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NMR NEWSLETTER

NO. 312

SEPTEMBER 1984

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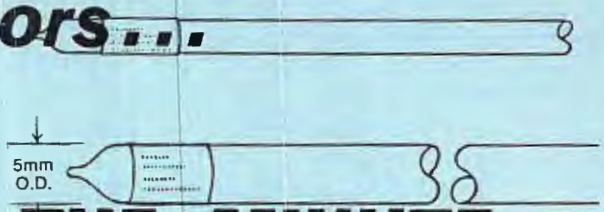

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	WGH-68	30% Orthodichlorobenzene in Benzene-d$_6$	Resolution	33.00
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TEXAS A&M UNIVERSITY
NMR
NEWSLETTER

All Newsletter Correspondence
Should be Addressed to:

Professor Bernard L. Shapiro
Department of Chemistry
Texas A&M University
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Prof. Dr. GERHARD HÄGELE
Institut für Anorganische Chemie
und Strukturchemie I
der Universität Düsseldorf
and Winfried Boenigk

4000 Düsseldorf, den May 17th, 1984
Universitätsstr. 1/26.42.U1.32
Telefon 0211 - 311-2288/2287
Telex 8 587 348 uni d

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

Automated Analysis of NMR Spectra

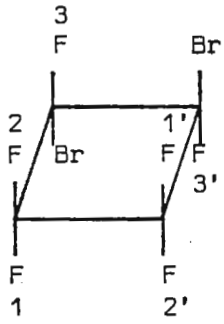
Dear Professor Shapiro,

our spectral synthesizer DSYMPLOT [1] was combined with principles of line-shape analysis (DAVINS [2]) to obtain DAVSYM [3] and DAVSYM2 [3]. These new programs allow a more efficient automated analysis of spectra in isotropic and anisotropic solvents using chemical equivalence in a general form to factorize the hamiltonian matrix. We can tackle systems up to 10 spins I=1/2 if there is sufficient symmetry factorization. While DAVSYM uses one coherent digitized line-shape, DAVSYM2 is able to omit irrelevant (empty) parts. Up to six spectral regions can be dealt simultaneously in one iteration.

We demonstrate our new comfortable tools with the ^{19}F-spectrum of trans-1,2-dibromohexafluorocyclobutane. The results confirm the time consuming analysis done by M. Engelhardt [4] using LACX.

Yours sincerely

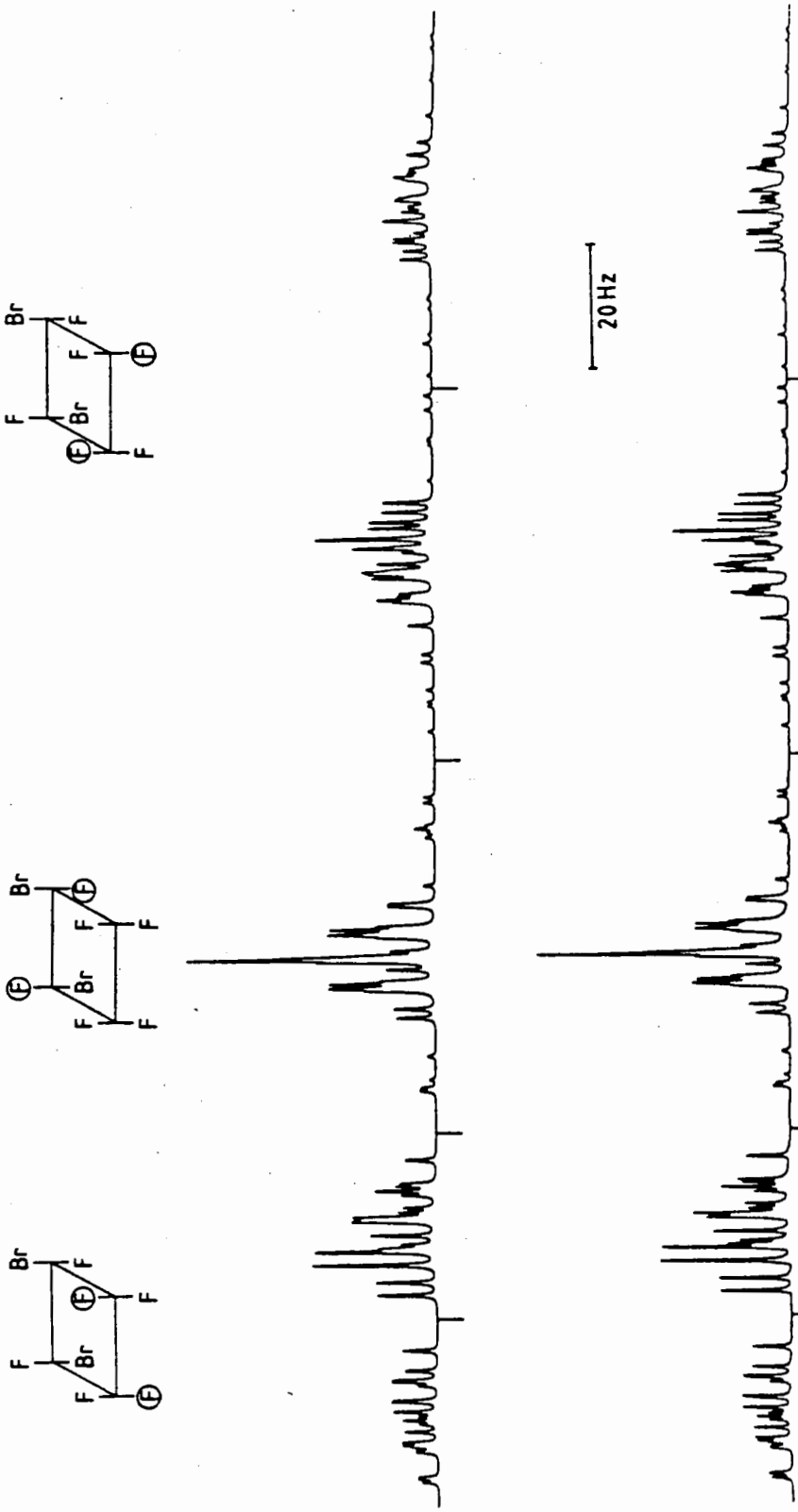
Winfried Boenigk



$[ABC]_2$-spin system

a) vs. C_6F_6
HW half width
DR digital resolution

	LACX	DAVSYM2	
ν_1	3926.099	3926.034(3)	Hz a)
ν_2	3291.189	3291.753(3)	Hz a)
ν_3	3540.658	3540.589(3)	Hz a)
$J_{11'}$	3.385	3.381(6)	Hz
J_{12}	213.803	213.737(4)	Hz
$J_{12'}$	-8.405	-8.424(4)	Hz
J_{13}	-5.219	-5.227(5)	Hz
$J_{13'}$	-4.916	-4.923(4)	Hz
$J_{22'}$	2.418	2.418(6)	Hz
J_{23}	-9.312	-9.290(5)	Hz
$J_{23'}$	9.160	9.122(5)	Hz
$J_{33'}$	-16.809	-16.88 (1)	Hz
HW	-	0.310(3)	Hz
DR	-	0.1	Hz/point
rms	3.5	3.33	%



84.665 MHz ^{19}F-spectrum of Trans-1,2-dibromohexafluorocyclobutane
upper trace: calculated spectrum with improved parameters
lower trace: experimental spectrum (five spectral regions)

References:

1) W. Boenigk and G. Hägele, QCPE Program No. 470 (1984)
2) D.S. Stephenson and G. Binsch, QCPE Program No. 378 (1979)
3) Dissertation W. Boenigk, Universität Düsseldorf 1984
4) Diplomarbeit M. Engelhardt, Universität Düsseldorf 1983

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Pr. Bernard L. SHAPIRO
TAMU NMR Newsletter
Texas A & M University
Dpt of Chemistry

COLLEGE STATION
Texas 77843 USA

N./Réf. 84 07 259 CB/MCH
V./Réf.

Wissembourg, le July 31, 1984

Dear Barry,

Spin playing, Tungsten NMR and inorganic chemistry :

It is well recognize now that multinuclear NMR plays an important role in organometallic and inorganic chemistry. Besides direct observation of the metallic center, a new and exciting area to be developped is the application of the entire pulse sequence artillery.

I present some interresting results obtained in the heteropolyanion field. The model compound is $[H_2W_{12}FO_{39}]^{5-}$, a Keggin type derived polyanion where one O_a oxygen has been replaced by one fluorine atom (black circle in the figure). The "normal" W183 spectrum of this compound (Fig 1A) shows the expected 3 resonances (1/2/1) with one resonance split by a $^2J_{W-O-F}$ coupling (32 Hz) (1).

Applying a $\{^{19}F\} \rightarrow {}^{183}W$ INEPT sequence, spectrum 1B is recorded in the same amount of time. One clearly sees AB type pattern of the homonuclear $^2J_{W-O-W}$ coupling of the fluorinated site (1).

It was tempting then to try, after the INEPT polarization scheme, a (W183) → W183 magnetization transfer from the 19F labelled tungsten to the homonuclearly coupled non fluorinated adjacent site at - 107 ppm, following R. Freeman original sequence on C13 (2).

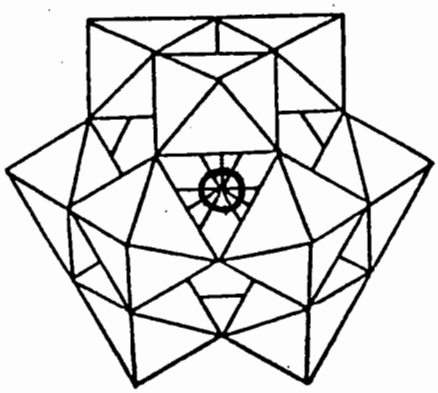
The idea and sequence came out nicely as shown in Fig. 1C you get a 1D, $\delta - \delta$ Tungsten correlated spectrum which gives at once the structural features around the fluorinated site. Extension to 2D, INEPT/ INADEQUATE is evident.

Best regards,

christian
C. BREVARD

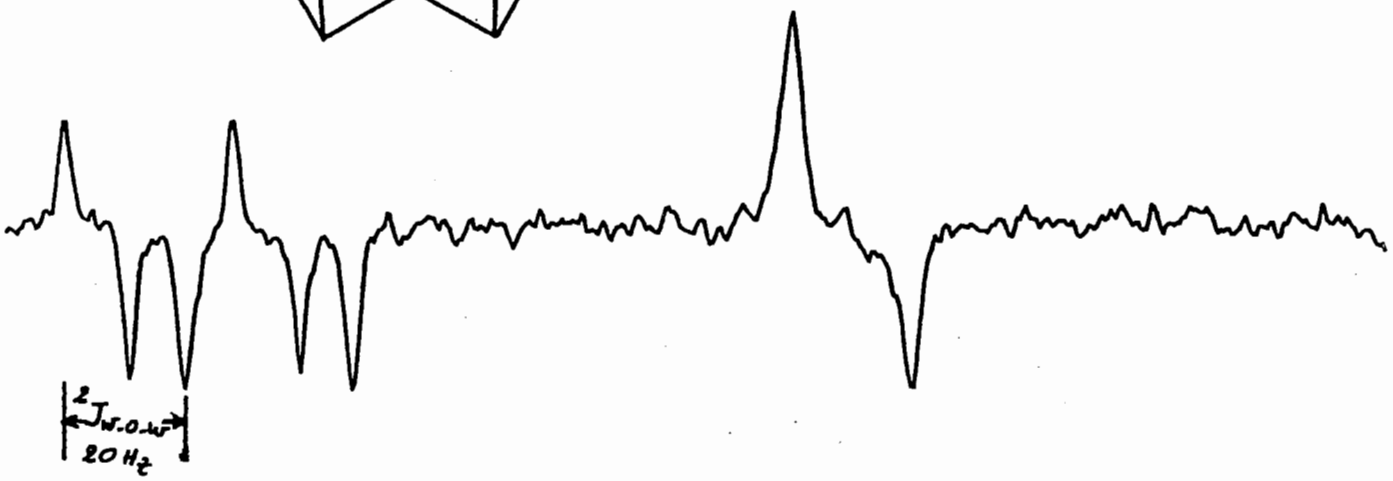
(1) J. Lefebvre, F. Chauveau, P. Doppelt, C. Brevard
JACS 103 4589 (1981)

(2) O. Sorensen, R. Freeman, T. Frenkiel, T. Marecci, R. Schuck,
J.M.R. 46 180 (1982)

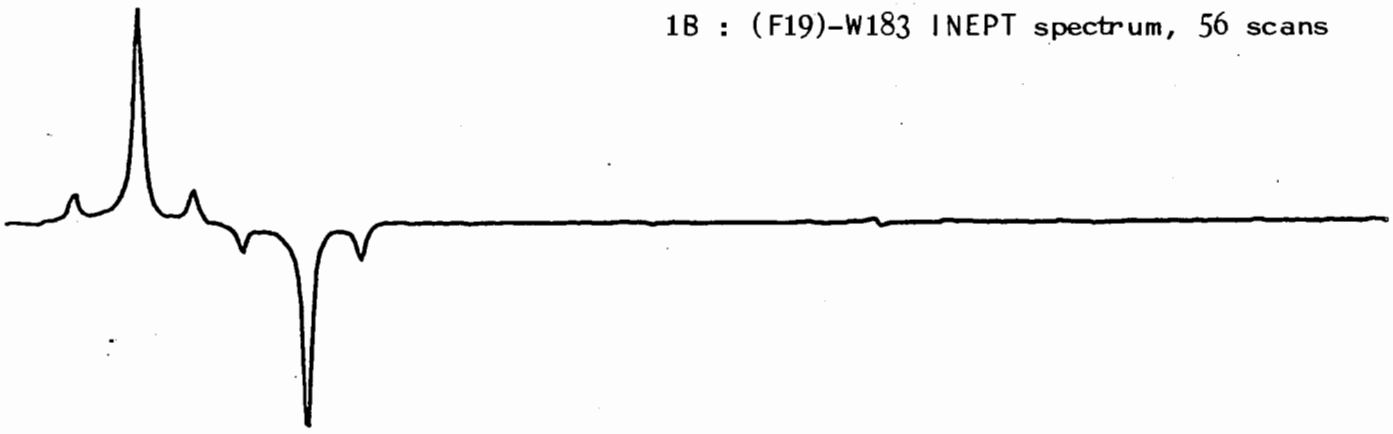


$$\left[H_2 \, W_{12} \, O_{39} \, F \right]^{5-}$$

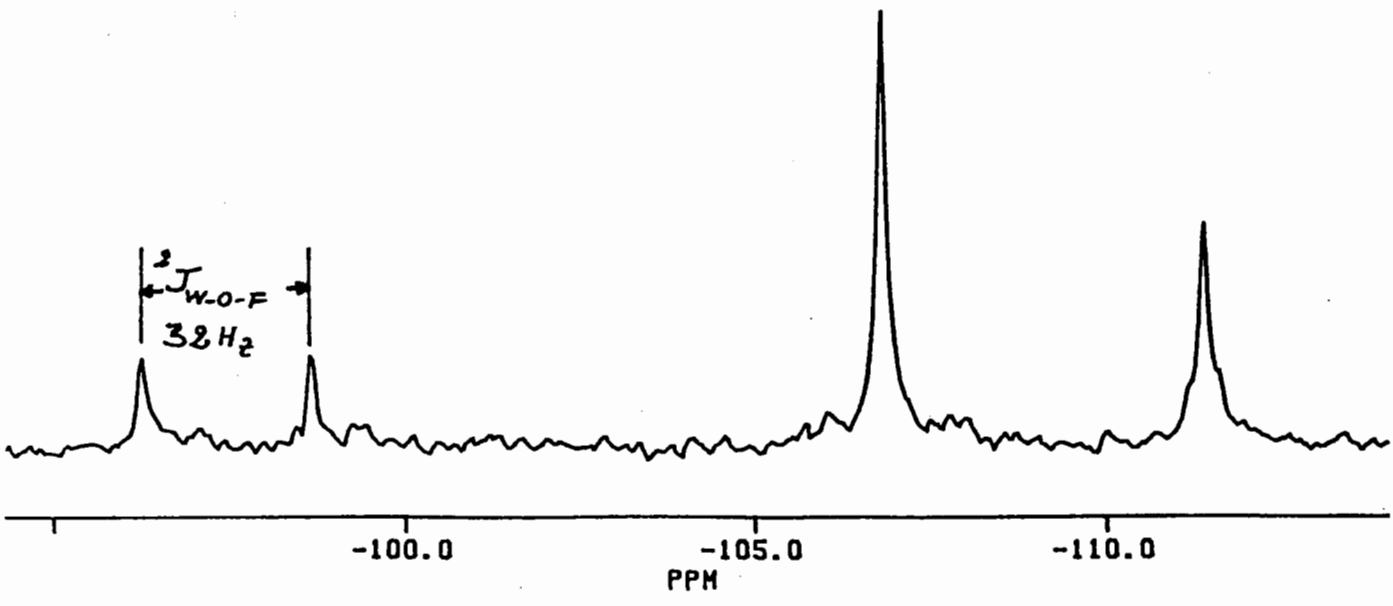
1C : (F19)-W183/(W183)→W183 INEPT/INADEQUATE
double transfer, 256 scans



1B : (F19)-W183 INEPT spectrum, 56 scans



1A : W183 spectrum, 56 scans



312-5

DEPARTMENT OF HEALTH & HUMAN SERVICES

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August 16, 1984

Professor Barry Shapiro
Texas A & M University
College Station, Texas 77843

Dear Professor Shapiro:

The Laboratory of Cellular and Molecular Biology will have another opening for an NMR spectroscopist, starting as early as October, 1984, or at a later date, depending on the availability of the candidate. We are presently conducting molecular studies on metal interactions with biochemical substances, and structural studies on nucleic acids and RNA polymerase, using a Varian XL-200 spectrometer, which is also used for cellular aging studies. We are about to have delivered an Oxford Research Instruments Biospec 1.9/300 spectrometer for in vivo studies on the aging of intact animals.

