TEXAS A&M UNIVERSITY

NMR NEWSLETTER

NO. 309

JUNE 1984

DEADLINE DATES

No. 311 --------- 6 August 1984

No. 312 ------ 3 September 1984

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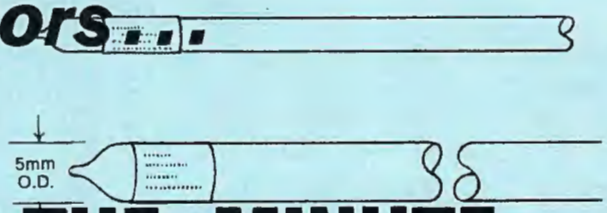

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	WGH-02	Acetaldehyde (Neat)	Resolution	32.00
	WGH-21	20% Acetaldehyde + 2% TMS in Benzene-d$_6$	Resolution	33.00
	WGH-23	20% Acetaldehyde in Acetone-d$_6$	Resolution	33.00
	WGH-22	20% Acetaldehyde in Benzene-d$_6$	Resolution	33.00
	WGH-24	20% Acetaldehyde + 2% TMS in Acetone-d$_6$	Resolution	33.00
	WGH-17	Orthodichlorobenzene (Neat)	Resolution	32.00
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CONTRIBUTORS

Chemagnetics, Inc.
Intermagnetics General Corporation

All Newsletter Correspondence
Should be Addressed to:

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

AUTHOR INDEX ------ TAMU NMR NEWSLETTER NO. 309, JUNE 1984

Xerox Corporation
Joseph C. Wilson Center for Technology
Webster, New York 14580

May 4, 1984

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

XEROX

Dear Barry:

Signals from High Resolution Sample Tube Caps

Recently, we have been looking at the proton NMR of a number of low solubility materials (ca. 10^{-4}–10^{-5} molar) in $CDCl_3$ solution on the University of Rochester's WH-400 spectrometer. At first we were puzzled why the dominant component in each of these supposedly aromatic materials was a long aliphatic chain. The explanation for this appeared to be that chloroform was extracting out low molecular weight polyethylene oligomers from the sample tube caps. Since these samples were prepared a day in advance and were stored in a container for transporting to the U of R, overnight contact of solvent with the caps was not unlikely. To test this possibility we put Aldrich 99.8% $CDCl_3$ in a number of Wilmad NMR tubes, some inverted and some kept upright overnight. Two spectra are shown below. Clearly the inverted tube (507 PP with a yellow cap, bottom figure) shows a very large polyethylene signal (concentration of ca. 24 parts per thousand by weight) as well as a number of smaller unidentified peaks, presumably from the cap as well. We obtain similar spectra from green caps. Even in the upright tube (top figure) chloroform vapors apparently leach out a small amount of polyethylene (ca. 0.7 parts per thousand) from the cap.

We do not know whether this problem is of general knowledge and therefore thought it appropriate to inform your readers of it. Possible solutions are to use teflon caps, seal the tube with teflon tape before inserting a standard cap, or flame seal the tube so that no cap is necessary.

Sincerely,

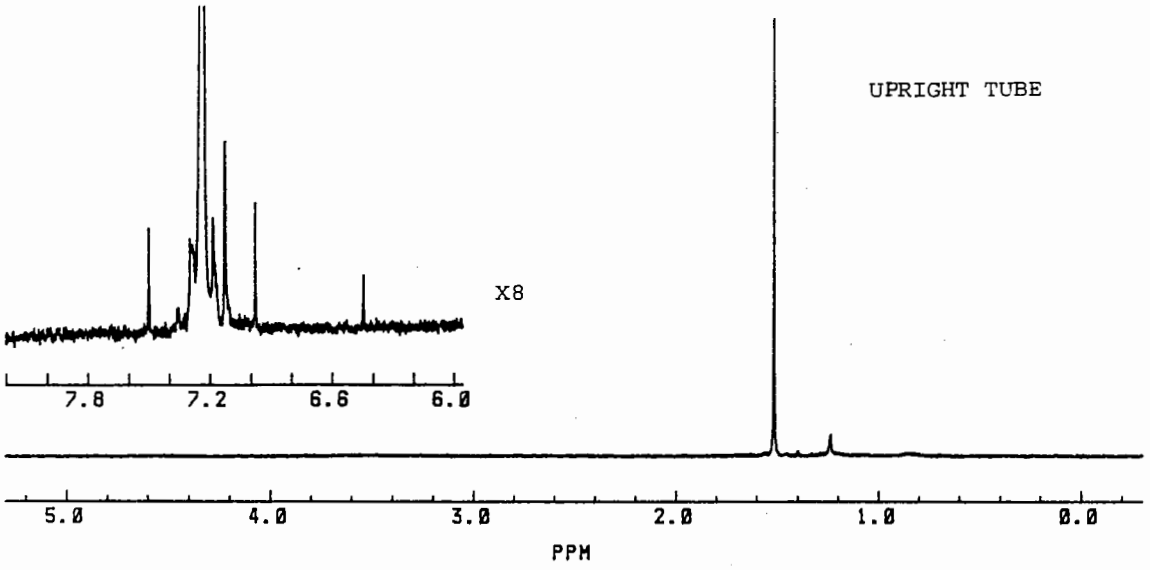
Sam

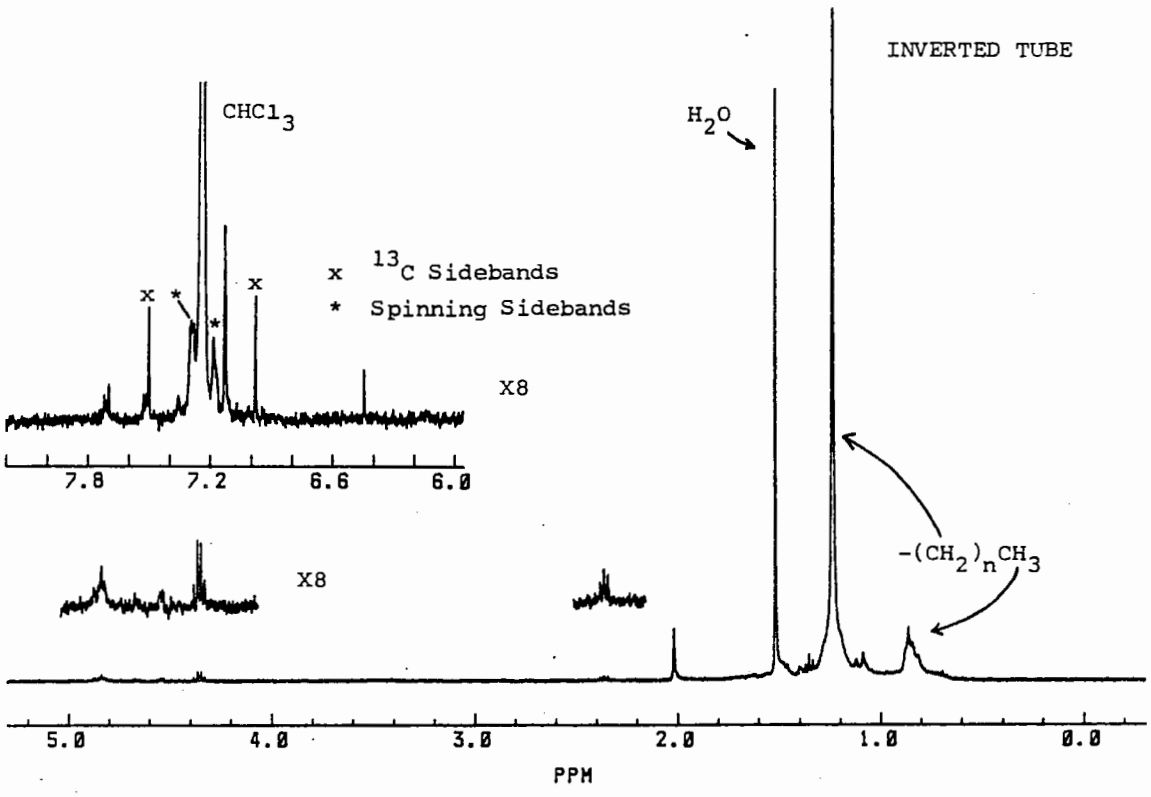
Samuel Kaplan

Ray

Ray Crandall

SK/RC/dc


UPRIGHT TUBE
X8
7.8
7.2
6.6
6.0
5.0
4.0
3.0
2.0
1.0
0.0
PPM


INVERTED TUBE
CHCl3
H2O
x 13C Sidebands
* Spinning Sidebands
X8
X8
-(CH2)nCH3
7.8
7.2
6.6
6.0
5.0
4.0
3.0
2.0
1.0
0.0
PPM

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1984 May 7

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

Dear Barry,

A Structure for the Catalytic Complex of
Ribulose Bisphosphate Carboxylase

In order to meet your deadline, we are rushing the following note. Please credit this contribution to the account of Donald D. Bly.

We have started some NMR studies in biological systems using an NT-360WB spectrometer. One of the outcomes of work is described.

