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Newsletter

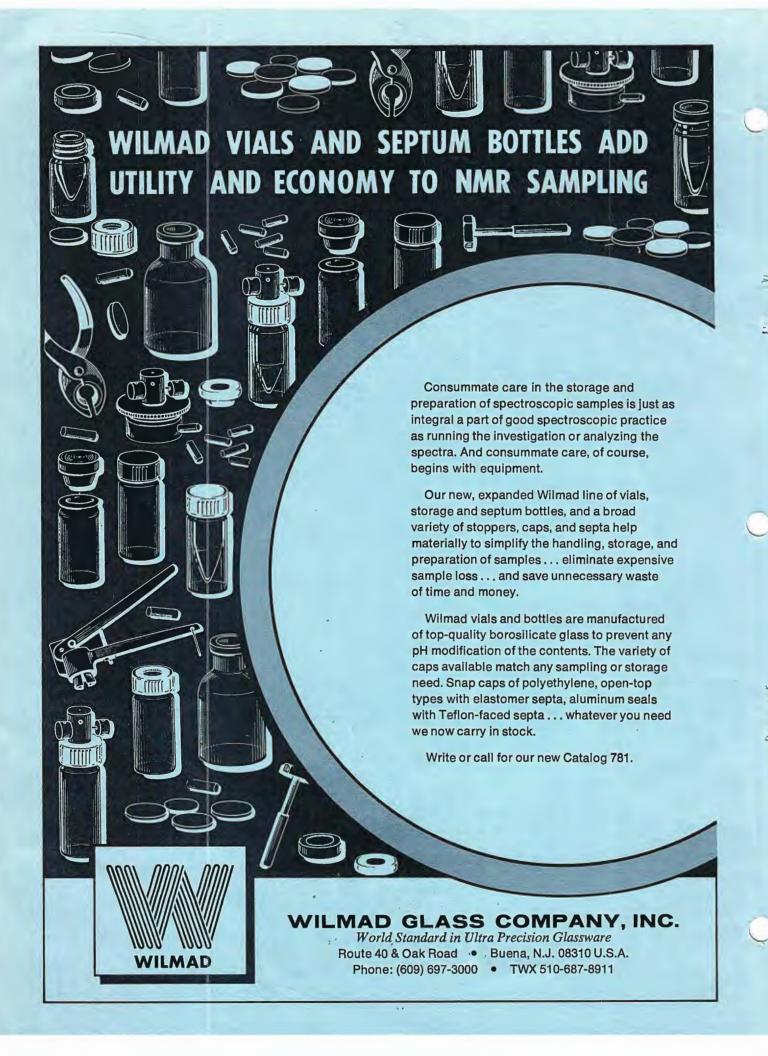
No. 2 5 2

September, 1979

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DEADLINE DATES: No. 253 1 October 1979 No. 254 5 November 1979

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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# University of Illinois at Urbana-Champaign

School of Chemical Sciences
Urbana, Illinois 61801

July 25, 1979

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

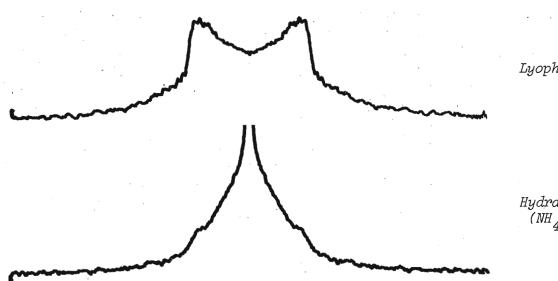
Dear Barry:

NMR of Protein Crystals; NMR of Deuterated Spin-Labels;

Phospholipid Headgroup Conformations; Postdoc Positions.

We thought we would report some results obtained in the above areas using our new wide-bore 360 spectrometer (which has the surprisingly good drift rate of  $5 \times 10^{-9} \text{ hr}^{-1}$ !).

First, we have obtained a number of  $^{13}\mathrm{C}$  and  $^{2}\mathrm{H}$  NMR spectra of specifically isotopically labelled protein crystals. These experiments are of course of great interest both from the point of view of crystal-solution structure comparisons, and from the standpoint of protein-hydration investigations. The following Figure shows the 55 MHz  $^{2}\mathrm{H}$  spectrum of ribonuclease A (EC 2.7.7.16) containing a Me-SCD<sub>3</sub> sulphonium group at position 29 of the polypeptide chain, as a lyophilized powder (A) and hydrated with a concentrated (NH<sub>4</sub>)<sub>2</sub> SO<sub>4</sub> solution (B).



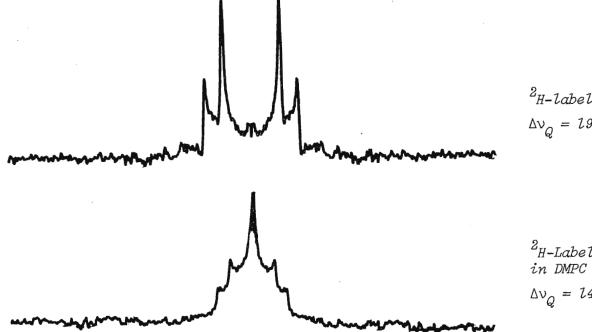
Lyophilised powder

Hydrated with Satd.  $(NH_4)_2SO_4$ 

These spectra were obtained in only  $\sim$  20 minutes of data accumulation, opening up the way to numerous studies of motions in protein crystals.

Second, we have been investigating the differences between phospholipid order parameters as determined by ESR (of spin-labelled molecules) and <sup>2</sup>H NMR (of <sup>2</sup>H-labelled species). One always has, of course, the problem of the different timescales between the two measurements, so that slow motions may easily reduce the <sup>2</sup>H NMR order parameter but might not have any effect on the ESR spin-label order parameter. Investigations aimed at finding out just how much of a perturbation the nitroxide radical causes, or whether it might be involved in some specific interactions (eg. hydrogen bonding with a protein) are clearly complicated by this problem.

We have therefore synthesized a <sup>2</sup>H-labelled phospholipid spin label and determined the <sup>2</sup>H quadrupole splittings of the labelled compound (in a DMPC bilayer) together with those of a pure <sup>2</sup>H-labelled DMPC bilayer. The results are shown in the accompanying Figure

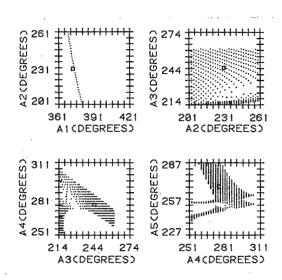


 $^{2}$ H-labelled DMPC  $\Delta v_{O} = 19.1 \ 11.5 \ kHz$ 

 $^{2}$ H-Labelled Spin-Label in DMPC Bilayer  $\Delta v_{O} = 14.2$ , 8.5 kHz

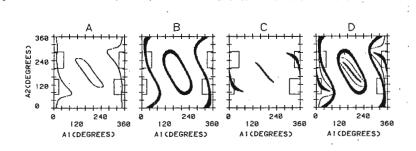
and indicate quite clearly that the spin-label  $\Delta v_{\rm Q}$  (or order parameter) values are some 20% smaller than those of the pure bilayer the label is supposed to be probing. These results are of course not subject to any "timescale" rationalizations.

Third, we have been calculating phospholipid headgroup structures using  $^2\text{H}$  NMR  $\Delta\nu_{Q}$  and  $^{31}\text{P}$   $\Delta\sigma$  values and a straightforward Cartesian transformation method first outlined by Seelig and co-workers (BBA  $\underline{467}$  (1977) 109). Contrary to our expectations there are exceedingly large numbers of possible headgroup conformations which can satisfy all the NMR observables. The following plots are representative:



Solutions for DPPE in excess water at  $49^{\circ}$ C using ± 30 degree torsion angle ranges, two degree increments, the tensor of Kohler and Klein, and the NMR parameters  $\Delta \sigma = -37.5$  ppm,  $\Delta v_{12}, 13 = \pm 9700$  Hz, and  $\Delta v_{10}, 11 = \pm 3700$  Hz.

It isn't possible to be unambiguous even when the results of Herzfeld et al. (Biochem. 17, 2711 (1978)) are included, as shown in the accompanying Figure.



 $\alpha_1,\alpha_2$  solutions for the three constraints:  $\Delta\sigma=47\pm1$  ppm(A),  $0_3$ -P- $0_4$  oriented at  $47\pm5$  degrees with respect to the bilayer normal (B), and  $\alpha_{33}$  of the Herzfeld phosphorus chemical shift tensor oriented parallel to the bilayer plane (C). D is a superposition of A,B and C. The outlined sub-regions are  $\pm$  40 degree spaces about each of the two crystallographic conformers.

Finally, I shall have several postdoctoral positions available in the coming year for outstanding candidates to work on "biological NMR" problems, in solids or liquids, using in addition to our current 150/220/360 MHz systems a new 500 MHz instrument.