I should appreciate recommendation of suitable applicants as well as distribution of this information to such applicants.

With the very best regards.

Sincerely,

Gunther L. Eichhorn/alb

Gunther L. Eichhorn
Chief, Laboratory of
Cellular and Molecular Biology

GLE/alb

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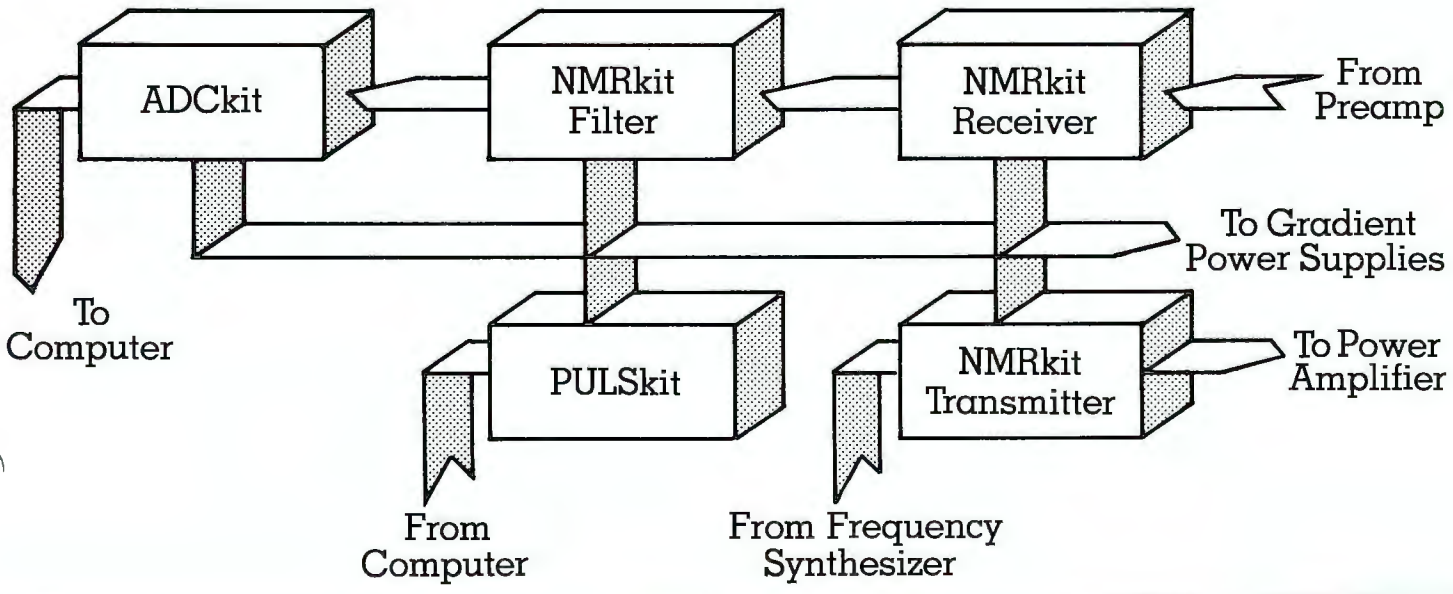

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—The **Filter** board is composed of two four-pole Butterworth or Bessel filters (one set of filters for each channel). The selection of the bandwidth and filter type (Bessel or Butterworth) is under computer control.

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

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—A 16-bit adder/subtractor to control the sign of the output signal

Each of these components is controlled by a separate bit in the control word. This simplifies the acquisition software and allows maximum flexibility in the choice of the acquisition mode.
Controlled directly by the pulse programmer, the **ADCkit** offers an elegant solution to fast acquisition of NMR signals.
For maximum convenience, a 12-bit Digital-to-Analog converter is also included on the board.

The **DECkit-2** is a fully broad-banded decoupler: it allows homo- or hetero-decoupling (WALTZ-16) on any nucleus from 3.5 MHz to 80 MHz. Optional ranges (2–32 MHz, 7–160 MHz or 50–400 MHz) are also available. In order to operate properly, it requires an external frequency synthesizer as well as an appropriate broadband power amplifier.

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August 23, 1984

Professor Bernard L. SHAPIRO
Texas A&M University
College of Science
COLLEGE STATION, Texas 77843
U.S.A.

RUNNING TITLE: ^{31}P T_1 VALUES

Dear Professor Shapiro,

Although ^{31}P NMR is now relatively routine, there is surprisingly little known with respect to spin-lattice relaxation times for this nucleus.

We have recently measured T_1's for the triarylphosphines, PR_3, R = p-XC_6H_4 as well as for several transition metal comlexes containing these ligands. Interestingly, the values for a given PR_3 change significantly (see below). Since we know that T_1 (dipole-dipole) is decreasing with increasing molecular weight of the complexes, T_1 measurements might prove an interesting way to estimate the size of the fragment to which the phosphine is complexed. Further work is in progress.

Please credit this contribution to the account of Prof. L.M. Venanzi.

Sincerely

Paul Pregosin

Compound	T_1, sec.
PPh_3	26
$AuCl(PPh_3)$	16
$IrCl(CO)(PPh_3)_2$	9

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DEPARTMENT OF BIOLOGICAL CHEMISTRY

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August 1, 1984

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

Measurement of Absolute Interproton Distances By Frequency-Dependent Nuclear Overhauser Effects.

Dear Barry:

We have been studying the conformation of MgATP bound to adenylate kinase, and to a 45-amino acid synthetic peptide (residues #1-45) which binds MgATP with comparable affinity (Biochemistry 23, 3357 (1984)). Conformations are determined by acquiring a set of distances among the H1', H2', H3', H4', H5', H2 and H8 protons of ATP. These interproton distances (r_{AB}) are obtained by measuring NOEs between protons A and B using the two-spin equation

$$r_{AB} = D[(\frac{1}{\sigma_{AB}})(\frac{6\tau_r}{1 + 4\omega_I{}^2\tau_r{}^2} - \tau_r)]^{1/6} \qquad [1]$$

in which D is a constant, σ_{AB} is the cross-relaxation rate, τ_r is the correlation time, and ω_I is the proton precession frequency. We study the time-dependent development of the NOEs in order to more accurately calculate σ_{AB}, and to distinguish primary from secondary effects.

Equation [1] contains two unknowns, r_{AB} and τ_r. Hence measurements of τ_{AB} made at one frequency permit the determination of only relative interproton distances. Relative distances may be converted to absolute distances by using as a standard a pair of protons in the system whose distance apart is known and invariant with conformation. For ATP an invariant distance is approximated by that between H2' and H1', which remains within the range 2.9 ± 0.2Å regardless of conformation, as shown by x-ray and model building (Biochemistry 22, 3439 (1983)).

We have recently tried the novel and more rigorous approach of experimentally measuring absolute interproton distances by examining the frequency dependence of σ_{AB}. From equation [1] it can be seen that measuring σ_{AB} at two values of ω_I yields both unknowns, τ_r and r_{AB}. The data of Table I indicate that σ_{AB} values for peptide-bound ATP are lower at 500 MHz than at 250 MHz, in accord with equation [1]. Moreover, the correlation time determined from these data (2.3×10^{-10} sec) yields an appropriate value of 3.05 Å for the "invariant" distance from H2' to H1'. The complete set of distances led to an acceptable conformation for peptide-bound MgATP. Therefore, calculating τ_r from the frequency dependence of σ appears to be a valid technique, and should be

applicable to other systems, providing the correlation time is of the proper magnitude for the available frequencies. For a comparison between 250 MHz and 500 MHz, a suitable range would be approximately 1-9 x 10^{-10}s. The method should be particularly useful for samples which lack an invariant reference distance.

Sincerely,

Dave Al

David C. Fry Albert S. Mildvan

TABLE I. Calculation of τ_r and r_{AB} from the Frequency Dependence of Selected NOES

Peptide + MgATP

NOE	$\sigma_{250\ MHz}$	$\sigma_{500\ MHz}$	$\sigma_{250}/\sigma_{500}$	τ_r(s)	r (Å)
H2' to H8	0.080	0.025	3.2	2.3×10^{-10}	2.70
Peptide resonance (2.07 ppm) to H8	0.030	0.0088	3.4	2.4×10^{-10}	3.18
Peptide resonance (1.22 ppm) to H8	0.031	0.0096	3.2	2.3×10^{-10}	3.16
H2' to H1'	0.039	$\leq$0.012	$\geq$3.2	$(2.3 \times 10^{-10})^a$	3.05

[a]Assumed

CHEMICAL INSTITUTE
UNIVERSITY OF AARHUS

DEPARTMENT OF ORGANIC CHEMISTRY
HANS JØRGEN JAKOBSEN

8000 Århus C, Denmark
Telephone (06) 12 46 33

August 23, 1984
HJJ/EL

Professor BERNARD L. SHAPIRO
Department of Chemistry
Texas A and M University
COLLEGE STATION, TX 77843
USA

SEMINA : SEMUT Editing of INADEQUATE ^{13}C NMR Spectra

Dear Barry,

Recently we have evaluated the usefulness of incorporating SEMUT spectral editing techniques (1) into the INADEQUATE (2) experiment. A number of pulse sequences for this purpose may be thought of. The SEMINA pulse sequences edit $^{13}CH_n$-$^{13}CH_m$ fragments of INADEQUATE spectra according to the number of attached protons. ^{13}C-^{13}C double quantum coherence behaves very similar to ^{13}C single quantum coherence when subjected to a SEMUT editing sequence in that $^{13}CH_n$ - $^{13}CH_m \sim {}^{13}CH_{n+m}$. This is the basis for the SEMINA-1 pulse sequence which can decompose an INADEQUATE spectrum into 7 subspectra (n+m = 0,1, ..., 6). The SEMINA-2 sequence, which contains two editing steps, combines SEMINA-1 with normal SEMUT to provide a further decomposition into subspectra. The editing Θ = 0°,180° pulse variants of these sequences are especially useful in practical work since they do not significantly degrade the sensitivity of the experiments compared to INADEQUATE. Thus, the total time devoted to a usual INADEQUATE experiment may profitably be divided into the two and four subexperiments required for these variants of SEMINA-1 and -2, respectively. These experiments considerably simplify the assignment and accurate measurements of ^{13}C-^{13}C coupling constants and the determination of C-C connectivities compared to 1D INADEQUATE. In the Θ = 0°,180° version of SEMINA-2 the first editing pulse Θ_1 separates the $^{13}CH_n$-$^{13}CH_m$ fragments according to n+m being even or odd and the second editing pulse Θ_2 further discriminates according to n and m being even or odd giving a total of four subspectra. An illustrative example is shown in the accompanying Figure. Detailed reports on this work should appear in Journal of Magnetic Resonance in the near future.

Sincerely yours,

HANS J. JAKOBSEN ULLA B. SØRENSEN OLE W. SØRENSEN
ETH, Zürich

1. H.Bildsøe, S.Dønstrup, H.J.Jakobsen, and O.W.Sørensen, J.Magn.Reson. 53, 154 (1983); O.W.Sørensen, S.Dønstrup, H.Bildsøe, and H.J.Jakobsen, J.Magn.Reson. 55, 347 (1983).
2. A.Bax, R.Freeman, and S.P.Kempsell, J.Am.Che.Soc. 102, 4849 (1980).

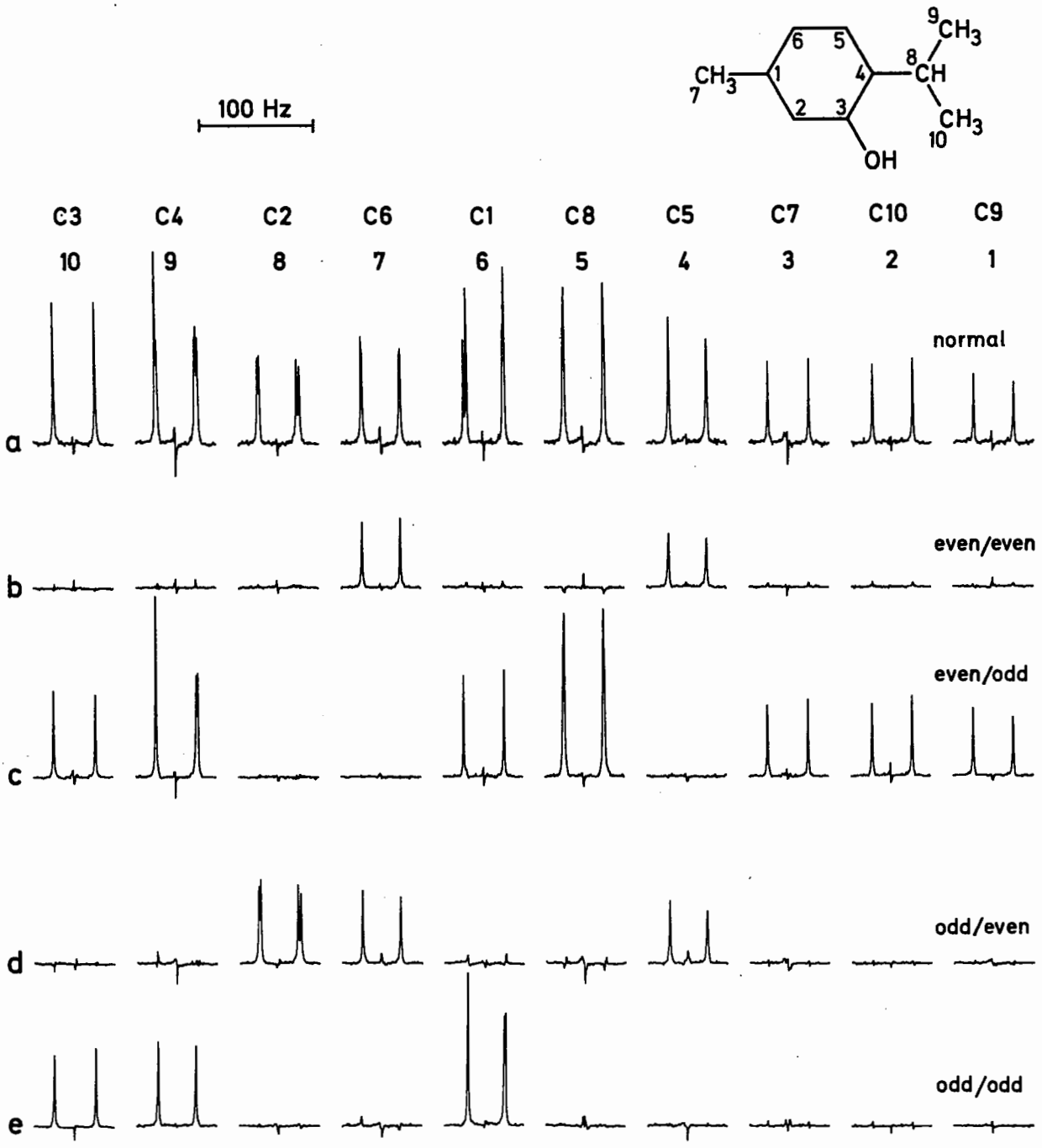


Figure. SEMINA-2 ^{13}C NMR spectra of menthol recorded on a Varian XL-300 spectrometer. For simplicity the double quantum excitation period was optimized only for the one-bond $^{13}CH_n$-$^{13}CH_m$ fragments. (a) $\theta_1 = 0°$, $\theta_2 = 180°$ subexperiment corresponding to a refocused INADEQUATE spectrum. (b-e) Edited ^{13}C-^{13}C satellite spectra with the vertical scale reduced by a factor of four compared to (a) and indicated by (n+m):even or odd/(n,m):even or odd. (b) Even/even, (c) even/odd, (d) odd/even, and (e) odd/odd subspectrum.

