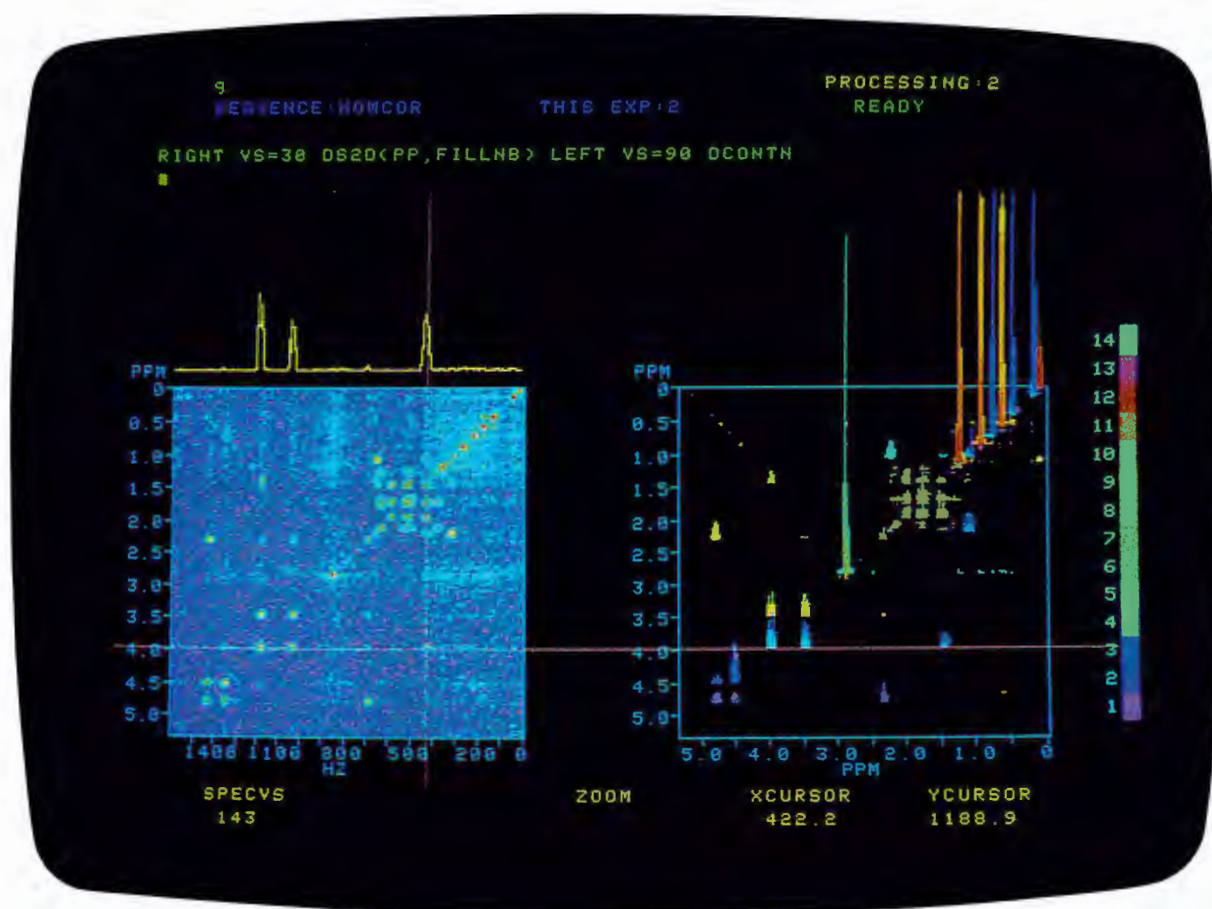
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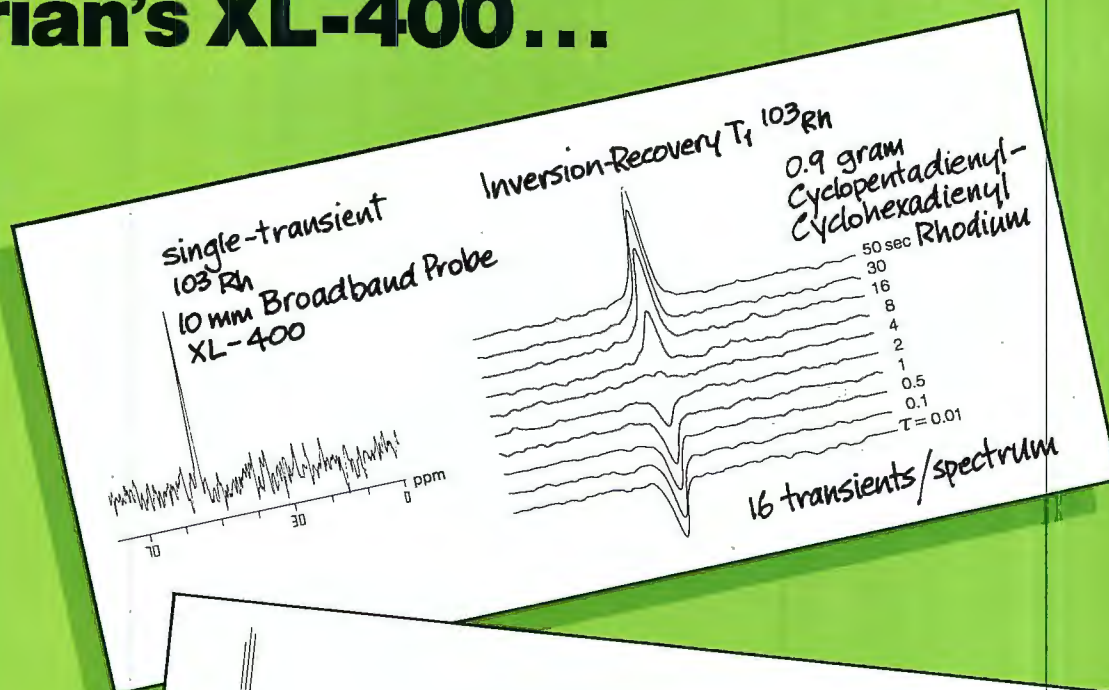
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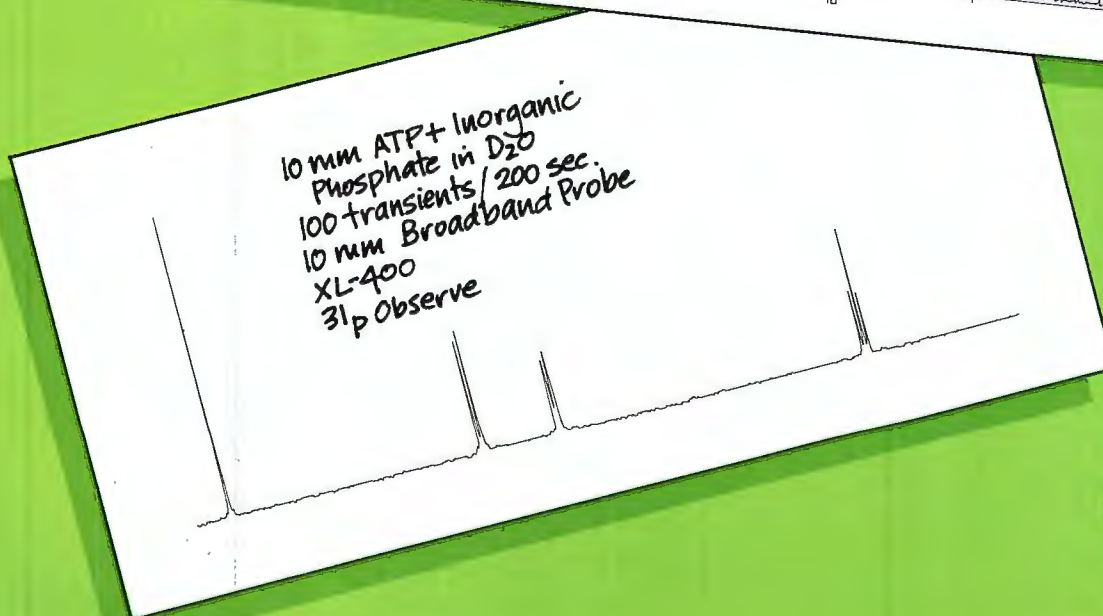


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November 8, 1984

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

³¹P NMR Studies of Inorganic Phosphate
Transport into Red Blood Cells

Dear Barry:

We have recently been working in collaboration with Dr. Reinhart Reithmeier on anion transport in red blood cells (RBCs), using ³¹P NMR spectroscopy. The resonance for intracellular inorganic phosphate (P_i^I) can be resolved from that of extracellular inorganic phosphate (P_i^O) by adjusting the extracellular pH (pH 6.4) to be appreciably different from the intracellular pH (pH ~7.4), since the ³¹P chemical shift varies considerably with pH. Upon addition of P_i (pH 6.4) to an equal volume of packed RBCs, the decrease in the intensity of the P_i^O resonance and increase in the intensity of the P_i^I resonance with time reflects the kinetics of the P_i influx (Fig. 1). The intracellular and extracellular pH were found to converge as P_i transport progressed, as determined from the change in chemical shifts of the P_i^I and P_i^O resonances with time (Fig. 1). These pH changes are consistent with transport of the $H_2PO_4^{-1}$ rather than the HPO_4^{-2} form of P_i . This work will soon be submitted for publication.

