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Newsletter

JUN 26 1981

No. 273

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DEADLINE DATES: No. 274 6 July 1981
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All Newsletter Correspondence, Etc., Should be Addressed To:

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

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Subject: pH-STATTING AND RAPID MIXING IN SPINNING NMR TUBES DURING
SPONTANEOUS PRECIPITATION

Dear Professor Shapiro:

We are using high-resolution ^{31}P NMR to follow the spontaneous precipitation of calcium phosphates from supersaturated solutions, a process accompanied by the release of protons. Since the phosphorus chemical shift is pH-dependent, we wish to maintain a constant solution pH. We have, therefore, developed a simple apparatus which permits the acquisition of spectra from spinning 20mm sample tubes while: (1) constantly monitoring the pH; (2) adding a solution of base to maintain constant pH (pH-statting); (3) efficiently mixing the added base with the sample by means of specially-designed mixing vanes. A diagram of the apparatus, which is lowered into a spinning (uncapped) NMR tube in our wide-bore CXP-300 magnet, is shown. The stationary mixing vanes promote turbulent mixing and also prevent vortexing when the tube is spun. In the absence of vanes, we found mixing in spinning 20mm tubes to be extremely slow (>>5 minutes).

There are other areas where features of this apparatus could prove very useful:

1. Automatic acquisition of data for chemical shift "titrations" (chemical shifts vs. pH);
2. Automatic acquisition of data as a function of sample dilution, or as a function of concentration of an additional reagent (e.g. metal ion complexation studies);
3. Kinetic studies of relatively fast reactions ($t_{1/2} \geq 10\text{s}$);
4. Studies of reactions while maintaining constant ion concentrations using ion-selective electrodes;
5. Decoupling at high-fields and accurate variable-temperature studies (temperature gradients can be minimized by using the mixing vanes).

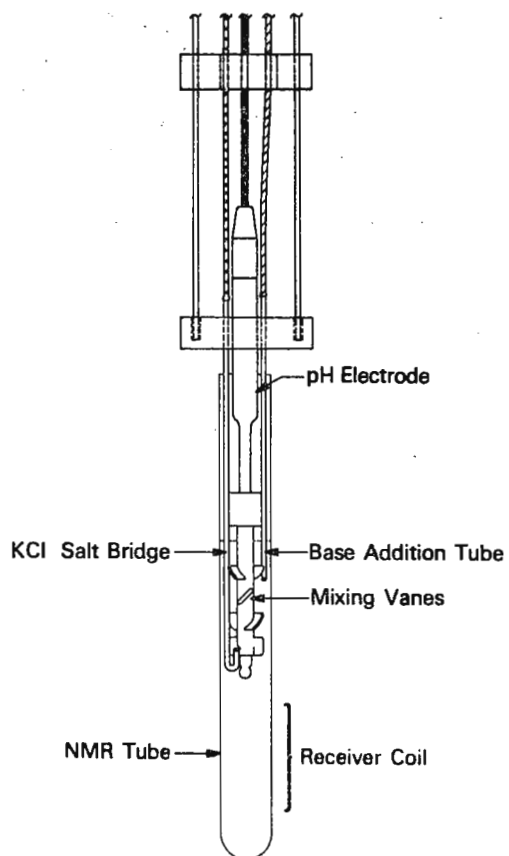
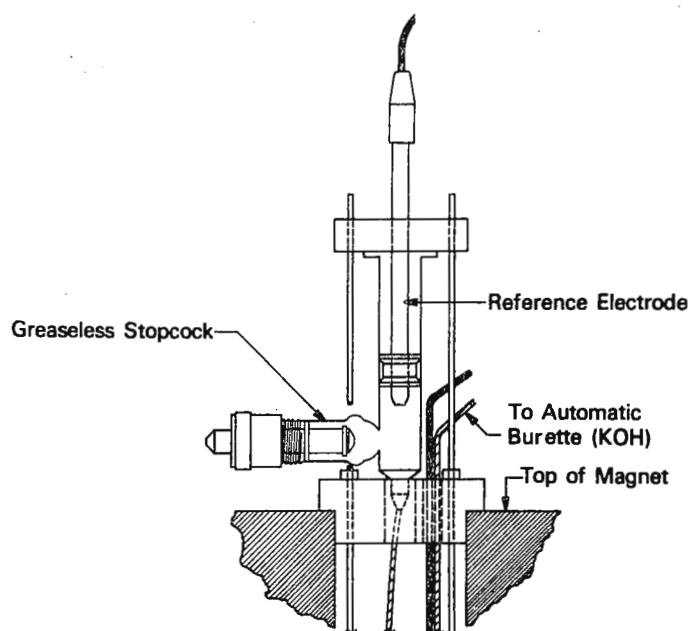
Very truly yours,

J.P. Yesinowski
J.P. Yesinowski

R.J. Sunberg
R.J. Sunberg

James Benedict
J.J. Benedict

st



International Business Machines Corporation

5600 Cottle Road
San Jose, California 95193

RESEARCH LABORATORY K34/281

May 7, 1981

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

Dear Barry:

Spurious ringing is common in pulsed NMR, and we have had our share recently. This has happened because we have built probe bodies from aluminum which was fine until we used the same probe at a lower frequency, for experiments where minimal dead time was required, or have used a larger sample coil. In all three instances, we have solved the ringing problem by using brass instead of aluminum; in the last case, changing only the probe sides was sufficient.

This remedy is one of several in Steve Roeder's useful note on these effects in J. Mag. Res. (33, 199 (1979)).

Best regards,

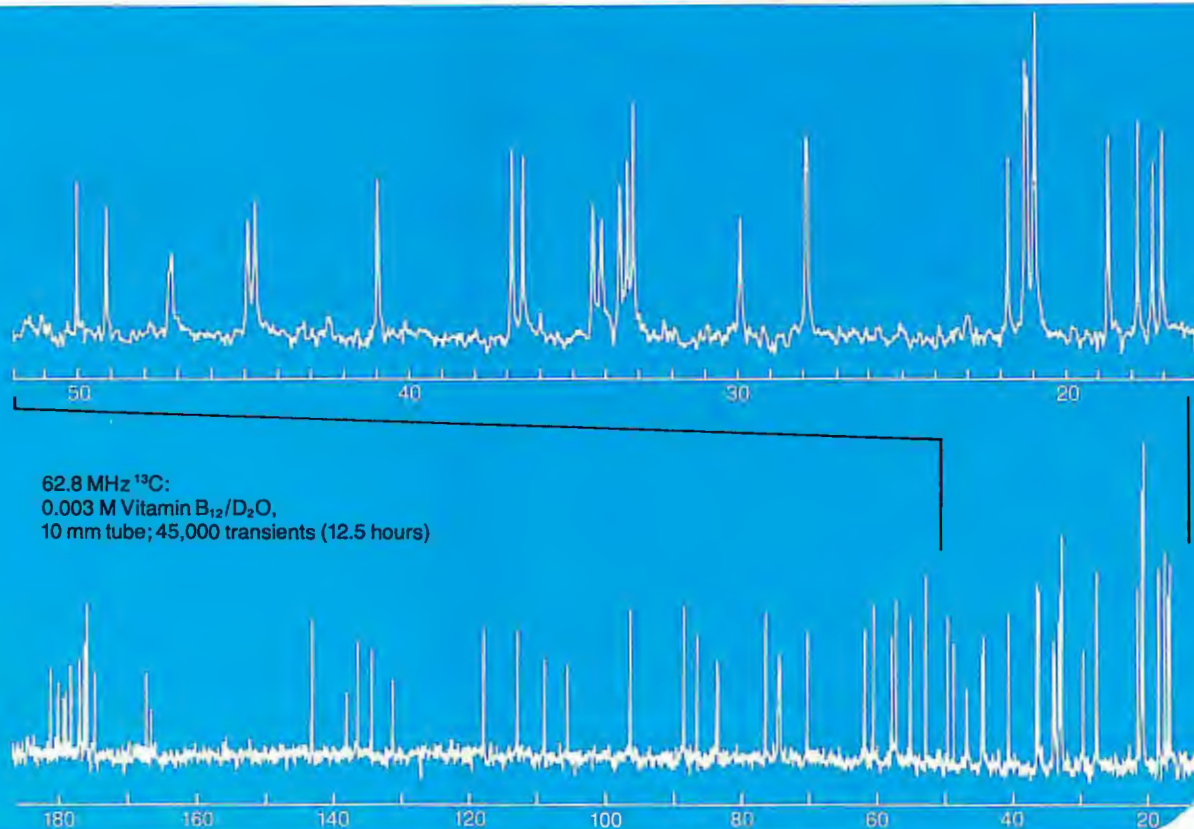


C. S. Yannoni

/cas

Short Title: Spurious Ringing

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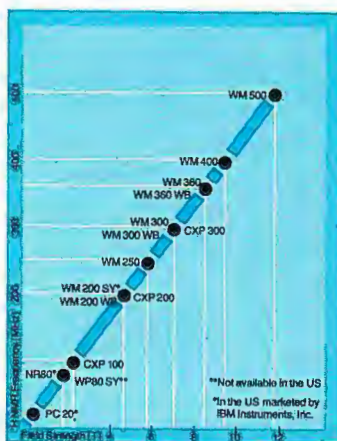
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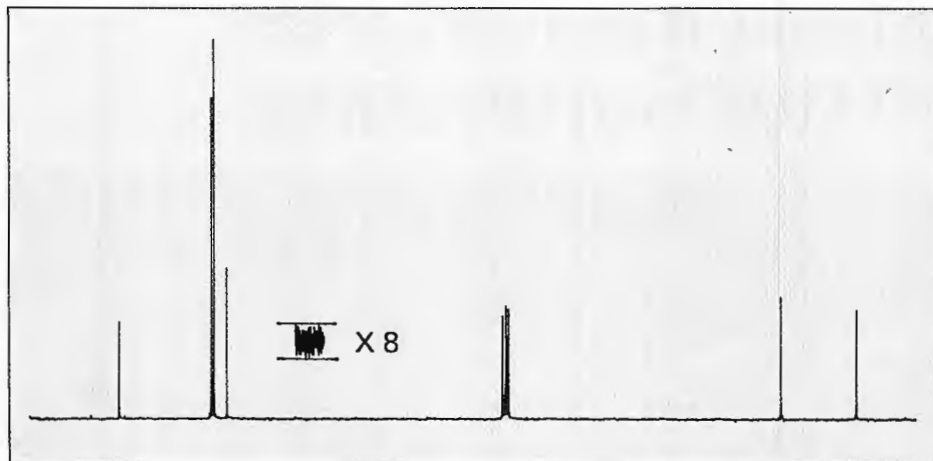
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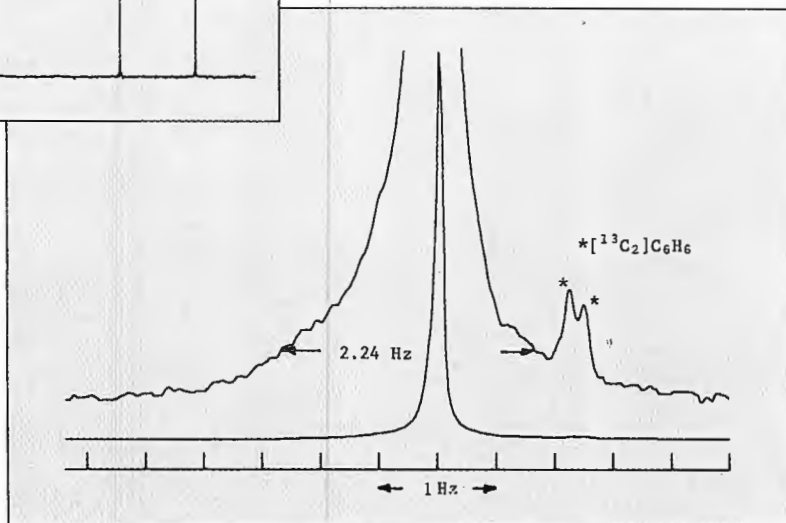


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8th May, 1981.