312-13

Department of Chemistry
University of Canterbury Christchurch 1 New Zealand

1 August 1984

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

Dear Barry,

Rotational Anisotropy of the 1-Adamantyl Cation

As you can see from the letterhead this response to your note of July 2nd is being written in scenic New Zealand where I am a guest of this Department prior to attending the IUPAC Physical Organic Chemistry Conference in Auckland on 20th August.

Following our earlier study of the relaxation behaviour of the t-butyl cation, we have investigated the 1-adamantyl cation, with the idea that whilst specific interactions between cation and counter-ion have not been indicated by chemical shifts and coupling constants (and thus the cations might be considered 'free'), such interactions may in principle be detected from T_1 data by the extent of anisotropic reorientation (ρ).[1]

1-Adamantyl cations were prepared as 0.25M solutions in SbF_5/SO_2 and SbF_5/SO_2 ClF from 1-adamantanol and the T_1 values determined at 213K. Application of the Woessner equations for an axially symmetric ellipsoid allowed us to determine best fit values of $D_\perp$ and $\rho(D_{11}/D_\perp)$.

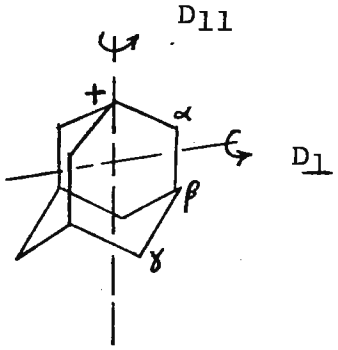
	$T_{1(\alpha)}^{DD}$ [a]	$T_{1(\beta)}^{DD}$	$T_{1(\gamma)}^{DD}$	ρ [b]
SO_2	3.8	6.6	3.6	1.0
SO_2ClF	2.2	3.8	1.9	1.7

[a] ±0.2 s. Values calculated from D_{11}, $D_\perp$ reproduced these values to ≤0.2 s.

[b] ±0.1

In SO_2 the 1-adamantyl cation reorients isotropically (ρ=1) consistent with a near-spherical solute not involved in a specific interaction with the surrounding media. However, significant anisotropy (ρ=1.7) occurs in the rotation of this cation in SO_2ClF indicative of specific interactions of the cationic carbon with the superacid medium.

Although we are unable to identify the exact nature of these interactions, they are independent of the nature of the leaving group. The anisotropy does not appear to be large enough to involve tight ion pairs but the results indicate that cations generated in superacids may not be completely 'free,' at least in some solvents. A similar suggestion was made by Ned Arnett from the results of his thermochemical studies of carbinols in superacids.[2]



Yours sincerely,

David Kelly

D.R. Leslie D.P. Kelly

Department of Organic Chemistry
University of Melbourne, Australia

1 Kelly, D.P.; Leslie, D.R.; Craig, R.A. J. Magn. Reson. 1983, 52, 480-491.

2 Arnett, E.M.; Hofelich, T.C. J. Am. Chem. Soc. 1982, 104, 3522-3524.

Book Review

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

Fourier Transform NMR Spectroscopy
by **Edwin D. Becker** (National Institutes of Health, Bethesda, Maryland)

American Chemical Society
Education Division
1155 Sixteenth Street, N.W.
Washington, D.C. 20036

Audio Course consisting of six audiotape cassettes (4.3 hours playing time) and a 90-page manual. Copyright 1983. $250.

Dr. Becker, who has written and/or co-authored two of the more successful NMR textbooks, has now turned his hand to the production of a new ACS audiotape course. This course on Fourier Transform NMR is designed for the beginning practioner who has had an introductory course on CW methods with spectral interpretation. It also serves those who have been away from the field for several years and now wish to find out what has been going on in the interim.

Divided into six sections covered in a little over four hours of listening time, it is obvious that the level of presentation must be highly descriptive and nonmathematical. The first section on the physical basis of NMR is a good review of fundamentals including the concept of the rotating frame of reference, nuclear relaxation, pulse NMR and the FID along with its Fourier transform. These subjects are dealt with in greater length in the second section, which also includes phase sensitive detection, relationships of spectral width, acquisition time, resolution and signal/noise, and decoupling.

The third section, applications, continues with material on data acquisition and signal/noise in time averaging. Examples are drawn from proton, carbon-13, and nitrogen-15 NMR, with other nuclei only briefly mentioned. Applications to rapid reactions, biological systems, and relaxation measurements are considered. The latter are given a more extensive treatment in Section Four, which also prepares for the subsequent discussion (Section Five) of the NMR of solids and the latest NMR developments which are considered in Section Six. Lacking hands-on experience with solids NMR, I found Section Five particularly informative both with regards to the current techniques and the limitations thereof. Becker is to be complimented on the way in which he introduces complex subjects briefly and then returns to them one or more times in subsequent developments.

In contrast, Section Six starts off with a superficial treatment of correlational spectroscopy and stochastic excitation. However, the general unavailability of these techniques on commercial instruments suggests that the time would have been better spent on a more detailed account of 2D NMR, the penultimate subject covered. After further development of spin echo formation, are both J-spectroscopy and 2D correlation NMR are discussed briefly with one example each. For the organic chemist, there may be some disappointment that the double quantum coherence experiment for determining carbon connectivity schemes was not mentioned. Perhaps this tape was prepared a little too soon for this. The course concludes with a brief look at NMR imaging.

Becker's goals for the course are very well achieved. Persons needing this sort of introduction to FT-NMR will find the course well worth the time and effort.

8/31/84 W.B.S.

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IBM Instruments Inc.

School of Science
Department of Chemistry

August 2, 1984

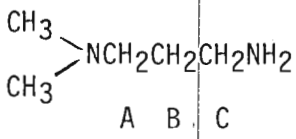
Rensselaer Polytechnic Institute Troy, New York 12181

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

Title: Spin Coupling Inequivalence in N,N-dimethyl-1,3-diaminopropane

Dear Barry:

I wanted to report on some interesting work that I am collaborating on with Drs. S. Bunce and D. Aikens here at RPI. In an ongoing study of microprotonation constants of various polyamines,[1] our latest compound of interest has been N,N-dimethyl-1,3-diaminopropane.



A B C

The pH titration was studied using ^1H NMR (200 MHz) using the following conditions. Samples were 0.01 M in 90% H_2O 10% D_2O. The water resonance was greatly suppressed using decoupler preirradiation on the H_2O resonance.

Several spectra are shown in the accompanying figure. The H_A and H_C multiplets cross each other at about pH 7.8. One puzzling aspect of these spectra was the unusual multiplet for proton H_A.

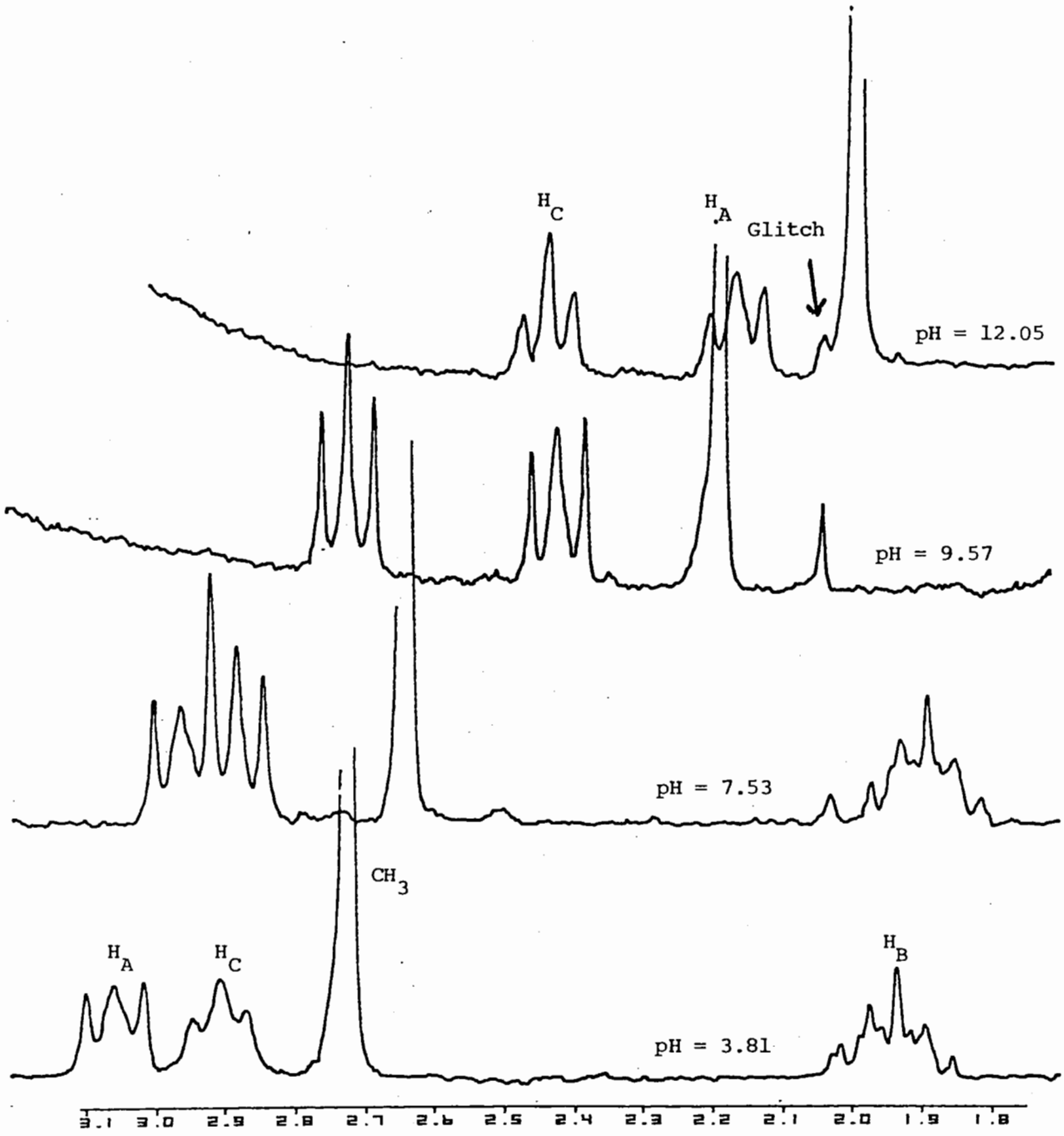
A recheck of the spectrum using a pulsed method of solvent elimination to check for artifacts due to strong irradiation of water was performed and did not affect the multiplet. The relative intensities and structure of the central transition indicated that the coupling interaction of H_A was more complex than an A_2X_2 system with protons H_B. After several attempts at computer simulation, the only satisfactory fits required assuming methylene spin coupling inequivalence and an AA'XX' spin system. In fact, to approach a satisfactory simulation of the entire spectrum, all six methylene protons must be considered as inequivalent with respect to spin coupling (ie. an AA'MM'XX' system). After thinking this over we realized that this should indeed be the case for this molecule which contains in effect two spin systems of the classic $Y\text{-}CH_2\text{-}CH_2\text{-}Z$ pattern which is known to be an AA'XX' system that often gives deceptively simple spectra. We are not aware of any previous examples of polyamines for which the spin coupling has been discussed in detail in the literature and would appreciate any such references your readers may be aware of.

Sincerely,

Dr. H. M. Schwartz

[1] F. Onasch, D. Aikens, S. Bunce, H. Schwartz, D. Nairn and C. Hurwitz, Biophysical Chemistry, 19, (1984), 245-253.

Variation of pH for N,N-dimethyl-1,3-diaminopropane



^{1}H Chemical Shift (200 MHz)

TEXAS CHRISTIAN UNIVERSITY
Box 32908
Fort Worth, Texas 76129
817-921-7195

Department of Chemistry

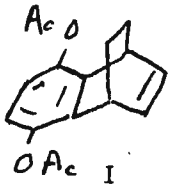
Unknown Structure Via a Double Quantum Coherence Experiment

8/13/84

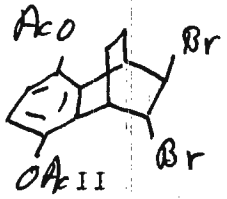
Dear Barry:

While there are now a number of papers dealing with $^{13}C-^{13}C$ connectivity determinations, these are largely of the "how to" variety with applications to known structures. Recently, a problem arose here in which conventional NMR simply did not supply an unambigueous answer. The problem arose as follows.

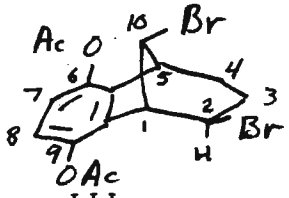
The bromination of the olefin I potentially can give a variety of structures involving normal addition(cis or trans to the double bond, syn or anti to the aromatic ring) or with Wagner-Meerwein rearrangement of either the ethano bridge or aryl group to bromonium or carbocation intermediates. There is one related case in the literature which suggests II as the most likely product. However, the structure proof in this example was



I



II



III

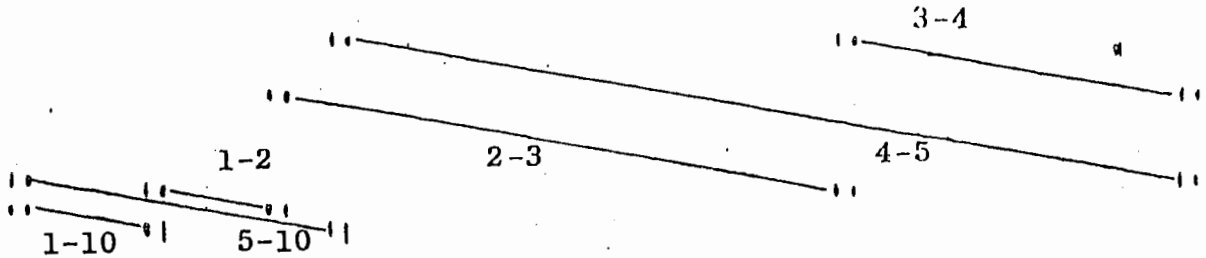
entirely based on an incomplete analysis of the ^{1}H NMR spectrum. Any consideration of vicinal coupling constants, dihedral bond angles and the effects of substituent electronegativity on same would suggest that this is a highly risky way to go. Nor can proton or carbon chemical shifts provide a unique answer to the many possibilities.

Ultimately Ralph Hurd and LeRoy Johnson of General Electric Company, NMR Instruments kindly obtained 300 MHz proton spectra with selective proton decoupling allowing a complete analysis of the proton system along with a 2D-$^{1}H-^{13}C$ correlation spectrum so that each proton could be assigned to its respective carbon. The final solution was provided by Roy who did the $^{13}C-^{13}C$ double quantum coherence experiment according to the method of Turner (J. Magn. Reson., 53,259(1983)). The data were acquired on the prototype GN-300. The partial results showing the aliphatic carbons only are given in the accompanying figure. This is a two level contour plot showing pairs of peaks which arise from ^{13}C nuclei which are spin coupled to directly bonded ^{13}C nuclei. Shown at the bottom is a plot of the projected 2-D data. The contours corresponding to the central peaks are along the diagonal at the bottom. Based on these data the correct carbon skeleton is shown to be as III above. Proton and ^{13}C data then allow the details of conformation and configuration to be ascertained. Complete parameters are available for interested parties.

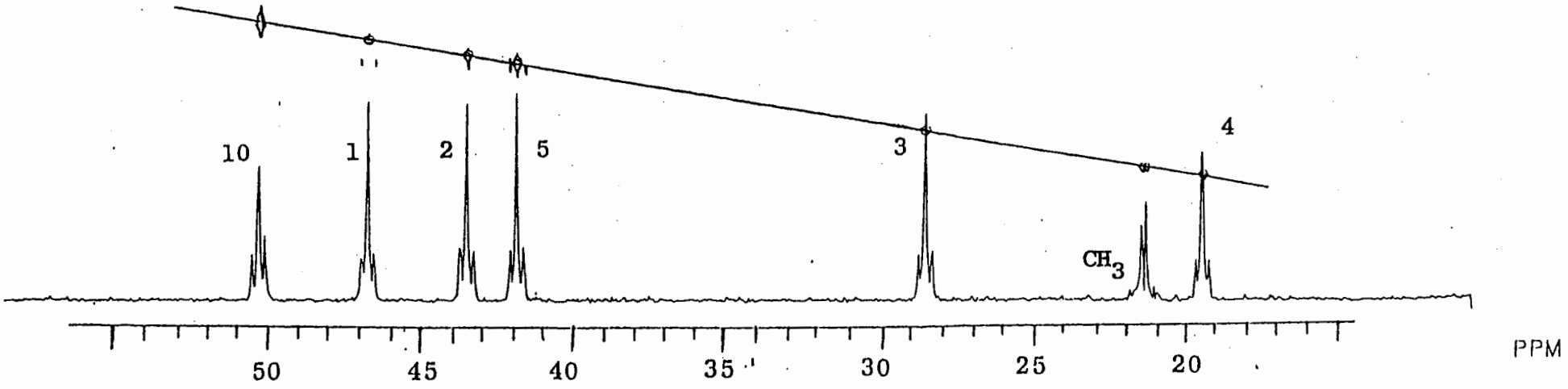
Best regards,

Bill

W. B. Smith



Numbers relate to carbons in III.