One very interesting and initially confusing aspect of this study was the appearance of an additional resonance in addition to the P_i^I and P_i^O resonances (Fig. 2). This third resonance's appearance was quite reproducible between experiments, blood samples and P_i concentrations. It did not appear at the beginning of the experiment, but only about 20-30 minutes after the erythrocytes had been mixed with the P_i solution and put into the spectrometer. The NMR samples were always spun at 20-25 Hz to average out B_0 field inhomogeneities during data acquisition. If the blood samples were not spun in the NMR spectrometer or if the sample was spun but was taken out and remixed each 6 minutes, no third resonance occurred. RBCs settle very slowly (2-3 days) in a 50% hematocrit; if, however, they are spun at the low spinning rate of 20-25 Hz, the cells settle in about 30 minutes. Thus, the mysterious third resonance seemed to be an experimental artifact generated by P_i^O within the settled RBC "pellet", having a different magnetic susceptibility from P_i^O in the clear supernatant. Indeed, if a non-transportable compound such as adenosine 5'-monophosphate (AMP) was added to RBCs and ³¹P NMR spectra were taken of the spinning sample, two resonances did appear after about 30 minutes (Fig. 3). Therefore, caution should be exercised in doing NMR experiments with cells which can readily spin down and have magnetic susceptibilities significantly different from the extracellular solution.

Sincerely yours,

Manfred

Manfred Brauer, Ph.D.

Brian

Brian D. Sykes, Ph.D.

Figure 1.

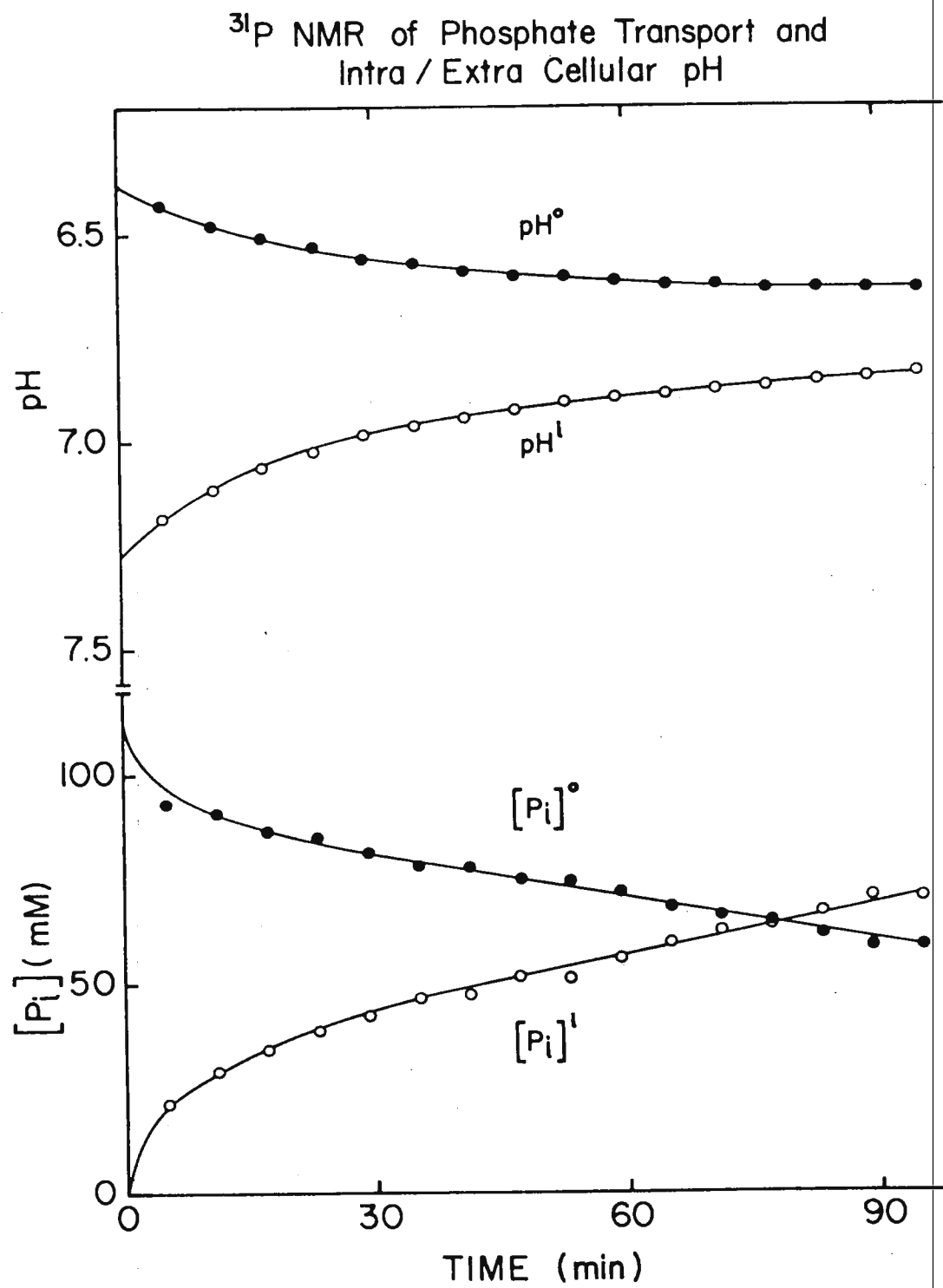


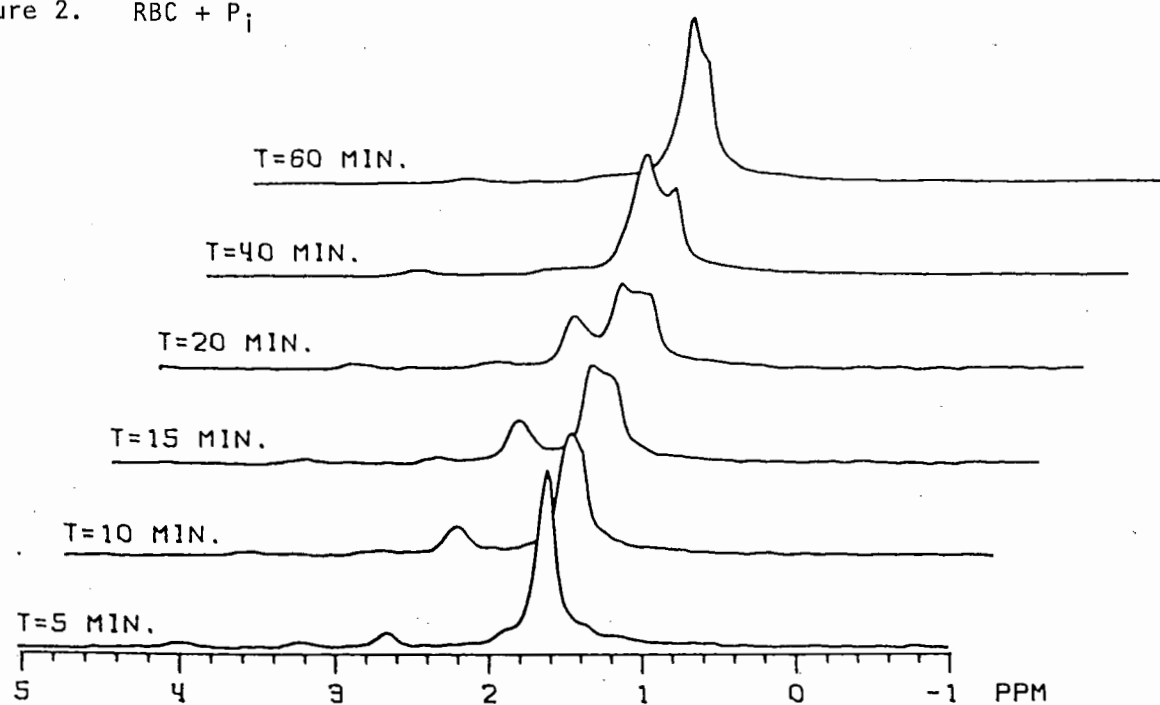
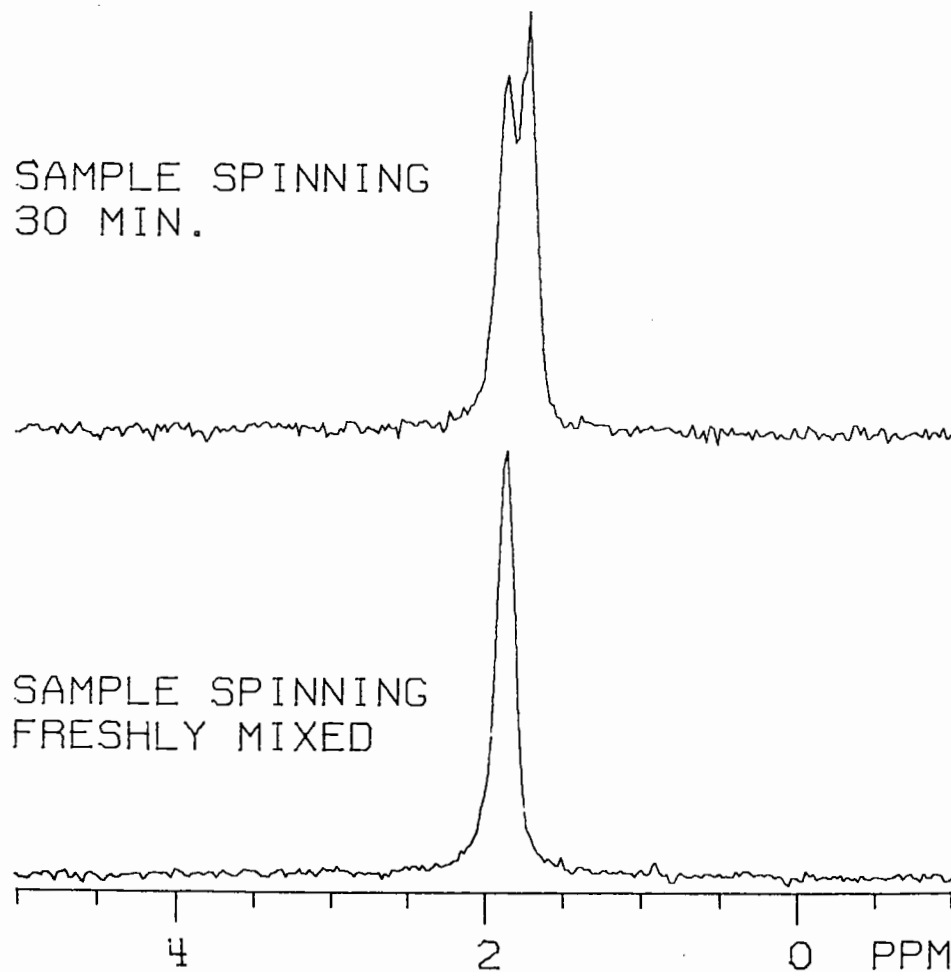
Figure 2. RBC + P_iRBC + AMP