Sincerely yours,

Bob Skarjune

Eric Oldfield "

Dave Rice

Mike Rothget

Andrew Scheinman

Gene DeRose

msh



#### **University of Nottingham**

Department of Chemistry

UNIVERSITY PARK NOTTINGHAM NG7 2RD TEL NOTTINGHAM 56101

HB/JL

2nd August 1979

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

Dear Professor Shapiro,

#### 12C/13C Isotope Effect on a Conformational Equilibrium ?

Recently we attempted to decide whether the degenerate conformational equilibrium in cis-1,4-dimethylcyclohexane is altered by a change of the  $1^{-12}\text{CH}_3$  to  $1^{-13}\text{CH}_3$ . As a consequence of such an alteration, the room-temperature, and C-4' in  $(1\rightleftharpoons 2)$  would be different from the room-temperature averaged shift  $(\delta_1)$  of carbons C-1" and C-1'.

The signal for C-4',4" is a singlet, whereas that for C-1',1" is a doublet, being the low-field half of an AB system ( $J_{AB} = J_{13}_{C-13}_{C}$ ). Now the observed  $\delta_1$  must be corrected in view of the well-established, direct, 1-bond isotope effect; 1 however, we assumed that a direct isotope effect over 4-bonds would be negligible and that the observed  $\delta_4$  needed no correction.

$$CH_3$$
 $(1)$ 
 $^{13}CH_3$ 
 $CH_3$ 
 $(2)$ 

The 1-bond isotope effects were determined from the  $^{13}$ C spectrum of (1  $\rightleftharpoons$  2) at 172 K, where the rate of conformational interconversion is so slow that the spectra of both (1) and (2) are observed. In the F.T. experiments at 25.15 MHz we employed' 16 K data points over 1000 Hz, for a digital accuracy of + 0.06 Hz. In the event, the direct 1-bond isotope effects were -0.14 Hz (i.e. an upfield isotope effect) for both equatorial and exist

substitution of methine carbon.

The  $^{1\,3}\text{C}$  spectrum at 300 K gave  $\delta_1$  -  $\delta_4$  as -0.15 Hz (i.e. upfield shift). Consequently an isotope effect on the position of equilibrium is either non-existent, or is too small to be measured by the chosen method of analysis.

Yours sincerely,

JABOOTE -

Dr. H. Booth, J.E. Everett. \*

cf. J. Jokisaari, Org. Mag. Res., 1978, 11, 157.

of Present address:

Dept. of Chemistry, McMaster University, Hamilton, Ontano.

CH-4056 Basel (Schweiz) July 30. 1979

# INSTITUT FÜR PHYSIK DER UNIVERSITÄT BASEL EXPERIMENTELLE KERNPHYSIK

Klingelbergstrasse 82, Telefon 061 - 44 22 80

Prof. Dr. P. Diehl

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

#### Postdoctoral Position Available.

Dear Barry,

I will have funds available for a postdoctoral position for one year beginning November 1st 1979 (or later). The work will involve NMR-research in oriented molecules. We have a multinuclear FT-spectrometer (WH 90 DS, Bruker).

The salary amounts to Swiss Francs 37'000.— per year. There is no travel allowance. Any person interested should write to me as soon as possible.

Sincerely yours

Prof. Dr. P. Diehl

#### THE SWEDISH INSTITUTE FOR SURFACE CHEMISTRY

Drottning Kristinas väg 45 S-114 28 Stockholm - Sweden Tel. 22 25 40

July 31, 1979

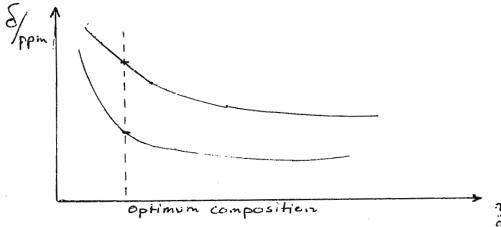
Dr Bernard L Shapiro
Dept of Chemistry
Texas A&M University
College Station, TX 77843
USA

Dear Professor Shapiro,

#### Resolution optimization by proper choice of environment

When studying the three component systems  $\rm H_2O$  - Potassium cis 9, 10, Oleat-Pentanol we found the  $^{13}{\rm C}$ -chemical shifts to be concentration dependent in the  $\rm L_2$ -phase, which contains inverted micelles.

By plotting chemical shifts as a function of cencentration we got the results given in Fig. 1 (the figure is just ment to clarify the idea).



on Potassium cleat

Fig. 1. The chemical shift as a function of the mole ratio water to Potassium Oleate at constant Pentanol to Potassium Oleate ratio.

From the diagram you can choose the optimum chemical composition, i.e. the largest chemical shift difference for the carbon atoms of greatest interest. As an example we have been able to separate the shifts of all carbon atoms of Potassium Oleate (see Fig. 2).

We think that in this system the main reasons for changes of the chemical shifts are

- entropy changes, which are connected to the motional freedom of alkylchains
- electric field effects, induced by changes in hydrogen bonds to the carbonyl group.

As a conclusion we think that this kind of chemically induced shifts can be very helpful in assigning spectra.

Sincerely yours,

Thomas Ahlnäs

PS Please credit this letter to the account of Professor Pekka Pyykkö.

21st ENC, TALLAHASSEE, FLORIDA, MARCH 16-20, 1980 21st ENC, TALLAHASSEE, FLORIDA, MARCH 16-20, 1980 21st ENC, TALLAHASSEE, FLORIDA, MARCH 16-20, 1980

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

Dear Barry:

The next annual meeting of the Experimental NMR Conference (ENC) will be held at Tallahassee, Florida. Registration will begin on Sunday, March 16, and the program will run through Thursday, March 20. Sessions will focus on new experimental techniques and developments in a variety of nmr areas, probably including: Cross Polarization and Magic-angle Spinning; Refocussing and 2-Dimensional Techniques; NMR Imaging; Probe Design for Superconducting Magnets; Progress in Very High Field Spectrometers; Multiple-Pulse Line-narrowing Techniques for Solids; Developments in High Resolution Techniques for Liquids; Quadrupolar Nuclei.

Most of the talks will be given by speakers invited by session chairmen. There will be some time reserved for shorter contributions, and poster sessions will be held. Anyone who has not been invited by a session chairman by November 1, but believes strongly that his or her talk would be more appropriate for one of the above-mentioned sessions than for a poster session, should contact me before November 15, 1979. Those interested in presenting a poster talk should contact the chairman of the poster session, Dr. James Frye, Regional NMR Center, Department of Chemistry, Colorado State University, Fort Collins, CO. 80523, before January 15, 1980.

Everyone who attended the ENC in 1975, 1976, 1977, 1978 or 1979 is on the current mailing list, and will receive registration information and a copy of the preliminary program in mid-December. Anyone who is not on the mailing list or for some other reason does not receive the registration material during December should contact the ENC Secretary, Dr. A.A. Bothner-By, Department of Chemistry, Carnegie-Mellon University, 4400 Fifth Avenue, Pittsburgh, PA. 15213. The local arrangements chairman for the 21st ENC is Dr. George C. Levy, Department of Chemistry, Florida State University, Tallahassee, FL. 32306.

Sincerely,

Gary E. Maciel

Chairman, 21st ENC

Department of Chemistry Colorado State University Fort Collins, CO. 80523 APPLIED CHEMISTRY LABORATORIES

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P.O. BOX 4331, MELBOURNE, VIC. 3001 - 506 LORIMER ST., FISHERMEN'S BEND, TELEPHONE 640251. TELEGRAMS CHEMIRO MELBOURNE

9 August 1979

Dear Professor Shapiro,

TITLE 19 F Centre Band Decoupling

For many years we have used our HA100 to obtain  $^{19}\mathrm{F}$  spectra and in the process solved the problem of successfully performing  $^{19}\mathrm{F}$  homonuclear decoupling. The extended chemical shift range and large  $\mathrm{J}_{\mathrm{FF}}$  values (\$200 Hz) makes it very difficult to transfer sufficient decoupling power into a side band generated some 5000 Hz or more from the centre band.

The answer is to place the peak to be decoupled, directly under the 94.1 MHz centre band, where up to a maximum of 0.5 watt decoupling power is available. This may require extending the lock oscillator frequency range which can be achieved by its substitution with an external audio oscillator e.g. Wavetek # 111.

The recorder swept oscillator now requires a presetable frequency start point as well as usual chart sweep widths. We have achieved this by using either a Varian C-1024 coupled to a voltage controlled oscillator e.g. Wavetek # 111 or a Varian spectrosystem with a programme patch to bypass any frequency measurement.