The complexation of ribulose bisphosphate carboxylase (Rubisco) with CO_2, Mg^{2+}, and carboxyarabinitol-P_2 (CABP) to produce the quaternary complex, ECM-CABP, closely mimics the formation of the catalytically competent ECM-3-keto-CABP form during enzymatic catalysis. We have prepared large quantities of the enzyme from Rhodospirillum rubrum via large scale fermentation of E. coli containing the Rubisco gene on a high copy number plasmid. This enzyme was used in the preparation of quaternary complexes with various metals (Mg^{2+}, Cd^{2+}, Mn^{2+}, Co^{2+}, and Ni^{2+}) and with specifically C-enriched ligands. ^{31}P and ^{13}C NMR studies of these complexes demonstrate that the activator CO_2 site (carbamate site), the metal binding site, and the substrate binding site, and the substrate binding site are continguous. The results indicate that the metal ion is intimately involved in both enzyme catalysis and enzyme activation through binding to the carbamate oxygen ligands and to O-2 and the carboxyl oxygens of the reaction intermediate, 3-keto-CABP. This structure shown on the next page suggests a mechanistic unification of the carboxylase and oxygenase reactions in which the metal ion acts both as a scaffold for group transfer reactions and as an electron sink for the C-2, C-3 bond breaking steps in the reactions. It

follows that the carboxylase and oxygenase activities are influenced by the structures of both the catalytic and activation sites.

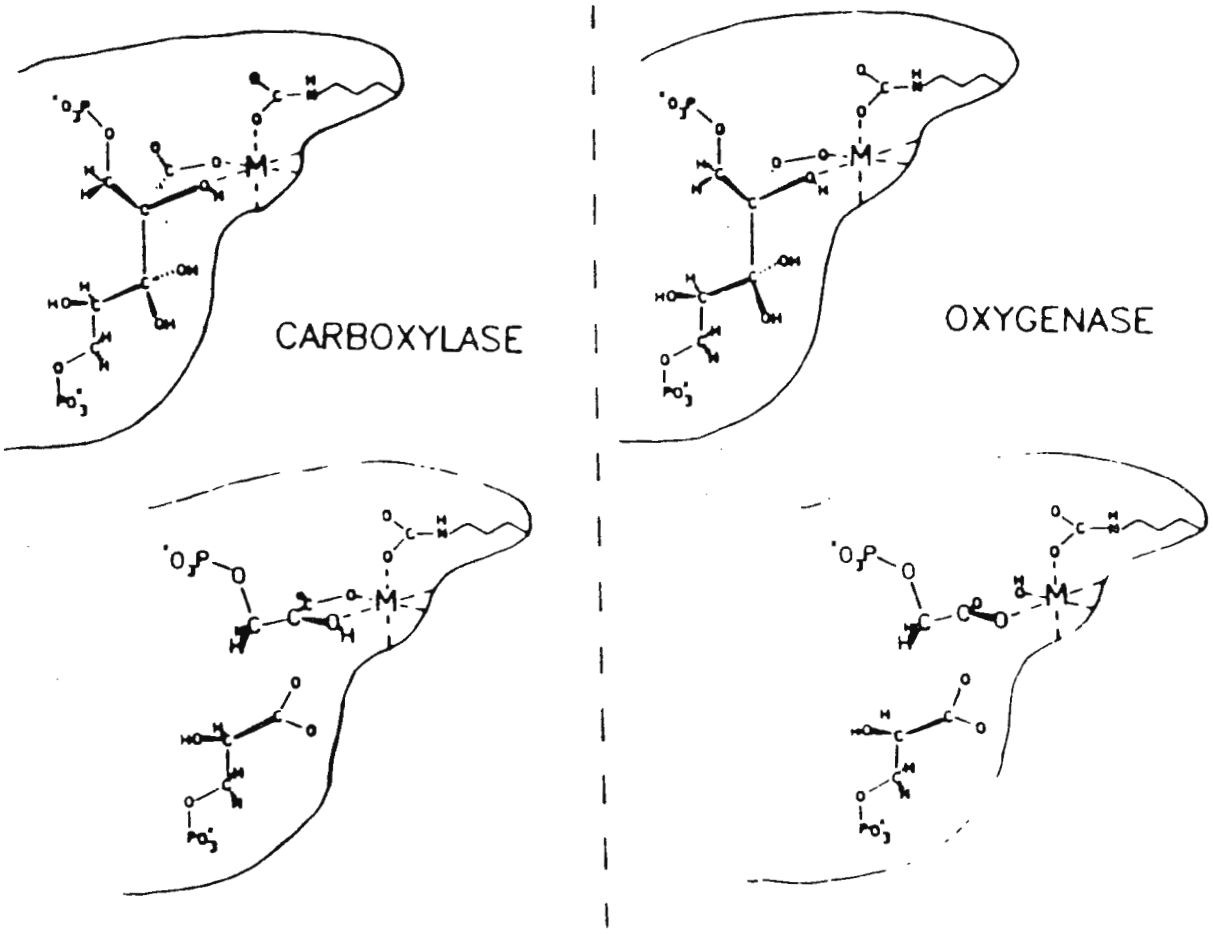
These results will be presented at the "Molecular Biology - Photosynthetic Apparatus" Conference to be held at Cold Spring Harbor Laboratory during May, 1984, and also will be communicated to the "Journal of Biochemistry" shortly.

Sincerely,

Gade S. Reddy

John Pierce

GSR:JP/dew



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UNIVERSITE D'AIX-MARSEILLE
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TEL. (91) 62.15.54

Professeur P. J. COZZONE

Marseille, le May 7, 1984

Professor SHAPIRO
Department of Chemistry
College of Science
Texas University
College Station TX 77843
U.S.A.

^{31}P NMR investigation of post mortem ATP catabolism in different muscle types

Dear doctor Shapiro,

We have used P-31 NMR at 80.9 MHz to compare post mortem catabolism of phosphorylated metabolites in different rabbit muscle types. In particular, we have monitored the fall of intracellular pH and the inter-related catabolisms of inorganic phosphate, ATP and phosphocreatine (PC) in pure fast (αw) and slow twitch (βR) muscles as well as in muscles with mixed fiber types (αw, + βR, + αR).

The main results are as follows :

1. Initial levels of ATP and PC in Conoïde (βR) are lower than in Psoas (αw).
2. ATPase activity and ATP resynthesis from PC have been studied. The longest half-life of ATP is observed for Psoas. The lower initial concentration of PC in slow twitch Conoïde is catabolized more rapidly than in the fast twitch Psoas.
3. For all muscles, the rates of decrease in intracellular pH were similar. However, there is some indication that the buffering capacity might vary to some extent according to fiber types.
4. In addition, heterogeneous Pi signals have been recorded for all muscle types suggesting a distribution of inorganic phosphate among several compartments (e.g. sarcoplasm and sarcoplasmic reticulum) with different internal pH values.

We are currently studying further the nature of this compartmentation.

Sincerely yours,

P. Cozzone — P. Canioni

Ph. Gatellier* — J.P. Renou* — C. Valin*

* Institut National de la Recherche Agronomique - Station de Recherches sur la Viande - Theix 63122 CEYRAT

		Psoas	Gastrocnemius	Conoïde
pH	pH_0	7,14	7,2	7,02
	Rate 10^{-3} pH min^{-1}	- 3,3	- 2,7	- 3,0
Pi	$(Pi)_0$ μmg^{-1}	11	13	13,5
	Rate 10^{-3} μmg^{-1} min^{-1}	113	57	30
PC	$(PC)_0$ μmg^{-1}	30	21	7
	Rate 10^{-3} min^{-1}	9,8	13,6	25,2
ATP	$(ATP)_0$ μmg^{-1}	8,7	7	3,8
	$t_{\frac{1}{2}}$	326	300	240

Post mortem changes in rabbit muscles stored at 25°C

309-7

VRIJE UNIVERSITEIT

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telefoon 020 - 548

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

uw kenmerk	uw brief van	ons kenmerk	datum	bijlage(n)
			May 4, 1984	

onderwerp

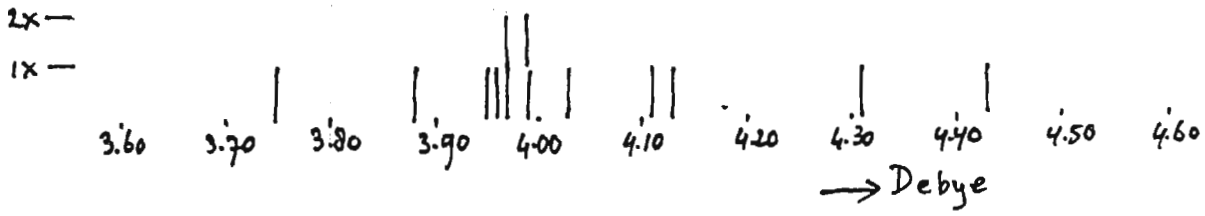
"The dipole moment of nitrobenzene"

Dear Barry,

McClellan's invaluable compendium [1] of electric dipole moments of molecules contains more than 11,000 entries. Perusal shows that dipole moments of numerous molecules have been investigated by more than one author, often by different methods. In many cases the scatter of the numerical results is small, say $\pm$ 0.1 D.

Remarkably, the literature values of the dipole moment of nitrobenzene scatter widely, even if measurements in one and the same solvent are considered. For nitrobenzene in benzene, sixteen values are reported which cover the range of 3.33 - 5.17 D. The data can be represented by the stick diagram of fig. 1.