Figure 3.



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**Twenty-sixth Experimental Nuclear Magnetic Resonance Spectroscopy Conference
Asilomar, California, April 21-25, 1985**

November 27, 1984

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

Dear Barry:

Subject: Twenty-Sixth Experimental NMR Conference

The 26th Experimental NMR Conference (ENC) will be held at the Asilomar Conference Center near Monterey, California from April 21 to April 25, 1985. Information for Registration will be sent to all attendees of the conference during the past five years. Registration forms may also be obtained from the conference secretary, Dr. R. C. Bryant, at the address shown in this letterhead. The registration packet includes instructions on the presentation of posters on current research. The Poster Chairman for 1985 is Dr. Charles G. Wade of IBM Instruments, 40 W. Brokaw Road, San Jose, CA 95030.

Very truly yours,



P. Mark Henrichs
Research Laboratories
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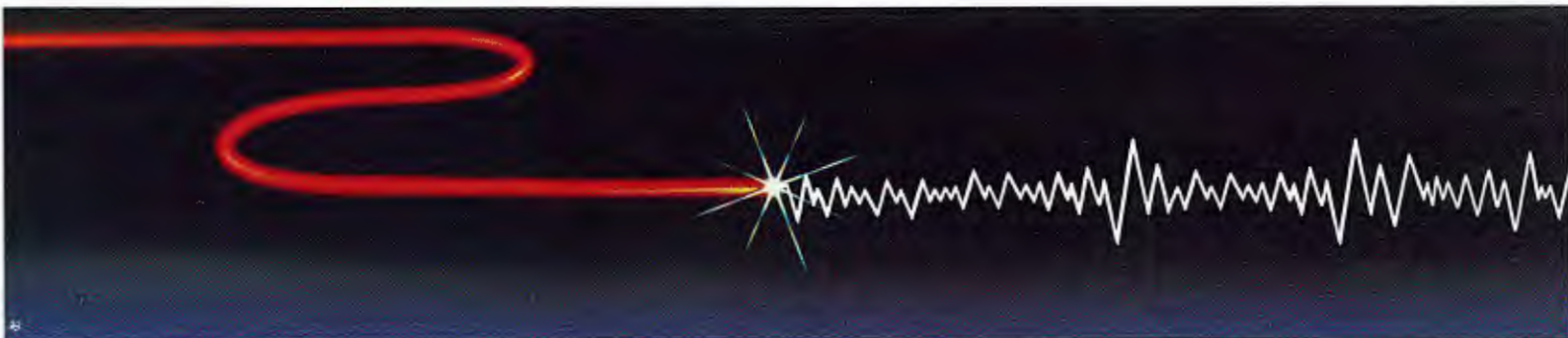
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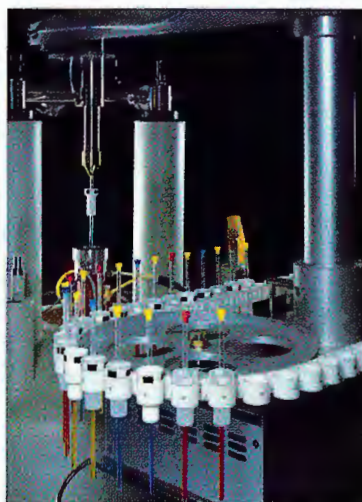
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Texas A & M University,
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³¹P NMR of plant cell suspension cultures

Dear Professor Shapiro,

In our continuing work with ³¹P NMR of plant cell suspension cultures, we were interested to see if we could identify more unambiguously the resonances in the region -8 to -12 ppm of the spectrum where the nucleoside diphosphosugars occur. Recent chromatographic work on various culture extracts has shown that uridine diphosphosugars are major phosphorus-containing components in the cell, although the HPLC separation of the glucose and galactose containing compounds was very difficult. Although the in vivo ³¹P NMR signals for these compounds are broad, spectra at 162 MHz of the extracts, after careful treatment for the removal of paramagnetic ions¹, allowed immediate identification and quantification of these components for various cultures (see figure).