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

Dear Barry,

Graphics ^{13}C n.m.r. Data-Terminal

We have recently made a start on a project which I have had in mind for some time, namely to provide ourselves with either our own ^{13}C -data retrieval/data prediction system or (as seems more likely) the local front-end which would afford us access to a network system. We have managed to purchase second-hand (for £3000) a vector graphics system (GEC 928) to form the basis of this.

We have got to the stage in programming where we are able to draw a structure on the display screen and code the structure into a form where it can be transferred to a larger machine for further processing. We have also managed to do some sub-structure identification in the local machine but the store available (32K words) is insufficient for the depth of searching necessary.

The programs have been arranged to be portable requiring only that the 928-Fortran-Graphics subroutines can be emulated in any machine to which the programs are transferred.

Yours sincerely,

Peter Bladen

Binod Acharya

Peter Bladen

Acharya



The Guelph-Waterloo Centre for Graduate Work in Chemistry

Guelph Campus, Department of Chemistry, University of Guelph, Guelph, Ontario, N1G 2W1 519/824-4120

April 30, 1981

Dr. Barry Shapiro
Department of Chemistry
Texas A and M University
College Station, Texas
U.S.A.

Southwestern Ontario NMR Center

Dear Barry:

The Southwestern Ontario NMR Center began operation on December 4, 1980. Since that time we have been running virtually full time (24 hours a day) on our Bruker WH-400. This instrument is fully broadbanded between 8 and 177 MHz. Changeover time is ca 15 min between nuclei. In the short few months that we have been in operation we have already observed ^1H , ^2H , ^{10}B , ^{11}B , ^{13}C , ^{14}N , ^{15}N , ^{17}O , ^{29}Si , ^{31}P , ^{39}K , ^{41}K , ^{47}Tl , ^{49}Tl , ^{53}Cr , ^{57}Ni , ^{67}Zn , ^{89}Y , ^{103}Rh , ^{119}Sn , ^{129}Xe , ^{195}Pt , ^{199}Hg and ^{207}Pb .

We have also run some interesting non high resolution spectra. One of these is illustrated by the accompanying ^{31}P powder pattern. We are currently experimenting with a "solid echo" microprogram to improve this spectrum. We hope to be able to report on this in the near future.

Sincerely,

Bob

R. E. Lenkinski,
Manager

D. Siminovitch

D. Siminovitch

B. McDonald

B. McDonald

^{31}P

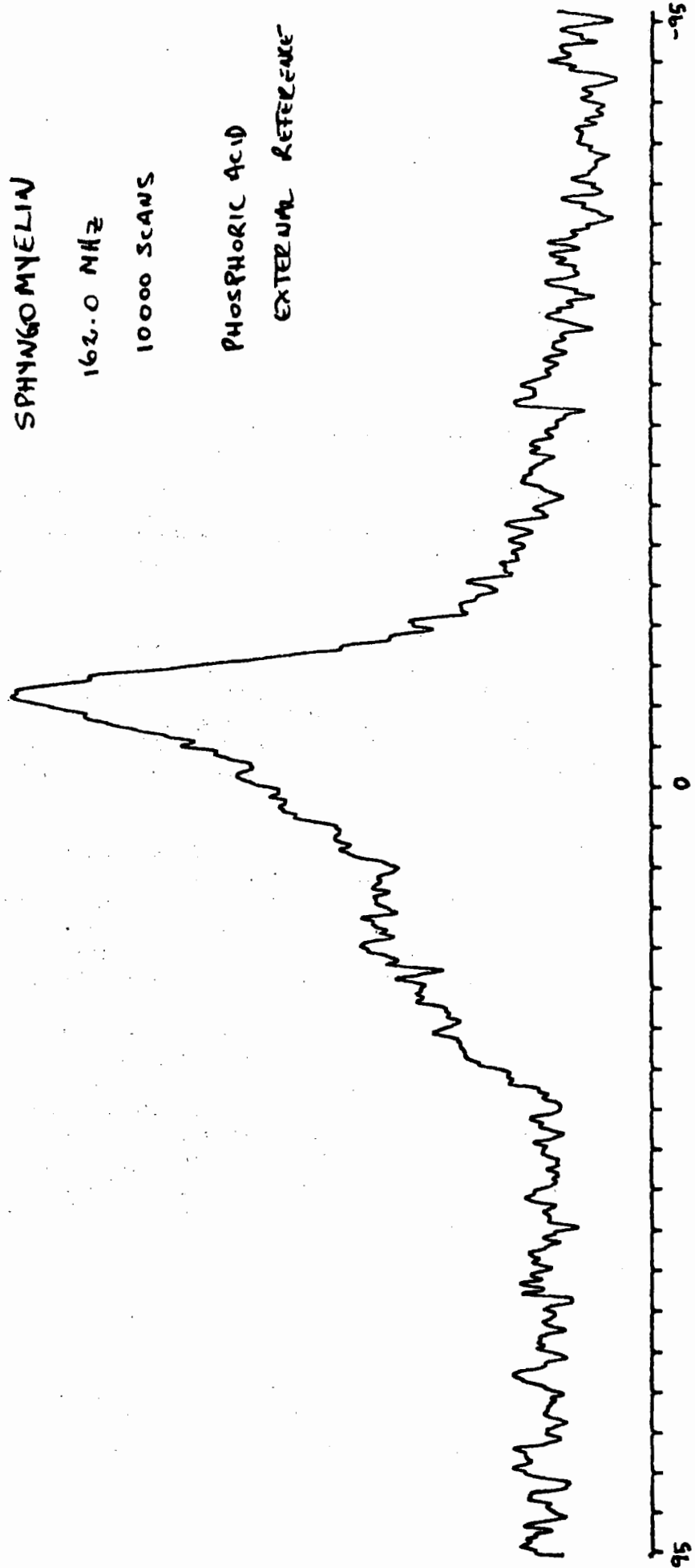
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DEPARTMENT OF CHEMISTRY

May 14, 1981

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

Dear Barry:

Spiropentane Spectrum and Structure

Spirohydrocarbons have been the subjects of numerous detailed theoretical studies of structure, energy, strain, and pi-electron delocalization. However, experimental structural information has been limited to electron diffraction and liquid crystal NMR studies of spiro[2,4]-4,6-diene.

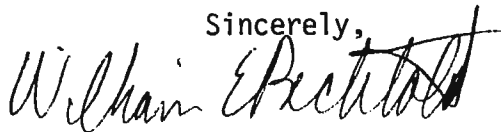
Unfortunately, initial structural information for spiro[2,4]-4,6-diene appears to have been somewhat contradictory. The electron diffraction study found the four equivalent H-C-H bond angles to be 118.4° , compared to 115.22° determined by liquid crystal NMR^{1,2}. Later ab initio and maximum overlap calculations predicted the angle to be 113.15° and 114.55° ^{3,4}. As the angle is sensitive to the hybridization of the carbon, the wide variation of its observed and calculated values appears unusual.

Neither the liquid crystal nor the electron diffraction structures were corrected for the effects of vibrational motion, which can be substantial. Subsequent to the two investigations, the normal coordinate analysis necessary to perform corrections for vibration has appeared⁵. We have used them to apply corrections for harmonic vibrational motion to the two previous experimental studies, and a second liquid crystal study, in an effort to account for the discrepancies.

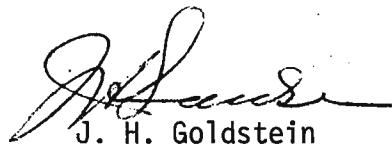
Results are presented in Table 1, along with ab initio and maximum overlap calculations. The electron diffraction carbon coordinates and C-H bond length have been used to scale the NMR distance ratios. The results indicate general agreement between liquid phase and theoretical calculations, although the gas phase structure still appears to be different. While slight distortions are to be expected in different media, the magnitudes in the present case seem unusually large, especially in light of the previous agreement for cyclopropane studied by the two techniques. The reproducibility of the values in separate liquid crystal phases confirms the condensed phase bond angles.

Please accept this as our "subscription" fee.

Sincerely,



Wm. B. Bechtold



J. H. Goldstein
Professor of Chemistry

Table 1. Comparison of Structural Information for Spiropentane
by Different Methods

Proton Distances				
	Phase IV ^a	Previous NMR ^{a,b}	ED ^c	
r ₁₂	1.817 ± 0.003	1.812 ± 0.003	1.860 ± 0.007	
r ₁₃	2.518 ± 0.004	2.523 ± 0.004	2.467 ± 0.033	
r ₁₄	3.105 ± 0.006	3.106 ± 0.006	3.090 ± 0.026	
r ₁₅	3.131 ± 0.006	3.138 ± 0.006	3.139 ± 0.042	
r ₁₆	3.792 ± 0.007	3.797 ± 0.008	3.801 ± 0.037	
r ₁₈	4.353 ± 0.008	4.358 ± 0.008	4.364 ± 0.034	
H-C-H				
Phase IV ^a	Previous NMR ^{a,b}	ED ^c	ab initio ^d	Max. Overlap ^e
115.0° ± 0.7°	114.5° ± 0.7°	119.4 ± 1.1°	113.9°	114.6°

a. Calculated using vibrationally corrected electron diffraction C-H bond length (1.077 Å) and carbon coordinates

b. Direct couplings from Ref. 2

c. Uncorrected distances from Ref. 1

d. Ref. 3

e. Ref. 4

References

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May 20, 1981

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

Postdoctoral Position Available

Dear Barry:

A postdoctoral position is immediately available in our project concerning magnetic resonance of bioluminescent proteins from jellyfish. We are looking for someone either with NMR experience and interest in biological NMR or with biochemical or biophysical expertise and interest in applying NMR to biological problems. The initial appointment is for one year at an annual salary in the range of \$12,000 to \$13,000. The term may be extended for one or two additional years contingent upon reasonable progress and mutual satisfaction.