FIG 1 Nitrobenzene in Benzene



It is obviously of interest to understand the reason of the above scatter and to select the "best" value. For example in ref. 2 the dipole moment is stated to be 4.24 D; from this value the Kirkwood g-factor is calculated as 0.87 at 25 ^{o}C and 0.83 at 200 ^{o}C. A g-value < 1.0 is thought to be typical for molecules whose dipole axis is the long axis of the molecule, as in nitrobenzene.

In an independent approach we have measured the alignment of nitrobenzene in benzene as a function of the mole fraction. The NMR spectrum of perdeuterobenzene was recorded while the liquid was exposed to a strong electric field. The deuterium splitting is directly related to the magnitude of the alignment [3]. Making assumptions which are common in the interpretation of dielectric polarization, one has [4]

$$\langle \tfrac{3}{2}\cos^2\theta - \tfrac{1}{2}\rangle_E / E^2 = \frac{1}{15}\left\{\frac{\varepsilon_{12}(n_1^2+2)}{2\varepsilon_{12}-n_1^2}\right\}^2 \left[\frac{m}{kT}\right]^2 \qquad (1)$$

E is the applied field, m is the dipole moment, ε_{12} is the static dielectric constant of the mixture, n_1 is the index of refraction of the polar component (nitrobenzene).

The numerical value of the l.h.s. of eq. 1, extrapolated to infinite dilution in benzene is 1.09×10^{-9} e.s.u. at room temperature. Substituting $\varepsilon_{12} = 2.28$; $n_1 = 1.55$, one finds m = 3.6 D. The Kirkwood g-factor then is 1.21.

An important point is assessing this result is the following: induced polarization plays no role in our considerations. The alignment is practically not affected by the anisotropy in the polarizability. On the contrary, to relate the dielectric constant to the dipole moment, the first point is strongly relevant. It is well known that the separation of induced polarization from orientational polarization is not always straightforward. This may explain part of the scatter in the dipole moments tabulated by McClellan. However, the following is probably of major importance.

The alignment of nitrobenzene in benzene depends strongly on the mole fraction [4] ; for the pure liquid the l.h.s. of eq. 1 amounts to $0.52\ 10^{-8}$ e.s.u! Even at a mole fraction of 0.1 no plateau is measured. This suggests that, even at small concentrations, intermolecular interactions between nitrobenzene molecules play a role. Polarization measurements should thus be carried out as a function of concentration, the smallest concentration giving the best value. According to McClellan, such experiments have been reported in ref. 5. Depending on concentration m varies between 3.33 - 3.80 D!

Experiments in several solvents are under way to settle this problem.

References

[1] A.L. McClellan, "Tables of Experimental Dipole Moments", vol. 2, Rahara Enterprises, 1974.

[2] A.D. Buckingham, Discuss. Farad. Soc., 43, 205 (1967).

[3] P.C.M. van Zijl, B.H. Ruessink, J. Bulthuis and C. MacLean, Acc. Chem. Res. (in press).

[4] J. Biemond and C. MacLean, Mol. Phys. 26, 409 (1973).

[5] S.K.K. Jatkar and V.K. Phansalkar, J. Univ. Poona, Sci. Technol. 22, 57 (1962).

Yours sincerely, C. MacLean B.H. Ruessink

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April 30, 1984

Dr. B. L. Shapiro
TAMUNMR Newsletter
College of Science
Texas A&M University
College Station, TX 77843

"Data Transmission between the Aspect and the IBM System 9000"

Dear Barry:

In answer to your multicolored reminders, I wish to report that before (and after) having changed "hats," I have been spending some time studying the problem of communication between the Aspect computers and the IBM System 9000 series computers. We developed a number of ways which are now embedded in the released software for the System 9000.

The first is the TRSB or TRSC (Transfer to Specnet) commands in DISNMR, DISCXP and the TM command of FTQNMR. These all produce a well-defined data stream out of the Channel B or Channel C RS-232 port, which can be listened for by the System 9000. These standard Specnet format files are then sign-extended to 32-bits and stored on the System 9000 floppy or hard disk for processing using the nmr package.

A second way is by simply using system calls to reprogram the System 9000 floppy diskette drive to read Aspect diskettes. This has resulted in the FDCONV program which is now available from IBM.

Best wishes from incipient Connecticut.

Sincerely,

Jim

James W. Cooper, Ph.D.

/lw

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9th May, 1984

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

Dear Barry,

"Stereodynamic properties of perylenequinones with helical chirality, studied by NOE"

Cercosporin, phleichrome and the elsinochromes are metabolites of some species of fungi, of which several are pathogenic for many plants. Recent interest has arisen in the photodynamic antibacterial activity and in the mechanism of the phytotoxic activity. An unusual feature of these molecules is the complex stereochemistry since the perylenequinone system is inherently dissymmetric: it possesses only a C_2 axis; additional asymmetric centers are on the side chains. The twisting of the aromatic ring, forced into a helical shape by the steric constraint of the methylenedioxy group and of the side chains, is clearly observed in the CD spectra and in the X-ray diffraction pattern. This latter analysis, performed on a derivative of cercosporin 1 by the oxygen anomalous dispersion method, gave the absolute configuration (R) of the side chain carbons and the helicity of the ring. Assuming the axis C_{6a}—C_{6b} as a chirality axis, the axial chirality of this compound is (R)[1]. The inversion of the helicity of the ring for 1 and 2 to give the iso-compounds 2 and 4, occurs by heating. Simultaneously a change in the conformation of the side chains takes place: they assume a different orientation toward the perylenequinone system.

The preferred conformation for all the compound 1-8 has been estabilished through extensive use of nuclear Overhauser enhancement (NOE) experiments: the irradiation of the methyl protons of the side-chains induces different NOE effects on the protons at C-13 and C-16. This allowes to know the preferred conformation of the chains and their relationship with the helical perylenequinone system.

In particular the torsion around the bonds C_1—C_{13} and C_{12}—C_{16} resp. for compounds 1-4 has been studied by proton chemical shifts and coupling constants ($^2J_{CH}$ and $^3J_{CH}$); the torsion around the bonds C_{13}—C_{14} and C_{16}—C_{17} resp. by NOE experiments and by $^3J_{HH}$/dihedral angle relation. For compounds 5-8 the equatorial-equatorial orientation of H-13 and H-16 has been deduced from $^3J_{HH}$ values.

The absolute configuration thus obtained for the chains and the C-13,16 centers are reported in the Table.

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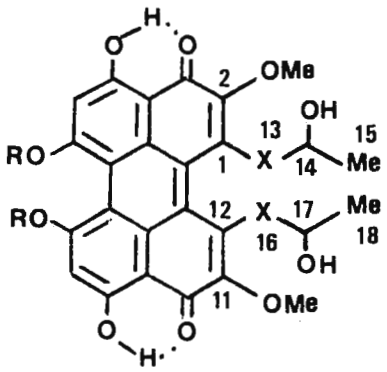
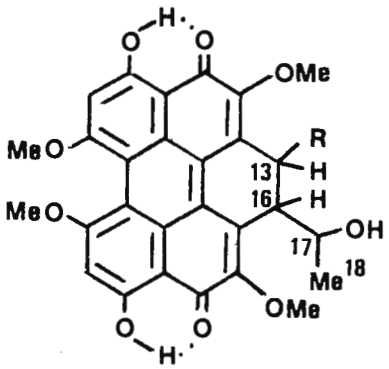
[1] L.Merlini and G.Nasini, 2nd Int. Conf. on Chemistry and Biotechnology of Biologically Active Natural Products, Budapest, 1983, Akademiai Kiadó, in press.





1	cercosporin	R-R = CH_2	5	elsinochrome B_1 [(b)]	R = COMe
2	isocercosporin	R-R = CH_2	6	elsinochrome B_2	R = COMe
3	phleichrome	R = Me	7	elsinochrome C_1	R = CHOHMe (14) (15)
4	isophleichrome	R = Me	8	elsinochrome C_2	R = CHOHMe

	absolute helicity	configuration chains	C-13,16	conformation	NOE% [(a)] of H-13A	H-13B
1	R	R R		"OH exo"	0	3.2
2	S	R R		"OH endo"	2.0	1.7
3	S	S S		"OH exo"	0	2.3
4	R	S S		"OH endo"	2.6	2.5
5	R	S	S S	"Me-exo"	2.0	
6	R	R	S S	"Me-exo"	7.6	
7	R	S S	S S	"Me-exo	8.7	
8	R	R R	S S	"Me-exo	10.9	

(a) NOE effects were measured on a Bruker CXP-300 spectrometer, by employing the difference spectroscopy method. For compounds with C_2 symmetry (1-4, 7 and 8) the irradiation of Me-15 and Me-18 induces NOE of the equivalent protons at C-13 and C-16; but in the case of 1-4 the two protons (A and B) of each methylene group are not equivalent and experience different effects. For 7 and 8, NOE experiments exclude only the trans conformations Me-18/H-16 and Me-15/H-13 respectively; thus the absolute configuration of the chains (C-14,17) has been obtained by shift values. Finally the irradiation of Me-18 induces different NOE effects for 5 vs 6 compound.

(b) The previously described elsinochromes B and C (U.Weiss and coll., J. Chem. Soc.(C), 1219 (1969) are mixture of diastereoisomers.