Yours sincerely,

Victor Wray
Victor Wray

Jochen Berlin
Jochen Berlin

Otmar Schiel
Otmar Schiel

1. V.Wray, O.Schiel and J.Berlin, Z. Pflanzenphysiol. 1983, 112, 215.

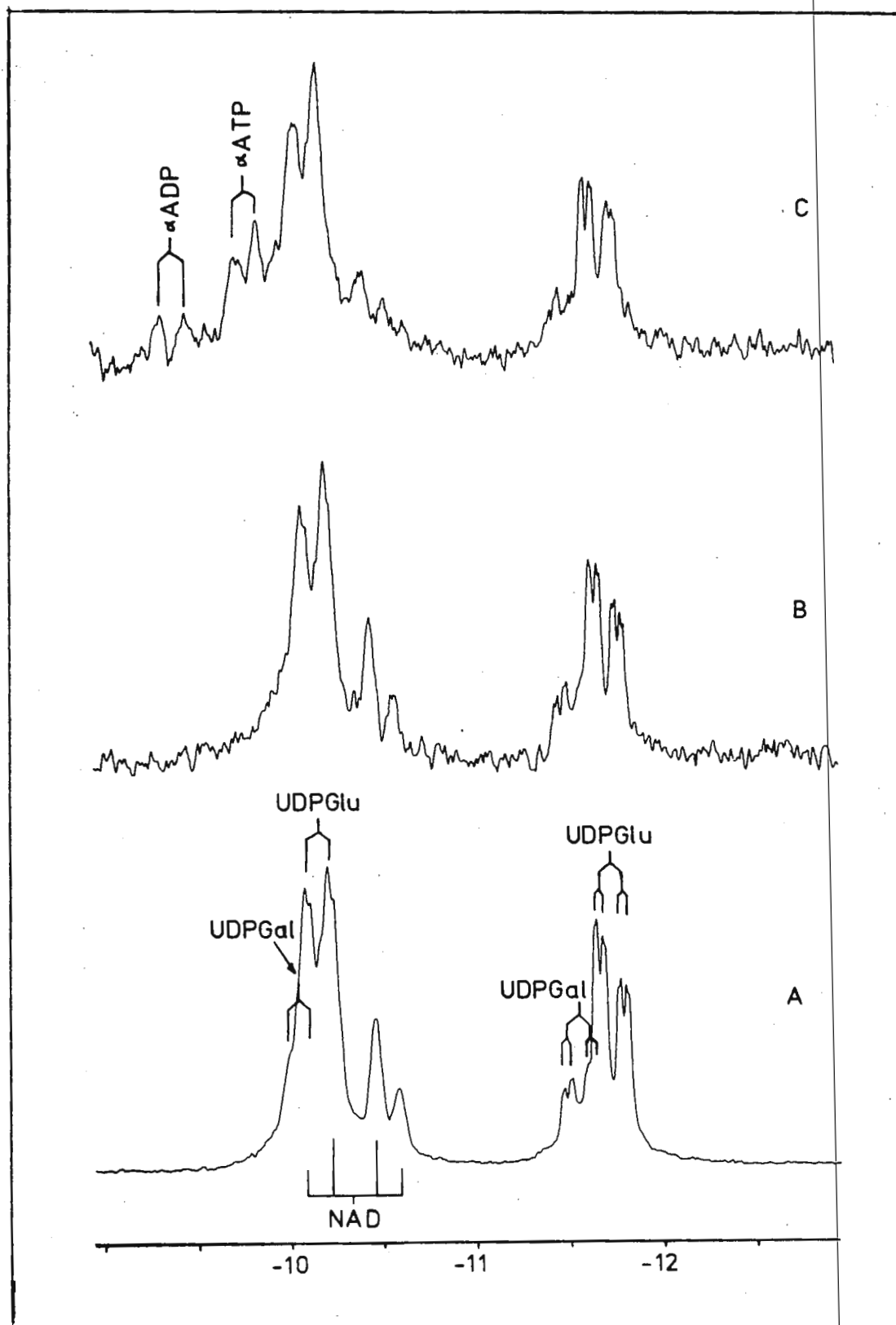


Figure. ^{31}P NMR spectra of A. Synthetic mixture of uridine diphosphoglucose, uridine diphosphogalactose and NAD (4:1:2), B. Extract of Solanum demissum culture, and C. Extract of Catharantus roseus culture.

Monsanto Company
800 N. Lindbergh Boulevard
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November 28, 1984

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

Dear Barry,

We find ourselves with a supply of analog chart paper for which we have no use. Perhaps one of your readers might be interested in obtaining one of the following items f.o.b. Monsanto.

- Bruker analog chart paper - 500 mm. MHz style : 4 boxes (2000 sheets)
- Varian analog chart paper - 500 mm. part no. 9499908-49 : 10 boxes (5000 sheets)

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With best wishes,



William C. Hutton
Research Specialist

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ÚSTAV PŘÍSTROJOVÉ TECHNIKY
612 64 BRNO, KRÁLOVOPOLSKÁ 147

9th November 1984

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

Dear Professor Shapiro,

recently we have described (1,2) a modified version of the 1 $\overline{2}$ 3 inversion pulse, which can be used for construction of broadband decoupling sequences with extended bandwidth of efficient performance. As a part of the study, we have also tested the applicability of these sequences to the real time J-scaling experiment (3). The performance was checked both theoretically (using exact Waugh theory) and experimentally. The results confirmed our anticipation and may be shortly summarized as follows:

Periodical application of a supercycle $C_{ab} \overline{C}_{ab}$ (bar denotes phase inversion of all elements), interrupted by delays t_p with decoupler power gated off, in pulse scheme for real time J-scaling, with

$$C_{ab} = \overline{b(4-a+b)(4-a+b)2b(1+b)(3-a)(4-a+b)b} \overline{b(1+b)(3-a)(4-a+b)2b(1+b)(3-a)(4-a+b)b}$$

enables to extend the performance from offset range $\pm 1.0 \Delta B/B_2$ for original WALTZ cycle ($a = 1.0$ $b = 0.0$) to the value of $\pm 1.45 \Delta B/B_2$ for the modified version having $a = 1.5$ and $b = 0.25$. Experimental verification and computed variation of residual coupling constant J^R for the values $a = 1.33$ and $b = 0.33$ are documented on accompanying figures.

Please credit this contribution to the subscription of Dr. M. Hájek, Institute of Chemical Technology, Prague.

With kindest regards

Sincerely yours

Vladimír Sklenář

Zenon Starčuk

Institute of Scientific Instruments
of Czechoslovak Academy of Sciences

- (1) Z. Starčuk and V. Sklenář, J. Magn. Reson. in press
- (2) V. Sklenář and Z. Starčuk, Org. Magn. Reson. 22, 662 (1984)
- (3) G.A. Morris et al., J. Magn. Reson. 58, 155 (1984)

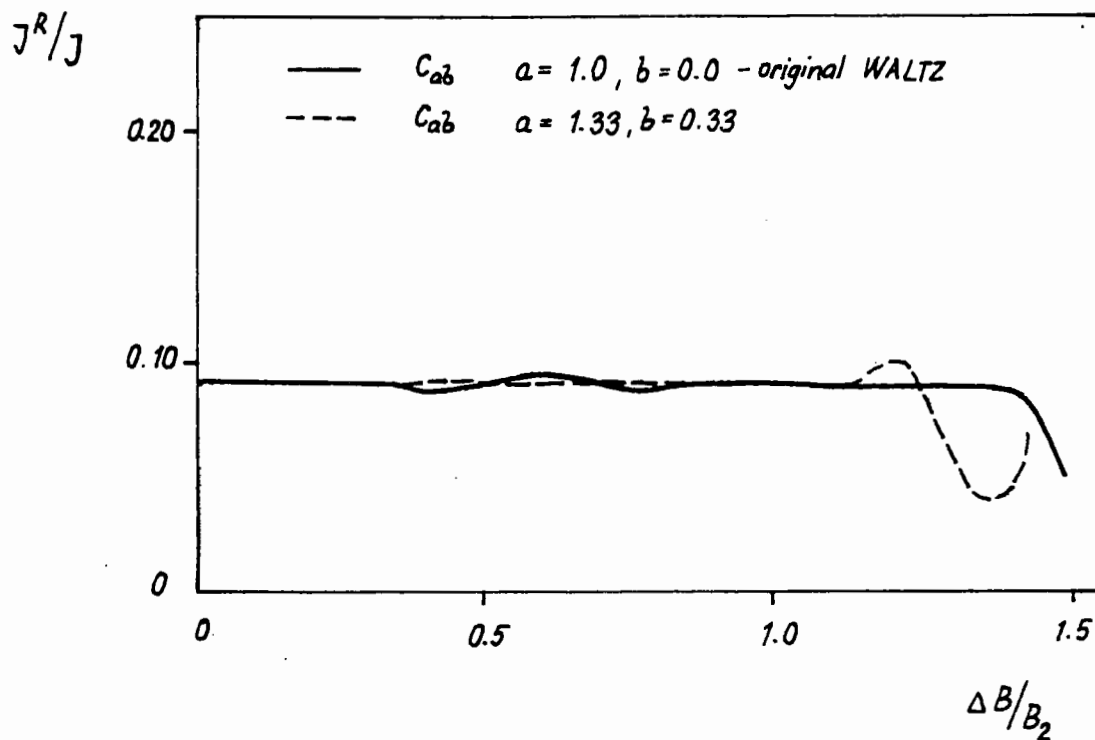


Fig. 1. Theoretical performance of the cycle $C \bar{C}$ in the J-scaling experiment with $a = 1.33$ and $b = 0.33$.

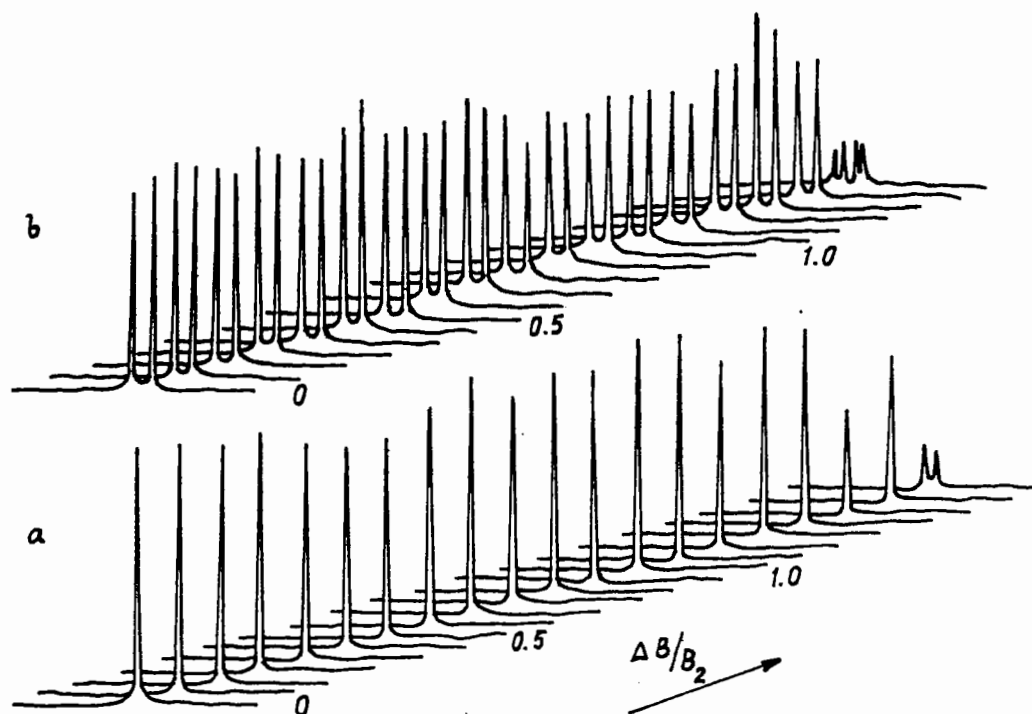


Fig. 2. Experimental spectra of the decoupling (a) and J-scaling (b) efficiency using the cycle $C \bar{C} \bar{C} \bar{C}$ (a) and $C \bar{C}$ with $a = 1.33$ and $b = 0.33$.