The emphasis of the work will be on experimental NMR although some EPR may also be involved. In the beginning the NMR experiments will be done at the Purdue University regional NMR facility in West Lafayette, IN. Within a year, however, we will have a 250-300 MHz high resolution NMR spectrometer in our laboratory in Indianapolis available for use on this and other projects. EPR experiments will be done in our laboratory. Interested individuals should send a curriculum vitae and arrange for two letters of recommendation to be sent to me.

Sincerely yours,

B. D. Nageswara Rao
Professor of Physics

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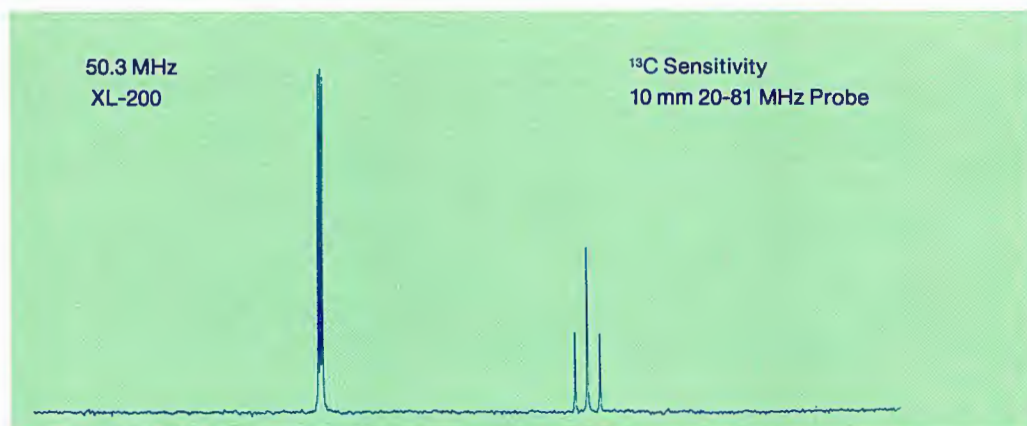
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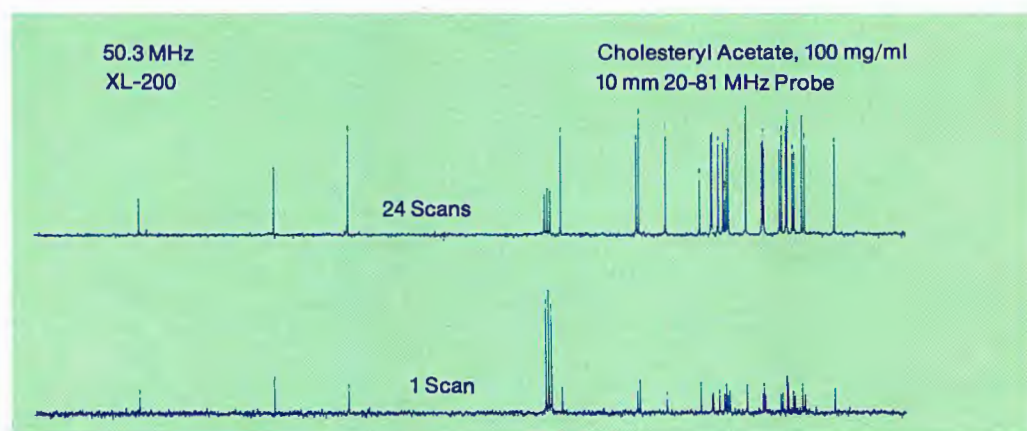
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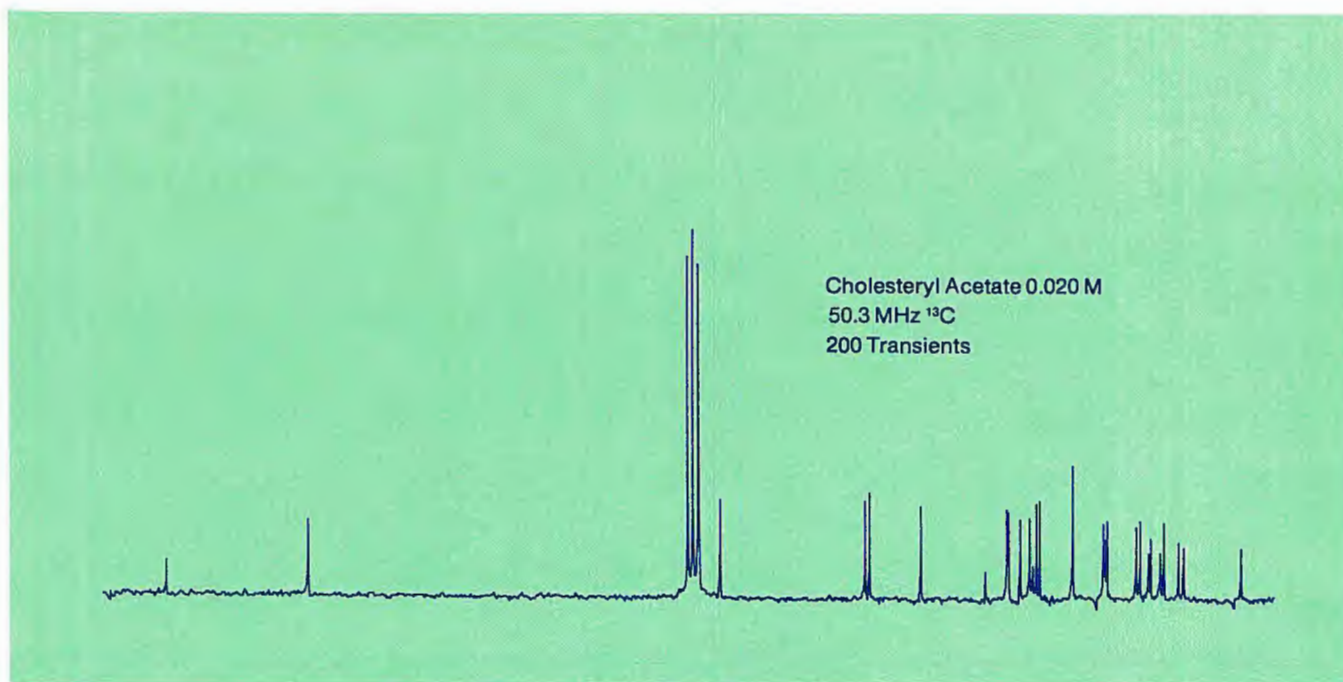
^{13}C Sensitivity Test: Single transient following 90° pulse on 60% C_6D_6 /40% dioxane using the 10 mm 20-81 MHz broadband probe.



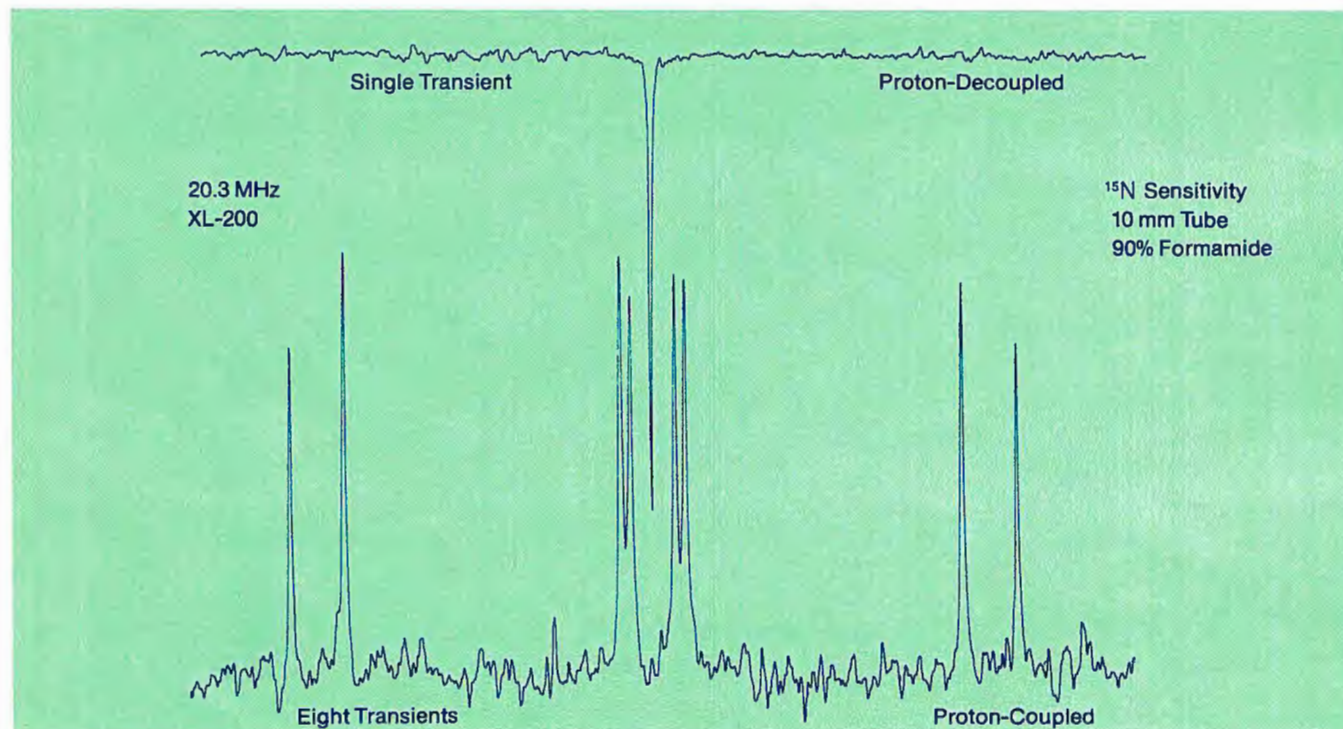
^{13}C Sensitivity Test: Cholesteryl acetate, 100 mg/ml, 10 mm broadband probe. Transients accumulated using 90° pulses every 2.28 seconds with 0.5 Hz line-broadening.

**Additional spectra appear
on the following page**





¹³C Sensitivity Test: 0.02 molar cholesteryl acetate in a 16 mm tube, 200 transients.