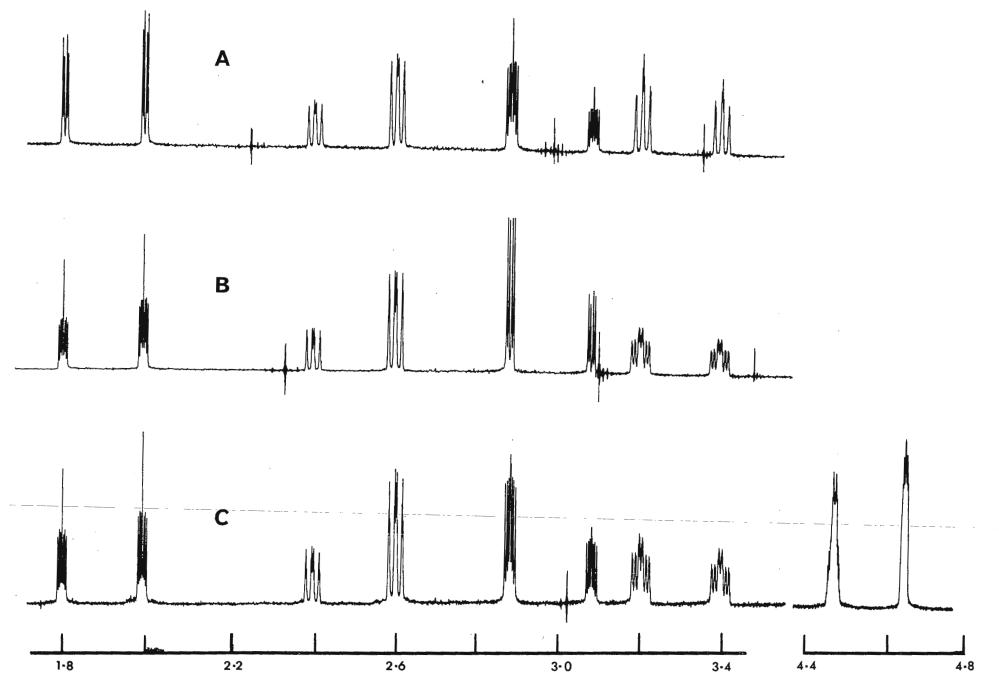
The technique is illustrated by the <sup>19</sup>F spectrum of the cistrans isomer mixture:

Spectra A and B show "centre band decoupling" of peaks at 4480 and 4650 Hz respectively at 20 db RF attenuation.

Please credit this contribution to Dr. D.P. Kelly's subscription.

Yours sincerely,

WILLING S.R. JOHNS



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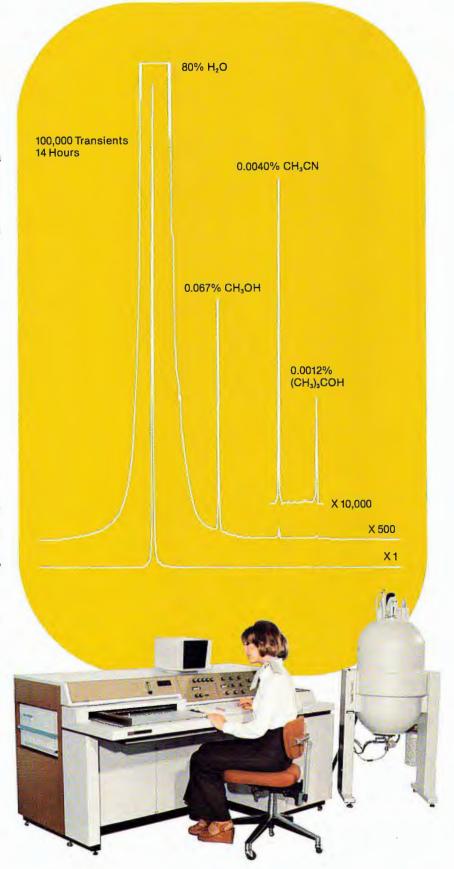
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# Papanicolaou Cancer Research Institute at Miami, Inc.

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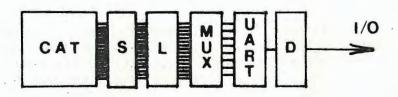
August 11, 1979

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

Dear Dr. Shapiro:

#### Progress on Minicomputer Interface for NMR Signal Averager

We wish to report progress on building a special purpose minicomputer interface for an NMR signal averager. Basically, we wanted an interface which could take data from a Varian C-1024 signal averager in one part of the building, and send the data to a minicomputer about 60 feet away for analysis. We decided to go with a serial data interface in order to avoid having to string 16 or 17 wires through the building. Serial data transmission can be accomplished with just 1 or 2 lines, and need not be as slow as teletype baud rates. In fact our interface can theoretically send data at up to 9600 baud, which is less than two seconds for complete memory transmission.



The CAT parallel output is first shifted to TTL compatible levels using the level shifter (S). In order to insure stability, each 16 bit word then goes into latches (L). Then 2:1 multiplexers (MUX) split each word into two 8 bit bytes. The 8 bit bytes are then converted from parallel to serial in the universal asynchronous receiver/transmitter (UART). Lastly, the UART output is shifted to RS 232-C levels by the line driver (L). Timing circuits not shown here change the CAT address, set the latches, control the MUX, and strobe data through the UART. Preliminary testing has been carried out at 1200 baud. R. Thomas, D. Dubin, and N. Lefkove made major contributions to this project.

Sincerely yours,

Ron Block

Ronald E. Block, Ph. D.

A non-profit institution for medical research and education—supported by tax exempt contributions

REHOVOT'ISRAEL

רחובות י שראל

ISOTOPE DEPARTMENT

מחלקת איזוטופים

July 31, 1979

Prof. Barry Shapiro
Department of Chemistry
Texas A and M University
College Station, Texas 77843
USA

Dear Prof. Shapiro:

#### Proton and oxygen-17 NMR studies of water permeability of membranes

We have been recently interested in studies involving the very fast process of water diffusion through membranes. Employing a vesicle suspension of DDPC (dipalmitoyl phosphatidylcholine), as a model membrane system, we have studied the exchange of water between the external and the intravesicular media by <sup>1</sup>H and <sup>17</sup>O NMR relaxation measurements of the water nuclei. In order to differentiate between the nuclear relaxation rates in the two media, the vesicles were prepared in such a way that paramagnetic ions (e.g. Mn<sup>2+</sup>) were entrapped in the intravesicular medium, thus enhancing markedly the relaxation rate of the inner water nuclei. Since paramagnetic ions diffuse very slowly across the membrane the nuclear relaxation rate of the immer water remain enhanced relative to that of the external water throughout the experiments.

The transport of water across the membrane induces an exchange of water between the fast relaxing and slow relaxing media which can be treated in an analogous way to a two sites chemical exchange process. The fraction f, of the fast relaxing water is small ( $\sim$ 5%), therefore the Swift and Connick treatment for exchange could be applied giving:

$$1/T_2 = 1/T_2^0 + f/(T_2^{i0} + \tau^i)$$

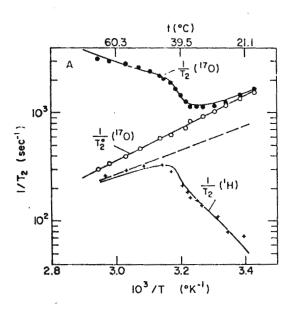
where 1/T<sub>2</sub> and 1/T<sub>2</sub><sup>0</sup> and 1/T<sub>2</sub><sup>io</sup> are respectively the measured relaxation rate, the relaxation rate of the external water and the relaxation rate in the internal medium without exchange. Fig. 1 presents the temperature dependence of <sup>1</sup>H and <sup>17</sup>O relaxation rates of water in a DPPC vesicle suspension containing MnCl<sub>2</sub> inside the vesicles. The water exchange rates derived from the results in Fig. 1 are presented in Fig. 2. Both Figs. exhibit a conspicuous change at about 40°C, which is the temperature in which the lipid chains undergo a phase transition. This change indicates that water permeability is sharply reduced at the phase transition on going from the liquid crystalline phase to the gel phase. It is also clear from Fig. 2 that the activation energy for water diffusion in the gel phase is much higher than in the liquid crystalline phase. The diffusion rates calculated from the <sup>1</sup>H and <sup>17</sup>O measurement

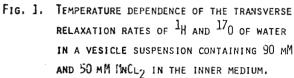
were the same (within experimental error) thus indicating that the rates measured correspond to a transport of a whole water molecule.

Similar investigations into other transport processes are currently in progress in our laboratory.

Please credit this contribution to Dr. R. Poupko's account.

Sincerely yours,
Hadassa Degani





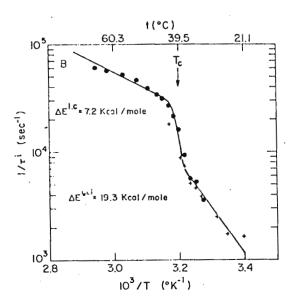


Fig. 2. Temperature dependence of the water diffusion rates across DPPC vesicles.