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DEPARTAMENTO DE QUIMICA

November 8, 1984

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

The ^{13}C nmr spectra of α -cedrene and of cedrol

Dear Professor Shapiro:

These are two reports^{1,2} on the assignment of the ^{13}C nmr spectrum of α -cedrene (1), the pertinent data being summarized on the Table. Evaluation of these data reveals that the assignments of C(4) and C(9), and of C(7) and C(8a) are interchanged and that in one study² no specific distinction of C(12) and C(13) was attempted.

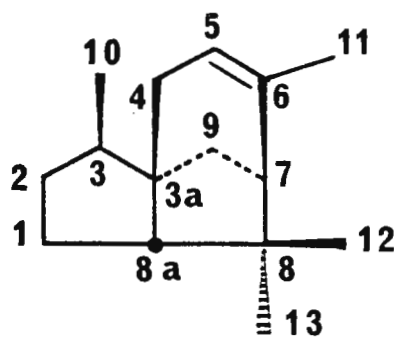
In order to gain conclusive proof for the distinction of the C(4)/(9) pair, a sample of 4-deutero- α -cedrene was prepared: Treatment of a mixture of α - and β -cedrene with *m*-chloroperbenzoic acid gave *exo*-5,6-epoxycedrane and *exo*-6,11-epoxycedrane, respectively. $\text{Et}_2\text{O}:\text{BF}_3$ catalyzed rearrangement of the 5,6-epoxide gave 5-cedranone which was equilibrated with MeOD/MeONa to deuterate position 4. Reduction of the carbonyl group with LiAlH_4 gave a mixture of 4-deutero-5-cedranol and 4-deutero-5-neocedranol. Finally, dehydration of the epimeric mixture of alcohols with *p*-TsOH in toluene gave 4-deutero- α -cedrene.

On the other hand, treatment of the *exo*-6,11-epoxide with LiAlD_4 gave 11-deutero-6-isocedrol which was dehydrated with *p*-TsOH to provide a sample of 11-deutero- α -cedrene, thus allowing confirmation of the assignment of the vinyl methyl signal.

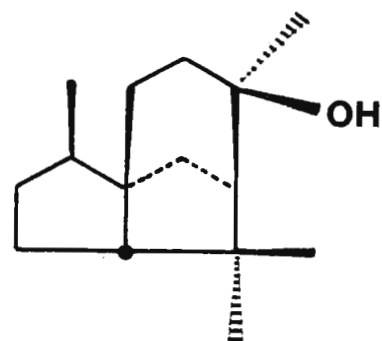
The distinction of the C(7)/(8a) pair of signals was done from a two-dimensional $^{13}\text{C}/^1\text{H}$ heteronuclear chemical shift correlation diagram³, which directly allows to assign H(7) as the X part of an ABX system in which the AB portion corresponds to the pair of protons attached to C(9). Since C(9) was ascertained by distinction from C(4) by deuteration, the assignment of C(7) follows directly.

For the specific assignment of the two carbon signals owing to the *gem*-dimethyl group, we also resorted to regiospecific deuterium labeling. A sample of cedrol was converted by transannular $\text{Pb}(\text{OAc})_4$ oxidation into 6,12-oxycedrane. The tetrahydrofuran ring was cleaved with $\text{Et}_2\text{O}:\text{BF}_3/\text{Ac}_2\text{O}$ to yield α -cedren-12-ol acetate and the ester hydrolyzed with base to afford α -cedren-12-ol. The primary alcohol was oxidized to α -cedren-12-al with $\text{Na}_2\text{Cr}_2\text{O}_7/\text{H}_2\text{SO}_4$ and reduced with LiAlD_4 to generate α -cedren-12-ol deuterated at C(12). A reoxidation as above gave 12-deutero- α -cedren-12-al which was converted into 12-deutero- α -cedrene by formation of the dithioethyleneketal followed by Ni (Raney) desulfurization.

Alternatively a ^1H gated-decoupling ^{13}C spectrum of α -cedrene shows that the long range (^3J) multiplicity of C(12) is a quintet while that of C(13) appears as a sextet. This is due to the fact that C(12) is almost 90° with respect to H(7). This methodology also allowed distinction of the *gem*-dimethyl signals of cedrol (2). All other carbons of cedrol were assigned from double quantum coherence measurements^{3,4} and show that the original spectral interpretation of cedrol is correct⁵. Therefore reversing of some pairs of signals for cedrol, as proposed later⁶, is not justified.



1



2

Atom	from ¹	from ²	our	from ¹	our
1	24.8	24.9	24.9	25.0	25.0
2	36.1	36.3	36.2	36.6	37.0
3	41.5	41.7	41.6	41.0	41.5
3a	53.8	53.9	53.8	53.7	54.1
4	40.6	38.8	38.9(b)	31.2	31.6
5	119.1	119.3	119.0	34.8	35.3
6	140.2	140.5	140.1	73.9	75.0
7	54.9	59.2	55.0	60.6	61.0
8	48.1	48.1	48.0	43.0	43.4
8a	59.0	56.1	59.0	56.2	56.6
9	38.8	40.8	40.7	41.5	42.0
10	15.1	15.4	15.5	15.2	15.6
11	24.7	24.7	24.8(b)	30.2	30.2
12	25.6	25.7(a)	27.7(b)	27.7	27.7
13	27.6	27.7(a)	25.7	28.6	28.9

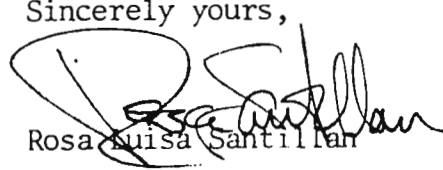
(a) not specifically assigned.

(b) labeled with deuterium.

- 1.- E. Wenkert, B.L. Buckwalter, I.R. Burfitt, M.J. Gasic, H.E. Gottlieb, E.W. Hagaman, F.M. Schell and P.M. Wovkulich, "Carbon-13 Nuclear Magnetic Resonance Spectroscopy of Naturally Occurring Substances", G.C. Levy, Ed., "Topics in Carbon-13 NMR Spectroscopy", Wiley-Interscience, New York, Vol. 2, p. 81 (1976).
- 2.- F. Bohlmann and C. Zdero, Chem. Ber., 112, 427 (1979).
- 3.- P. Joseph-Nathan, R.L. Santillan and A. Gutiérrez, J. Nat. Prod. (in press).
- 4.- J.N. Shoolery, J. Nat. Prod., 47, 226 (1984).
- 5.- A.F. Thomas and M. Ozainne, Helv. Chim. Acta, 62, 361 (1979).


 Pedro Joseph-Nathan

Sincerely yours,


 Rosa Luisa Santillan



THE PROCTER & GAMBLE COMPANY

MIAMI VALLEY LABORATORIES

P.O. BOX 39175
CINCINNATI, OHIO 45247

November 5, 1984

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

Si-29 NMR of Organosiloxanes

Dear Dr. Shapiro:

We have been utilizing Si-29 polarization transfer techniques routinely to assign structures of organosiloxanes, taking advantage both of the enhanced sensitivity and the suppression of the unwanted signal from the sample tube. The measurement of Si-H spin-spin coupling constants in polydimethylsiloxanes with terminal long-chain alkoxy groups is of current interest to us.

As an example, the accompanying figures show proton-coupled and selectively-decoupled Si-29 INEPT spectra of a dialkoxy tetramethyldisiloxane $(\text{RO}(\text{CH}_3)_2\text{Si}-\text{O}-\text{Si}(\text{CH}_3)_2\text{OR}, \text{R} = -\text{CH}_2(\text{CH}_2)_n\text{CH}_3)$ obtained on our JEOL FX-270 spectrometer. The proton-coupled Si-29 INEPT spectrum in Figure 1 consists of a complex multiplet that cannot be readily analyzed. On the other hand, the coupled INEPT with a Si-29 refocussing pulse spectrum in Figure 2, shows a heptet of triplets due to couplings to the six methyl ($^2\text{J}_{\text{Si-H}} = 7.3$ Hz) and the two methylene protons ($^3\text{J}_{\text{Si-H}} = 2.9$ Hz). In order to simplify the spectrum and confirm the assignments of the coupling constants, we applied a version of the SPINEPTR pulse sequence for the selective decoupling of protons, recently described by J. Schraml [1]. The advantage of the technique is that no hardware modification is required. Only the decoupler power and H-1 pulse length were adjusted until sufficient proton-decoupling selectivity and polarization transfer was achieved.