¹⁵N Sensitivity Test: 90% Formamide in dms_o-d₆, 10 mm 20-81 MHz broadband probe. Upper trace: single-transient (with NOE) proton-decoupled. Lower trace: eight transients, coupled (with NOE) 8-second acquisition time, 20-second delay time.

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Universidad de Buenos Aires
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Buenos Aires, may 19, 1981.

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

GOOD RESULTS WITH AN INDOR MODEL.
LOOKING FOR AN OLD NICOLET ON SALE.

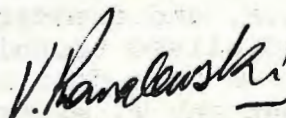
Dear Dr. Shapiro:

After several years of circumstantial silence, I am now very glad to be able to renew my subscription to TAMUN.

After our succesfull attempt to describe INDOR spectra as a simultaneous superposition of, splitting due to tickling and change of intensity due to redistribution of populations, in an AX and an AB cases, (J.Magn.Reson. 41, 61-73, (1980), we applied the method to the degenerate AX_n case (in particular the AX_3 case) with equal success. A paper on the subject is to appear shortly in the same magazine.

Now: Since we have here an old Nicolet NMR-812 Data System and we need to keep it operating as long as possible, and also wish to expand it if possible, we are looking for some old similar system on sale, say, an NMR-820 or the 1080, with or without disks. Perhaps some reader may find this offer interesting. This would help him, for instance, to buy a more modern system, like the 1280. For eventually interested people, my adress is given below.

Yours, sincerely



V.J.Kowalewski.
 Professor.

Dr. V.J.Kowalewski.
 Facultad de Ciencias Exactas.
 (1428) Buenos Aires.
 Argentina.



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Department of Chemistry

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1st May 1981

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

Dear Professor Shapiro,

Operation and Use of Bruker WM-250;
breakage of probe glassware

It is approximately 18 months since we acquired a Bruker WM-250 Spectrometer and the way in which the instrument has been used may be of interest to some of your readers.

Time is allocated to Inorganic and Organic sections in the ratio of 1 (week) to 3 (weeks). Inorganic applications have used the following nuclei:

^1H , B, ^{13}C , ^{14}N , ^{17}O , ^{19}F , ^{27}Al , ^{29}Si , ^{31}P , ^{35}Cl ,
 ^{51}V and ^{207}Pb .

Organic applications have been largely confined to ^1H and ^{13}C , with some work on ^{17}O and ^2D . As expected, the availability of a high field magnet has had a considerably greater impact on ^1H than on ^{13}C work. Apart from some ^1H - ^1H n.O.e. experiments, and a moderate amount of variable temperature work, specialised techniques have not been employed. For example, we have done no 2-dimensional NMR, and have not carried out any T₁ experiments! We have been able to solve our problems by the routine methods of ^1H observation (with ^1H decoupling, where needed) and ^{13}C observation with ^1H noise decoupling, with ^1H O.R. decoupling and with specific ^1H decoupling. Shift reagents have found some applications. When there is a queue of problems from up to 50 students and staff, there is no justification for using time-consuming and superfluous (though interesting) techniques. Whilst admitting that this situation is not always to our liking, we have to recognise that the instrument was purchased to

solve problems in Chemistry - not to collect NMR parameters as such! Indeed, most chemical problems can be solved without the need for the complete interpretation of spectra.

The most serious instrumental problem has been the breakage of glassware sections of several probes - due, we believe, to eccentric wobbling of the sample *tube* during insertion and/or ejection.

Yours sincerely,

A handwritten signature in dark ink, appearing to read "H. Booth", with a horizontal line underneath the name.

Dr. H. Booth.

Dr. H. Booth
R. Fleming
Dr. M.A. Healy.

¹ for example, the interaction between lead and adenosine phosphates had been studied (P.G. Harrison, M.A. Healy and M. Aslam).

UNIVERSITÉ CLAUDE BERNARD LYON I
LABORATOIRE DE SPECTROSCOPIE HERTZIENNE

43, boulevard du 11 Novembre 1918
69622 VILLEURBANNE Cedex

Tél. (7) 889.81.24 / (secrétariat poste 3210 ou 3370)

Villeurbanne, le 7 Mai 1981

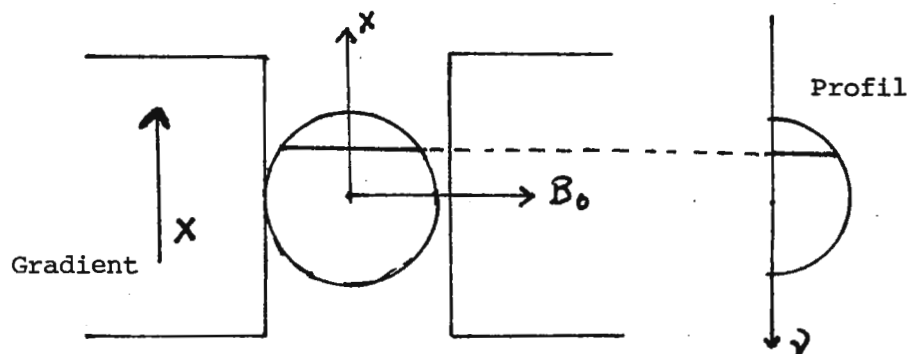
CINETIQUE DE LA PENETRATION DE L'EAU
DANS UN ECHANTILLON POREUX

Cher Dr SHAPIRO,

En marge d'expériences d'imagerie que nous développons en vue de l'étude de milieux biologiques, il nous est apparu intéressant d'utiliser directement un XL 100 pour étudier la pénétration de l'eau dans des échantillons de plâtre nécessairement de petite dimension (10 mm d'épaisseur).

La largeur de la raie de l'eau absorbée est d'environ 300 Hz, aussi pour une résolution du mm faut-il des gradients supérieurs à 3000 Hz/cm.

Nous avons pu aller jusqu'à des gradients de 3500 Hz/cm selon X en faisant débiter dans les bobines de correction x des courants qui restent tolérables pour ces dernières (cf. TAMU NMR n°259). La linéarité des gradients ainsi réalisés sur le volume étudié a été testée sur un échantillon cylindrique de 10 mm. Nous avons bien vérifié que le profil est proportionnel à $\sqrt{R^2 - \rho^2}$, ρ variant de $-R$ à R .



Nous avons pu étudier la cinétique de la pénétration de l'eau dans le plâtre. En définitive il est intéressant (et presque étonnant) de constater qu'une méthode aussi rudimentaire conduit à des applications intéressantes.

Sentiments les plus cordiaux.

A. BRIGUET

J. DELMAU

J.C. DUPLAN

Delmau

Duplan

gradient
X

B_0

Echantillon
de platre

100 Hz

t > 1 heure

face humidifiée

t ≈ 10 mm

Prof. Dr. P. Diehl

CH-4058 Basel (Schweiz) May 20, 1981

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

PASCAL Version of program SHAPE

Dear Barry,

The program SHAPE¹⁾ which iteratively derives the geometry and order parameters of oriented molecules from the measured direct couplings by a weighted least-square-fit-method is the most commonly used program of this type. We have therefore thought it worth-while to write a PASCAL version which can be used e.g. on the Bruker Computer Aspect 2000.

The program works with up to 9 nuclei with a possibility of 9 symmetry relations between the coordinates. Any internuclear distance may be kept constant. The program is written as a dialogue with the user so that in principle no manual is needed. Individual input parameters may be changed during the run. Approximate memory requirement is 15 k on a 24-bit computer.

The program will finally be available through the Aspect Users Club but if somebody is very eager to have it earlier he should contact us and will receive a listing.

Yours sincerely

Peter *F. Moia*
Peter Diehl Franco Moia

1) P. Diehl, P.M. Henrichs and W. Niederberger,
Mol. Phys. 20, 139 (1971)

THE SOLID LEADER in NMR

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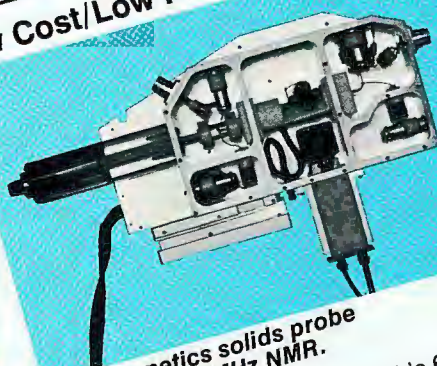
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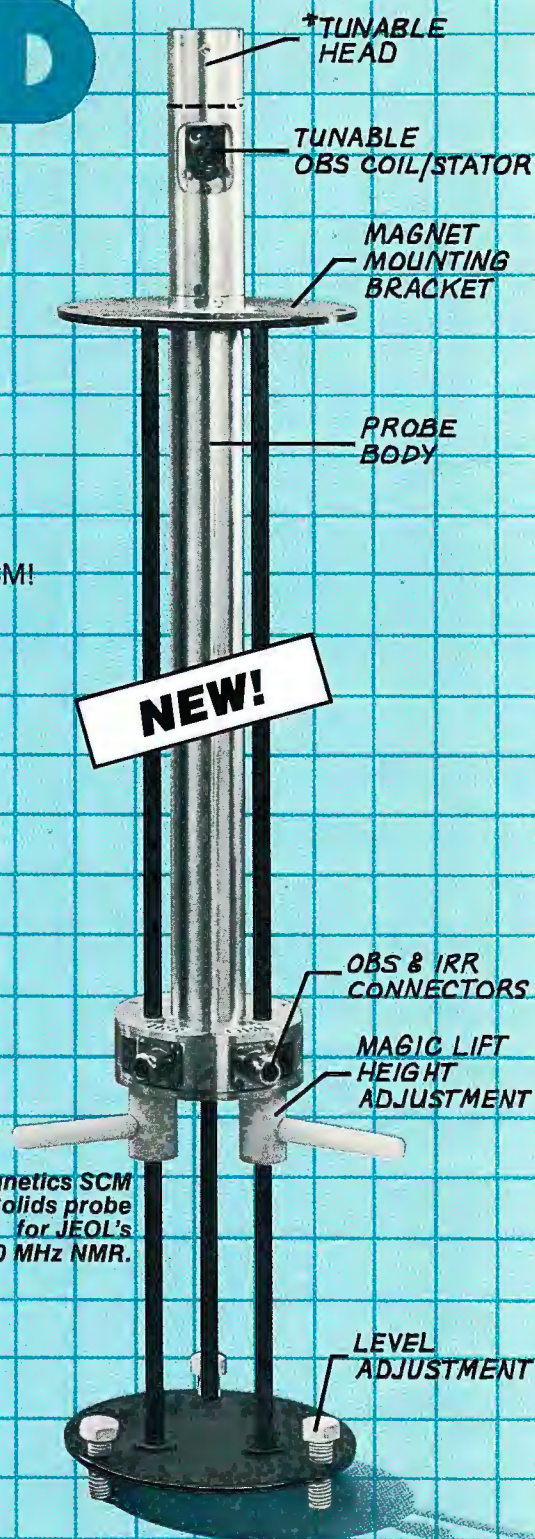
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DEPARTMENT OF CHEMISTRY
SANTA BARBARA, CALIFORNIA 93106