HD/sn

#### UNIVERSITY OF CALIFORNIA, SANTA BARBARA

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SANTA BARBARA · SANTA CRUZ

DEPARTMENT OF CHEMISTRY
SANTA BARBARA, CALIFORNIA 93106

August 3, 1979

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

- (1) Studies of Enzyme-Polymer Conjugates
- (2) Positions Available

#### Dear Barry:

(1) Synthetic polymers can easily be attached to proteins to provide materials which have new chemical and physical properties. We have linked homopolymers of N-acryloyl- $\beta$ -alanine to the enzyme  $\alpha$ -chymotrypsin and are exploring the effects of the conjugated polymers on the structure and function of the enzyme's active site. p-Fluorocinnamic acid binds tightly to the enzyme in a manner that makes it a good competitive inhibitor of the enzyme and the binding is not appreciably modified when the polyanion is attached to the protein. Dynamics of molecular motion of the complexed fluorocinnamate have been studied by fluorine nuclear relaxation rate measurements at 51.0 and 94.1 MHz. Using an ellipsoid of revolution as a model for the small molecule, the data allow us to extract a diffusion coefficient for rotational diffusion parallel to the long (major) axis of the ellipsoid and a coefficient for motion perpendicular to the major axis. 1,2 Some results are given in Table I. The diffusion coefficients for p-fluorocinnamate complexed to native enzyme are those expected if the small molecule has no freedom of motion beyond the tumbling of the protein. The interesting result is that molecular motion is only slowed slightly in the protein-polymer conjugate even though the molecular weight of the attached polymer is as large as six million. Localized sidechain motions in polymers are generally rapid and there must be enough flexibility in the conjugates that the tethered enzyme molecule scarcely knows that it is linked to a large polymer.

Table I. Diffusion Coefficients for p-Fluorocinnamate System

System	D <sub>11</sub> , s <sup>-1</sup>	D <sub>⊥</sub> , s <sup>-1</sup>
Free molecule <sup>2</sup>	2.1 x 10 <sup>10</sup>	4.6 x 10 <sup>9</sup>
Chymotrypsin complex 3	1.1 x 10 <sup>7</sup>	$1.1 \times 10^{7}$
Chymotrypsin-polymer complex	$0.5 \times 10^7$	$0.3 \times 10^7$

(2) We have several postdoctoral positions that we would like to fill before the end of 1979. Candidates with interests in nmr spectroscopy, synthetic chemistry, protein isolation and characterization or polymer chemistry (any or all of these!) are invited to apply; contact me for further information.

Sincerely yours,

D. T. Loehr

J. T. Gerig

#### References

- 1. D. E. Woessner, J. Chem. Phys. <u>37</u>, 647 (1962).
- 2. J. T. Gerig, B. A. Halley and J. A. Reimer, J. Am. Chem. Soc. <u>99</u>, 3579 (1977).
- 3. J. T. Gerig, B. A. Halley and C. E. Ortiz, J. Am. Chem. Soc. 99, 6219 (1977).

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# FACHBEREICH CHEMIE DER PHILIPPS-UNIVERSITÄT

Dr.Stefan Berger

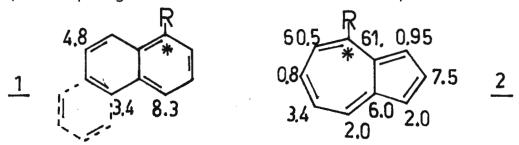
Fachbereich Chemie - Lahnberge D-3550 Marburg 1
Professor B.L.Shapiro
Texas A&M University
College of Science
College Station, Texas 77843

Telex: 4 82 372

den 16.8.79

# 13<sub>C</sub>13<sub>C</sub> Spin Coupling Constants in Azulenes

Dear Professor Shapiro, in earlier work we have proposed that the  $^{13}\text{C}^{13}\text{C}$  spin coupling constants in naphthalines  $^{1}$  and phenanthrenes  $^{2}$  are mainly transmitted by  $^{1}$  electrons. However, the large spin coupling constant to C-4 in these compounds could also



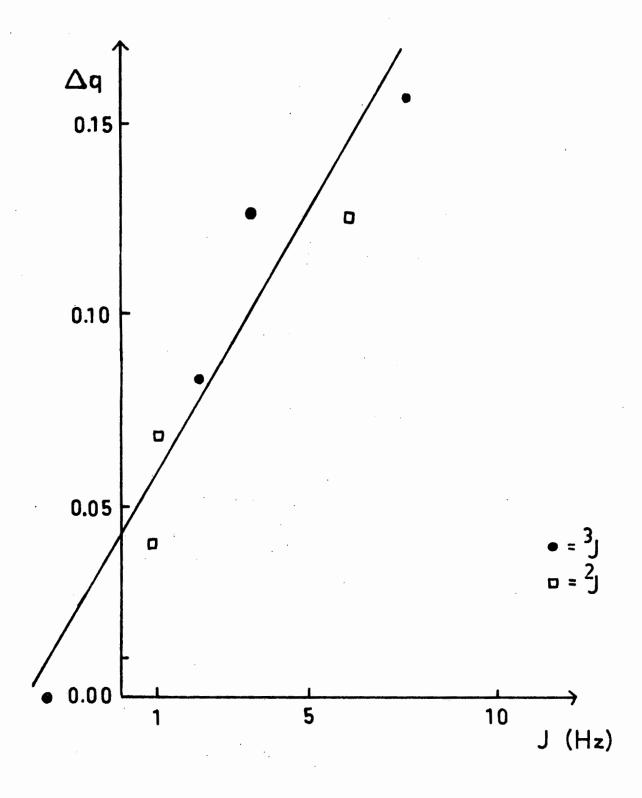
result from an addition of the spin coupling transmitted through the skeleton by two similar pathways. To get further insight into the mechanism of spin coupling in aromatic compounds we have synthesized azulenes 2 (R=H,CH<sub>3</sub>) with <sup>13</sup>C at C-4. These compounds provide a critical test for the reachanism since they exhibit four independent and unequivocal vicinal coupling constants. In the figure a plot of these coupling constants vs the reachance differences between the 4-azulenylmethylradical and the 4-azulenylmethylanion <sup>3)</sup> from a PPP calculation is given. These results clearly suggest the dominance of the rechanism for <sup>13</sup>C<sup>13</sup>C spin coupling in aromatic compounds.

- 1)S.Berger and K.P.Zeller, J.C.S.Chem.Comm. 1975, 423
- 2)S.Berger and K.P.Zeller, Org.Magn.Res. <u>11</u>, 303 (1978)
- 3)S.Berger and K.P.Zeller, to be submitted, preprints available

Sincerely yours

Chola. max

(University Tubingen)





#### UNIVERSITY OF GRONINGEN DEPARTMENT OF PHYSICAL CHEMISTRY

ZERNIKELAAN, PADDEPOEL, GRONINGEN, THE NETHERLANDS

Tel.: 050 -

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

GRONINGEN. August, 14th 1979

"Photo-CIDNP of Nucleotides - Postdoctoral position available".

Dear Barry,

In my last letter<sup>1</sup> I described an application of the laser photo-CIDNP method to the protein ribonuclease.<sup>2,3</sup> We have since looked at a variety of proteins. The method seems to work fine; the enhancements are large and specific and yield a lot of information on the surface structure of proteins. Furthermore, it aids in making NMR assignments of specific amino acid residues.

It may be of interest that we have now found that the same trick can be used to study nucleotides as well. The Figure shows the photo-CIDNP difference spectrum of adenosine-5'-monophosphate (AMP) in the presence of flavin as a sensitizer. Enhanced absorptions are observed for the H(8) and H(2) protons of the adenine ring. A similar effect is observed for the H(8) proton of guanine in GMP. The CIDNP effects arise from reversible electron-transfer from the nucleic acid bases to triplet flavin, photo-excited by laser irradiation in the probe of the Bruker HX-360. As in the case of the amino acids the photo-reaction is highly reversible and hopefully can be used to generate CIDNP in naturally occurring polynucleotides. We are currently extending the experiments to larger nucleotides and trying to determine the effect of base pairing and base stacking on the CIDNP intensities.

#### Postdoctorial position

A postdoctor/al position is available in my laboratory for a one year period renewable for another year. The work involves NMR studies on biological macromolecules, in particular further development and applications of the laser photo-CIDNP method. Candidates should have a strong background in biophysics or biochemistry and an interest in applying optical techniques in NMR.

The annual salary is in the range \$ 19500 - 2800**9** depending on experience. Potential candidates should send me a resume and arrange to have forwarded three letters of recommendation.

Best wishes,

yours sincerely,

Robert Kaptein

Please credit this contribution to the account of W.D. Weringa.