I apologize for the delay, with many thanks,

Yours sincerely,

dr. A. Arnone Prof. R. Mondelli

Drexel University • Philadelphia, Pennsylvania 19104

Drexel University

College of Science
Department of Chemistry
(215) 895-2638, 2639

May 16, 1984

Dr. B. Shapiro
TAMU Newsletter
Department of Chemistry
Texas A & M University
College Station, Texas 77843-3255

Dear Barry,

I wanted to inform your readers of some recent studies we have made using the pulsed-field gradient spin-echo NMR technique. We have been using PGSE to study microemulsions of various types. A microemulsion is a 1 phase system containing water, oil and emulsifier. An interesting system is one made up of didoceyldimethylammonium bromide, water and hydrocarbons from hexane through tetradecane. These novel 3-component systems have been characterized by D.F. Evans et.al.(1) Plotted in the figure are the self-diffusion coefficients of water as a function of water content for hexane, decane and tetradecane systems. The unusual feature of the hexane and decane data is that the diffusion of water gets slower as water is added. This occurs as the macroscopic viscosities decrease and the solutions go from conducting to non-conducting. This behavior is just opposite of that in other microemulsions whose water diffusion coefficients increase with added water.

On the basis of these and other observations we have concluded that the systems studied are both water and oil continuous at low water contents. The water is contained in a continuous tube structure. As water is added the system forms more typical water in oil spheres. As can be seen from the figure there is also oil specificity and the tetradecane system does not become non-continuous in water. The PGSE method is a very powerful structural tool for these systems..

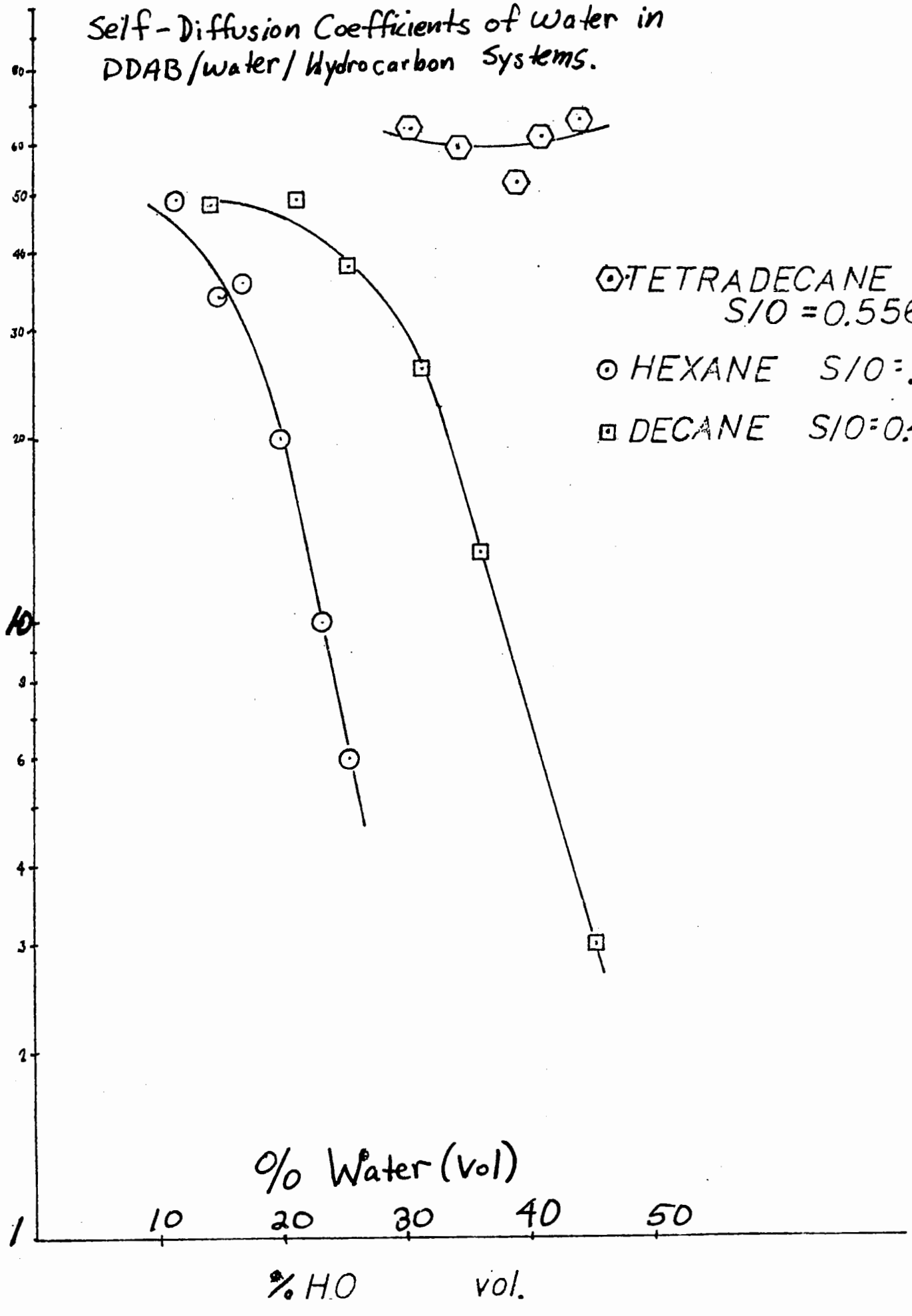
Sincerely,

Frank D. Blum
Assistant Professor of Chemistry

Steve Pickup

1. S. J. Chen, D.F. Evans, B.W. Ninham, J. Phys. Chem., 1984 in press.

FDB/dc
Enc.


Self-Diffusion Coefficients of Water in DDAB/water/Hydrocarbon Systems.
TETRADECANE S/O = 0.556
HEXANE S/O = .447
DECANE S/O = 0.486
$10^{10} \times D$ (m^2/s)
60
50
40
30
20
10
9
6
4
3
2
1
% Water (vol)
10
20
30
40
50
% H.O vol.

309-15

PHILIP MORRIS

U.S.A.

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

May 21, 1984

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

Dear Barry:

Bugs in the Magic Angle Spinning Probe

Recent notes in the TAMU NMR Newsletter by G. W. Buchanan (1) and J. N. Shoolery (2), as well as some experiments that I have been doing, prompted me to try observing cigarette beetle larvae by 13C NMR using a magic angle spinning probe. Many solid materials of biological origin contain liquids or highly mobile components that exhibit 13C signals which are not significantly broadened by dipolar interactions or chemical shift anisotropy. Thus, Prof. Buchanan was able to detect triglyceride fatty acids in insect larvae by 13C NMR using a conventional high resolution probe. As Jim Shoolery has pointed out, however, there is still some resonance broadening in such materials which results from variations in the magnetic susceptibility within the sample. Since this broadening is at most a few ppm, slow spinning at the magic angle can be employed to obtain solution quality spectra (3). The improvement in resolution is easily seen by comparison of the attached spectrum with Buchanan's results (1), but it is important to realize that the susceptibility effects are field dependent and MAS is even more crucial at higher field strengths. This was a rather crudely done experiment; consequently, I have liberally applied Shoolery's recommended method for removing spectral artifacts (4). I was not scrupulously concerned about the safety of the larvae, but since the spinning speeds required are not great, it may be possible to do the experiment without harming the bugs.