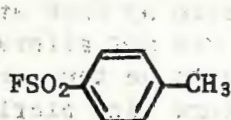
May 20, 1981

Professor B. L. Shapiro
TAMU NMR Newsletter
Texas A & M University
College Station, Texas 77843

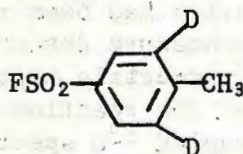
"Deuterium NMR Studies of Tosylchymotrypsin"

Dear Barry:

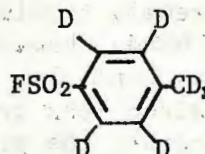
We have been engaged for sometime in studies of the protein which is formed when the enzyme α -chymotrypsin is treated with tosyl fluoride. This reagent forms a sulfonate ester with a serine residue at the active site which is essential for catalytic activity and X-ray crystallography shows that in the solid the aromatic ring of the tosyl-group is partially inserted into a cleft of the enzyme surface. We have explored the motion of the tosyl group in solution by using deuterium



I



II



III

spectroscopy and the enzyme derivatives prepared from II and III. At 76.8 MHz (using the WM-500 at Caltech) the aromatic deuterium signals are 320 ± 50 Hz wide while the methyl deuterons have a linewidth of 150 ± 25 Hz. Predictions made using reasonable quadrupolar coupling constants and the known correlation time (~ 15 nsec) for the protein suggest the aromatic linewidth should be at least three times larger than the observed value if the aromatic ring is immobilized within the protein structure. Given the experimental observation we conclude that the tosyl group is moving rapidly and independently of the protein molecule. Analysis of the data in terms of a two-state (flip-flop) model indicate that aromatic ring is rotating at a rate greater than 5×10^{10} times/sec. Thus, it seems unlikely that the tosyl group spends all of its time wedged in a protein cleft when the protein is in solution.

The resolution and sensitivity of the WM-500 make deuterium spectroscopy almost a pleasure and we will be looking at several other derivatives of chymotrypsin by this method in the future.

Sincerely,

M. E. Ando

J. T. Gerig

E. F. Weigand

M. E. Ando



of The City University of New York

Department of Chemistry • 695 Park Avenue, New York, N.Y. 10021 • (212) 570-5666

May 19, 1981

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

Title: ^{13}C Chemical Shifts of Dehydroquinuclidine

Dear Barry:

Dehydroquinuclidine (**1**) is the simplest example of a non-conjugated enamine. For use as a synthetic intermediate, we required more than trivial quantities of (**1**), whose apparently routine preparation had been reported by Grob et. al. in 1957.¹ In our hands, however, the procedure described did not afford identifiable product, and only low yields of materials expected to be the corresponding salts were obtained upon treatment of the reaction mixture with picric acid or oxalic acid. Indeed, the proton-decoupled ^{13}C spectrum of the reaction mixture obtained from base-induced elimination of 3-tosyloxyquinuclidine had all the appearances of a wheat field after optimum climatic conditions, although some stalks were in regions optimistically expected for (**1**). Gas-liquid chromatography of the mixture displayed at least nine components including three major ones. The first two of these were collected as colorless mobile oils and the third initially condensed as a white solid, but rapidly turned into a colorless oil on exposure to air. This third one was identified (by preparation of its picrate salt) as dehydroquinuclidine. The ^{13}C chemical shifts are given in Scheme 1. They have been assigned by comparison with values (shown in parentheses) calculated by correcting the chemical shifts for quinuclidine (**2**)² with anticipated effects of the 2,3 double bond. The latter was derived from comparison of bicyclo[2.2.2]octane and bicyclo[2.2.2]-2-octene.³ Except for C-2, the agreement is very good. Assignment of C-2 was confirmed from $^1\text{J}_{\text{CH}}$ values, 183 Hz vs 165 Hz for C-3. The higher value for C-2 is consistent with the effect of the adjacent electronegative atom.

The smaller difference between the C-2 and C-3 chemical shifts of **1** (7.0 ppm) compared with other enamines (30-50 ppm)⁴ is consistent with reduced (or the absence of) nitrogen lone pair delocalization through the adjacent orthogonal p_i system. Similarly, the difference between the chemical shifts of C-3 and C-4 in *N,N*,2,6-tetramethylaniline is smaller (3.4 ppm) than the corresponding difference (6.7 ppm) in the primary aniline.⁵ In the former compound, nitrogen lone pair delocalization is reduced owing to steric interactions between the ortho methyls and the dimethylamino group.

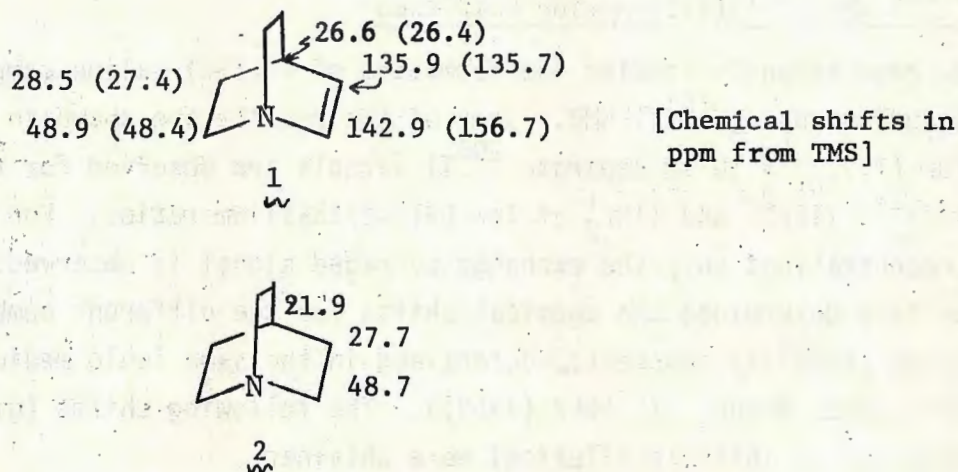
A full report of the isolation and spectral properties of (1) will appear shortly.

Sincerely yours,

Stanley Daniello
Stanley Daniello

Bob
Robert L. Lichter

Scheme 1



1. C.A. Grob, A. Kaiser and E. Renk, Helv. Chim. Acta., 1957, 40, 2170-85
2. G.C. Levy, R.L. Lichter and G.L. Nelson, "Carbon-13 Nuclear Magnetic Resonance Spectroscopy," Second Edition, Wiley-Interscience, New York (1980) p. 70.
3. J.B. Stothers, J.R. Swenson, and C.F. Tan, Can. J. Chem., 1975, 53, 581-8.
4. E.g., R. Stradi, P. Trimarco, and A. Vigevani, J. Chem. Soc., Perkin Trans. I, 1978, 1-4.
5. P.C. Lauterbur, J. Chem. Phys., 1963, 38, 1415.

P.S. We have just obtained the ^{15}N chemical shift. Want to guess?

Stockholm, May 21, 1981

Professor B L Shapiro
Texas A&M University
Department of Chemistry
COLLEGE STATION
Texas 77843

U S A

Dear Prof. Shapiro,

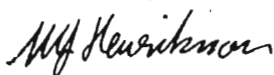
 ^{205}Tl NMR of Tl(III)-Halide Complexes

We have recently studied the formation of Tl(III)-halide complexes in aqueous solutions using ^{205}Tl -NMR. Some of the results are shown in Figs 1 and 2. For $[\text{Tl}]_{\text{tot}} = 50 \text{ mM}$ separate ^{205}Tl signals are observed for the species Tl^{3+} , TlCl^{2+} , TlBr^{2+} and TlBr_2^+ at low halide/thallium ratios. For higher halide concentrations only the exchange averaged signal is observed. In this region we have determined the chemical shifts for the different complexes by using known stability constants, determined in the same ionic medium (Ahrland *et al.* Acta Chem. Scand. 17, 1567 (1963)). The following shifts (given down field from Tl^+ at infinite dilution) were obtained:

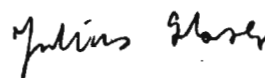
n	$\delta_{\text{TlCl}_n}^{3-n}$ ppm	$\delta_{\text{TlBr}_n}^{3-n}$ ppm
0	2086	2086
1	2198	1538
2	2201	766
3	2412	1184
4	2645	1318

When the total concentration is increased for the low halide/thallium ratios the observed signals from the separate complexes broaden and finally coalesce as shown in Fig. 2. This shows that the rate of exchange increases rapidly with increasing thallium concentration. In the bromide system the big difference between the chemical shifts for the different species gives rise to very broad signals (up to $\sim 50 \text{ kHz}$ at 21 kG) for intermediate exchange rates.