DE GUIGNÉ
TECHNICAL CENTER
WESTERN RESEARCH

Stauffer Chemical Company
1200 S. 47th St. / Richmond, CA 94804 / Tel. (415) 231-1000 / TWX (910) 382-8174

August 17, 1984

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

SUBJECT: "Carbon-13 NMR Analysis of Lipid Biosynthesis in Corn Suspension Cells"

Dear Barry:

In the course of investigating the effects of various herbicides on fatty acid biosynthesis in suspensions of Zea mays, a necessary prerequisite was the characterization of the lipid components prior to herbicide treatment. Growth of the cells for 2, 4, 6, 8 (Fig. 1), and 24 hr in the presence of 2 mM sodium [1-^{13}C] acetate or [2-^{13}C] acetate followed by NMR analysis of the extracted lipids displayed an initial incorporation of acetate into oleate (18:1) via palmitate (16:0) and stearate (18:0). After 24 hr, NMR analysis suggested that >90% of the labeled oleate had been converted to linoleate (18:2). Integration of the C_1 enriched carbonyl resonances of the neutral and polar lipid's fatty acids showed an even distribution of the label between the two lipid groups. Separation of the lipids into polar and neutral fractions followed by P-31 NMR spectroscopy showed the polar lipids to be primarily represented by phosphatidylcholine with lesser amounts of phosphatidylethanolamine and phosphatidylserine also observed.

Sincerely,

CKT

C. K. Tseng

D. J. Ashworth

CKT/DJA/ls
Attachment

(continued p. 24)

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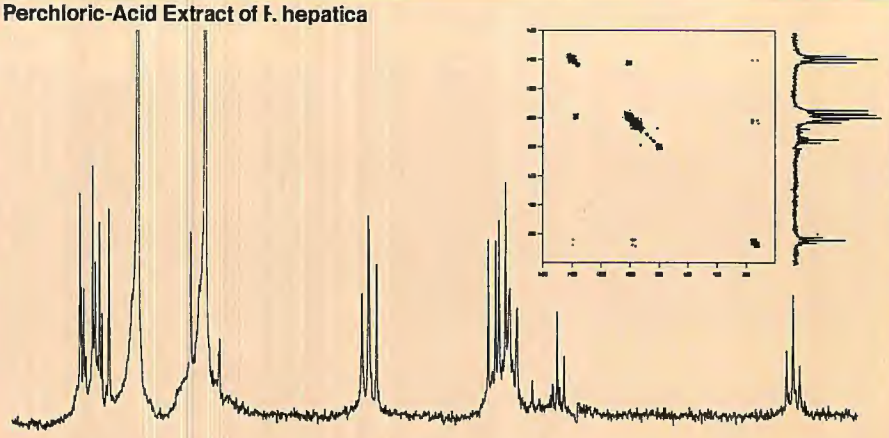
XL performance for demanding biological NMR studies

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

Perchloric-Acid Extract of F. hepatica

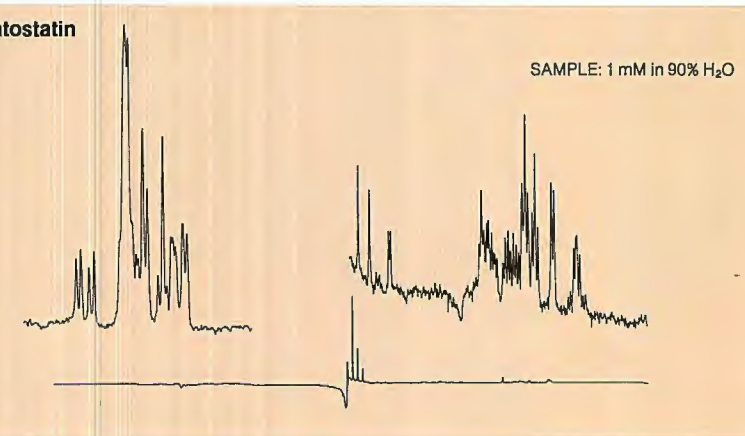


The high-sensitivity probes of the XL-200 allow spectra to be obtained from biologically relevant compounds at low concentration. The ^{31}P spectrum above is of a perchloric-acid extract of F. hepatica *(bovine liver flukes) and was obtained in 4 hours (1500 transients) at 81 MHz, using a 10-mm probe.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

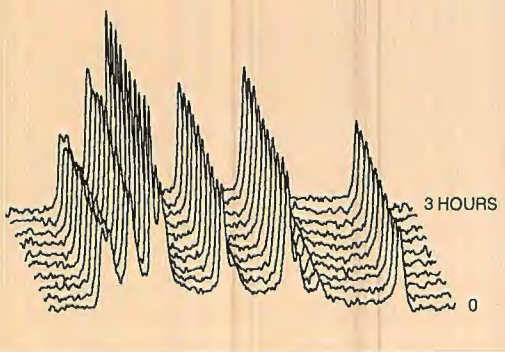
Two-dimensional NMR is a powerful method for analyzing complex mixtures. The contour plot shown above the spectrum is the result of a ^{31}P homonuclear shift correlation experiment carried out on a portion of the fluke extract using a 5-mm ^{1}H broadband switchable probe at 81 MHz. The total experiment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

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

^{1}H Somatostatin

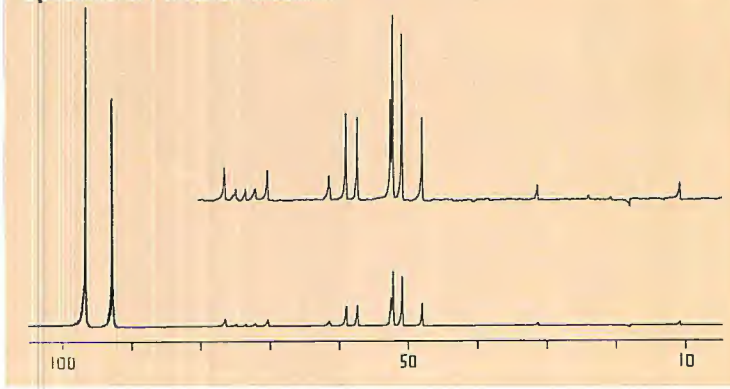


Perfusion of Tissue



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

Spectrum of Perfusion Medium



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

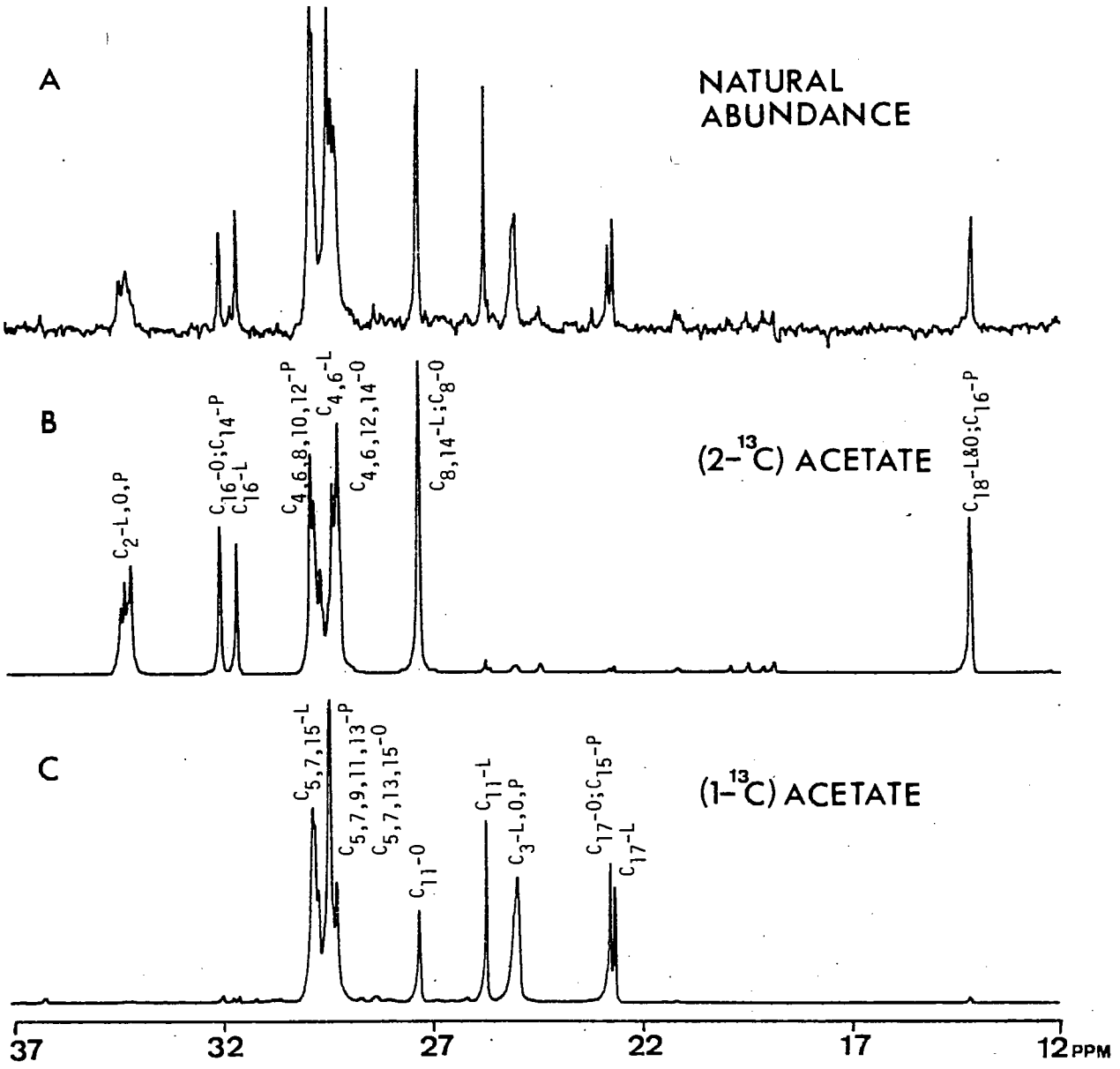


Figure 1. Carbon-13 NMR spectra of isolated corn cell lipids following culture growth for 8 hr in (A) normal medium, (B) medium and 2.0 mM sodium [$2^{-13}C$] acetate, and (C) medium and 2.0 mM sodium [$1^{-13}C$] acetate. Resonance assignments correspond to major labeled fatty acids present. P = palmitate, O = oleate, L = linoleate.

312-25

Department of Organic Chemistry

La Trobe University
Bundoora Victoria
Australia 3083.
Telephone (03) 478 3122
Telegraphic address
Latrobe Melbourne
Telex No. (AA) 33143

15 August 1984

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

Dear Barry,

^{95}Mo Sensitivity

We have been attempting to improve our sensitivity in order to detect broad line (2000Hz) ^{95}Mo signals at less than 5 x 10^{-3}M on our JEOL FX-200 spectrometer. ^{95}Mo, with a spin of 5/2 and abundance of 16%, resonates at 13MHz (H1 at 200MHz) and so the problems of detection are typical of low frequency nuclei in that low sensitivity and acoustic ringing make signals hard to observe.

We, like others, have constructed a transverse probe, consisting of a single coil wound using enamelled copper wire 22mm in length with a 15mm i.d. This coil accepts a specially constructed cell made for a 15mm nmr tube that can be sealed and having an effective volume of 4mls. The 90° probe is 28μsecs and with no provision for sample spinning or locking, the natural linewidth of a sample of sodium molybdate is 4Hz. The effective real signal to noise gain over our tuneable probe, where this inhomegeneity broadening is negligible, is about 3:1 for the same volume of sample.

Acoustic ringing can be overcome by introducing a relatively long dead time, but this, of course, reduces the nmr information for fast relaxing broad line signals. For low concentration, where a large number of pulses are required (10^7), dead times of 750μsecs were required. We have found that the ACOUSTIC pulse sequences using a very short delay (0.5μ)secs between the 180° and 90° pulses were effective in reducing the acoustic ringing, thus allowing a large reduction in the delay time to 250μsecs for runs of 10^7 pulses (in conjunction with the lead shielding, see below).

It appears that the main source of the acoustic ringing was the copper outer cover of our probe. We sought a number of ways of eliminating this source of ringing including: removing the cover (very noisy, good acoustic properties); winding a cover out of braided copper wire (less noisy, good acoustic properties); no cover and room temperature shims removed!! (good noise properties, good acoustic properties, inconvenient); and finally, and using a lead cover (good noise and acoustic ringing properties). (The lead came from the roof of my house - RB).

Our standard method is now to use the transverse probe with the lead cover, and an ACOUSTIC pulse sequence. The overall signal to noise improvement is many fold for a broad line signal as can be seen from the figure.

Please credit this contribution to Ian Rae at Monash University.

Yours sincerely,

R.T.C. Brownlee — Bob

M.J. O'Connor — Max

B.P. Shehan — Philip Shehan

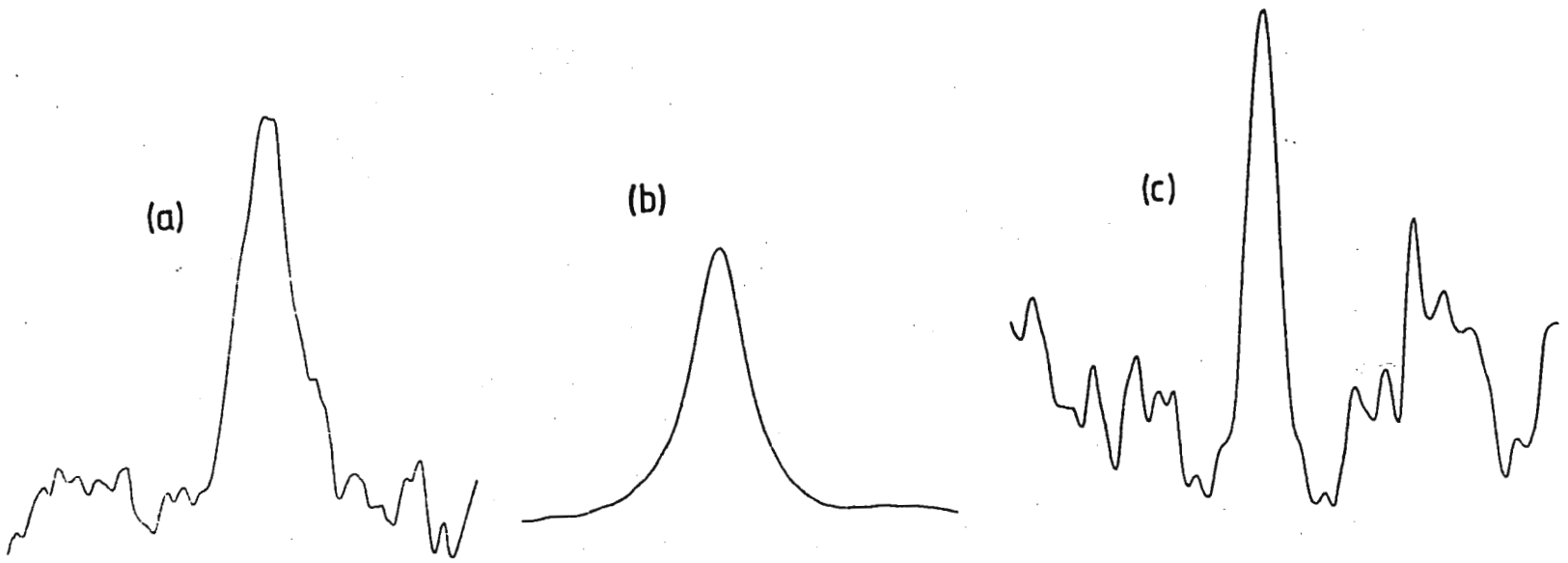


Figure ^{95}Mo NMR Spectra of $[Mo_2(O_2CnBu)_4]$ in THF

(a) [Mo] = 1M; 1.36×10^6 transients; dead time 800 μs; FX200 multilow probe.

(b) [Mo] = 1M; 1×10^4 transients; dead time 100 μs; transverse probe.

(c) [Mo] = 5×10^{-3}M; 9.6×10^6 transients; dead time 350 μs; transverse probe.