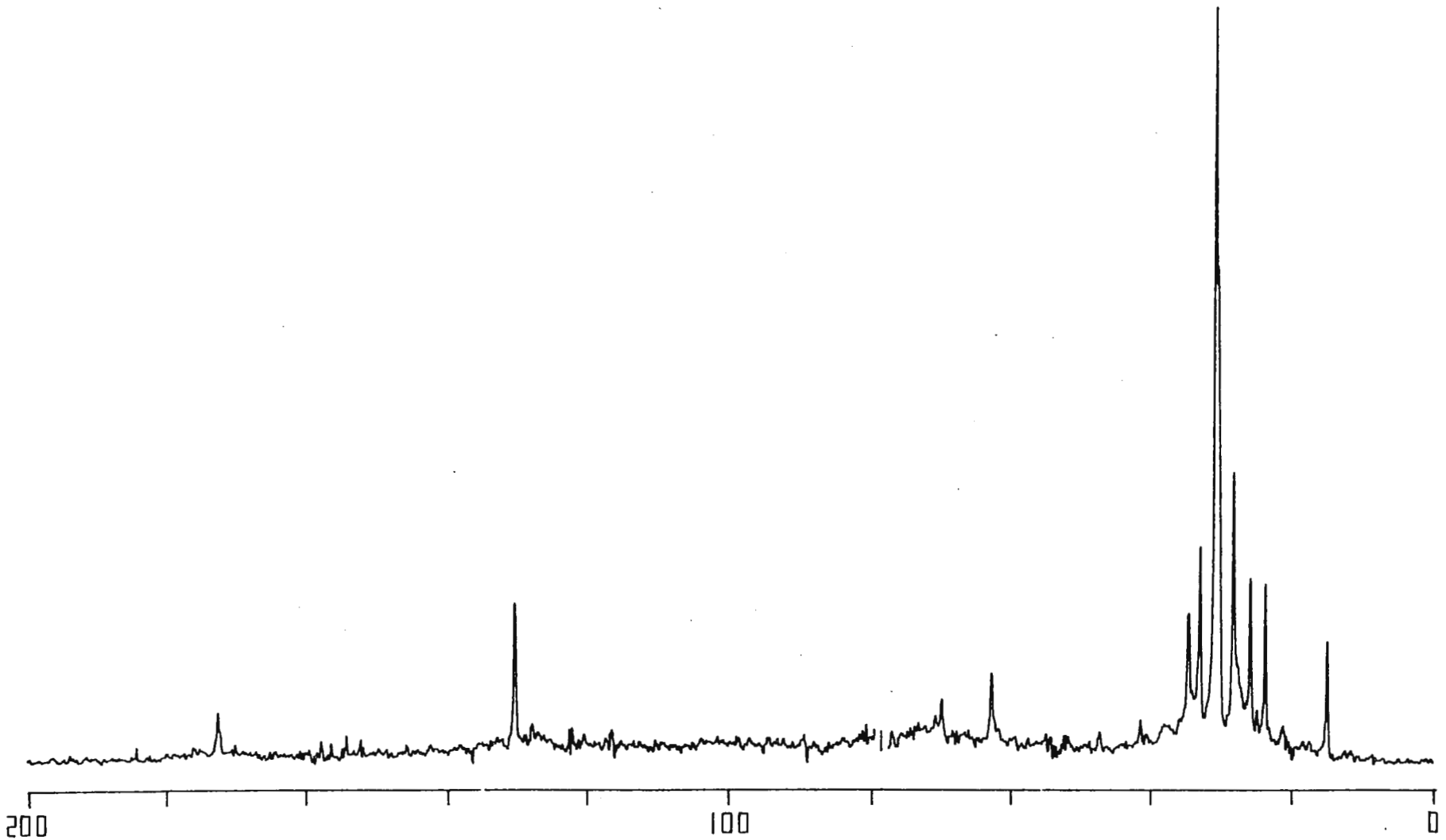
Sincerely,

Jan

Jan B. Wooten
Research Scientist

References: 1) G. W. Buchanan, TAMU NMR Newsletter no. 298, July, 1983.
2) J. N. Shoolery, TAMU NMR Newsletter no. 301, October, 1983.
3) A. N. Garroway, J. Mag. Res. 49, 168 (1982).
4) J. N. Shoolery, Varian Users Conference, Palo Alto, 1983.

JBW/clc



50 MHz ^{13}C MAS spectrum of cigarette beetle larvae.

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May 23, 1984

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

Dear Barry:

The study of isotopic multiplets in the ^{13}C NMR spectra of polyols with partially deuterated hydroxyls is progressing well. Partial deuteration is achieved by adding calculated amounts of D_2O to a DMSO-d_6 solution of the material. The multiplets are due to upfield isotope effects on the ^{13}C shifts: 0.09-0.12 ppm for directly bonded hydroxyls (β-effect) and 0.07 ppm or less for hydroxyls on vicinal carbons (γ-effect).

In cyclic vicinal diols the magnitude of the γ-effect is sensitive to the relative geometric relationship, cis or trans, of the hydroxyls: the trans-effect is larger (JACS, 1984, 106, 2461). This led to the realization that the multiplet pattern for carbons 3 and 4 of pyranoses can serve as a fingerprint of molecular structure at the pentopyranose level. Examples are shown in the Figure (from the top: β-D-allopyranose, α-D-galactopyranose, α-D-lyxopyranose, α-D-xylopyranose). Note that the relative arrangement of hydroxyls at carbons 2, 3 and 4 is reflected in the multiplet pattern for C3. However, the arabino and lyxo structures are symmetry related. Therefore, in order to distinguish them from one another, the multiplet of C4 is needed as well. In oligosaccharides, the kind and number of isotopic multiplets for the nonanomeric methines (carbons 2, 3, 4, and 5 of aldohexoses; carbons 3, 4, and 5 of ketohexoses) form sets characteristic of the position of substitution and can be used in tracing the sequence of glycosidic linkages. An approach for the ab initio analysis of di- and trisaccharides was devised. The new approach is based on the isotopic multiplets and on some well known features of the ^{13}C chemical shifts of carbohydrates.

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With kindest regards,

Sincerely,

Jacques Reuben
Analytical Division

JR:mew
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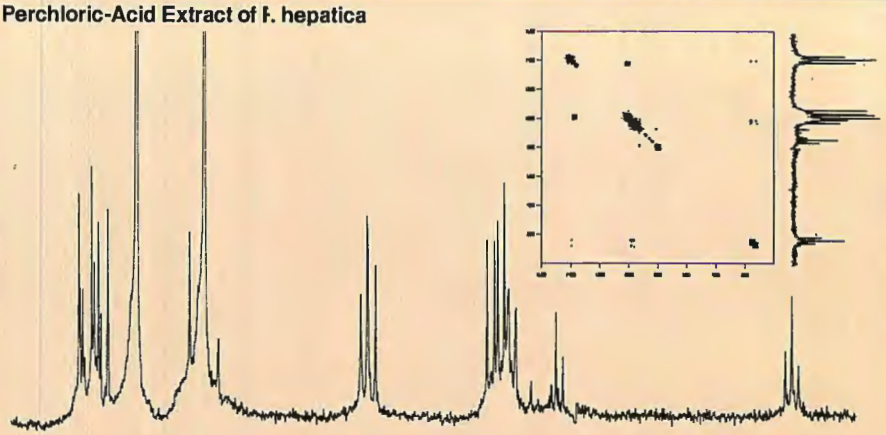
XL performance for demanding biological NMR studies

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.

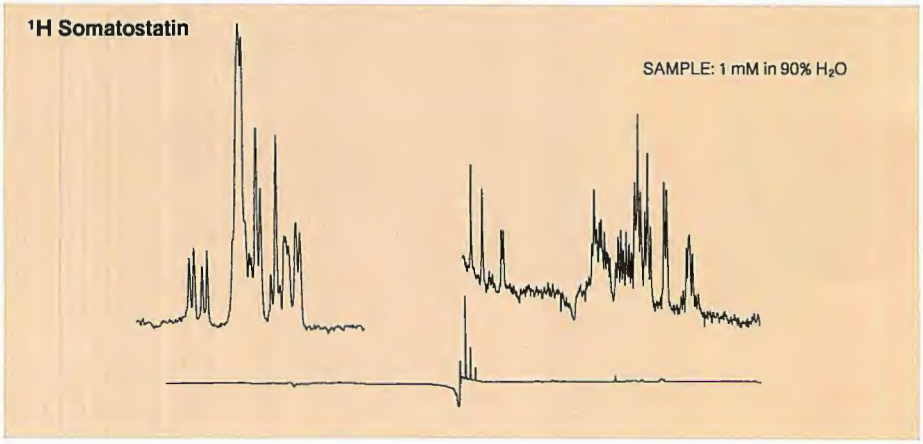
Perchloric-Acid Extract of F. hepatica



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.*

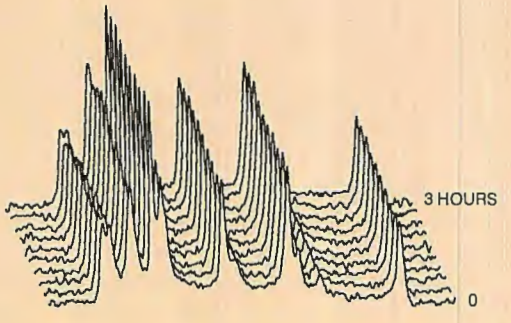
Two-dimensional NMR is a powerful method for analyzing complex mixtures. The contour plot shown above the spectrum is the result of a ^{31}P homonuclear shift correlation experiment carried out on a portion of the fluke extract using a 5-mm ^{1}H broadband switchable probe at 81 MHz. The total experiment 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.

^{1}H Somatostatin



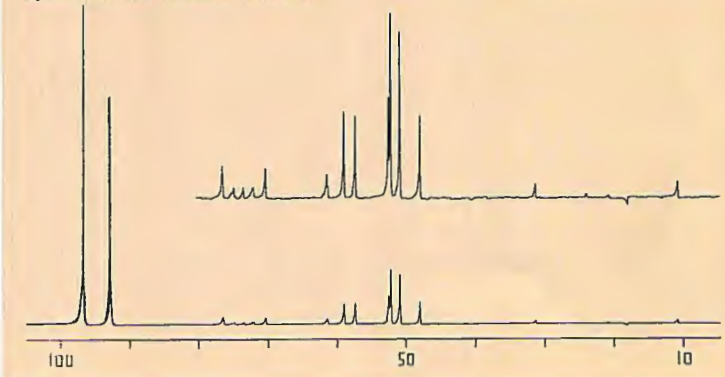
Biological NMR often requires the observation of protons in H_2O, particularly for observation of exchangeable protons. The strong signal from the solvent can be suppressed effectively by pulse sequences such as time-shared Redfield 2-1-4 or, as here, the Jump-and-Return pulse sequence. This XL-400 spectrum 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.