1. TAMU NMR Newletter 238, 1 (1978).

2. R. Kaptein, in "NMR in Molecular Biology", B. Pullman (Ed.), Reidel, 1978, p. 211.

3. B.G.J.M. Bolscher, J.A. Lenstra and R. Kaptein, J. Magn. Res., 35, 163 (1979).

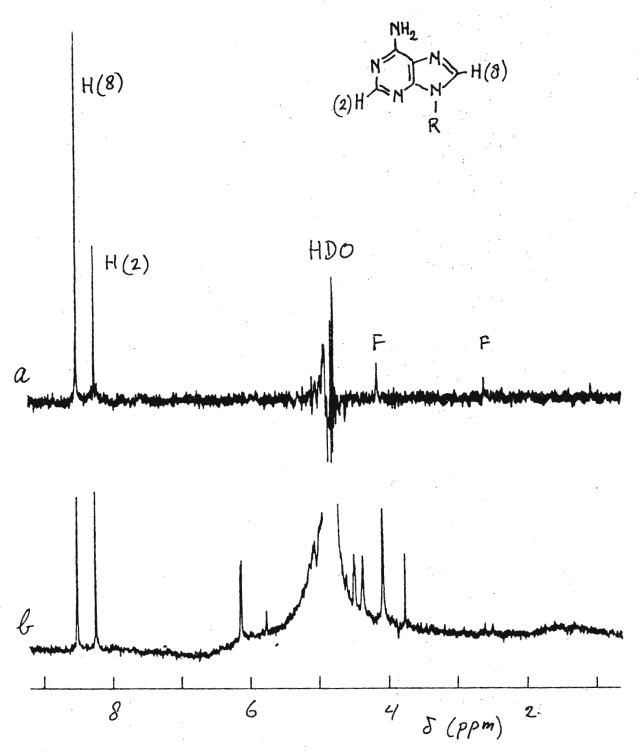


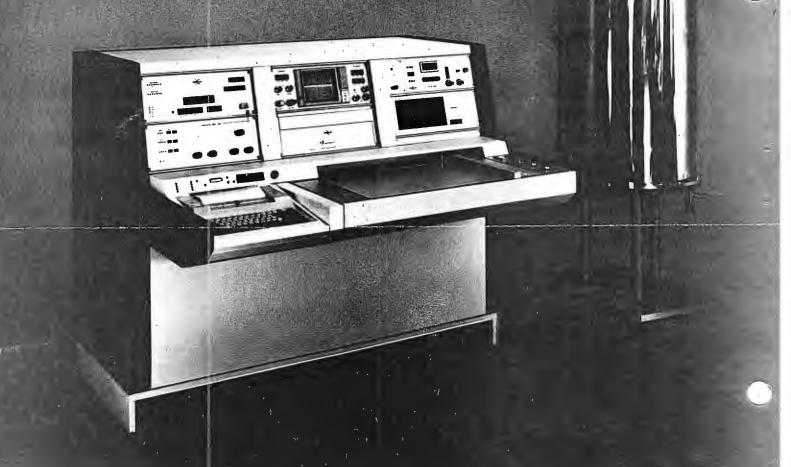
Figure (a) 360 MHz photo-CIDNP difference spectrum (light minus dark) of 5 mM AMP (R=ribose-5-phosphate) and 0.4 mM flavin in  $\rm D_20$ , pH 6.2,  $\rm 22^{oC}$ ,  $\rm 10$  scans.

(b) dark spectrum lines indicated by F belong to the flavin. The light spectrum was obtained by irradiating the sample by 0.6 sec light pulses from an argon laser prior to data acquisition.



BRUKER announces a new landmark in low-cost high-resolution superconducting NMR spectrometers with

<sup>1</sup>H frequency of 250 MHz





RESEARCH CENTER: P.O. BOX 26583, RICHMOND, VIRGINIA 23261 TELEPHONE (804) 271-2000

August 6, 1979

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

Dear Barry,

The Philip Morris Research Center is seeking applications from highly qualified NMR spectroscopists to fill a position in the NMR Laboratory. The applicant should have a Ph.D. in Chemistry; postdoctoral experience is preferred, but not essential for consideration.

The NMR Laboratory is presently equipped with a multinuclear Varian XL-100 and a Bruker WP-80. We are involved in a wide variety of fundamental research problems; to mention a few: conformation of naturally occurring alkaloids,  $^{1\,3}\text{C}$  and  $^{2}\text{H}$  T<sub>1</sub> investigations of molecular motion, kinetics and mechanism of reactions.

Interested applicants should contact me directly.

Sincerely,

T. P. Pitner, Ph.D.

Research Scientist

TPP/bpp

# University of Illinois at Urbana-Champaign

School of Chemical Sciences
Urbana, Illinois 61801

July 25, 1979

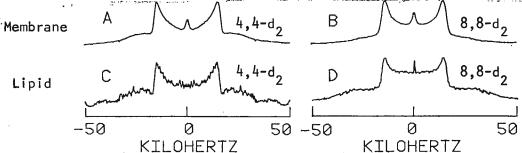
Professor B. L. Shapiro Department of Chemistry Texas A & M University College Station, TX 77843

Dear Barry:

### <sup>2</sup>H and <sup>31</sup>P NMR of Membrane Systems

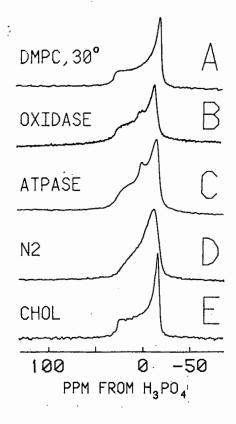
We have recently been investigating protein-lipid interactions in a variety of model and intact biological membranes, using  $^2{\rm H}$  and  $^{31}{\rm P}$  NMR spectroscopy.

We have found surprisingly small differences between the <sup>2</sup>H NMR spectra of (protein-containing) intact cell membranes and (protein-free) total lipid extracts of the micro-organism <u>Acholeplasma laidlawii</u> B (PG 9), grown on specifically <sup>2</sup>H-labelled tetradecan-l-oic acids in the presence of the fatty acid synthesis inhibitor avidin, as shown in the accompanying Figure.



This result may be a reflection of the presence of lipid-rich regions in the membrane and the relatively high lipid content of the membrane. We also found that these cells grew rapidly even with  $\sim 95\%$  of their lipids in the crystalline gel-state!

Our  $^{31}P$  NMR studies (at 60.7 MHz) have concentrated on investigating the "role" of the phospholipids polar headgroup in protein-lipid "interactions." We find that in most instances protein-phospholipid interactions are characterized by considerably enhanced  $^{31}P$  T<sub>1</sub> and T<sub>2</sub> relaxation rates, presumably a result of an immobilization of the phosphate group due to polar group protein-lipid interactions. There are only minor changes in the  $^{31}P$   $\Delta\sigma$  values. Typical results are shown in the accompanying Figure.



These results are the first to demonstrate by NMR the likely importance of protein-lipid polar group interactions leading to lipid immobilization.

Oldheld

Sincerely yours,

S. Y. Kang

S. Rajan

H S Gutowsky

Bob Kinsey

Eric Oldfield

# NEW England Nuclear®

August 3, 1979

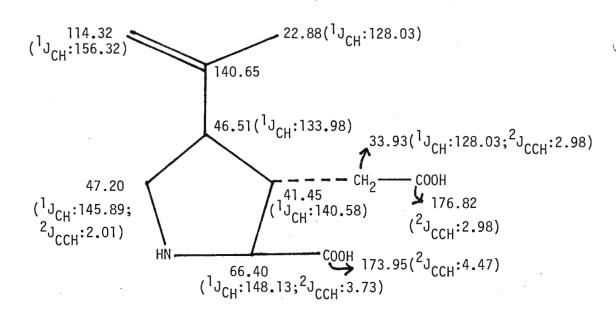
Professor B. J. Shapiro Dept. of Chemistry Texas A & M University College Station, TX 77843

# $\frac{\text{Carbon-13 NMR Spectral Parameters of}}{\text{L-}\alpha\text{-Kainic Acid}}$

Dear Barry:

hist Film Dave akerd

In connection with some of our work on the synthesis of radio-labelled L- $\alpha$ -kainic acid and its derivatives, we needed to study their  $^{13}\text{C}$  NMR spectral characteristics. Given below are the  $^{13}\text{C}$  chemical shifts and the C-H coupling constants that could be extracted for L- $\alpha$ -kainic acid; assignments have been made unambiguously utilizing the general theories of  $^{13}\text{C}$  chemical shifts and the C-H coupling patterns in spectra where proton decoupling is gated to retain NOE and C-H couplings:



[Spectra were run in  $D_2D$  with 1,4-dioxane as internal standard; chemical shifts converted to TMS scale using  $\delta$  dioxane =  $\delta$  TMS + 67.40;  $\underline{J}$  values are in Hz].

Sincerely,

Crist Filer Dave Ahern Richard Seguin P.R. Srinivasan

Larry Thomas

ANALYTICAL RESEARCH

August 10, 1979 Ref: A/R 2528

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

"13C SPECTRUM OF QUINOXALINE MONOXIDE"

#### Dear Barry:

As part of a program to establish a spectroscopic method for distinguishing 2-substituted quinoxaline oxides from the 3-isomer, the <sup>13</sup>C nmr spectrum of quinoxaline monoxide was measured and the values are indicated in Table 1. The substantial difference in the chemical shifts of C-2 and C-3 indicated that these resonances could be used for this purpose. In several alkyl, aryl and acyl derivatives in which isomers were available, the resonance for C-2 was always less than 140 ppm whereas the shift for C-3 was greater than 145 ppm. In addition, <sup>1</sup>J<sub>CH</sub> for C-2 was approximately 192 Hz in all cases whilst for C-3 the <sup>1</sup>J<sub>CH</sub> was closer to 187 Hz.

Sincerely,

Mike

Michael L. Maddox

TABLE 1

1 3 C NMR DATA FOR QUINOXALINE OXIDE<sup>a</sup>

a) Measured at 22.62 MHz in dimethylsulfoxide-d6,  $305^{\circ}$ K, digital resolution 0.73 Hz/pt.