Sincerely yours,



Ulf Henriksson



Julius Glaser

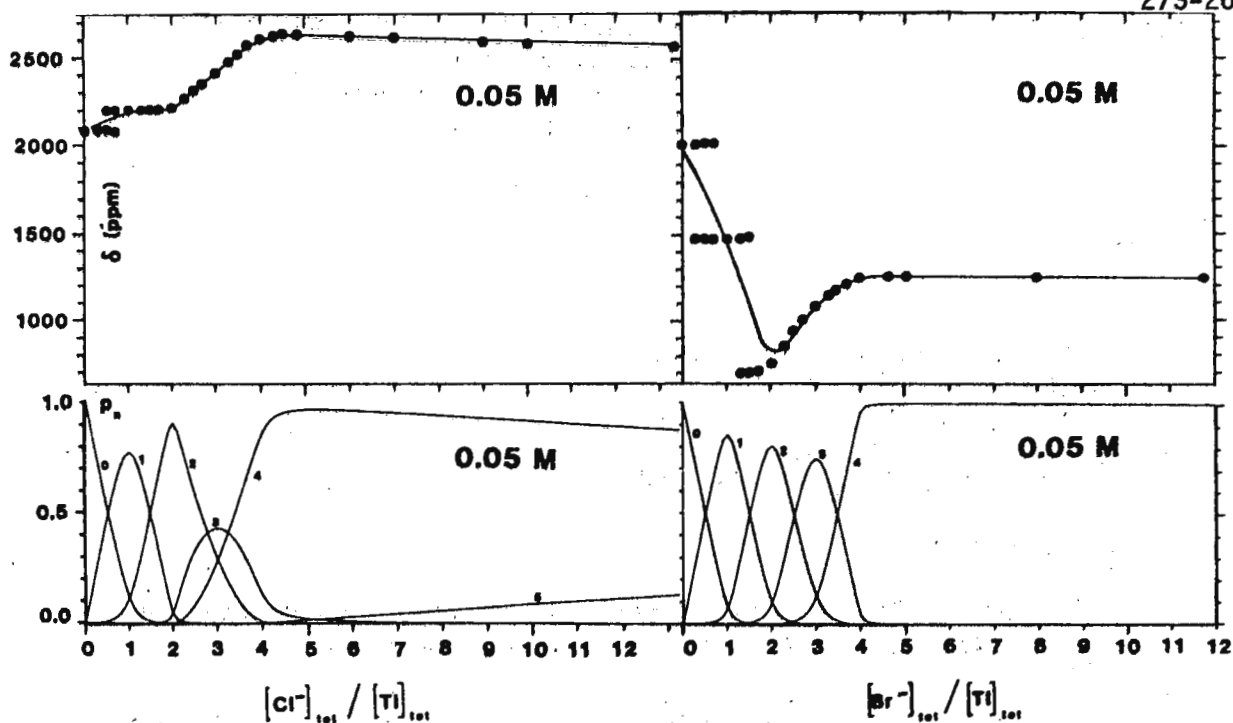


Fig. 1: ^{205}Tl shifts from 50 mM aqueous solutions of Tl(III) as function of the halide/thallium ratio. • indicates experimental points; full lines are calculated using stability constants and shifts for individual complexes.

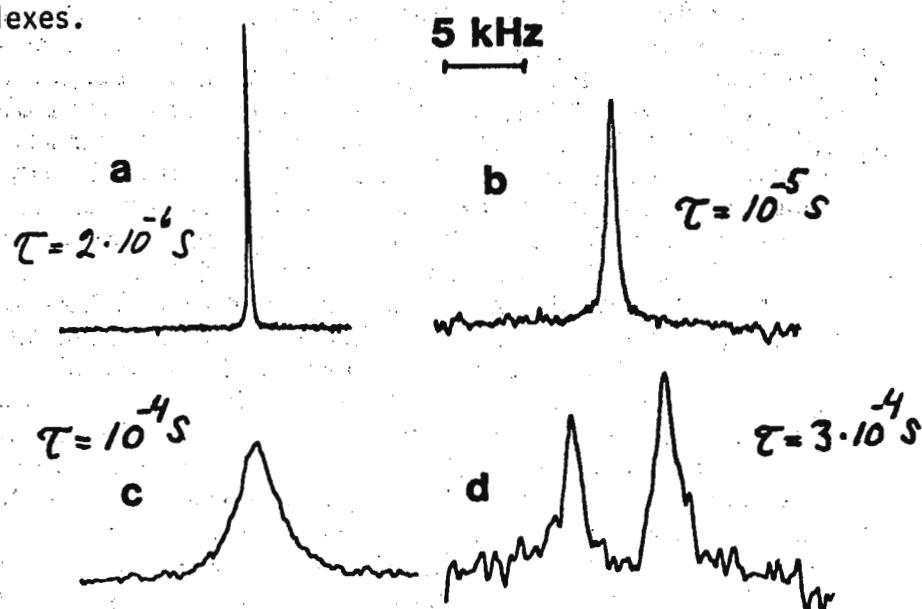


Fig. 2: 51.9 MHz ^{205}Tl spectra from solutions with composition $\text{TlCl}_{0.5}$ for a: $[\text{Tl}]_{\text{tot}} = 2.2 \text{ M}$; b: $[\text{Tl}]_{\text{tot}} = 1.0 \text{ M}$; c: $[\text{Tl}]_{\text{tot}} = 0.31 \text{ M}$ and d: $[\text{Tl}]_{\text{tot}} = 0.05 \text{ M}$. Approximate life times are shown in the figure.

DEPARTMENT OF HEALTH AND HUMAN SERVICES
PUBLIC HEALTH SERVICE
NATIONAL INSTITUTES OF HEALTH
BETHESDA, MARYLAND 20205

May 22, 1981

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

Dear Barry:

"Efficient detection of paramagnetically shifted resonances in solution with a spectrometer designed for solids"

One of the persistent problems in interpreting proton NMR Spectra of large proteins is disentangling the thousands of overlapping signals. In heme enzymes and other proteins containing paramagnetic ions we are fortunate in having many of the signals shifted by contact interactions. However, the disadvantage of having widely shifted resonances (from about 120 to -50 ppm) is that at high field they cover a large frequency range--often larger than can be readily accommodated with standard high resolution spectrometers.

Recently we have found that a 250 MHz proton NMR spectrometer for solid samples, which has been built by Dennis Torchia and his co-workers in NIH, is very effective in detecting hyperfine-shifted heme signals of some heme enzymes with small sample volumes and low concentrations. This system has a transverse solenoid detecting coil fitted to 5 mm sample tube, and an EBN power amplifier. The signal is collected in a Nicolet Explorer oscilloscope and is transferred to NIC 1080 computer and processed in it. Usually a 1.5 μ s pulse provides a 90° flip. Typical sample condition we used was 50 μ l of 1 mM heme enzyme (MW: ca. 50,000), which is 30 times less than the usual condition for a commercial NMR, 0.3 ml, 5mM solution. Conventional WET (180°- τ -90°) pulse sequences are found to be helpful to eliminate the residual H₂O signal. Careful adjustment of the τ value can reduce the huge H₂O signal found in water instead of D₂O. Undesired buffer signals, such as 0.5M acetate and 0.1M tris buffers, and some part of protein signals were also effectively suppressed by this pulse sequence. To obtain a good signal to noise ratio 4K transients were accumulated, which took about 10 minutes. The only disadvantage encountered on this system is relatively poor resolution. But this can be overcome by using a capillary (Wilmad #529D, 1mm ID) in which 5 μ l of sample was placed. By this method, 3 mM of metmyoglobin could be detected with 1K transients, in about 3 minutes, as shown in the attached figure.

Professor Bernard L. Shapiro

So far we have worked on cytochrome c peroxidase to prove two sets of heme signals in the unitary enzyme. The tremendous reduction of sample volume and concentration is crucial to handle such enzymes because it is usually laborious to obtain a large amount of enzymes and to purify in large scale. From this viewpoint the sample used in the above capillary is truly comparable with the amount usually used for spectrophotometry. By utilizing this advantage, we hope to be able to work on some respiratory enzymes in the near future.

Sincerely,

Toshiro Inubushi

Toshiro Inubushi

Ted

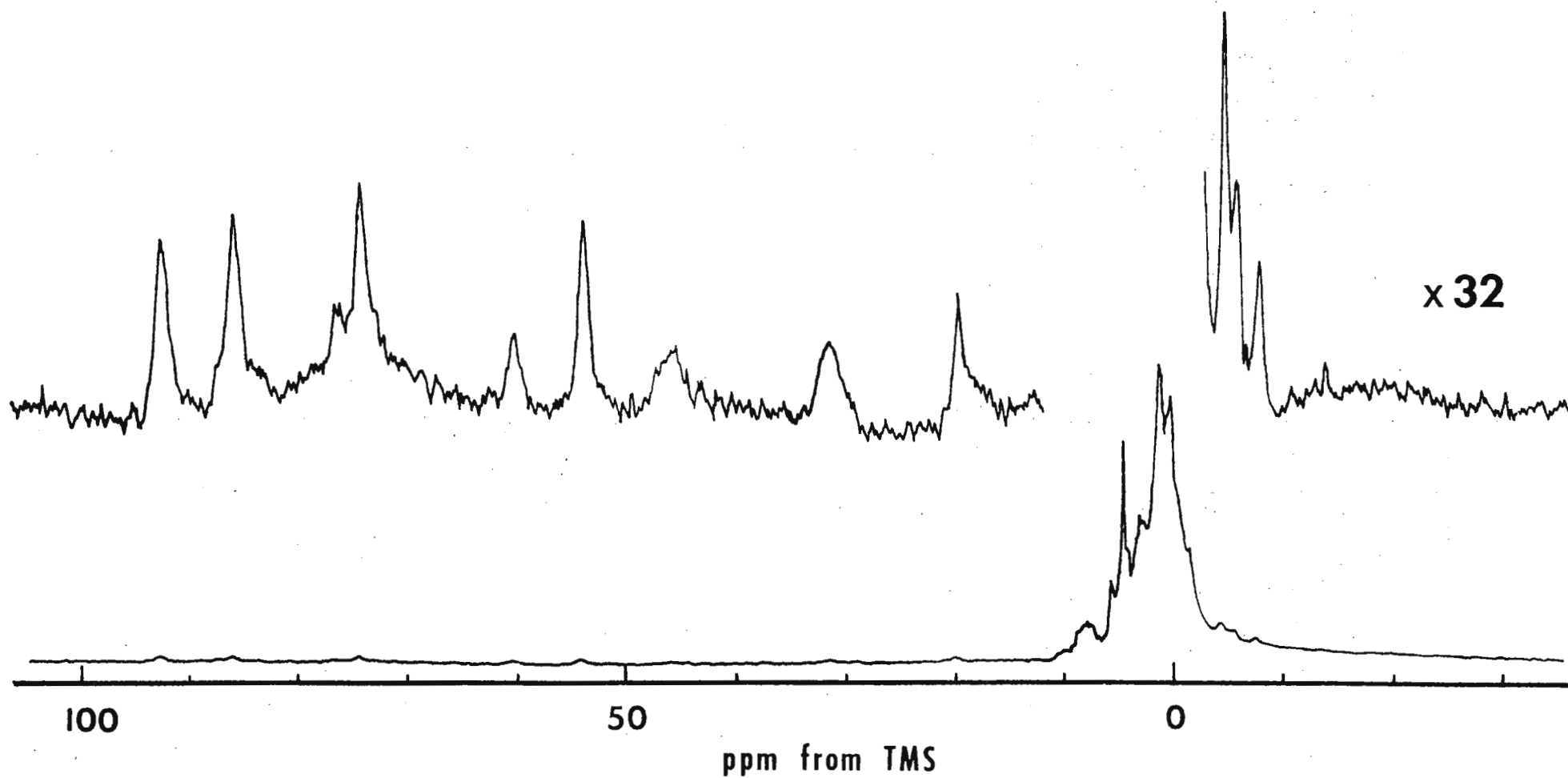
Edwin D. Becker

Attachment

METMYOGLOBIN, 3 mM, 5 μ L

1024 SCANS

273-29



Varian / 611 Hansen Way / Palo Alto / California 94303 / U.S.A.