Perfusion of Tissue

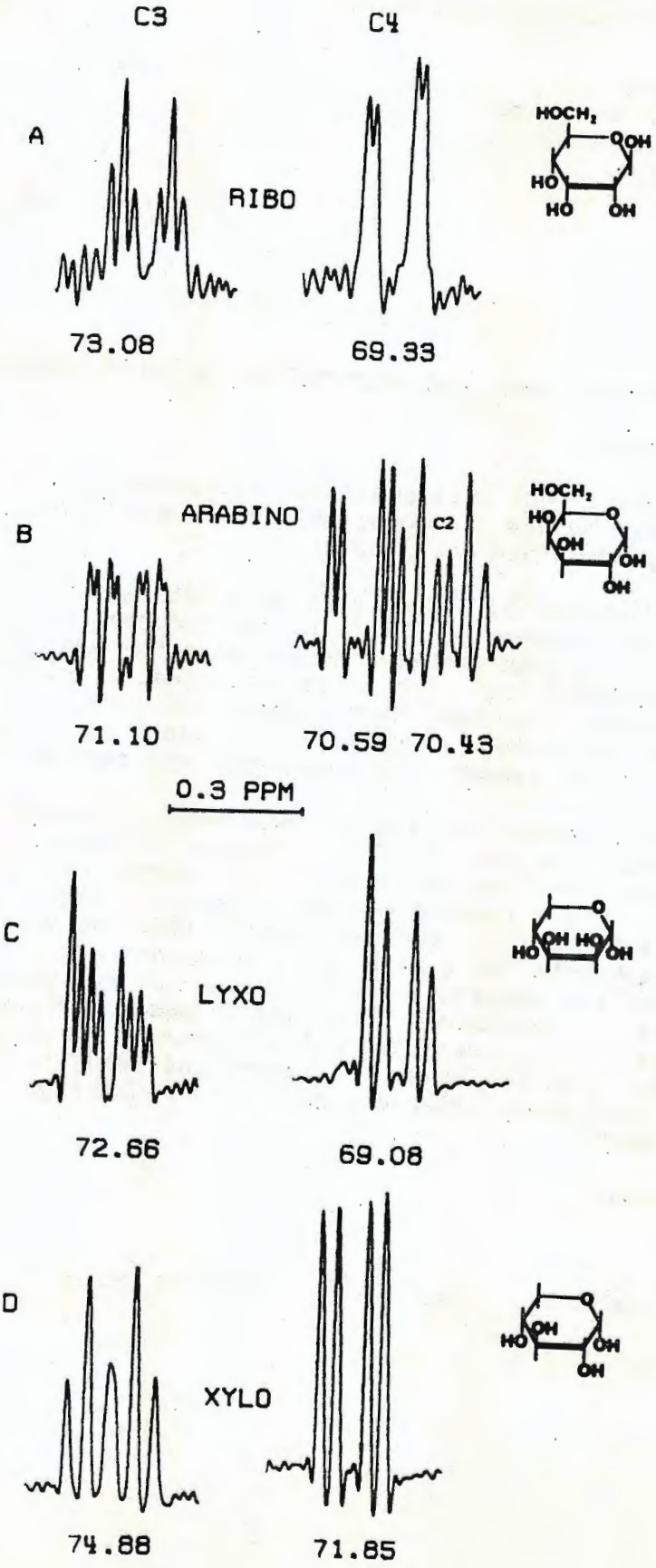


Use of a modified 10-mm tube permits the NMR study of intact tissue while perfusing with temperature-controlled nutrient. No hardware modification of the spectrometer is necessary. The stacked plot shows the time course of ATP resonances during tissue perfusion with a nutrient medium and illustrates 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.


C3
C4
A
RIBO
73.08
69.33
B
ARABINO
C2
71.10
70.59 70.43
0.3 PPM
C
LYXO
72.66
69.08
D
XYLO
74.88
71.85

309-21

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

SMB/AJ-3

24th. May, 1984

Subject: VARIABLE TIME BASE ACQUISITION: A PULSE PROGRAM.

Dear Prof. Shapiro,

Our colleagues at the Port Sunlight laboratory have asked us to reply to your, by now infamous, multi-cloured letters. This shows that even Sponsors are not immune.

In TAMU Newsletter no. 232 we gave some details of a variable time base interface between an SXP pulse spectrometer and a signal averager. Since then the SXP has become a CXP and the signal averager an ASPECT 2000 with pulse programmer. However the need to acquire variable time base data remains the same, so we have written a pulse program to do the job of the original hard-wired interface. In case any other readers are interested the rational is as follows.

Any CXP Spectrometer can perform this sequence without any modification. The sequence is reproduced in Figure 1 and runs as follows. The spectrometer is set up as normal for a CPMG T2 measurement before loading the VTB pulse programme. The interface is then set to external address advance (XA) and you are then ready to run. As before the time base is synchronous with the CPMG pulse sequence and the sampling rate is changed at two points. The change over points are determined by C1 and C2 and the decreased sampling rates by C3 and C4. External address advance pulses are supplied by the -W pulse from the Pulse Programmer and triggers the ADC on the negative going edge. Please credit this "subscription" to the Unilever account.

Yours sincerely,

Steve Bociek Steve Ablett Arthur Darke David Welti

S.M. Bociek S. Ablett A. Darke D. Welti

```
; Variable Time Base

;A  D C B
 0  1      (                    +W     ; Trigger pulse for sweep ADC
 1  1      (+X                         ; Proton 90 degree pulse
 2  A      (                           ; Pulse spacing/2
 3  2      (        +Y                 ; Proton 180 degree pulse
 4  A 1 2  (                       -W  ; Trigger plse for XA
 5  A E 7  (                           ; First break point
 6  3      (                           ; Timing delay 2*DA
 7  2 3 6  (        +Y                 ; Do C3 pulses before next XA Pulse
 8  A 2 5  (                       -W  ; XA Trigger pulse
 9  A E B  (                           ; Second break point
 A  3      (                           ;
 B  2 4 A  (        +Y                 ; Do C4 pulses before next XA Pulse
 C  A 2 9  (                       -W  ; XA Trigger pulse
 D  O F    (                           ; Recycle Delay


;SA=0              ;ST= +W         ;SB OFF          ;SX -W
;QP OFF            ;TRE +X         ;RLS +X



;DO=3.0000E0       ;D1=4.000E-6    ;D2=8.000E-6
;D3=1.920E-4       ;DA=9.600E-5
;C1=624            ;C2=200         ;C3=10           ;C4=100
```

Figure 1. A Pulse Program for Variable Time Base Acquisition. Suitable for any CXP series spectrometer equipped with a Pulse Programmer.

The University of Connecticut

STORRS, CONNECTICUT 06268

THE COLLEGE OF
LIBERAL ARTS AND SCIENCES
Department of Chemistry

24 May 1984

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

Deuterium NMR of Flexible Chains in Nematic Solvents

Dear Dr. Shapiro:

We continue to study the way in which flexible solutes adapt to the uniaxial constraints imposed on them in a nematic solvent via DMR. In Fig. 1 we show the quadrupolar splitting pattern exhibited by diglyme-d_{14}. It is reminiscent of that of the corresponding analogue alkane (nonane) even though the hetero atoms in diglyme cause substantial differences in its conformer probabilities. In contrast with an alkane, the gauche states are energetically more favorable than trans of the C-C bonds of the diglyme chain. The assignment and relative quadrupolar splittings are well accounted for using a phenomenological model wherein the probability distribution is shifted to favor the more elongated chain conformations. (Fig. 2). We anticipate learning more about the internal energetics of flexible molecules in liquid phases by continuing these types of DMR studies.