#### UNIVERSITY OF CALIFORNIA, SAN DIEGO

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SANTA BARBARA · SANTA CRUZ

DEPARTMENT OF CHEMISTRY, B-017 LA JOLLA, CALIFORNIA 92093 August 14, 1979

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

TITLE: "Extending the useful life of the worn and torn Teletype: an electronic keyboard and interface for NTCFT on Nicolet 1080-NIC-80 computers"

Dear Professor Shapiro:

The Teletype ASR- or KSR-33 terminal is a common terminal for use with a mini-computer running a pulse-FT nmr spectrometer, but it has a number of disadvantages. It is slow, noisy, and under heavy usage, likely to wear out and malfunction.

The "control S" mode of Nicolet's NTCFT software can be used to bypass the speed and noise problems whenever hard copy is not necessary (most of the time, in our experience). The teletype must be left running, which means that much of it is still wearing out, even though only the keyboard is in use.

For about \$100 a solid-state keyboard can be purchased and interfaced to a Nicolet 1080 or NIC-80 computer; the TTY can then be left off, giving it a complete rest except during the (usually) short intervals when some sort of permanent record is desired. We have used two different keyboards: from Electronics Warehouse, Inc., 15820 Hawthorne Blvd., Lawndale, CA 90260 (\$77 kit, \$93 assembled, metal enclosure \$27.50), and George Risk Industries, GRI Plaza, Kimbal, Nebraska 69145, (GRI 753, \$72 assembled, plastic enclosure \$15, available from NCE/Compumart and other computer suppliers). Both provide full ASCII encoding, with upper-case only selectable (necessary for use as a TTY substitute).

A schematic of a keyboard interface to the Nicolet computer, using a UART (AY-5-1013A, TR 1602, COM 2502, etc.) for the necessary parallel to serial conversion, is shown below. We brought the necessary connections out the unused accessory power connector in back of the NIC-80 computer; either the unused "I/O device 2" or 80-pin "R2" connectors can be used on the 1080. The computer signals FDATA-and ENABTTY- are easily accessible on the wire-wrap bed at the rearmost 80-pin connector of board 11, on pins 42 and 60 respectively. (Board 11 is the outermost, short board.) The CLOCK signal is clearly labelled on board 11 and can be taken off at an unused pin of either the 80-pin wire-wrap connector, or of the 17-pin connector at the rear of the board. Ground, +5 and -15 volts are easily accessible at a number of places inside the computer.

To use the keyboard, either the Teletype must be disconnected (inconvenient), or the Nicolet interface card inside the Teletype must be modified slightly by cutting

August 14, 1979

the vertical foil trace between the blue/white wire at the top of the card and the diode directly beneath it. A 510 ohm 2 watt resistor is then soldered across the cut. (This card is easily accessible when the plastic cover is lifted off the Teletype.)

Please credit this contribution to Dr. Ed Dennis' account.

John Wright

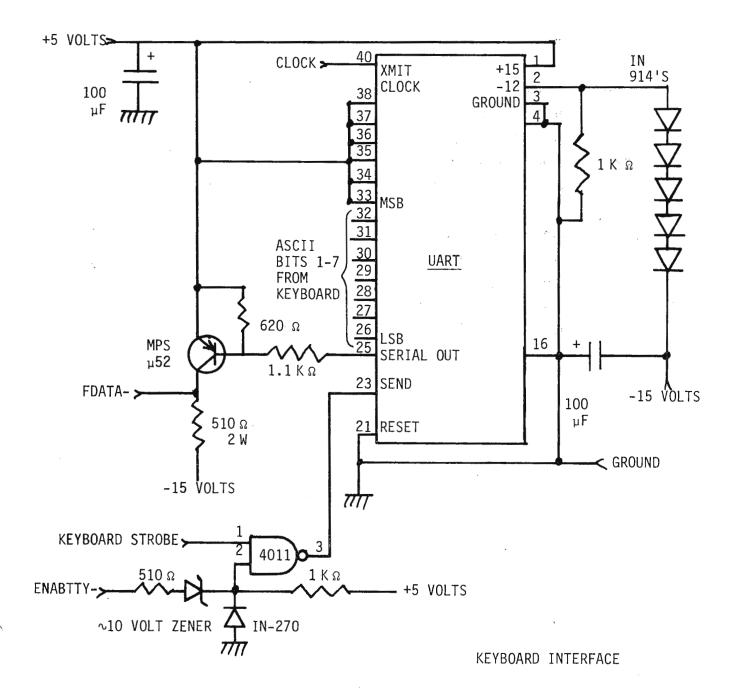
Operations Director

UCSD NMR Research Resource

Stephen aubent

Stephen Aubert

UCSD NMR Research Resource





# The University of Sydney

Department of Organic Chemistry N.S.W. 2006

Professor B.L. Shapiro Department of Chemistry Texas A and M University College Station Texas 77843 USA TELEPHONE: 692 1122.

17 August 1979

Dear Barry,

#### NON-BONDED INTERACTIONS FROM DYNAMIC NMR RESULTS

The relationsip between the magnitudes of repulsive non-bonded interactions (steric hindrance) and the size of interacting groups is known to be complex  $^{l}$  as it depends not only on size and energy-penalty but also on the geometry of the system.

The properties required from a system which would be useful in such investigations can be specified in terms of certain design requirements and we have synthesised about 40 compounds in series (1) for this purpose. It should be obvious that under conditions of slow exchange the prochiral geminal methyl groups should give rise to separate signals in  $^1\text{H}$  or  $^{13}\text{C}$  NMR spectra and hence it should be possible to obtain the activation parameters for the inversion process. In practice there are problems connected with small and inherently temperature-dependent values of  $\Delta v_{\text{Slow}}$ , but painstaking work gave accurate values for  $\Delta G^{\not=}$  for all compounds in the series, which proved to be isoentropic.

Indeed, as expected from original reasoning, the energy penalty for groups of well-defined size gives a smooth monotonic curve when plotted against the van der Waal's radii<sup>2</sup> of X. This permits the evaluation of effective van der Waals radii of less symmetrical groups and, via arguments too long to detail here, leads to the surprising conclusion that the system behaves like an elastic material (stress is proportional to strain).

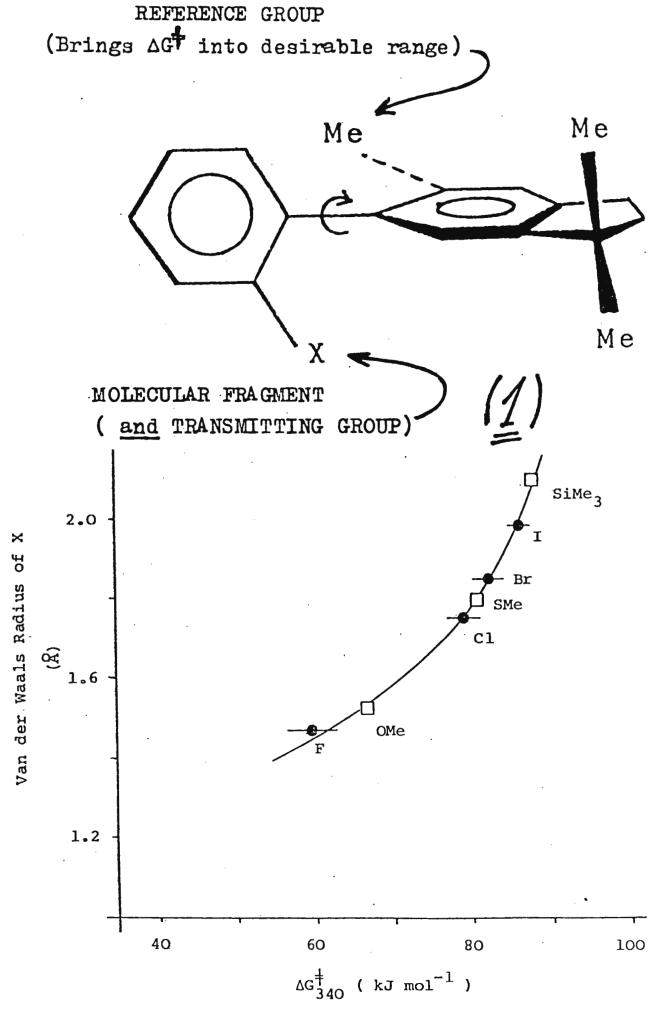
This work was carried out by Dr L.D. Field and Mr G. Bott and will be submitted for publication shortly.

Yours sincerely.

. Sternell

<sup>1</sup>Förster and Vögtle, Angew.Chem.Int.Ed., 16, 429, (1977)

<sup>2</sup>Bondi, J.Phys.Chem., 68, 441, (1964) and





U.S. Department of Energy Laramie Energy Technology Center P.O. Box 3395, University Station Laramie, Wyoming 82071

Professor Bernard L. Shapiro Department of Chemistry TAMU NMR Newsletter Texas A&M University College Station, Texas 77843 August 20, 1979

13C Chemical Shifts Assignments of Gammacerane Isolated from Oil Shale Bitumen

Dear Barry:

The organic materials found in sedimentary deposits, such as shales, are believed to be of biological orgin. Evidence for such a belief is the identification of "biological markers" which are organic compounds that have retained enough of their chemical and structural characterization so as to be identified with compounds produced by familiar biosynthetic organisms. Cummins and Robinson have isolated a small amount of a pentacyclic triterpane from Green River oil shale bitumen which was identified by Hills and Whitehead using mass spectroscopy as gammacerane (C<sub>30</sub>H<sub>52</sub>). The gammacerane structure is shown in Fig. 1. Since the material is not readily available and the C information on saturate polycyclics is limited, I believe the following information may be useful to some readers.