Tel. (415) 493-4000

Telex 34-8476



May 27, 1981

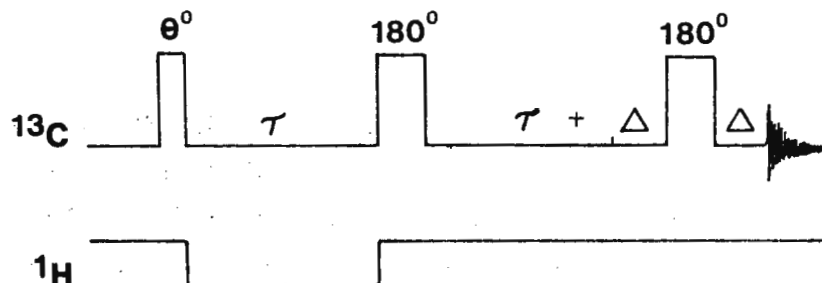
Professor Bernard L. Shapiro
 Department of Chemistry
 Texas A & M University
 College Station, TX 77843

Attached Proton Test (APT)

Dear Barry:

Our attention was turned recently to a very nice experiment first described in 1980¹ and elaborated upon in 1981^{2,3}, but which has, as far as we know, not been taken up by very many ¹³C NMR spectroscopists as a welcome replacement for the single-frequency off-resonance decoupling (SFORD) experiment. If one uses a 90°-τ-180°-τ pulse sequence with the decoupler gated "off" during the first τ period, and with $\tau = \frac{1}{J_{CH}}$, J-modulation of the ¹³C precession results in a spectrum with the signals from CH₂ and non-protonated carbons displaying opposite phase from those arising from CH₃ or CH groups, as shown in Figure 1. The advantages of this way of determining the number of protons attached to each carbon atom are obvious to anyone who has struggled with the interpretation of a badly split-up and overlapped SFORD spectrum from a complex molecule with many closely spaced spectral lines.

The simple spin-echo experiment requires the use of 90° pulses, since smaller tip angles leave a component of magnetization along the +z axis which is then inverted by the 180° pulse. Refocussing of the x and y components occurs normally, but recovery of the magnetization for nuclei with long T₁'s requires an additional equilibrating delay with attendant inefficiency of data acquisition. We have overcome this problem by using a double spin echo sequence as shown below:



The purpose of the second 180° pulse is to re-invert the z-component of magnetization, leaving it where it would normally be found after a single pulse experiment.

With this sequence, the only change in the spectra is that carbons with even or odd numbers of attached protons give signals which point in opposite directions. Figure 1 shows a crowded spectrum which presents formidable problems for the SFORD experiment but which is readily interpretable with the double echo sequence. Even with 45° pulses at 0.8 sec intervals, the slowly-relaxing but narrow quaternary carbon signals from carbons 5, 10, and 13 are not suppressed.

Some confusion could arise between methyl groups and CH groups in the cross-over region of the chemical shift scale. In practice, this is unlikely. The smallest chemical shift listed for a CH group in a ^{13}C spectral catalog⁴ is 24.8 ppm. The only methyl groups with chemical shifts greater than 24.8 in reference(4) are a small number on aromatic rings or double bonds, adjacent to carbonyl functions, or on highly branched quaternary carbons.

Quaternary carbons and CH_2 groups with sp^3 hybridization are overlapped over a larger chemical shift region. Distinguishing between these may require a second experiment in which use is made of the different time evolution of the various signals, shown in Figure 2. If $\tau=4$ msec, only the quaternary carbons should have appreciable amplitudes, except for protonated carbons with $J_{\text{CH}} \geq 160$ Hz. Noise-modulated off-resonance decoupling will also identify quaternary carbons.

Although similar information can be obtained from the refocussed INEPT sequence such experiments cannot readily be performed on many FT-NMR spectrometers, and quaternary carbons can be eliminated entirely. The pulse sequence described in this letter preserves the NOE intensity gain which is nearly as great as that achieved through polarization transfer by INEPT. Consequently, it appears that for all practical purposes this method may become the method of choice for multiplicity determination.

Since all experiments seem to have ~~to have~~ a name, we have pondered long and hard, seeking a suitably descriptive one, simple enough for routine use. At first, regarding it as an INEPT experiment with Absent Polarization Transfer, we thought of APT, but it didn't seem to be. But then we realized that the experiment is truly an Attached Proton Test and we felt better about it. Therefore, we formally propose that spectra run with this pulse sequence be called APT spectra and hope that our colleagues will agree that they are.

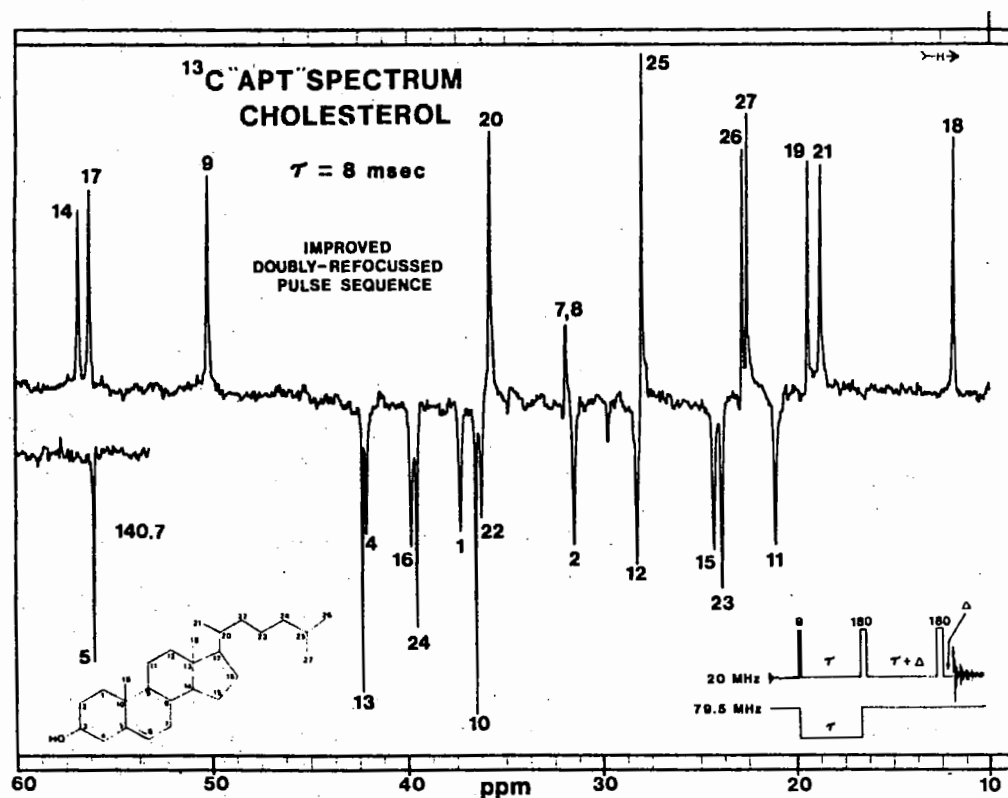


Figure 1

Owners of XL-200's can implement this pulse sequence immediately in the usual way. Owners of FT-80A spectrometers will need a simple patch to the standard program⁵.

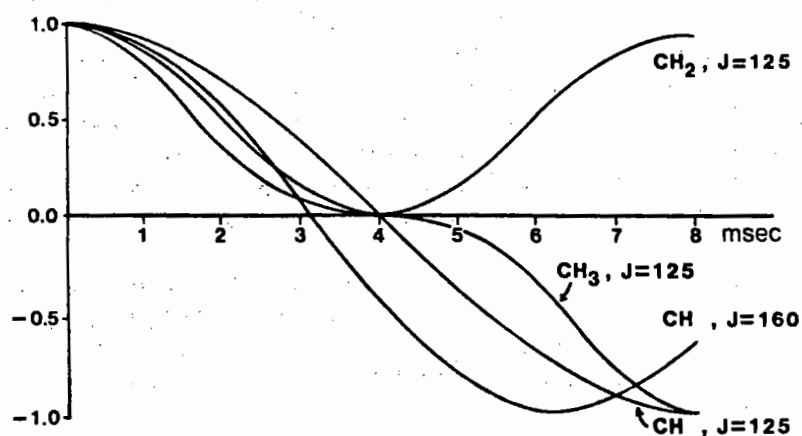


Figure 2

With best regards,

James N. Shoolery
J.N. Shoolery
Varian NMR Applications Lab
Palo Alto, California

Steve Patt
Steve Patt
Varian NMR Applications Lab
Florham Park, New Jersey

¹F.A.L. Anet, N. Jaffer, and J. Strouse, 21st Experimental NMR Conference (1980)

²C. Le Cocq and J.-Y. Lallemand, J.C.S. Chem. Comm. 150, (1981)

³D.L. Rabenstein, T.T. Nakashima, and D.W. Brown, 22nd Experimental NMR Conference (1981)

⁴L.F. Johnson and W.C. Jankowski, "Carbon-13 NMR Spectra," Wiley-Interscience, New York, 1972

⁵Obtainable by writing to either of the authors

Department of Chemistry
State University of New York at Stony Brook
Long Island, NY 11794
telephone: (516) 246-5057

Stony Brook

26 May 1981

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

Re: Ionophore-Catalyzed Cation Transport Between
Phospholipid Inverted Micelles: Manifest in DNMR

Dear Barry:

The figure is taken from the work of Dr. Shuenn-Tzong Chen when he was in my laboratory. It depicts the $^{31}\text{P}\{^1\text{H}\}$ spectrum of a dispersion of hydrated dipalmitoyl lecithin (DPL, 70 mM) inverted micelles in benzene ($T = 52^\circ\text{C}$). The preparation was such that some of the micelles contain one Pr^{+3} ion each (and three NO_3^- counter ions) in their aqueous cores, some contain one La^{+3} ion, and some contain no ions. Thus, the sharp resonance in spectrum a) is assigned to the lipids in those micelles with no Pr^{+3} ions and the broader resonance downfield to those in Pr^{+3} containing micelles (S.-T. Chen and C. S. Springer, Chem Phys. Lipids, **23**, 23 (1979)).

Spectrum b) results when the solution is made 0.91 mM in the sodium salt of the antibiotic lasalocid-A. Both resonances have become noticeably broadened. If the NaX concentration is doubled to 1.82 mM, the two lines coalesce into a single broad resonance; spectrum c). Since the ionophoretic properties of lasalocid-A are well known, a logical assessment is that the ionophore is catalyzing the equilibrium exchange of Pr^{+3} ions among the micelles to the point where it becomes fast on the NMR timescale. Consistent with this is the fact that dilution of the NaX only (spectrum d); obtained by mixing equal aliquots of the solutions of a) and b); thus $[\text{NaX}]$ is 0.91 mM again) causes reappearance of the two broadened lines. Resuming the increase in $[\text{NaX}]$ (e) 1.82 mM, f) 2.73 mM, and g) 5.45 mM) causes recoalescence and subsequent sharpening.