As is shown, the multiplet in Figure 2 collapses into a triplet when the decoupler frequency is set at the methyl resonance (Figure 3) and clearly appears as a seven-line pattern when the methylene resonance is irradiated (Figure 4). For this particular setting of the decoupler power, the spin-spin couplings are reduced by 50% ($^2\text{J}_{\text{Si-H}}(\text{reduced}) = 3.6$ Hz, $^3\text{J}_{\text{Si-H}}(\text{reduced}) = 1.4$ Hz).

Sincerely,

THE PROCTER & GAMBLE COMPANY
Research & Development Department

Fouad S. Ezra
Fouad S. Ezra

Jack D. Wendel
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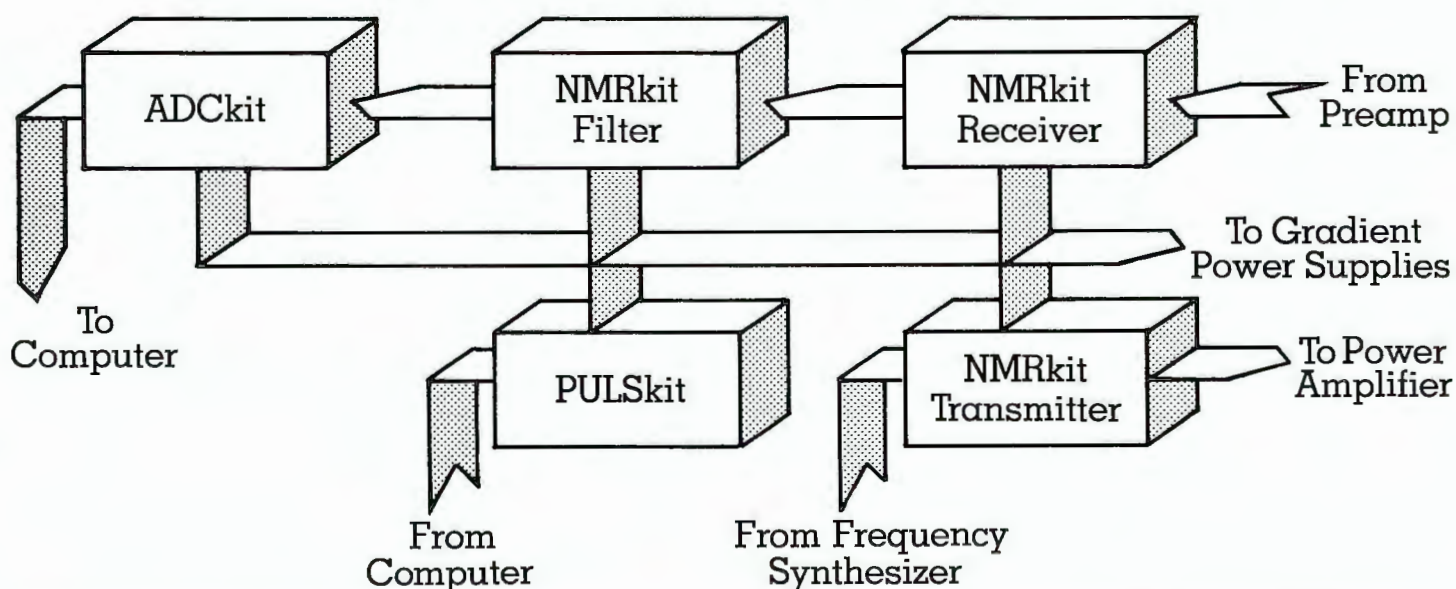
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—The **Filter** board is composed of two four-pole Butterworth or Bessel filters (one set of filters for each channel). The selection of the bandwidth and filter type (Bessel or Butterworth) is under computer control.

The **PULSkIt** is a universal pulse programmer specifically designed for NMR applications. It generates the different intervals required in an NMR experiment, controls the magnetic field gradients and the shape of the selective pulse, and can also drive a two-channel analog-to-digital converter (the **ADCKit**, for example). Besides a time resolution of 100 ns and a minimum pulse width of 500 ns, the **PULSkIt** has five, independent, 16-bit loop counters and a memory of 2K x 128 bits, providing 76 control lines for your instrument. The **PULSkIt** can be interfaced to a VAX-11/750 or PDP-11 computer via a DR-11/W interface board, or any other 16-bit bus using the appropriate interface.

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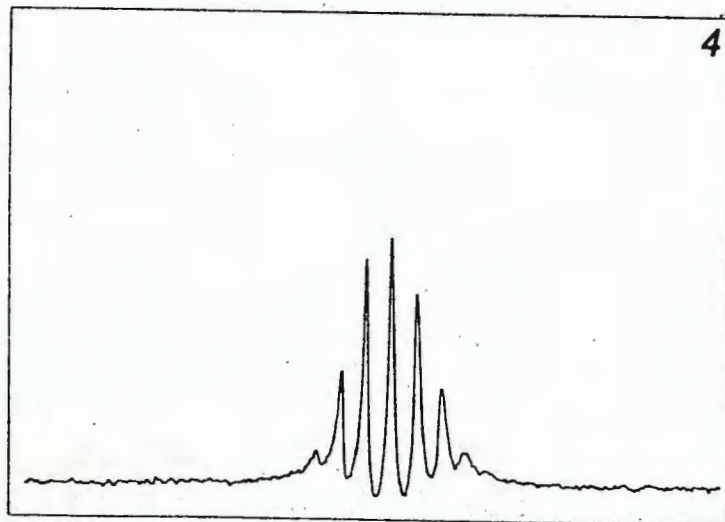
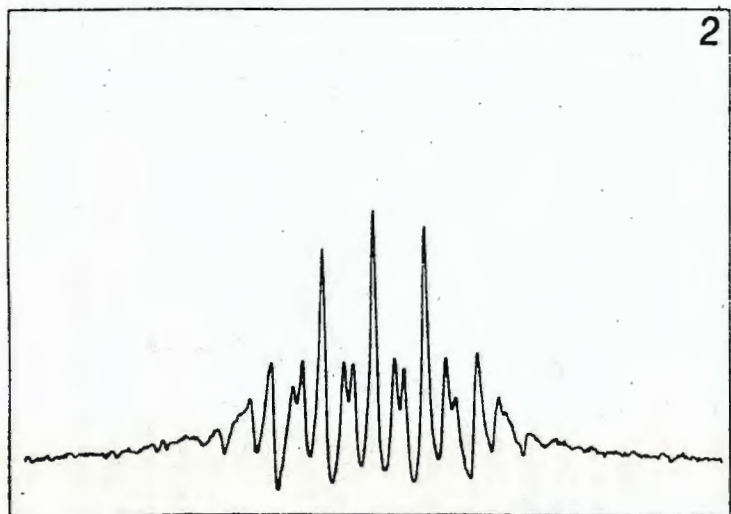
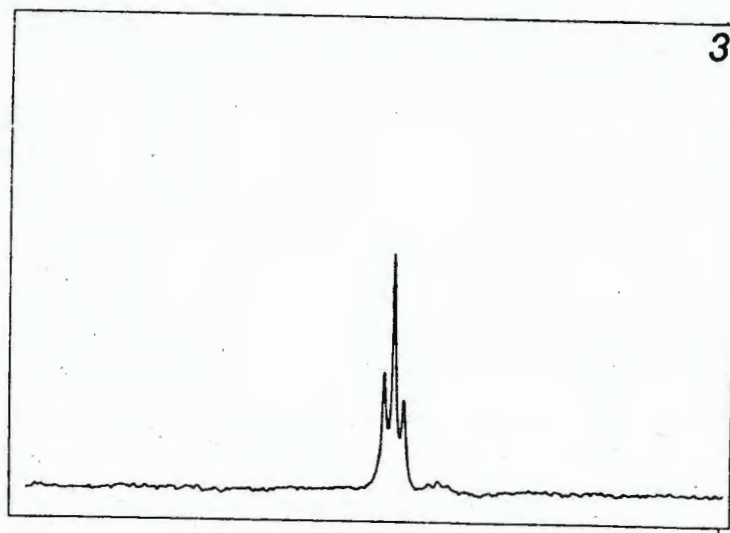
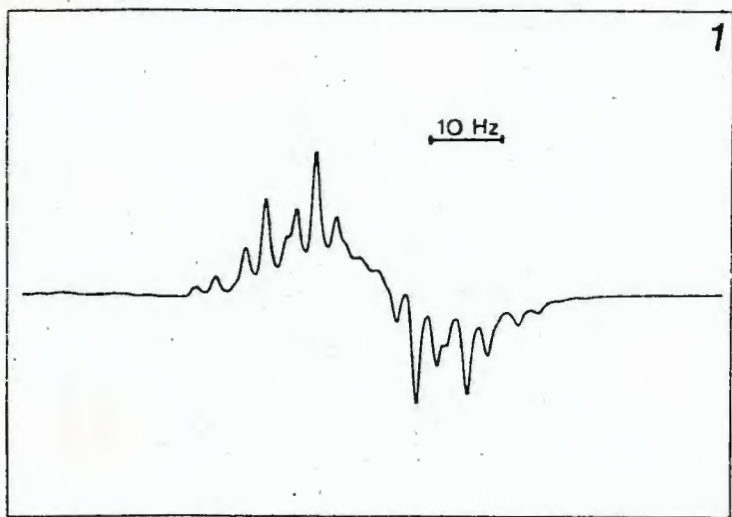
Controlled directly by the pulse programmer, the **ADCKit** offers an elegant solution to fast acquisition of NMR signals.