Fifteen  $^{13}$ C resonances are observed which indicates that it has the C<sub>2</sub>v symmetry proposed 13 The carbon-13 chemical shift resonances are listed in Table I. The  $^{13}$ C shifts for podocarpane (Fig. 2) are also listed for comparison. At present the shifts have been assigned based only on comparison with the shifts recorded for podocarpane and a pentacyclic triterpenoid. Ring carbons 1 through 6 of gammacerane can easily be related to the analogous carbon atoms of podocarpane since little perturbation of these chemical shifts is expected when additional rings are added or when a CH<sub>3</sub> group is substituted at carbon position 8 in the podocarpane molecule. The CH<sub>3</sub>'s were assigned 15.95, 33.39 and 21.61 ppm since these were the closest resonances to the methyl shifts observed in podocarpane. The CH<sub>3</sub> carbon (13) at position 8 was assigned the value of 16.59 ppm. Carbon resonances for positions 7 through 10 were tentatively assigned the values listed in Table 1 by analogy to similar carbons in a pentacyclic triterpenoid. The only remaining carbon (position 11) was given the chemical shift value of 29.71 ppm. Off-resonance and partially relaxed Fourier transform techniques will be attempted in the near future to confirm the assignments.

Sincerely,

Daniel A. Netzel

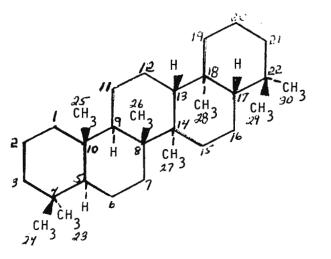


FIGURE 1

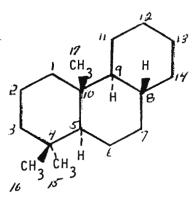


FIGURE 2

Table 1

Carbon Fig. 1	number Fig. 2	Gammacerane (1)	Podocarpane (2)	Difference
1,19		40.36	39.1	+1.26
2,20		18.74	19.0	-0.26
3,21		42.19	42.4	-0.21
4,22		33.28	33.3	-0.02
5,17		56.24	55.5	+0.74
6,16		21.18	21.8	-0.61
7,15		33.08	35.9	-1.82
8,14		41.97	36.8	+5.17
9,13		50.35	56.3	-5.95
10,18		37.38	36.9	+0.48
11,12	15	29.71	25.1	+4.61
23,29		33.39	33.6	-0.21
24,30 25,28 26,27	16 17	21.61 15.95 16.59	22.0 14.3	-0.39 +1.65

#### REFERENCES

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- 2. Hills, I. R., Whitehead, E. V., Anders, D. E., Cummins, J. J., and Robinson, W. E., Chem. Commun. 752 (1966).
- Wahlberg, I., Almqvist, S-O., Nishida, T., and Enzell, C. R., Acta Chem. Scan. <u>B29</u>, 1047 (1975).
- Ricca, G. S., Danieli, B., Palmisano, G., Duddeck, H., Elgamal, M.H.A., Org. Mag. Res. <u>11</u>, 163 (1978).

Case Institute of Technology

August 21, 1979

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

Dear Professor Shapiro:

Our spectroscopy laboratory has been interested for some time in structural studies of polyimides, primarily because of their excellent thermal stability and mechanical properties. With the recent development of a solid-state <sup>13</sup>C NMR spectrometer, we have applied cross polarization and magic angle spinning to several polyimides as well as relevant model compounds, all of which are insoluble in organic solvents. Results for N,N'-diphenylether pyromellitimide (left) and poly(diphenyl ether pyromellitimide) are shown below. Our goal is to use chemical shifts to obtain information concerning conjugation along the polymer chains. Preliminary results suggest increased conjugation as the diphenyl ehter tends toward coplanarity with the pyromellitimide subunit.

These spectra were recorded at a carbon frequency of 37.7 MHz on the Nicolet NT-150 solid-state spectrometer, modified for higher spinning speeds and higher decoupling power. Samples were spun in an Andrews type rotor machined from Kel-F, at a speed of 2.5 kHz. Dipolar decoupling of the proton spins was accomplished with a field of magnitude 20G, as determined by the 90° pulse width  $(t_\pi/2=(t_2\,\pi^-t_\pi)/2)$ . We have found that this amount of decoupling power is necessary for well-resolved spectra of the model compounds, which are essentially 100% crystalline. The spectra of the N,N'-diphenyl ether pyromellitimide reveals 12 resolved carbon resonances, indicating rotation of the phenyl rings about the ether oxygen. Linewidths are approximately 70Hz. The polymer spectrum shows considerable broadening of the peaks, caused by differences in conformation and packing along the polymer chain.

We have a position available for a post doctoral fellow, possibly developing into a research staff position, to add to the work being done in <sup>13</sup>C solid state NMR. The primary instrument would be our Nicolet 150 wide bore with cross polarization and MAS. Interested parties should write to me or phone (216) 368-4176.

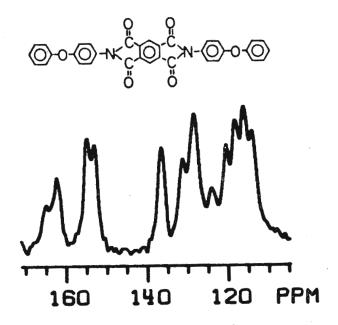
Sincerely yours,

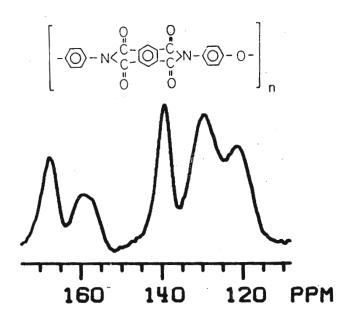
Jack L. Koenig

Professor of Macromolecular Science

and Physical Chemistry

JLK:bl





#### UNIVERSITY OF CALIFORNIA, DAVIS

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SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY

DAVIS, CALIFORNIA 95616

August 23, 1979

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

#### Proton Relaxation in Lanthanide Shift Reagents/ Postdoctoral Positions Available

Dear Barry,

A study of proton relaxation in lanthanide shift reagent complexes of the type,  $Ln(dpm)_3L_2$ , has shown that even the nonexchanging dpm (dipivaloylmethane) protons exhibit strong field-dependent linewidths, as shown in the figure. The field-dependence of linewidth is linear in  $H_0^2$ , and is present even in  $T_1$ 's, but to a lesser extent.

A similar field-dependence had been previously observed in high-spin ferrous hemoproteins, and interpreted as arising from the modulation, by the tumbling of the complex  $(\tau)$ , of the thermal equilibrium magnetization of the electron spin, and is known as the Curie-spin relaxation mechanism. The effect was expected to be important for molecules with large magnetic moments and a small value for the ratio  $\tau$ / $\tau$  ( $\tau$  = electron spin relaxation time). Thus the long  $\tau$  for a macromolecule can yield the appropriate condition.

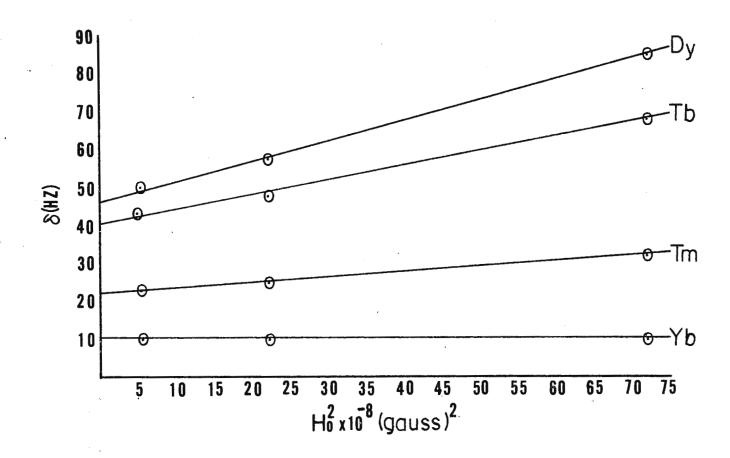
In the present shift reagent complexes,  $\tau$  is likely small in the non-viscous CDCl $_3$ , but  $\tau$  is known to be extremely short. The importance of the Curie spin mechanism increases with magnetic moment  $\mu(\text{eff})$  (see figure) as expected. Knowing the metal-t-butyl proton distance, the slopes of the lines in the figure can be used to determine  $\tau_r$ , yielding  $\sim\!1.4 \times 10^{-10}$  sec., which is very close to that predicted by the Stokes-Einstein relation.