312-27

Wageningen
Department of Molecular Physics

Your reference
Your letter of
Our reference 84/406 dJ/jbw
Date August 15, 1984
Enclosure(s) 2 figs.

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

Subject Improving the signal-to-noise ratio of CPMAS spectra by eliminating the effect of probe arcing

Dear Professor Shapiro,

One of the problems in CPMAS spectroscopy at high magnetic fields is probe arcing. While detecting the ^{13}C signals, the ^{1}H spins are irradiated with an intense r.f. field of about 14 Gauss to remove the strong C-H couplings. Probe arcing occurs, because the r.f. voltage to obtain this field is very high. If arcing occurs, the ^{13}C signal disappears and the noise level increases. On the other hand, it is not possible to reduce the r.f. field to prevent the probe from arcing, because this results in line broadening.

A typical spectrum of glycine, obtained on our CXP 300, equipped with a Z32DR-^{13}C-MAS probe in a 9 mm O.D. Andrew-type Boron Nitride rotor, is shown in fig. 1. The number of scans is 20, 2 scans had arcing noise. The spectrum of the 18 scans without arcing noise is shown in fig. 2. This spectrum has a higher signal-to-noise ratio, particularly around 0 Hz (the middle of the spectrum).

These spectra are obtained with a special version of the CXPNMR software. The memory is divided into three blocks. In an automated sequence, every single scan is recorded in block 1. After a scan is completed, the data of block 1 is added to the contents of block 3. During this transfer, the data of block 1 is examined to check whether or not more than 10 datapoints exceed a certain level. If not so, the data is accepted as normal and added to the contents of block 2. Thus, fig. 1 was obtained from block 3 and fig. 2 from block 2.

When all precautions are taken to reduce probe arcing, a CPMAS spectrum with improved signal-to-noise ratio is obtained, using a computer program that rejects the data of scans with arcing noise.

Please credit this contribution to the account of dr. T.J. Schaafsma.

Sincerely yours,

P.A. de Jager

R.F.A. Lukassen

Agricultural University/Transitorium, De Dreijen 11/6703 BC Wageningen/The Netherlands/Tel. (08370) 82044/82634

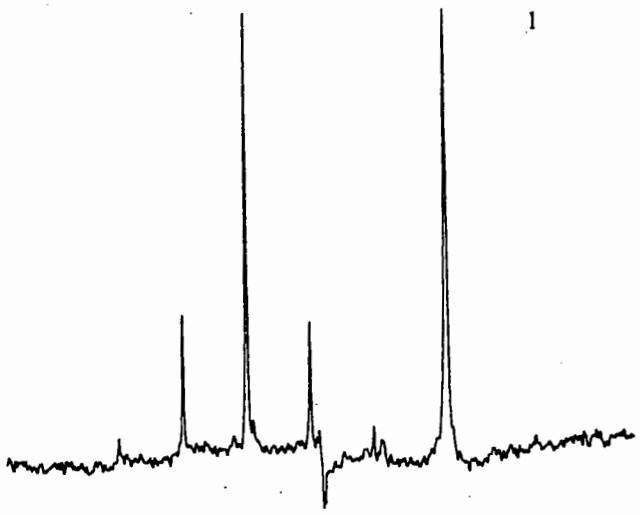


Fig. 1. 75 MHz ^{13}C-CPMAS spectrum of glycine in BN rotor, 20 scans (including 2 scans with arcing noise). SW = 31250 Hz LB = 30 Hz; no spin temp. alternation used.

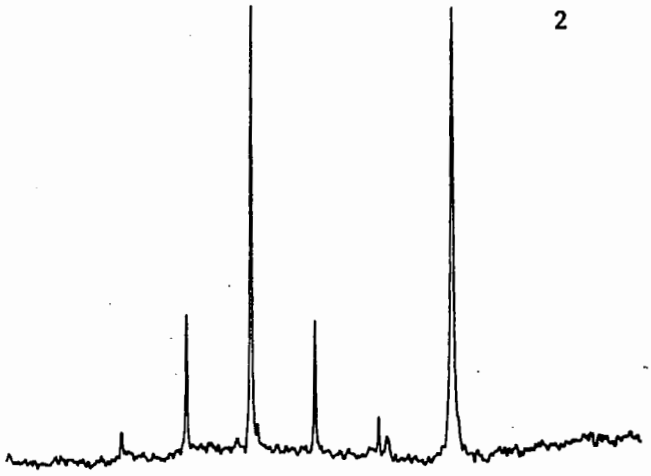


Fig. 2. See fig. 1. 18 scans (2 scans with arcing noise rejected).

The University of Texas
Health Science Center at Houston

MEDICAL SCHOOL
Department of Radiology

6431 Fannin
Room 2.132 M.S.M.B.
Houston, Texas 77030
(713) 792-5231

February 15, 1984

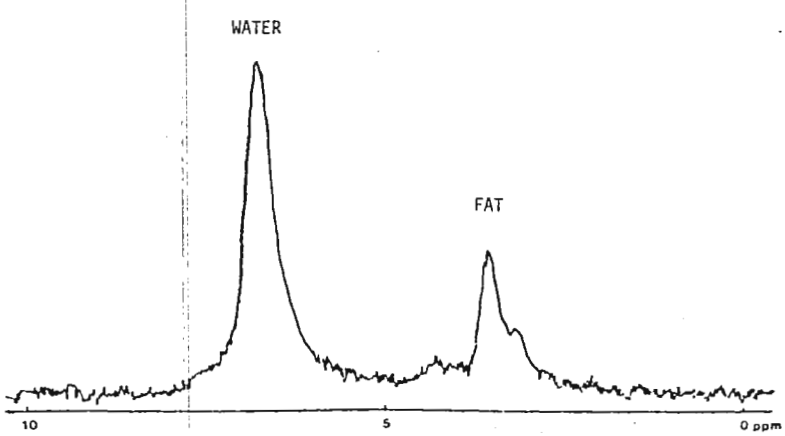
Professor B. L. Shapiro
Department of Chemistry
Texas A-M University
COLLEGE STATION, TX 77843

First results on an NMR Imaging Spectrometer (cont.)

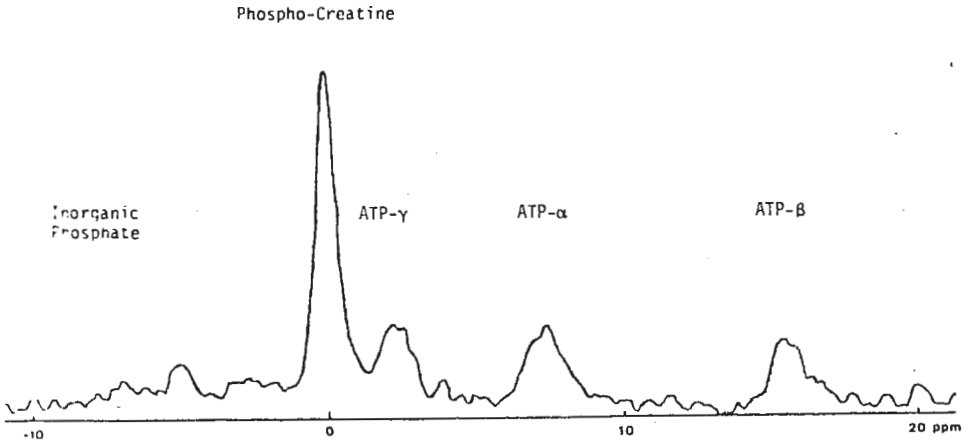
Dear Barry,

We finally got our Nalorac 2 Tesla 33 cm bore magnet (13") and it is now up and running. It was delivered at 1:00 am, 9 June 1984, and the installation was completed by July 9. The gradient and shim coils were also provided by Nalorac. Less than a month later, we were able to get our first results obtained with the system.

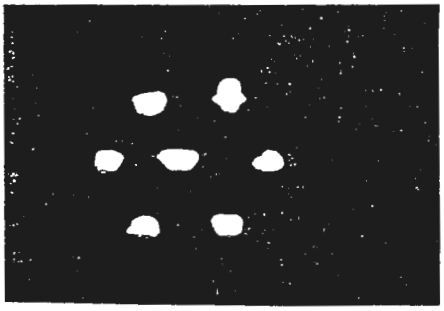
The spectrum to the right represents a Proton spectrum of the abdomen of a living rat. It was recorded in 4 seconds using a 2.5 cm diameter (1") surface coil. It clearly shows that our pet rat had a very peaceful life and grew a lot of fat in the past few months, judging from the height of the fatty peak right to the water peak!



The next spectrum is a phosphorus spectrum of the left leg of the same rat obtained in 7 minutes with the same surface coil tuned to the P-31 frequency. One can see easily the three small peaks of ATP, the large peak of Phospho-creatine, and the small peak of inorganic phosphate, further demonstrating that indeed our rat is in good health.



Finally the image below represents our first attempt at NMR imaging. It is a proton image of a phantom consisting of seven tubes filled with water. It is not the best NMR image in the world, and there is room for improvement.



Although these three examples do not exhibit a tremendous scientific value, they demonstrate that our system can perform both imaging and spectroscopy. It is our hope that we will soon be able to do both on a routine basis.

Yours sincerely,

Jean L. Delayre
Assistant Professor
Director, NMR program

312-31

University of Illinois
at Urbana-Champaign
505 S. Mathews Ave.
Urbana, IL 61801

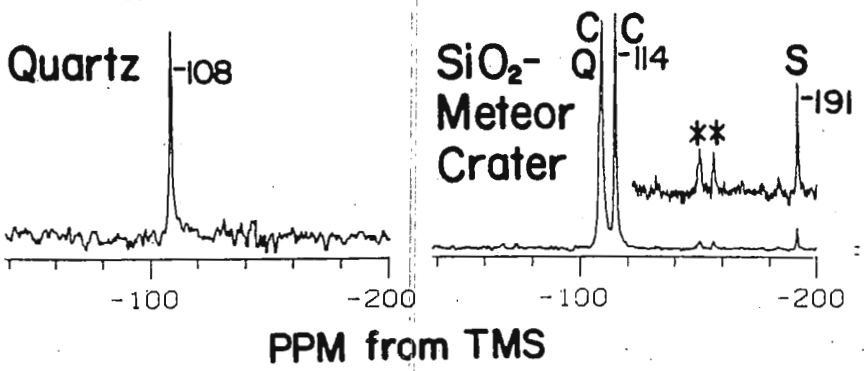
August 16, 1984

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

Dear Barry:

^{29}Si NMR of a Meteor Impact Site: ^{27}Al NMR of $Al_{13}O_{40}$ Clusters; ^{13}C NMR of Proteins at 11.7 Tesla

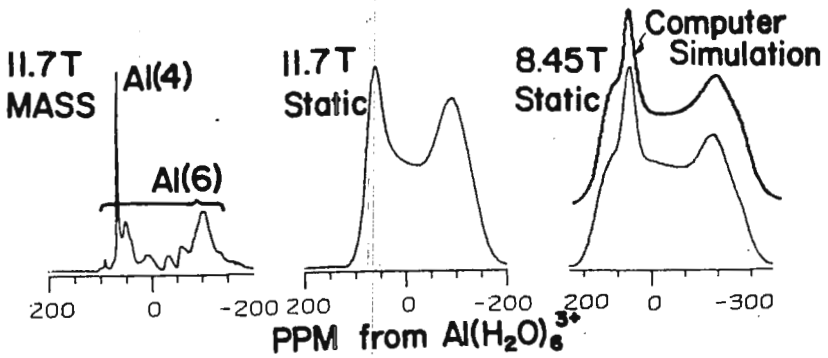
I report on a few recent results from our laboratories: First, we have observed the presence of the high pressure polymorphs of silica, coesite and stishovite, in whole rock samples from Meteor Crater, AZ, in collaboration with R. J. Kirkpatrick and colleagues in our Geology Department (1).



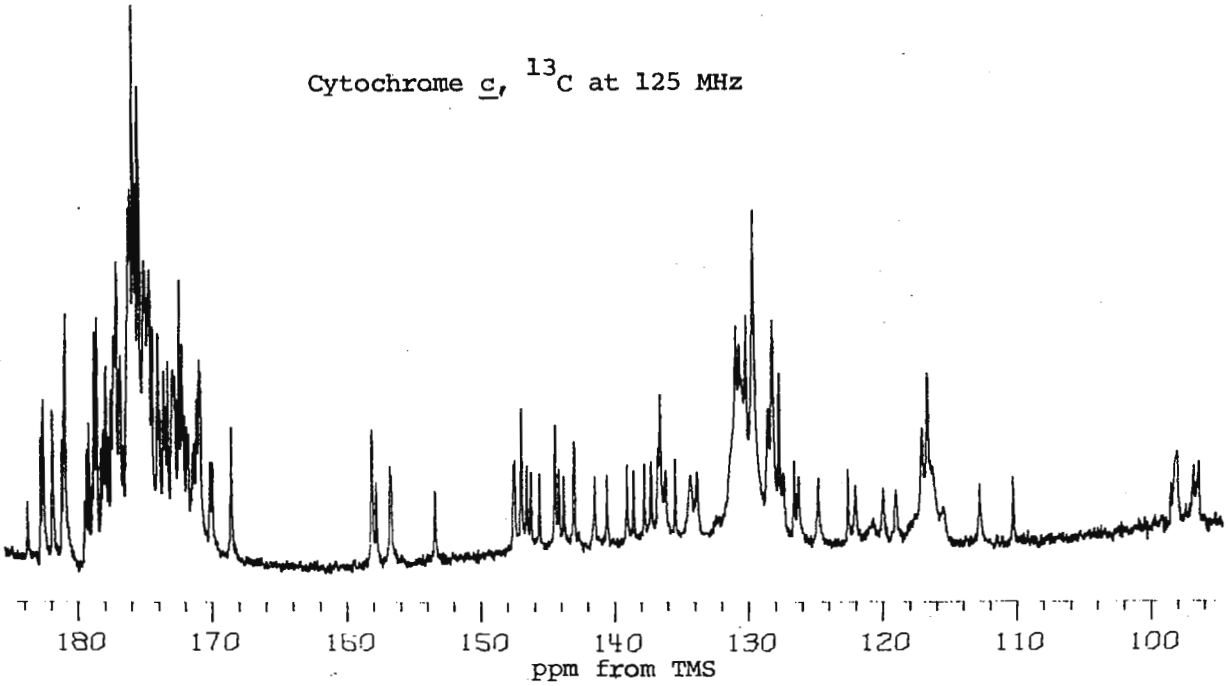
These species were presumably formed during meteor impact, and we are able to obtain good quantitation of coesite/stishovite ratios-a difficult task with XRD methods. We find a rather snort T_1 compared to that of quartz, which facilitates rapid data acquisition, and reduction of the otherwise intense quartz resonance.

Second, we have observed the six-coordinate Al sites in the tridecameric basic aluminum sulphate and selenates-previously unobservable at low field. The following Figure suggests that in the case of very large e^2qQ/h values (~10 MHz in this case)-it may be preferable to study static (rather than MASS or VASS) spectra (2).

$Na[AlO_4 \cdot Al_{12}(OH)_{24}(H_2O)_{12})](SO_4)_4 \cdot 13H_2O$



Third, we have recently obtained a number of high-resolution ^{13}C NMR spectra of proteins at 11.77 Tesla (500 MHz, ^{1}H resonance frequency). Contrary to our fears, it seems that ^{1}H decoupling can be carried out efficiently with as little as 2 watts of 500 MHz r.f. on a 10 mm tube (3). As expected, the nonprotonated and protonated aromatic carbons can have more similar widths at high field due to the increased CSA relaxation of the former, and reduced dipolar width of the latter, to that seen at low field.



Yours sincerely,

Eric Oldfield

1. W. H. Yang, R. J. Kirkpatrick, M. Vergo, J. McHone, T. I. Emilsson and E. Oldfield, submitted to Meteoritics.

2. A. Kunwar, A. Thompson, H. S. Gutowsky and E. Oldfield, submitted to J. Magn. Res.

3. R. Ramachandran, J. Bowers, and E. Oldfield, unpublished results.

BROCK UNIVERSITY
REGION NIAGARA

Department of Chemistry

416/688-5550

St. Catharines,
Ontario L2S 3A1
Canada

August 1, 1984.

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

Silicon-29 and Carbon-13 MAS Nmr of Silicon Carbide Polymorphs

Dear Barry:

In response to your recent ultimatum, we report some new nmr results on silicon carbide. This work started out as an attempt to interest the abrasives industry of the Niagara region in the possibilities of high-resolution solid-state nmr as an analytical tool (and hopefully to raise some money for a new nmr instrument). Silicon carbide (also known as carborundum) exists in numerous crystalline modifications based on hexagonal α-SiC (wurtzite-type ZnS) and there is also a cubic β-SiC structure (diamond or zinc-blende type). The complexity arises from the numerous possible stacking sequences in the crystal. In some respects silicon carbide is an ideal system for MAS nmr (e.g., the concentrations of both silicon and carbon atoms are 80 molar) but long spin-lattice relaxation times are a problem.