We have conducted total lineshape analyses on a number of such systems and found, in general, a first order dependence on antibiotic concentration. This has interesting implications as to the mechanism of transport across phospholipid bilayer membranes, We also find, as expected, inhibition by protons or other (diamagnetic) cations. We have recently submitted this work for publication.

Please credit this contribution to the Stony Brook subscription (Paul Lauterbur).

Best regards,

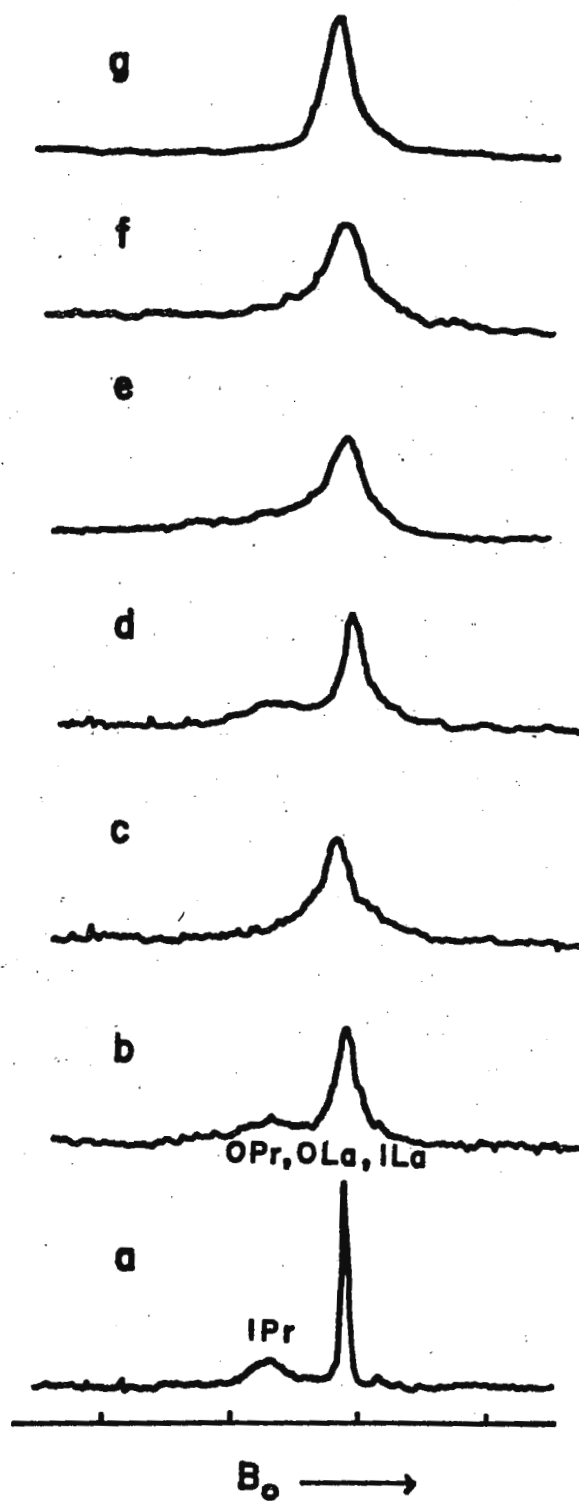


Charles S. Springer, Jr.
Assoc. Prof. of Chemistry

$^{31}\text{P}\{^1\text{H}\}$ NMR,

52°C

100Hz
2.47 ppm



University of Illinois at Urbana-Champaign

School of Chemical Sciences

505 S. Mathews
Urbana, IL 61801

Position Opening: OPERATIONS MANAGER, SPECTROSCOPY FACILITY

We seek a person to be in charge of the Molecular Spectroscopy Laboratory of the School of Chemical Sciences (SOCS). The position, with the title of Spectroscopist, requires a broad range of professional, research and educational skills. Minimum qualifications include an MS degree in physical science or engineering, and experience related to the responsibilities of the position.

The successful applicant will be responsible for the supervision, training and evaluation of the three Assistant Spectroscopists and one Engineering Technician who perform the day to day operations, and will also be the Operations Manager of the NSF sponsored Midwest Regional Instrumentation Facility, through 1983. He or she instructs students, post doctorates and faculty in the use of instruments, establishes policies so as to provide fair and general access of all SOCS research personnel to the instrumentation, and is responsible for the proper maintenance of all instruments. He or she should have a good understanding of the physical principles underlying the operation of lab instruments. Magnetic resonance spectroscopy is the major research function of the laboratory which contains 220, 250, 360 and 500 MHz FT instruments and five low field systems, together with FT-IR, UV, ESR and ORD/CD spectrometers. It is therefore especially important that the individual have a strong background in NMR techniques, and be able to help implement modifications and additions to existing instruments.

The desired starting date is September 1, 1981. The position is permanent. The starting salary is competitive and commensurate with experience. Those interested should apply at once. Application materials should be submitted promptly, preferably by August 1, 1981, in order to ensure consideration. Persons wishing to apply should send a resume to the address given at the end of this announcement and arrange for three letters of recommendation to be sent there also.

The University of Illinois is an Affirmative Action/Equal Opportunity Employer.

Inquiries should be addressed to:

Dr. H. S. Gutowsky, Director
School of Chemical Sciences
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Yours sincerely,



Eric Oldfield
Associate Professor of Chemistry

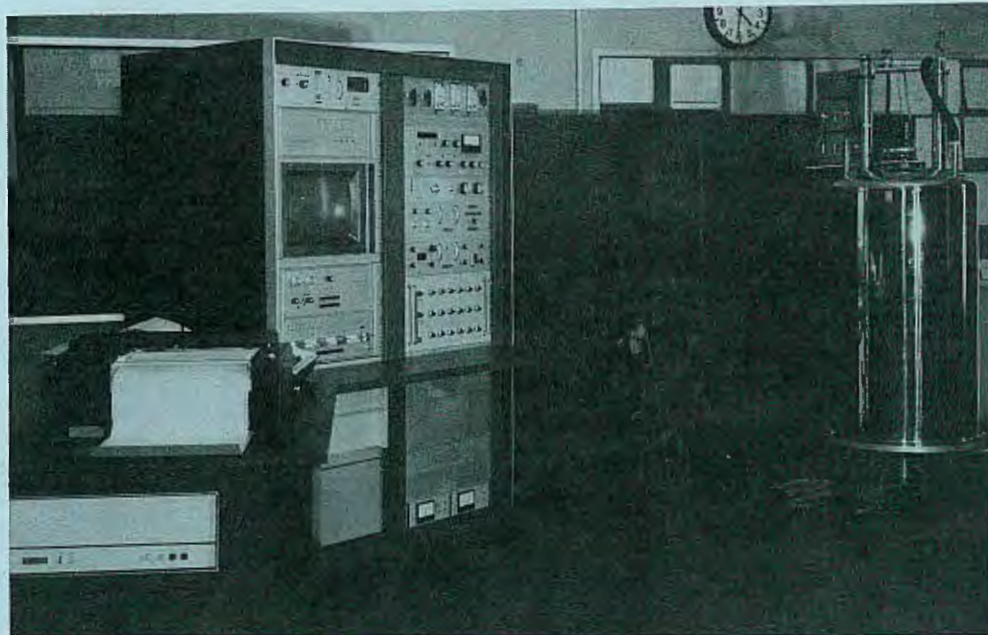
Nicolet Supercon FT-NMR Spectrometers

Uncompromising performance, limitless adaptability.

Our spectrometer systems have been conceived and designed to provide optimum performance while being fully adaptable to new techniques with minimal cost and difficulty. More than just a collection of instruments, they represent a completely modular approach to FT-NMR instrumentation that allows the user to expand his system as his research needs grow and to easily accommodate new experimental techniques as they develop.

Outstanding Nicolet features include these:

- A full range of superconducting magnets from 4.7T to 11.7T (200MHz to 500MHz proton frequency range), in both wide-bore and narrow-bore configurations.
- Multinuclear observation with a wide variety of fixed-tune and broadband probes.
- Simultaneous acquisition, processing, and plotting for greater sample throughput.
- Simplified control of spectrometer operations and parameters by using easy keyboard commands.



- Advanced Nicolet 1180E Data System with 128K/20-bit memory, 256-step pulse programmer, and the most comprehensive FT-NMR software package available.

- Extended dynamic range performance with 40-bit acquisition and floating-point processing.

- An expandable pulse-sequence library, including T_1 , T_2 , Redfield, INEPT, homo- and hetero- 2D-FT, etc.

- Convenient computer control of field shimming, observe and decoupling frequencies, sample temperature, and probe-tuning.

- Precise digital plotting with full annotation of spectral parameters and flexibility of hardcopy format.

The versatile Nicolet spectrometers provide the user with the ability to easily adapt to the newest techniques and experimental configurations.

Some of these are:

- High resolution studies of solids with Waugh-Pines cross-polarization and magic-angle spinning.
- High sensitivity wide-bore ^{13}C studies of high molecular weight polymers.

- Automated T_1 and T_2 measurements.
- Chemical dynamics studies.
- Temperature-programmed experiments.
- ^{31}P experiments on living organs.

NTE NICOLET MAGNETICS CORPORATION

A NICOLET INSTRUMENT SUBSIDIARY

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Mountain View, California 94041
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Telephone: 415-969-2076

FX SERIES OF FT NMR SYSTEMS

FX Features

- Light Pen Control System
- Bilevel Software Package
- 2-D Spectroscopy
- Auto T_1 , T_2 Meas./Calculation
- FX Series Work Station
- Programmable Multi-Pulser: INEPT, Selective Excitation, Cross Polarization, Bilevel Decoupling, etc.
- Digital Quadrature Detection
- Oxford SCM Systems
- Programmable Variable Temperature
- Double Precision (32 bit word length)
- Floppy; Moving Head Disc Systems

FX-60QS:

- CP/MAS
- ^{13}C , ^{31}P , ^{29}Si (examples)
- Routine Liquids/Solid State

FX-270:

- Dual Frequency Probes
- Broad-Band Probes
- "Tilt" Micro Probe

FX-90Q:

- OMNI Probe™ System
- 10mm, 5mm Micro Inserts
- Wide Band (^1H to ^{103}Rh)

FX-200:

- Dual Frequency Probes
- Broad-Band Probes
- CP/MAS Extension



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