The use of lanthanide ions to induce line broadening in enzymes and proteins suggests the possibility that in these macromolecules where  $\tau_{\rm c}$  is longer, the Curie spin mechanism may be even more important. If the binding site is known, the field-dependent contribution of the linewidth will lead directly to an estimate of  $\tau_{\rm c}$ .

Sincerely,

Serd N. La Mar Professor of Chemistry

GNL:qb



#### POSTDOCTORAL POSITIONS

Four postdoctoral positions, two in January 1980, and two in the Summer, 1980, will be available in my group. Work in one area involves nmr studies of peroxidases, cytochrome  $b_5$ , flavocytochromes, and the monomeric insect hemoglobins, particularly emphasizing protein dynamics, while the other area involves studies of metal-ion-induced dipolar relaxation as a structural probe in model system, proteins and peptide hormones. Available are both our new multinuclear wide-bore NT-200 and NT-360, to be upgraded to a NT-500 by the middle of 1980. The salary will be in the range \$9,500 - \$11,000, depending on experience. Interested persons should forward their C.V. and arrange to have sent to me two letters of reference.



#### DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

PUBLIC HEALTH SERVICE
NATIONAL INSTITUTES OF HEALTH
August 21, 1979

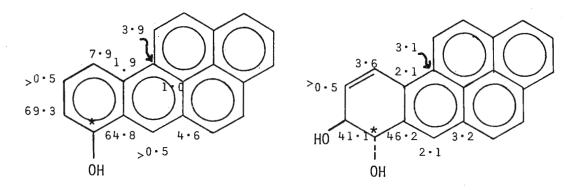
NATIONAL INSTITUTE OF ENVIRONMENTAL HEALTH SCIENCES P.O. BOX 12233 RESEARCH TRIANGLE PARK, N.C. 27709

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

Dear Barry:

#### <sup>13</sup>C-<sup>13</sup>C Coupling Constants in Benzo[a]pyrene Derivatives

With the continuing emphasis on  $^{13}\text{C}-^{13}\text{C}$  coupling constants,  $^1$  I thought you might be interested in the data that we have obtained for 7-hydroxy-benzopyrene-7- $^{13}\text{C}$  (I) and 7,8-dihydrobenzopyrene-7,8- $\frac{\text{trans}}{\text{color}}$ -diol-7- $^{13}\text{C}$  (II).



•

II

The magnitude of the coupling constants observed for I are similar to those observed for 1-hydroxynaphthalene-1-13C. We did not, however, observe (5000 Hz sweep, 32K FID) longer range coupling4into the other rings as has been observed for pyrene and phenanthrene derivatives.

The coupling constants observed for II are somewhat similar to those observed for benzyl derivatives. Examination of II with carbon-13 at the 10 position leads to the following couplings:  $J_{10}_{C-9}^{}$  = 66.3 Hz

and  $J_{10_{C-}6a_{C}} = >0.5 \text{ Hz}.$ 

Please credit this contribution to Dr. McKinney's account.

Sincerely yours,

Richard H. Cox

1. P. E. Hansen, Org. Magn. Reson., 11, 215 (1978). 2. S. Berger and K. P. Zeller, Chem. Commun., 423 (1975). 3. P. E. Hansen, O. K. Poulsen and A. Berg, Org. Magn. Reson., 7, 23 (1975). 4. S. Berger and K. P. Zeller, Org. Magn. Reson., 11, 303 (1978).



#### The Ohio State University

#### **Department of Chemistry**

140 West 18th Avenue Columbus, Ohio 43210 Phone 614 422-2251

August 24, 1979

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

Dear Barry;

Enclosed is a notice of a position in Dr. Berliner's group.

Yours sincerely,

Gi dem

Gideon Fraenkel Professor of Chemistry

GF:df

The proposed work involves magnetic resonance (NMR, ESR), fluorescence and chemical modification studies of two different enzyme systems as an approach to mapping their topography and characterizing their function(s) in more detail. Some enzyme isolation is also required.

- 1. Lactose synthetase: the enzyme galactosyl transferase has profoundly altered specificity when complexed in the mammary gland cellular environment with the "lysozyme like" protein,  $\alpha$ -lactalbumin. Our studies are attempting to define the structural changes induced in each subunit of this novel catalytic-regulatory two protein complex.
- 2. Human  $\alpha$ -thrombin: this key blood clotting enzyme in the coagulation cascade has several unique functions, many of which are yet to be defined in structural detail. While thrombin is very much a "trypsin-like" serine protease, its restricted specificity and yet unsolved crystal structure present a unique challenge to structural studies in solution.

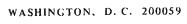
Starting date: September 15, 1979 or later. Salary: \$10,000/year (minimum) or up depending upon qualifications.

Interested applicants should forward a c.v. including copies of publications and two or three references to:

Dr. Lawrence J. Berliner
Department of Chemistry
The Ohio State University
120 McPherson Chemical Laboratory
140 West 18th Avenue
Columbus, Ohio 43210
U.S.A.

Telephone: 614-422-0134

#### HOWARD UNIVERSITY





DEPARTMENT OF CHEMISTRY

August 11, 1979

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

Dear Barry:

We have recently set up a Nicolet NT-200 spectrometer at the Chemistry Department at Howard University. The instrument is equipped with 20 mm 13C, 12 mm 31P and 5 mm 1H probes. We purchased the large 13C probe because of an interest in natural abundance 13C work on proteins and on some rather insoluble pyrimidines and pterins. We were curious as to whether we could use the 20 mm 13C probe with the 12 mm and 5 mm tubes without undue loss of sensitivity and/or resolution.

To test this we placed 200  $\mu$ l of dioxane in 7.0 ml of  $^2\text{H}_2\text{O}$ , tuned the cavity and shimmmed on the  $^2\text{H}_2\text{O}$  lock. With a single 90° pulse we obtained a signal to noise of 38:1, calculated by the sub-routine provided with the NTCFT program. The same amount of dioxane in the same height of  $^2\text{H}_2\text{O}$  in the 12 mm tube (2.8 ml) gave a signal to noise of  $^4\text{H}_2\text{I}$  and we obtained a somewhat narrower line. The same height of  $^2\text{H}_2\text{O}$  (0.5 ml) with 200  $\mu$ l of dioxane in the 5 mm tube also gave a signal to noise of  $^4\text{H}_2\text{I}$ .

The cavity was tuned and shimmed for each sample. For the 12 mm tube the lock signal was more than adequate for the shimming. For the 5 mm tube it was a bit noisy. It appears the for materials of relatively high solubility or low availability we can use the smaller tubes with no trouble. This will also save considerable expense on solvents when deuterated solvents are used.

Sincerely yours,

Carlyle B. Storm

Yohannes Teklu

## NT-150 A WIDE-BORE, FT-NMR SYSTEM FROM NICOLET





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- 31P experiments on living organs.
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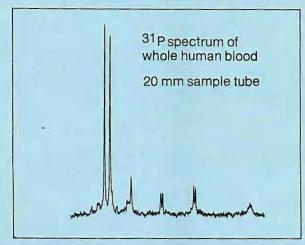
#### **FEATURES INCLUDE:**

- 3.5T superconducting magnet with 10 cm room-temperature bore.
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- Quick-disconnect probes for rapid changeover.
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- Nicolet 1180 data system with simultaneous acquisition, processing and plotting.
- Digital plotter with plot lengths selectable from 1 cm to 900 cm.

#### **OPTIONS INCLUDE:**

NT-150 MF: broad-band multi-nuclei observe for 4 to 60 MHz.

NT-150 CP: optimized system for Waugh-Pines cross-polarization studies.

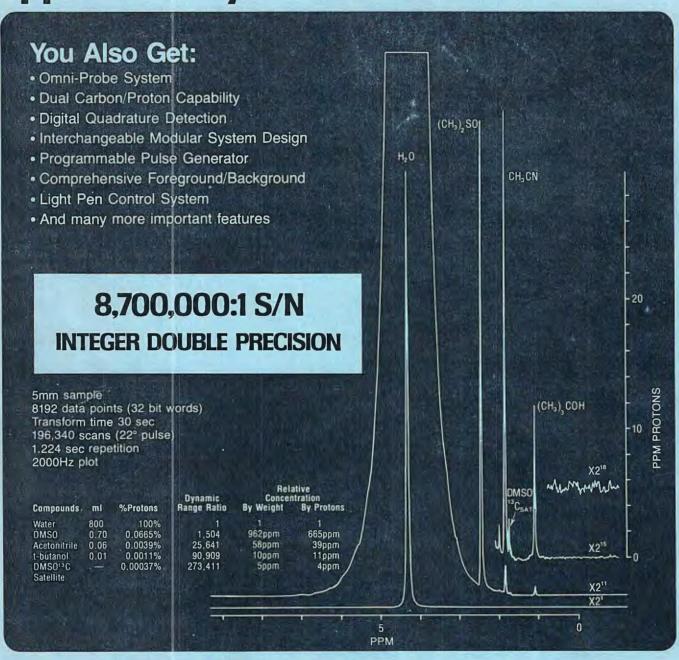


For more information or to discuss your applications, please telephone or write.



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Call or write for complete details...