Figures 1a and 1b are ^{29}Si and ^{13}C Magic Angle Spinning spectra of the common 6H hexagonal polytype of silicon carbide. The three equally-populated silicon environments give three well-resolved peaks of equal area, as do the three equally-populated carbon environments; the ^{13}C spectrum is an almost exact mirror image of the ^{29}Si spectrum, even to the chemical shift values in ppm from TMS (^{29}Si: -13.9, -20.2, and -24.5 ppm; ^{13}C: +15.2, +20.2, and +23.2 ppm). The carbon environments are isostructural with the silicon environments, and apparently the same crystal lattice factors determine chemical shift differences among the three distinct environments of both ^{29}Si and ^{13}C. Figure 1c shows the ^{29}Si spectrum of the cubic form, which has only a single silicon (and carbon) environment. We have not yet succeeded in obtaining a ^{13}C spectrum of this sample, apparently due to an exceedingly long T_1.

Figure 1d was obtained from a sample which by its X-ray powder pattern is a mixture of 6H and other polytypes. The same ^{29}Si peaks are present as in the 6H polytype, but in different proportions. This makes sense, as examination of several polytypes of known structure shows that there are only three distinctive types of silicon environment, out to 5 Å from the central silicon, although there may be many crystallographically independent silicon positions. For example the 15R form, with five crystallographically distinct silicon positions, contains the same three distinct types of silicon sites as the 6H form, but in a 1:2:2 ratio. Nmr studies of this polytype are in progress.

The best-resolved spectra are those obtained with the longest relaxation delays between pulses (up to four hours so far). Short delays give broadened peaks and also signals due to apparently-amorphous components. All this is consistent with lack of spin diffusion[1] so that different domains of the sample have very different T_1's. At short relaxation delays the signal from the bulk sample is apparently swamped by signals from minor, but fast relaxing, components near paramagnetic centres.

All spectra were obtained on the Bruker WH-400 instrument of the South Western Ontario High Field Nmr Centre with the assistance of Dr. Bob Lenkinski and colleagues, using our MAS probe built to specifications kindly provided by Prof. Colin Fyfe.

Yours sincerely,

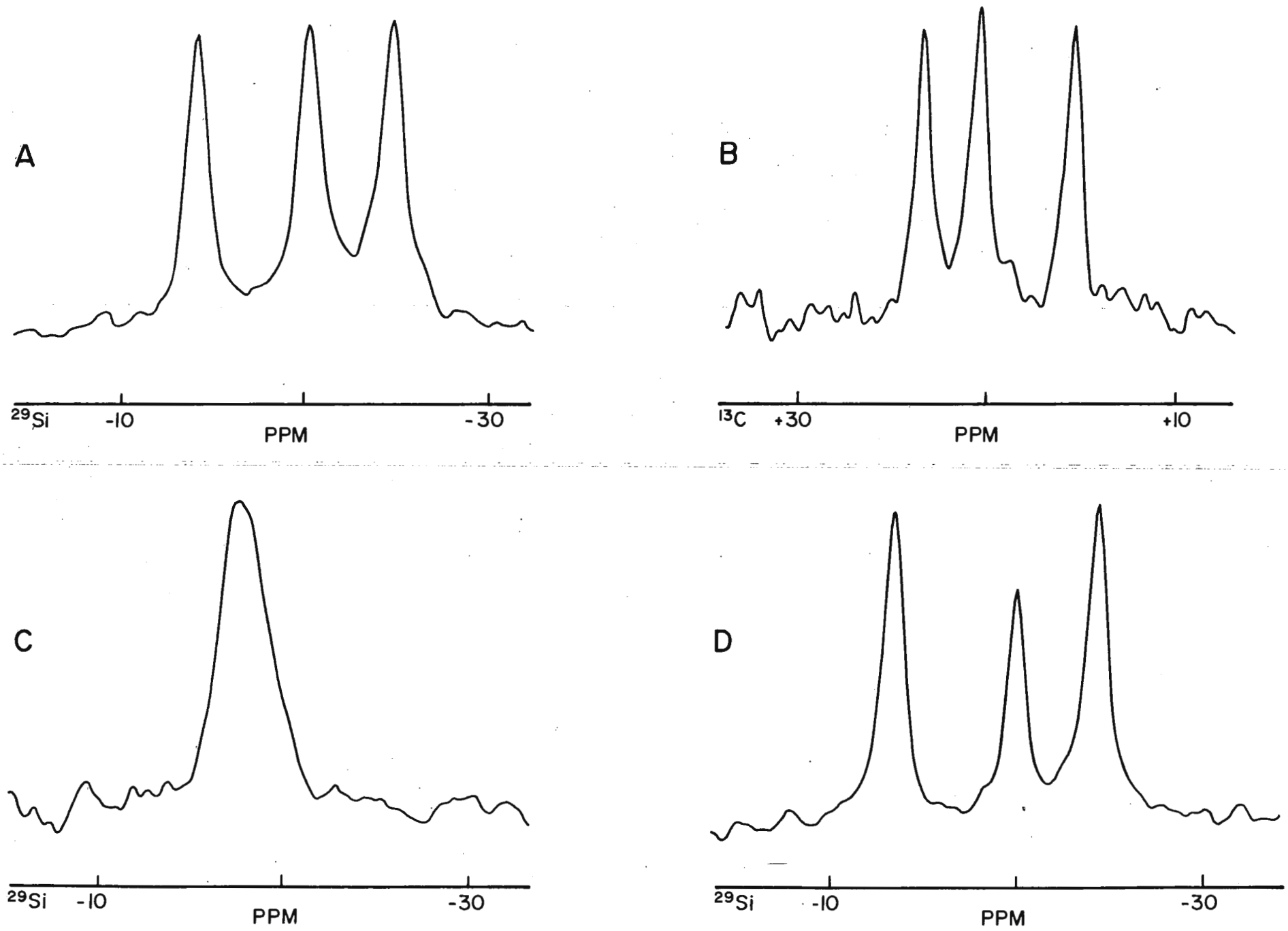
Gordon R. Finlay — G. R. Finlay

Steve Hartman — J. S. Hartman

M. F. Richardson

Barbara L. Williams — B. L. Williams

[1] P. M. Henrichs, M. T. Cofield, R. H. Young, and J. M. Hewitt, J. Mag. Res., 58 , 85 (1984).


A
^{29}Si
-10
-30
PPM
B
^{13}C
+30
+10
PPM
C
^{29}Si
-10
-30
PPM
D
^{29}Si
-10
-30
PPM

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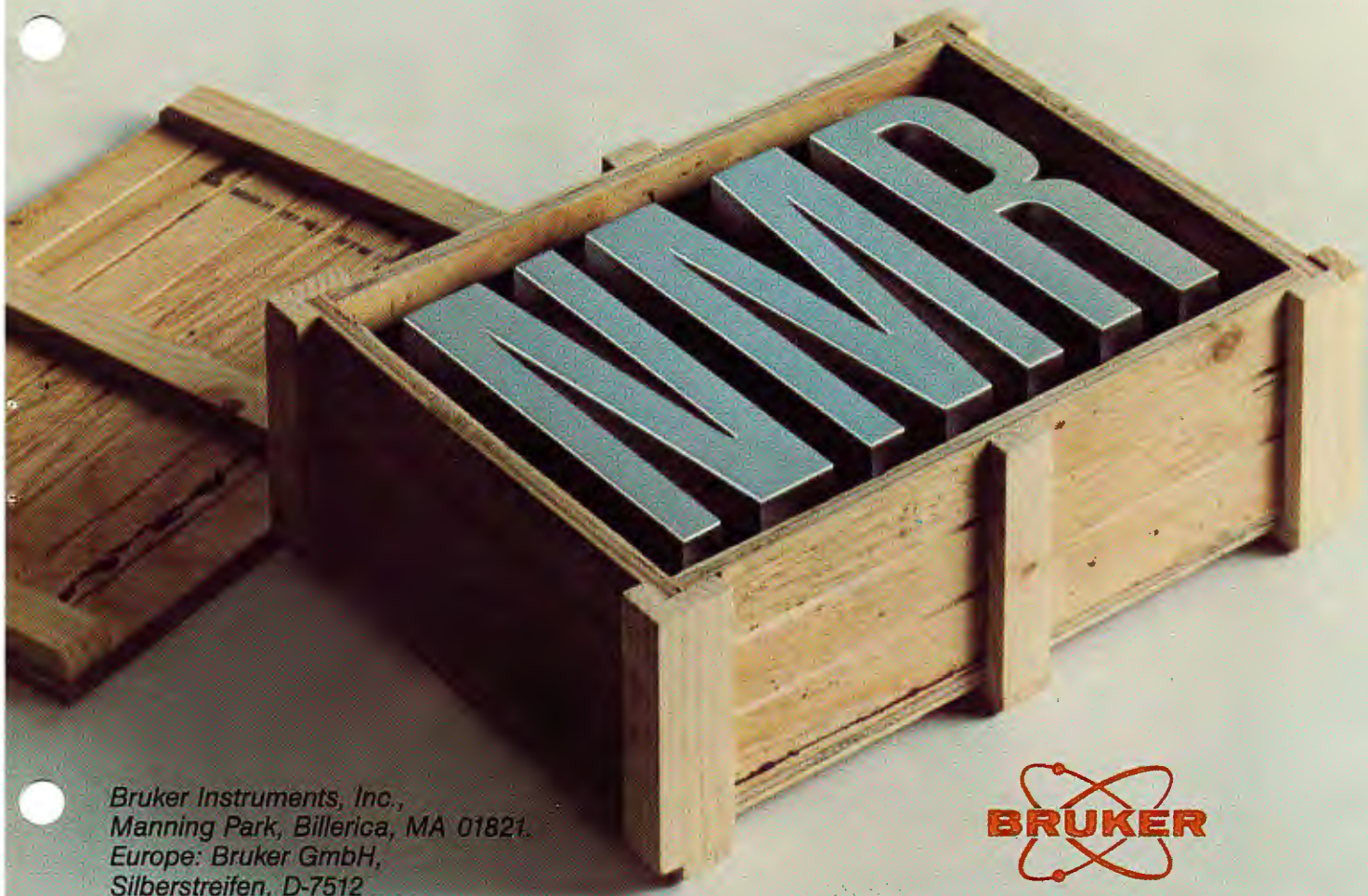

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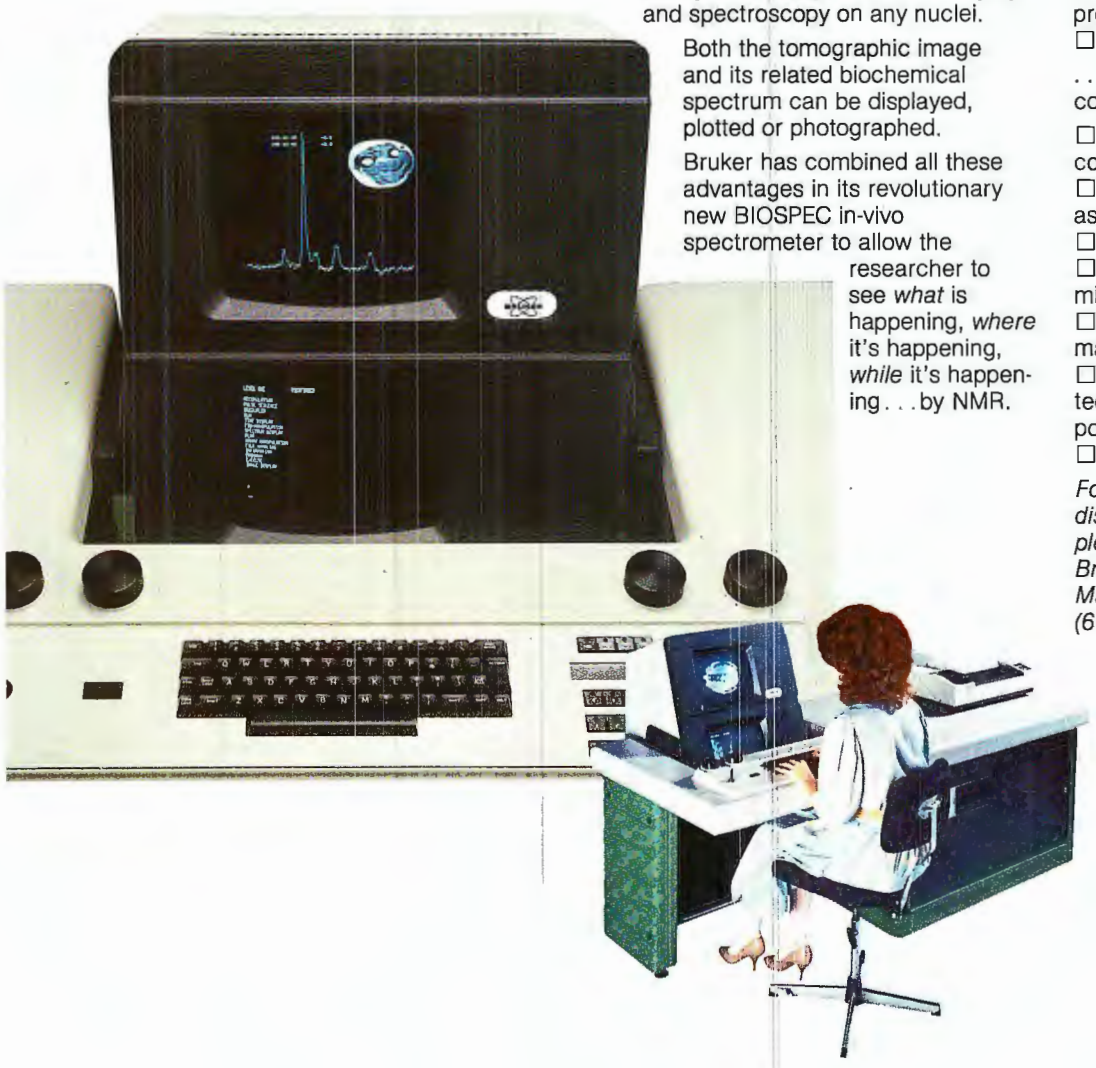

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20 August, 1984

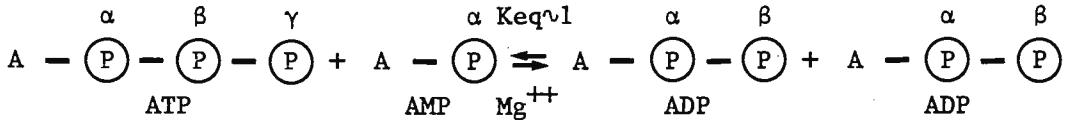
Professor B.L. Shapiro, TAMUNMR Newsletter,
Department of Chemistry, Texas A & M University,
College Station, Texas 77843, U.S.A.

Dear Barry,

"Analysis of 2-D ^{31}P-NMR spectra of the adenylate kinase reaction"

Our last letter (Feb 84; No. 305) dealt with ^{31}P-NMR inversion-transfer catalysed between 2- and 3-phosphoglycerate by phosphoglyceromutase. In those experiments we selectively inverted the 2- or 3-phosphoryl resonance with a DANTE[1] pulse sequence since our Bruker WM400 does not have a ^{31}P decoupling channel. We subsequently applied some tracer-exchange theory to the analysis of the data and the results will appear soon in the European Journal of Biochemistry.

When we began our NMR-based enzyme kinetic work we 'cut our teeth' on the adenylate kinase catalysed reaction which was the first one studied by Truman Brown[2]. We were able to reproduce his earlier results by using the specified buffer conditions for the reaction and performing selective inversions with the DANTE sequence. The enzyme catalyses the exchange of phosphoryl groups as follows:



Note that if the α-ATP resonance is inverted magnetisation is transferred to α-ADP, and to α-AMP via a "second pass" through the enzyme. On the other hand inversion of the γ-ATP resonance results in magnetisation transfer to the β- of one of the ADP's. 'Forbidden' exchanges in the above simple reaction are β-ADP → α-AMP and α-ATP → β-ADP; however if more complex exchange is occurring, say with an enzyme-phosphate intermediate, these 'forbidden' exchanges may then exist. They were not observed in our 1-D spectra but signal-to-noise was such that we could not conclusively decide

on the matter. Therefore we began some 2D-NOESY[3] experiments, and a spectrum from one such experiment is shown below. The experimental details are in the caption and the peak assignments are indicated on the figure. Note the following cross peaks that indicate exchange between the phosphoryl sites; α-ATP x α-AMP (a "2-pass exchange"), α-AMP x α-ADP (a "1-pass exchange"), β-ATP x β-ADP (a "1-pass exchange"); none of the 'forbidden' cross peaks are observable. We have quantified the exchange rates, something that hasn't been presented in the literature on 2D-NOESY of enzyme reactions, by determining the magnitude of the elements of the exchange matrix. We hope to publish the results very soon.