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Department of Chemistry
Telephone: 515-294-6342

November 8, 1984

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

Dear Dr. Shapiro:

"Computerized Instrument Access System"

The increased use of the departmental NMRs¹, and subsequent increased book keeping has necessitated the implementation of a computerized instrument access system (CIA) to keep track of users time on the instruments. For the past six months, we have been developing a system for this purpose. The overall concept of the system is to have each user log on to an instrument, using a "bar code" card, when they attempt to use it. When they log on, the users identification number, the instrument number, and the time will be stored. The instrument will then be enabled for use. When the user is finished with the instrument, the user will log off. Again the users identification number, the instrument number and time will be stored, and the instrument will be disabled. The users elapsed time on the instrument will then be calculated and stored.

The block diagram for the system is shown in Figure 1. Two computer systems are used. One computer system contains the monitoring program to control the system. This computer has a real time clock to time the use on the instruments, and is used to store the data onto floppy disk. The second computer is a small Motorola 6800 processor. The card readers and instruments are controlled by this computer. It serves to transmit data on the users and instruments to the controlling computer system, and to receive data from the controlling computer as to which instrument to enable or disable.

Function of the system is as follows: Each user is issued an identification card. In each room there is a card reader. When use of an instrument is needed, the user must read in their identification number and the number of the instrument they wish to use. The 6800 processor reads in the information and sends it to the controlling computer. This computer then searches a file of authorized users for that instrument. If the users identification number is found, then the users identification number, the instrument number and the time are written into a disk file. Then the code for the instrument requested is sent back to the 6800 processor. The 6800 processor subsequently sends a signal to the instrument to enable it.

Professor B. L. Shapiro
November 8, 1984
Page 2

The instruments are not actually turned off when disabled, but a necessary part of the instrument, like the data system, will be disabled. The controlling computer system used is the Kaypro 4+88, but almost any system could be adapted.

The use of this system allows much information about how and when an instrument is used to be tabulated easily, as well as monitoring who is using it and for how long. Thus it becomes an excellent management tool and saves many hours of time each month in billing and hand tabulations.

If information is requested on this system, we can be contacted at the above address, or at (515)294-5958.

Sincerely,

James A. Crowder
James A. Crowder

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Tom Lyttle
Thomas Lyttle

D. H. Huang
D. H. Huang

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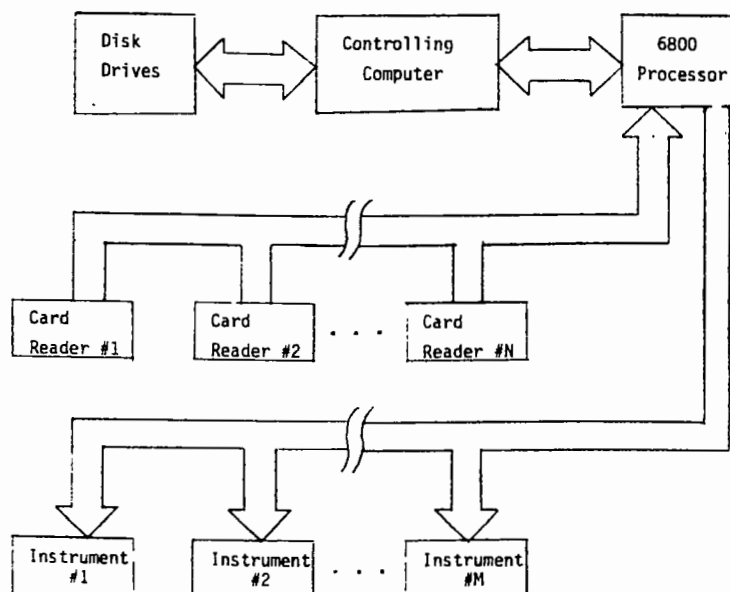


Figure 1
Block Diagram
Computerized Instrument Access System



The Ohio State University

Campus Chemical
Instrument Center116 Johnston Laboratory
176 West 19th Avenue
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Phone 614 422-3446

1 December, 1984

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

Postdoctoral Positions: NMR Imaging; 5S RNA

Dear Barry,

Several postdoctoral positions will be available in my laboratory, with start dates ranging from 1 January to 1 July, 1985, at a salary of up to \$18,000 plus benefits. The Ohio State University is an affirmative action, equal opportunity employer. The successful candidate(s) will be expected to participate in one of the following projects:

- (A) NMR Imaging. We will pursue three distinct approaches. First, our DISPA technique¹ has recently been used to distinguish normal from tumor cells from mouse spleen,² based on the proton NMR signal from water. We will examine the generality of that observation. Second, we have synthesized and demonstrated the tumor-localizing properties of water-soluble metallo-porphyrins. We will compare radio-labeled and paramagnetically-labelled porphyrins as contrast agents for γ -ray or NMR imaging. Third, we will develop stochastic excitation/detection³ for use in NMR images based on chemical shift differences.
- (B) 5S RNA. We are using proton 500 MHz intramolecular NOE's to determine the secondary structure (i.e., base-pair sequence) for small ribosomal RNA's of various primary sequences, with the goal of establishing a universal base-pairing scheme for 5S RNA's from all biological species. For the above experiments, we have access to modern FT-NMR instruments (80, 200, 300, 500 MHz), and a recently installed GE 1.5 tesla NMR imaging system.

1. Marshall, A. G. (1982) in Fourier, Hadamard, and Hilbert Transforms in Chemistry, ed. A. G. Marshall (Plenum, N.Y.), pp. 99-123.
2. Lin, D. P. et al. (1984) Soc. Magn. Reson. in Medicine 3, 474-475.
2. Blumich, B. (1984) J. Magn. Reson. 60, 37-45.

Interested candidates should submit a vita and graduate transcript, and have three letters of recommendation sent to:

Professor Alan G. Marshall
Department of Chemistry
The Ohio State University
140 W. 18th Avenue
Columbus, OH 43210

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Dear Barry:

Our system includes six probes for ^{13}C , ^1H and ^{31}P ; variable temperature controller. HP3320A frequency synthesizer and the 80 MHz magnet and spectrometer which are in good condition. The data system uses a 16K 620L computer with Sykes dual floppy drive.

Interested buyers should contact Claude Lucchesi at 312-492-3479 or 312-491-3479 as soon as possible.

Sincerely,

Claude A. Lucchesi, Director
Analytical Services Laboratory
312/492-3479 or
312/491/3479



SYRACUSE UNIVERSITY

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GEORGE C. LEVY, DIRECTOR (315) 423-1021

RESOURCE COMPUTER: (315) 422-3443, (315) 422-3447

POSITIONS AVAILABLE IN THE SYRACUSE UNIVERSITY NMR AND DATA PROCESSING LABORATORY.

Two continuing staff positions are currently open in our laboratory:

(1) Instrumentation Designer and (2) NMR Operator. Both positions have excellent salary and benefits plans, including TIAA/REF annuity; the first position can carry administrative functions and requisite salary.

Current instrumentation in the laboratory includes 150, 250 and 360 MHz NMR spectrometers and several 32 bit superminicomputers. In 1985, a 500 MHz NMR system and a DEC VAX 8600 will begin operations. In 1985, we will also initiate operations in a new joint Syracuse University/S.U.N.Y. Upstate Medical Center Biomedical Research Magnetic Resonance Laboratory, which will initially use a General Electric 2.0 Tesla 32 cm bore Chemical Shift Imager.



DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

November 15, 1984

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

National Institutes of Health
National Institute of
Environmental Health Sciences
P.O. Box 12233
Research Triangle Park, N.C. 27709

Dear Professor Shapiro:

I would like to announce the availability of several positions in the NMR workgroup in the Laboratory of Molecular Biophysics at the National Institute of Environmental Health Sciences. The group carries out a broad range of biologically oriented NMR studies, but has as its principal focus in vivo NMR investigations of metabolic regulation by environmental chemicals. NMR instrumentation includes a Nicolet QE-300 for routine analysis, a Nicolet NT-360 wide bore, multi-nuclear spectrometer, and two 33 cm bore, 4.7 tesla magnet based spectrometers scheduled for delivery in 1985. One of these will be equipped for both imaging and spectroscopic studies. Openings in the group include a permanent, civil service position for a Ph.D. level scientist with a background in NMR spectroscopy and a civil service position for a B.S. or M.S. level scientist to help operate and maintain the instruments. In addition, a visiting fellow position is currently available for a non U.S. citizen within 3 years of the Ph.D. degree. Interested candidates are invited to send a curriculum vitae and three letters of recommendation to Dr. Robert London, MD 17-05, LMB, National Institutes of Environmental Health Sciences, PO Box 12233, Research Triangle Park, N.C. 27709.