Very truly yours,

E. T. Samulski

Edward T. Samulski

ETS:mr

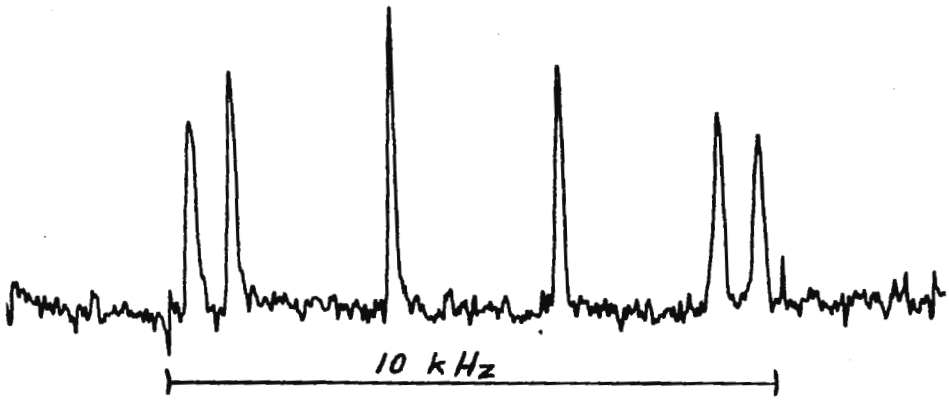


Fig 1

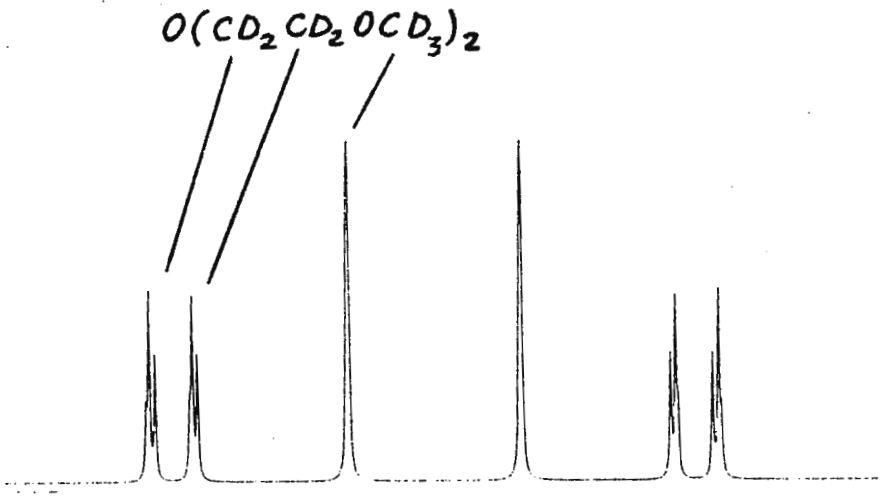


Fig 2

RESEARCH CENTRE

SUNBURY-ON-THAMES

MIDDLESEX

ALTERNATIVE METHOD FOR MEASURING 90° PULSES ON GX SPECTROMETERS

We would like to pass on to other GX users a new method for measuring 90°-pulses. It is much faster than the usual JEOL method of doing a stacked experiment, incrementing the observe pulse every time, and much easier to use and more accurate than watching the amplitude of the FID.

The method makes use of two commands in the RSX software (version 831030 V1.6), SAVEI and NEWYG. SAVEI instructs the computer to remember the values of YG (Y-gain) and NGAIN (internal scaling during FT etc), and NEWYG uses these to produce a display from a new experiment in which the peak intensity may be directly compared to those obtained before. The net effect is the same as setting the AI flag in Bruker and Varian software.

To use these commands to measure a 90° pulse follow the steps set out here:

i) Lock, shim and tune the sample, which should be concentrated enough to see a signal in one pulse.

ii) Find the signal, reduce FREQU to about 1000 Hz or less, set TIMES to 1, PW1 to about a 10° pulse and PD to several seconds to allow for relaxation. DUMMY must be zero.

iii) Collect one FID, transform and phase it and adjust YG so the peak is about half the height of the screen.

iv) Type SAVEI, from one of the command patterns. You must type it, as it does not appear on any pattern that we have found.

v) Increase PW1 by a few μs, then type in the following sequence of commands:

ACCUM #FTPH NEWYG REAL.

The signal will now be larger or smaller than it was before, depending on which side of the 90° pulse you are.

vi) As soon as you think you have a good idea of the value, double PW1 and start to look for the 180° pulse null. This may require an increase in YG to see. If you type SAVEI again after a YG increase, the program will remember the new value for next time.

When you have found the 180° pulse, check it by doubling PW1 for the 360° pulse null (usually cleaner) and then divide that value by 4 for the 90° pulse.

We use the following .GLG file to save typing:

```
;NDP.GLG
PW1
.KEY
ACCUM
#FTPH
NEWYG
REAL
```

The whole of step (v) is then accomplished by typing:

@NDP

(Ninety Degree Pulse)

We have found this method to be quick, easy and as accurate as you wish, and can recommend it.

Alan

A S F BOYD

Professor B L Shapiro
Chemistry Department
Texas A & M University
College Station
Texas 77843
USA

ASFB/LJC

24 May 1984

United States Department of Agriculture | Agricultural Research Service | North Atlantic Area Eastern Regional Research Center | 600 East Mermaid Lane Philadelphia, Pennsylvania 19118

May 21, 1984

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

Dear Dr. Shapiro:

Title: Evaluation of Phase Homogeneity Using Interrupted Decoupling/Inversion Recovery Sequence

As part of a study to evaluate the structure and interactions of potential grafting polymers with animal biomaterials, we explored the use of solid state ^{13}C CP-MAS NMR spectroscopy. The system under study was a composite consisting of chrome tanned leather (CTL) and emulsion polymerized polymethylmethacrylate (PMMA) which has been previously described (1). An initial evaluation of the unmodified CTL revealed that paramagnetic chromium, crosslinking the collagen chains had shortened the spin diffusion controlled proton T_1 values from 120 msec (untanned collagen) to 3.5 msec (CTL). We reasoned that the induction of such a short relaxation would be useful for evaluating the phase homogeneity in composites containing this biopolymer.

PMMA can be incorporated (2-80%) into the leather matrix through an emulsion polymerization process whereby it cannot be removed by solvent extraction (1). However, in order to clearly define the dynamics and level of interaction of the PMMA component in such a mixed polymer system, we had to contend with the problem of assessing the relaxation information associated with the overlapping resonances in the spectrum of these composites. In order to accomplish this measurement, we devised a pulse sequence that would allow us to measure the proton T_1's (via ^{13}C resonances) of the PMMA CH_3 and OCH_3 at 46 ppm and 53 ppm, respectively, that overlap with the broad region of collagen methylene resonances from 20 ppm to 69 ppm.

Scheme 1 depicts the interrupted decoupling/inversion-recovery sequence that was used to evaluate these composites. Relaxation time measurements made with this sequence and the normal inversion-recovery sequence on standard samples of PMMA gave identical results.

Because of their rapid reorientation time, CH_3 group resonances do not undergo severe dipolar broadening when the decoupling field is turned off in an interrupted decoupling experiment (2). However, the CH_2 resonances are generally lost through such a broadening mechanism and so spectra containing overlapping CH_2 and CH_3 groups will only exhibit the CH_3 resonance under these optimized conditions. By combining this methodology with the standard inversion-recovery sequence as in Scheme 1 we were able to independently evaluate the dynamics of the PMMA polymer imbedded in the CTL matrix.

The accompanying figure shows the results of one of our interrupted decoupling/inversion-recovery experiments performed on a composite containing 50% bound PMMA. These spectra clearly show heterogeneous proton T_1's at this level of PMMA add-on. However, spectra taken of composites at the early stages of the polymerization, 2% bound PMMA indicates that homogeneous domains of less than 100A dominate the material.

A full account of these studies will be submitted to the Journal of Applied Polymer Science.

Sincerely,

PHILIP E. PFEFFER
Research Leader
Physical Chemistry and Instrumentation Laboratory

DAVID G. BAILEY
Research Leader
Animal Biomaterials Laboratory

EDMUND F. JORDAN
Research Chemist
Animal Biomaterials Laboratory

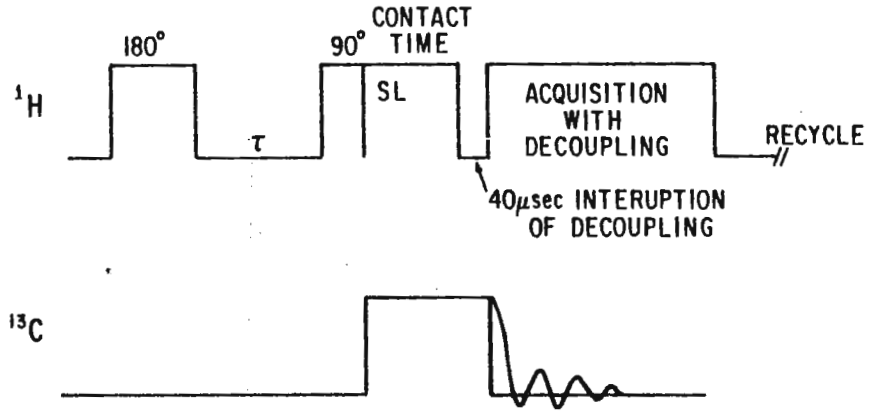
2 Enclosures

(1) E. M. Jordan Jr., B. Artymyshyn, A. L. Everett, M. V. Hannigan and S. H. Feairheller. J. Appl. Poly Sci., 25, 2621 (1980).

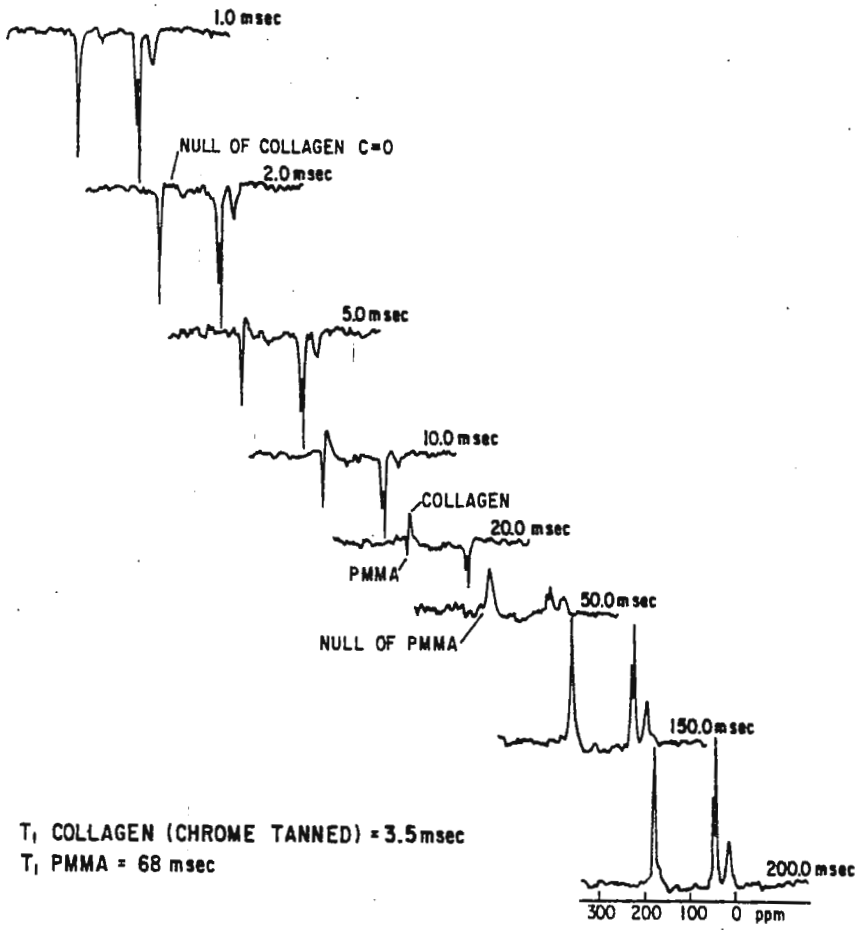
(2) S. J. Opella, M. H. Frey, and T. A. Cross, J. Am. Chem. Soc., 101, 5856 (1979).