Yours sincerely,

George L. Mendz

B E Chapman

Bogdan E. Chapman

Gae Robinson

Gae Robinson

Philip W. Kuchel

Philip W. Kuchel

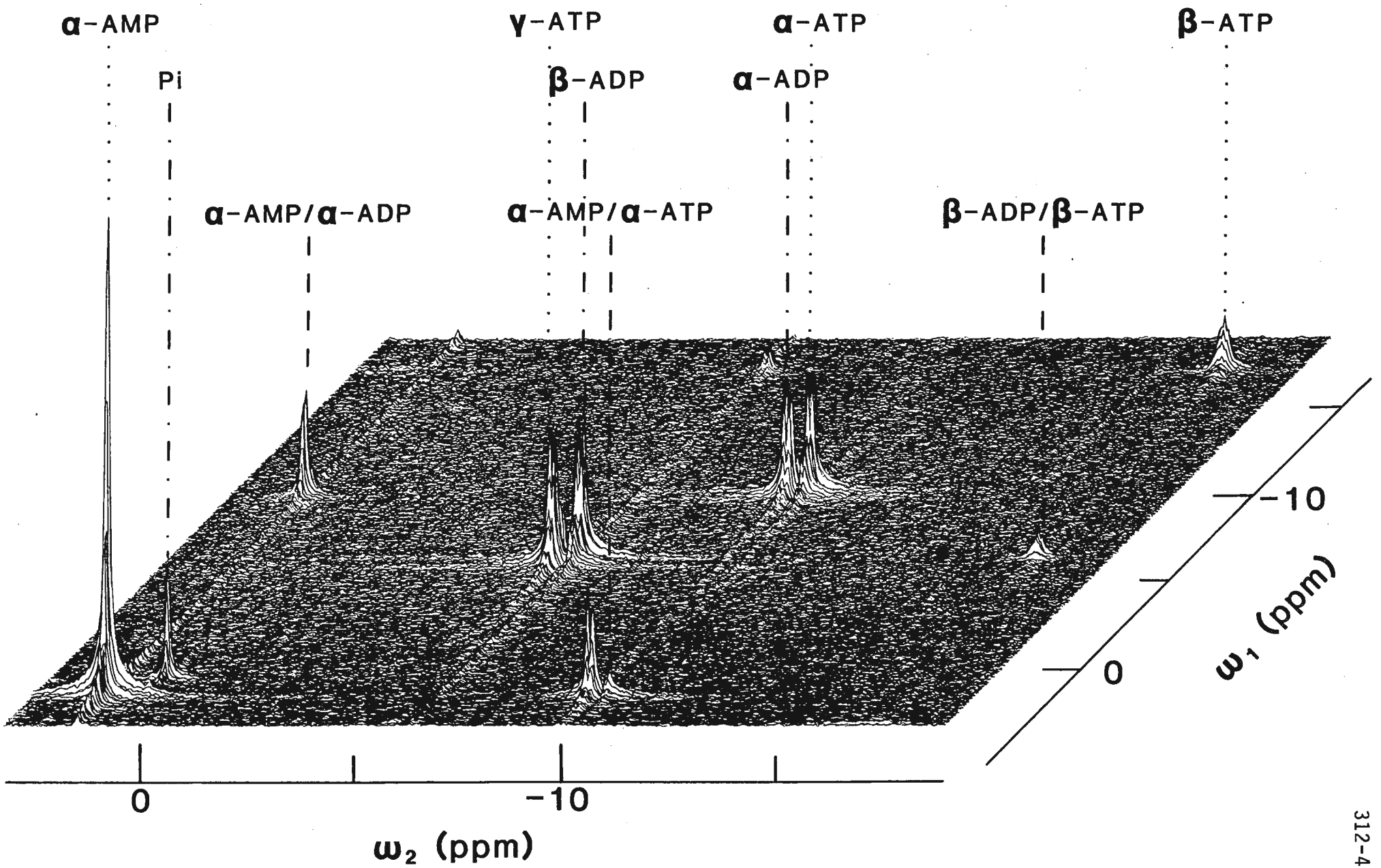
Figure caption: 2D-NOESY of the Adenylate Kinase Catalysed Reaction. Experimental details: NMR, 162 MHz, pulse sequence $(\pi/2\text{-}t_1 - \pi/2 - t_m - \pi/2 - t_2)_n$, n = 16, t_1 = 0.2 to 58.4 ms, t_m = 0.5s, t_2 = 0.2 s and recycling time 10s. Biochemicals, Sigma enzyme 310 IU/ml, buffers in (2), 37 mM ADP at start of reaction, 37°C.

---

[1]Morris, GA and Freeman, R (1978) J. Mag. Res. 29, 433-462.

[2]Brown, TR, Ugurbil, K and Shulman, RG (1977) Proc. Natl. Acad. Sci. USA 74, 5551-5553.

[3]Jeener, J, Meier, BH, Bachmann, P and Ernst, RR (1979) J. Chem. Phys. 71, 4546-4553.


α-AMP
Pi
γ-ATP
β-ADP
α-ATP
α-ADP
β-ATP
α-AMP/α-ADP
α-AMP/α-ATP
β-ADP/β-ATP
0
-10
ω2 (ppm)
0
-10
ω1 (ppm)

312-40

312-41

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

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

August 23, 1984

Professor B. L. Shapiro
Department of Chemistry
Texas A & M University
College Station, TX 77483

"New NMR Lab Setup and 2-dimensional NMR"

Dear Professor Shapiro,

Setting up a new NMR laboratory can be viewed as analogous to doing a contemporary 2D NMR experiment. To paraphrase two well-known Swiss spectroscopists:

"First, we have the PREPARATION period."

We arrived to find the NT-360 wide-bore magnet about 4 ft from a metal bench. We found we had to spend some time reshimming the non-spinning shims as we changed samples. The other half of the room was a wet lab. We corrected this installation, and were gratified to find virtually reproducible non-spin adjustments after correction. We then designed the adjacent room to accomodate a small elctronics bench, an FT-80 iron magnet system and a spanking new QE-300 spectrometer. The organic chemists were quite delighted with the MENU mode of the QE.

"Second, we have the EVOLUTION period."

We were barely settling in with these new-found riches, when the first of the series of tornadoes that devastated North Carolina hit our building. The roof was pulled back on itself and the rain poured in. After bailing water and sweeping, clearing and vacuuming the pieces of ceiling tile, it was damage assessment time. The NT-360 electronics checked out and the magnet retained field; however the field homogeneity was atrocious, and we had to shim and clean for three days before reasonable lineshape and field homogeneity were achieved. We were not as lucky on the QE-300. A floppy drive burned out and both transmitter and receiver sections needed attention. The General Electric service people were quite helpful to us, and we finally had the system up a few weeks later.

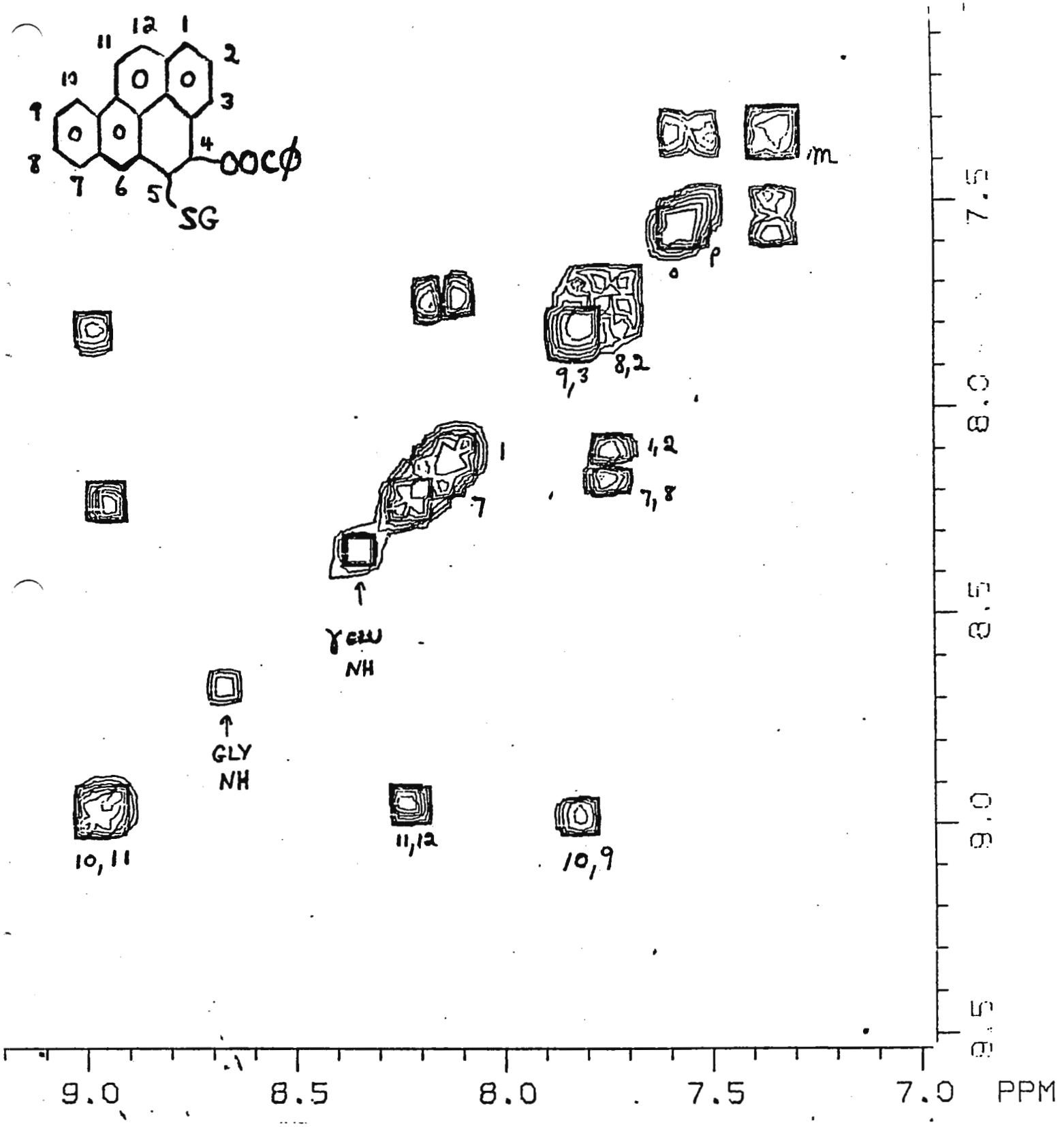
"Third, after the MIXING interval, we have the DETECTION period."

To show that after all that preparation, evolution and mixing above we still detect signals in the 2-dimensional manner, we give in the figure a 2D COSY spectrum of a glutathione-benzo(a)pyrene conjugate. Details on the appearance of the NMR spectra with the stereochemistry of the conjugates will be appearing soon elsewhere.

Sincerely,

Anthony Ribeiro

Anthony Ribeiro



360 MHz ^{1}H NMR 2D COSY spectrum of the AROMATIC and PEPTIDE NH region of a (4R, 5R)-Benzo(a)pyrene - 5 - Glutathione Conjugate in DMSO-d_6 (21°C).

312-43

Université de Nancy I

LABORATOIRE DE METHODOLOGIE RMN

D. CANET
Professeur

Professor B.L. SHAPIRO
Department of Chemistry
Texas A8M University
College Station, Texas 77843
U.S.A.

August 1, 1984

Title : the direct experimental determination of a dipole-dipole cross-correlation spectral density

Dear Professor Shapiro,

Longitudinal relaxation of an AX_2 spin grouping can be described by four magnetization modes. When $A \equiv {}^{13}C$ and $X \equiv {}^{1}H$, two of them are of special interest : $\nu_1 = \langle I_z^A \rangle$ and $\nu_3 = 4 \langle I_z^A I_z^X I_z^{X'} \rangle$. The time evolution of ν_1 yields the conventional relaxation time $T_1(A)$, whereas ν_3 can provide the more detailed information contained in the cross-correlation spectral densities. Previous approaches, to abstract this information, have used complete sets of complementary time evolution curves. The cross correlation spectral densities were deduced from non linear fitting procedures. In this letter, we mention a method that we have recently developed (full details to be published soon). It is based on the simple pulse sequence :

$$\pi({}^1H) - \tau - \frac{\pi}{2}\ ({}^{13}C) - 1/4\ J_{CH} - \pi({}^{13}C)\ ;\ \pi({}^1H) - 1/4\ J_{CH} - \underset{\text{Decoupling}}{\text{Acquisition}}$$

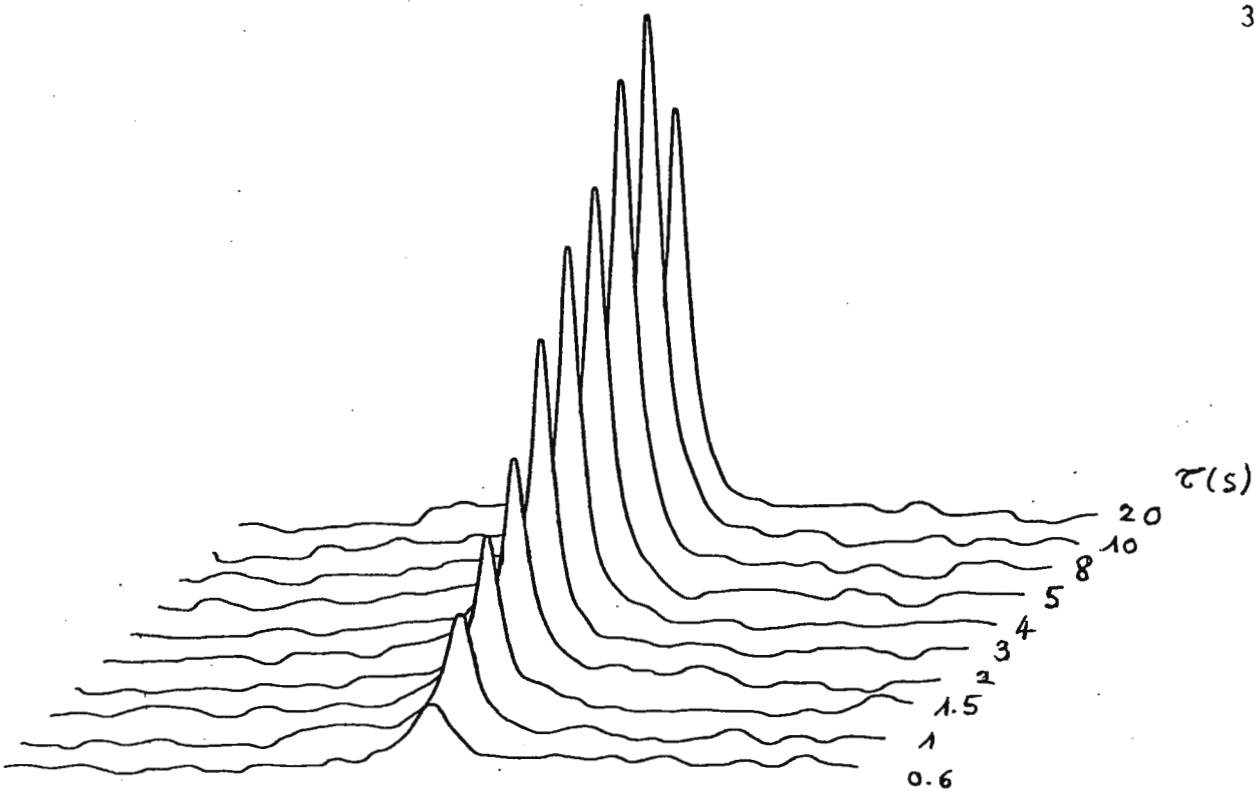
The cross correlation spectral density $K_{CH\ H'}$ is directly deducible from the initial slope of the detected quantity. The utility of this method is further enhanced by choosing a pulse sequence which sequesters one spin order (ν_1) and projects three spin order (ν_3) on to the observable. The result of an experiment performed an adamantane is shown in the accompanying figure (because of isotropic reorientation, adamantane provides a test case).

Yours sincerely.

D. CANET J. BRONDEAU C. MILLOT H. NERY L. WERBELOW

B.P. 239 - 54506 VANDOEUVRE les NANCY cedex Tel. (8) 328.93.93

Unité Associée au C.N.R.S. n°406 - Laboratoire d'Etude des Solutions Organiques Colloïdales



INDIANA UNIVERSITY

DEPARTMENT OF CHEMISTRY
Chemistry Building
Bloomington, Indiana 47405
(812)

April 24, 1984
812-335-1639

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

Dear Dr. Shapiro:

Indiana University has a Varian HR-220 Superconducting NMR Spectrometer for sale. The instrument is fully operational and includes CW ^{1}H and Pulsed FT ^{2}H and ^{11}B capabilities. Interested parties should contact me for additional details.