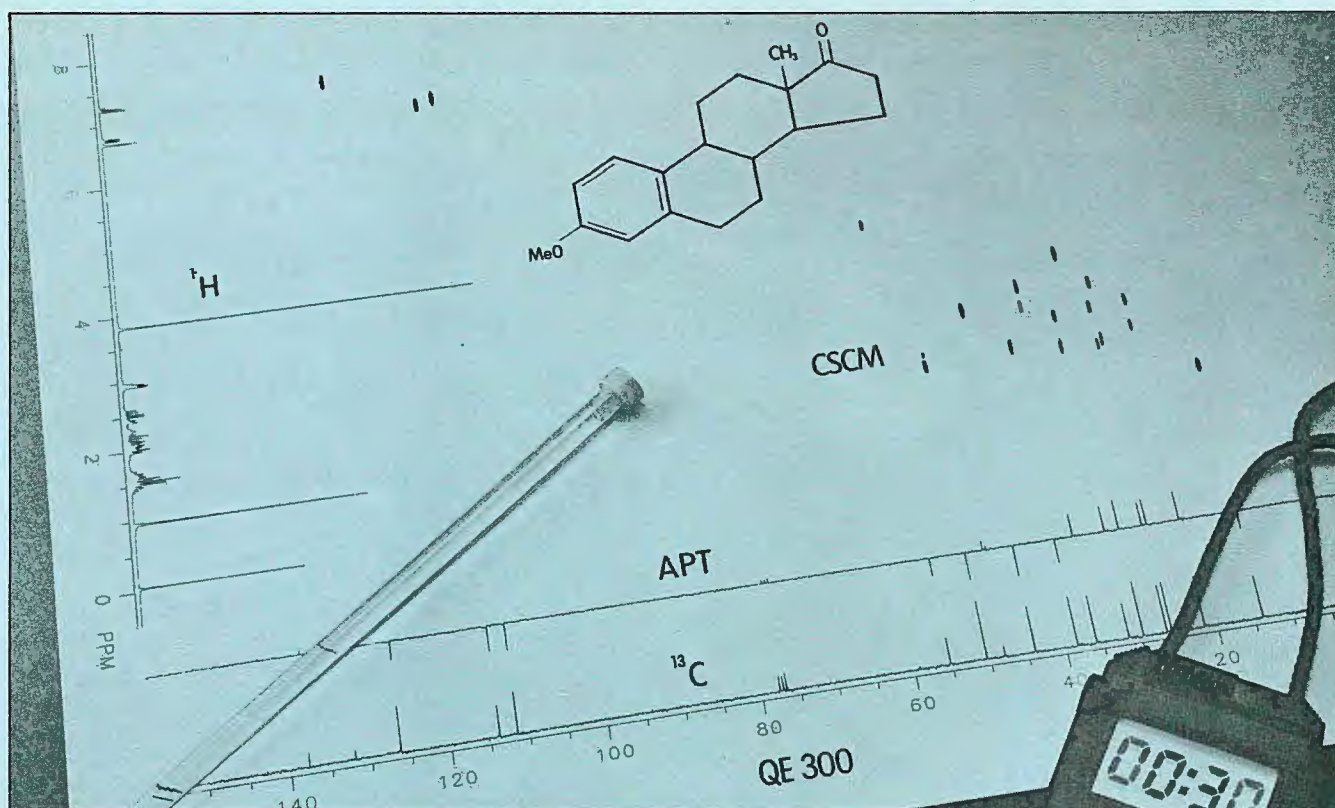
Sincerely

Robert London

Robert London, Ph.D.
Research Physicist

NUCLEAR MAGNETIC RESONANCE SPECTROSCOPIST. University of Lausanne,
Switzerland

A position is available for full time research in high pressure multi-nuclear magnetic resonance applied to chemical kinetics : solvent exchange on dia- and paramagnetic metal ions, bio- and inorganic reaction mechanisms, etc. Candidates should have a strong background in NMR theory and experience in multinuclear NMR. Ph.D. in chemistry or physics is required. The NMR laboratory consist in multinuclear WP-60, CXP-200 and AM-360 wide-bore Bruker spectrometers. The salary is commensurate with qualifications : 46'000.-- Swiss Francs (about \$ 19'000.--) and above. Send curriculum vitae and 2-3 letters of recommendations to : **Prof. A. Merbach, Institute of Inorganic and Analytical Chemistry, University of Lausanne, Place du Château 3, CH-1005 Lausanne, Switzerland.**



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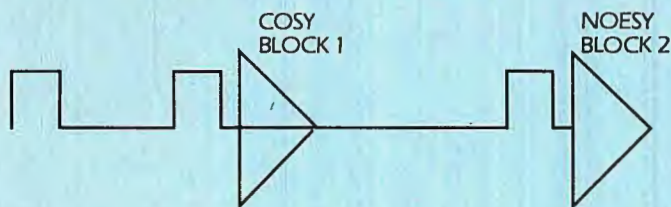
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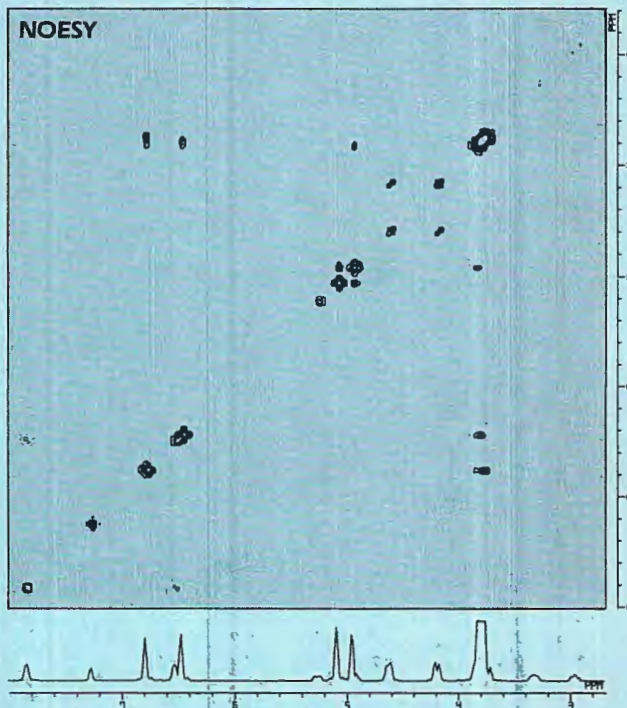
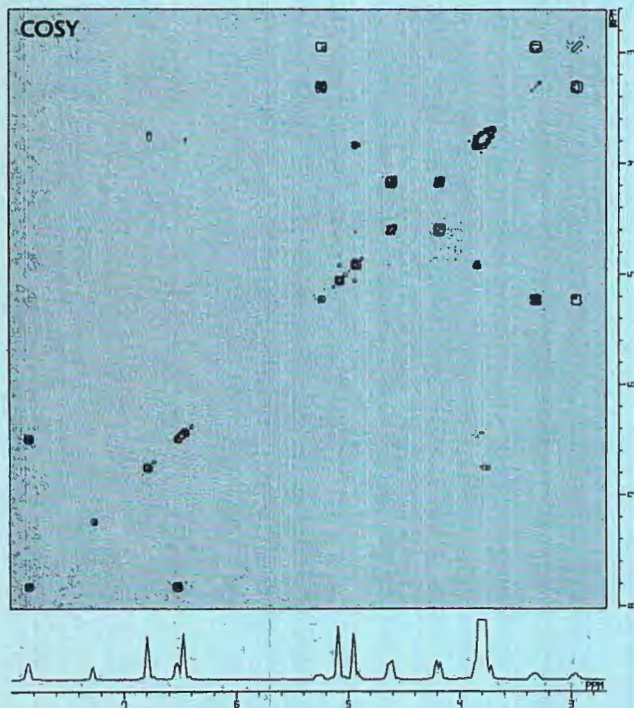
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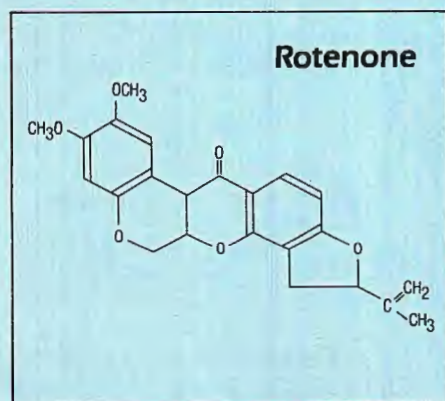


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*COCONOSY (Haasnoot, et. al., J. Magn. Reson., 56,343 [1984])



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