Sincerely,

Robert Addleman

Robert Addleman
NMR Supervisor

PURDUE UNIVERSITY

SCHOOL OF SCIENCE
at INDIANAPOLIS

PHYSICS DEPARTMENT
1125 East 38th Street
P.O. Box 647
Indianapolis, Indiana 46223
(317) 923-1321

August 30, 1984

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

^{1}H NMR of Aequorin

Dear Barry:

We are using magnetic resonance techniques to study the Ca(II)-sensitive bioluminescent protein aequorin which is isolated from jellyfish. Aequorin, an intriguing protein with a molecular weight of 20,000, emits light at 469 nm from an excited state of a substituted pyrazine which results from the oxidation of a low molecular weight chromophore which is non-covalently bound to aequorin. In the absence of Ca(II), spontaneous emission of light, referred to as Ca(II)-independent light emission, occurs at a rate much less (at least a factor of 10^6) than the rate of emission induced by Ca(II). ^{1}H NMR spectra taken in the course of time as the protein slowly becomes inactive (discharges) with no Ca(II) present show that the Ca(II)-independent emission is accompanied by a change in protein structure from a rigid form to a practically unfolded, inactive form in which a number of amino-acid residues are quite mobile. This is indicated by a substantial narrowing of many of the resonances of the slowly discharged protein as shown for the aromatic region of the spectrum in the Figure. Also shown is the aromatic region of the ^{1}H NMR spectrum of slow discharged aequorin from which the oxidized chromophore has been removed. The resonances of this apo-protein are intermediate in width. Apparently the slow discharged protein partially refolds when the chromophore is removed. The substantial changes noticed in the aromatic region suggest that some of the aromatic residues must be involved in the chromophore binding. These results will soon be submitted for publication in more detail.

Yours truly,

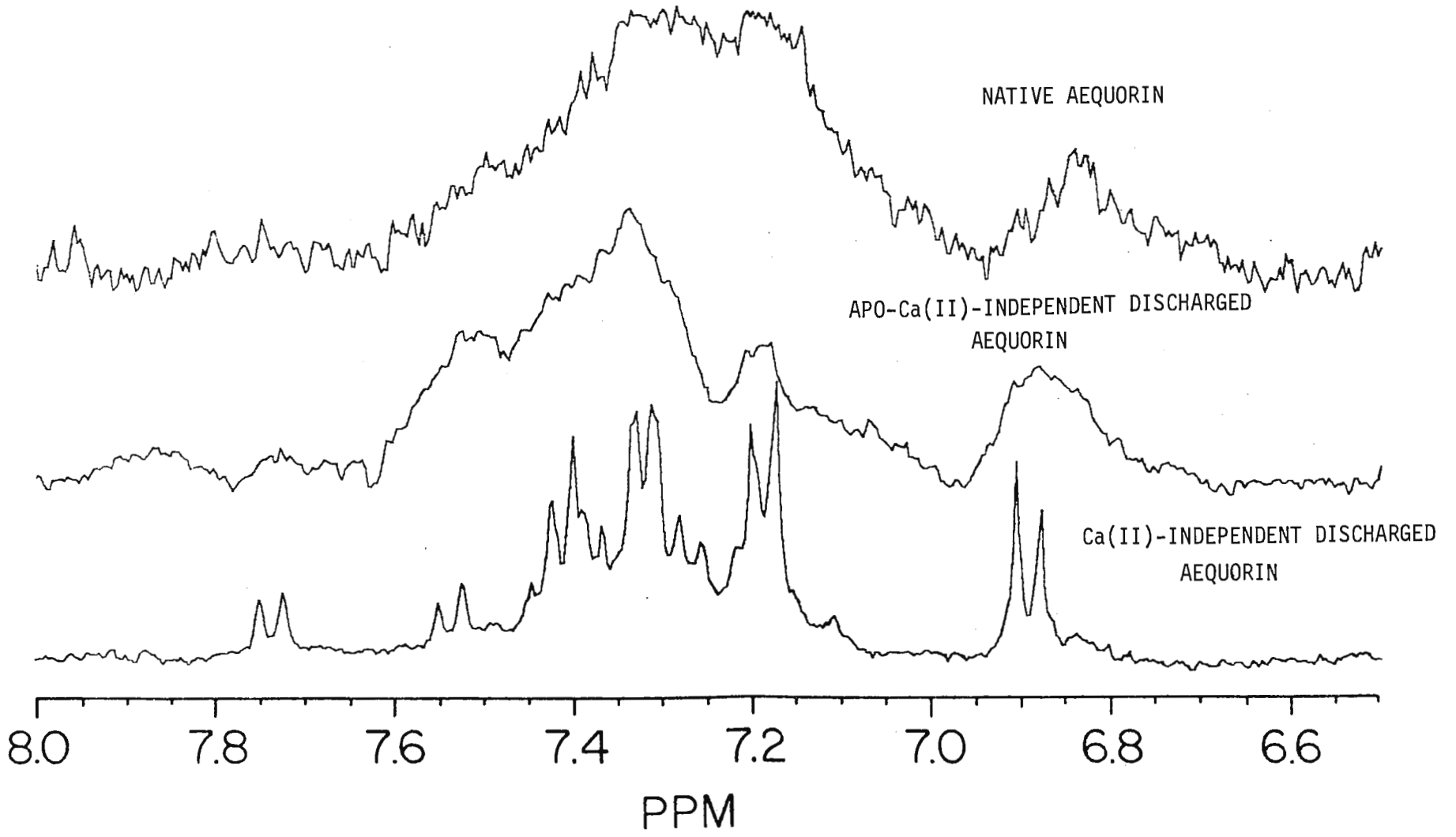
Marvin D. Kemple

Marvin D. Kemple

Please credit this to the account of B. D. Nageswara Rao.

Aromatic region of the 300 MHz ^{1}H NMR spectra

of various forms of aequorin taken at $5^{o}C$.



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D-8046 GARCHING, den 31.07.1984
Lichtenbergstraße 4
Ruf-Nr. (089) 3209/3080/3081 (Prof. Fischer)
3110 (Prof. Fritz)
3130 (Prof. Schmidbaur)
Telex: 17898174
Teletex: 898174

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

Title: Temperature Dependence of the ^{127}I-Nuclear Quadrupole Coupling in Tetramethylammonium-periodate $(CH_3)_4N^+IO_4^-$ (1)

Dear Professor Shapiro:

The ^{127}I-NMR powder spectra of (1), measured between 200 K and 300 K, are depictured in Fig. 1. The last spectrum at 300 K shows the typical ^{127}I-NMR signals of the II. order quadrupole splitting, while all other spectra with temperatures below 296 K have additional signals probably from a further quadrupole splitting. Lowering the temperature at least one of these coupling constants increases because there are two possibilities in calculating the quadrupole splittings $\Delta\nu_n$.

Case A with $z \rightarrow z''$ $(\Delta\nu_1)$, $z^* \rightarrow z'$ $(\Delta\nu_2)$ and
Case B with $z \rightarrow z'$ $(\Delta\nu_3)$, $z^* \rightarrow z''$ $(\Delta\nu_4)$

By knowing the Larmor precession frequency ν_L and the measured values of $\Delta\nu_n$ for the respective temperatures the quadrupole coupling constants can be calculated where the asymmetry parameter η disappears ($\eta = 0$). The test results for compound (1) are shown in Fig. 2.

Independent of the two possible cases the diagram shows the appearence of a second phase at $T < 296$ K. The quadrupole coupling constants of this low temperature phase are in order of 15 to 20 MHz. All ^{127}I quadrupole interactions of compound (1) have a normal temperature dependence with a negative temperature coefficient α and therefore a I. order phase transition is present. That means a discontinous change of the tetragonal crystal structure of the high temperature modification. This will change the symmetry properties in the lattice and create two inequivalent positions of the iodine atoms.

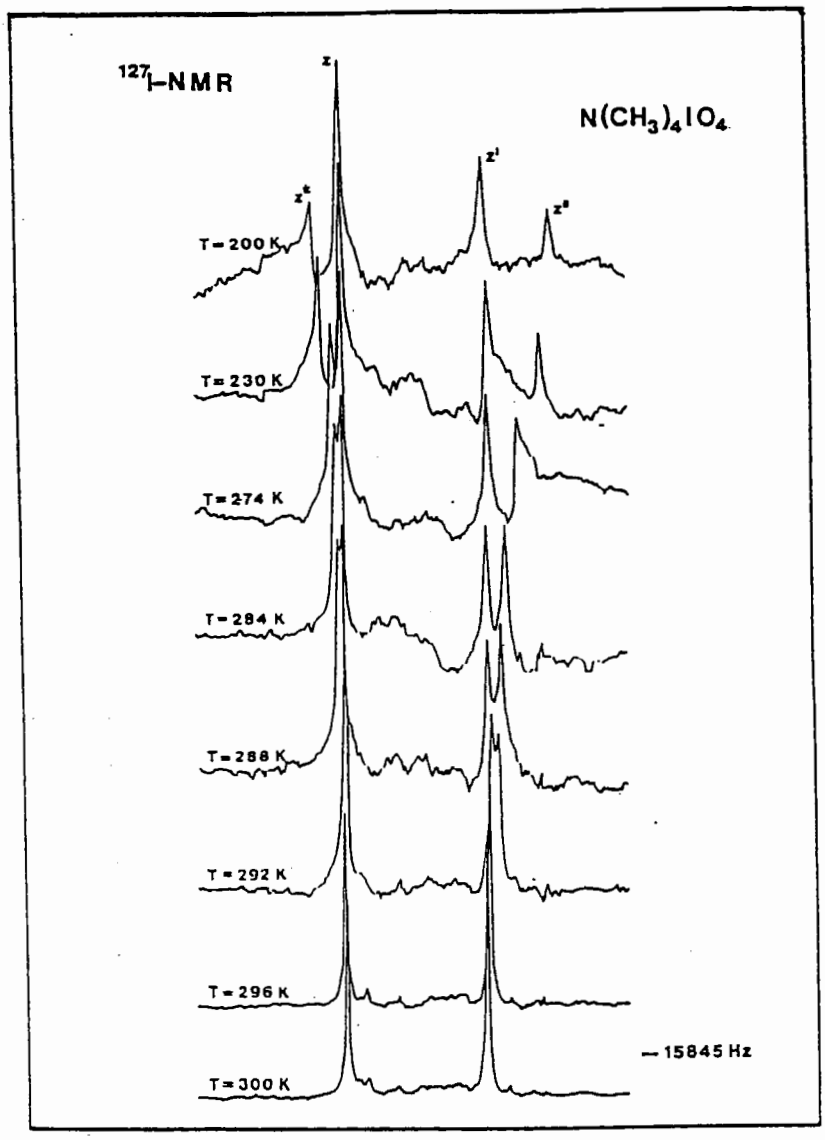

Fig. 1. ^{127}I-NMR signals of II. order quadrupole splittings $\Delta\nu$, measured between 200 K and 300 K.

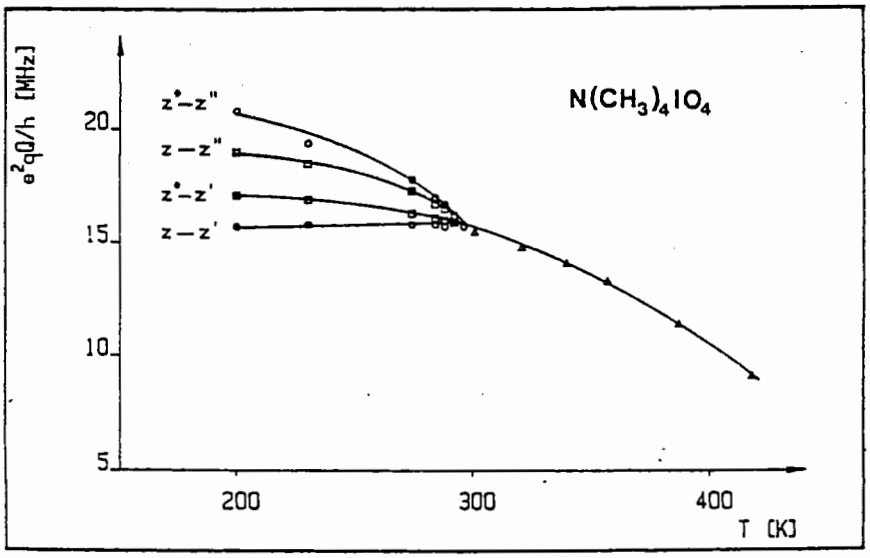

Fig. 2. Temperature dependence of ^{127}I quadrupole coupling constants e^2qQ/h, measured by II. order quadrupole effects in I-NMR powder spectra of $(CH_3)_4N^+IO_4^-$

Moreover it is remarkable that an extrapolation of the known e^2qQ/h (T) - curve (ΔΔΔ) into the region T < 300 K runs through the average values of the quadrupole coupling constants calculated from the two possible cases A and B.

From our measurements it is impossible to decide, which of the two cases correctly describes the behavior in the lattice, where obviously two sorts of distorted IO_4^--tetrahedrons exist. With case B one of the IO_4^--tetrahedrons is provided with a small electric field gradient, which is independent of temperature (lower curve ooo). The other has a larger electric field gradient, which is temperature dependent. Consequently the IO_4^--tetrahedron is more distorted.

If case A is the right, one group of the IO_4^--tetrahedrons changes its field gradient at the iodine atom in the same way as the high temperature modification. This can be seen in Fig. 2, where the Δ-curve passes continously into the lower □-curve. For the other IO_4^--tetrahedron one observes an insignificantly larger electric field gradient. In this prefered version there are smaller deviations in the lattice.

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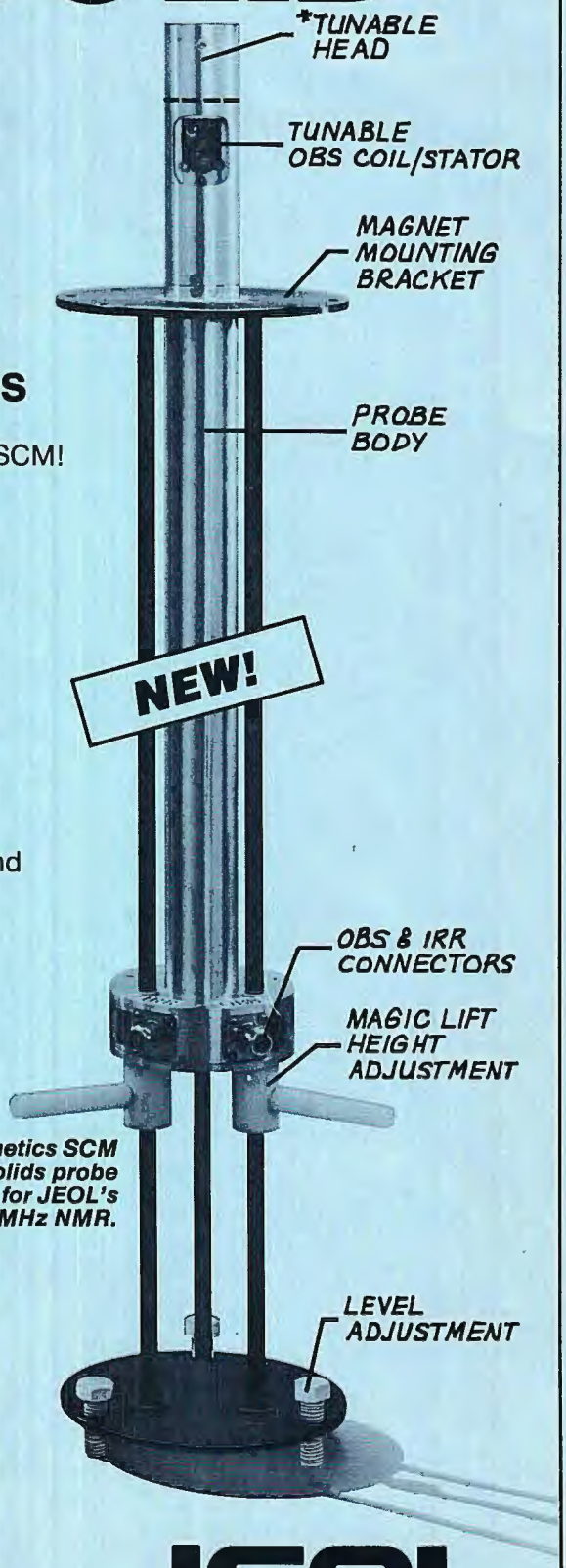

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