United States Department of Agriculture
Agricultural Research Service
Northeastern Region Eastern Regional Research Center
600 East Mermaid Lane
Philadelphia
Pennsylvania
19118

PROTON T_1 INTERRUPTED DECOUPLING / INVERSION-RECOVERY PULSE SEQUENCE



PROTON T_1 WITH INTERRUPTED DECOUPLING OF 50% PMMA LEATHER COMPOSITE



T_1 COLLAGEN (CHROME TANNED) = 3.5 msec
T_1 PMMA = 68 msec

At last, for solids and liquids:

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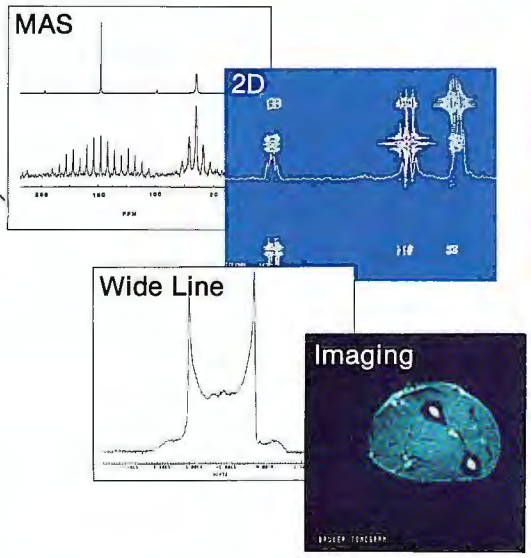

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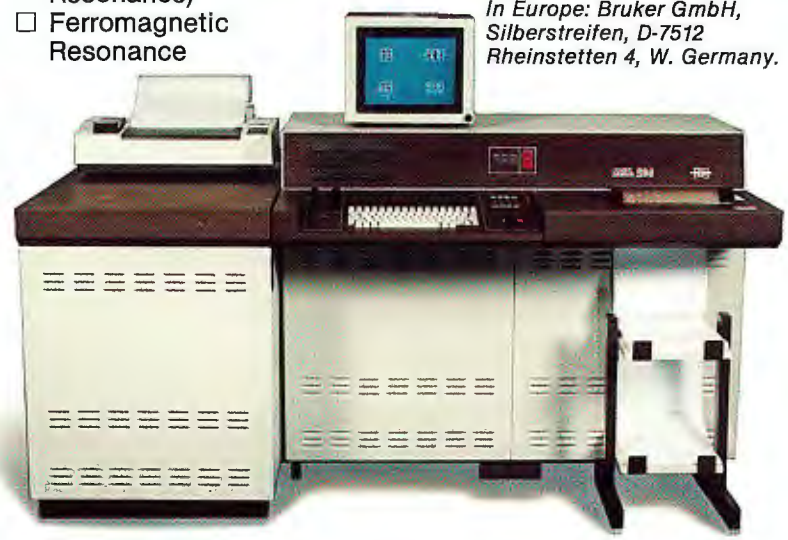

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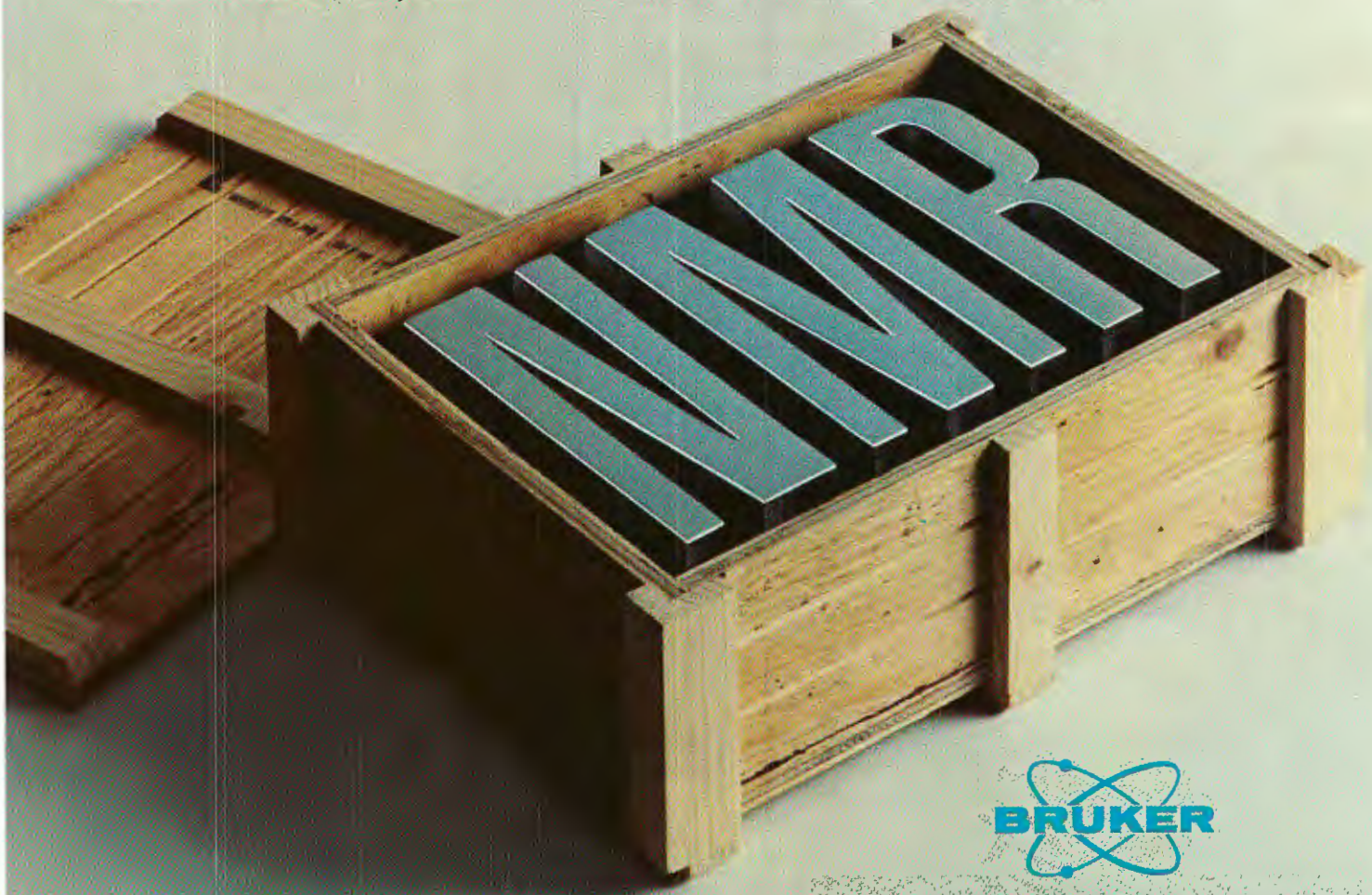

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NMR Systems designed to solve problems.

Koninklijke/Shell-Laboratorium, Amsterdam

Shell Research B.V.

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

Uw ref.:

Onze ref.: IS/22 0766

Amsterdam, 30th May 1984
Badhuisweg 3
Tel. via telefoniste (020) 30 9111
Tel. rechtstreeks (020)
Hr/Mw

Re.: Solid State NMR of clathrasils.

Dear Professor Shapiro,

Thank you for your coloured reminders!

Recently, we published a short note on the characterization of some synthetic silicates structurally related to clathrate hydrates, the so-called clathrasils (J.C.S. Chem. Commun., 1983, 1360).

We used solid-state ^{13}C NMR to probe the fate of the organic template molecules and ^{29}Si NMR to study the silicate framework.

For clathrasil-1H we were able to show that 1-aminoadamantane remains intact and occluded within the cages after synthesis, and that the four crystallographically distinct silicon T sites in the structure were represented by three peaks in the silicon solid-state spectrum.

To bring the story up to date, we have now obtained spectra from clathrasil-1H showing all four sites as separate peaks in the correct intensity ratios 12:4:12:6. Moderate artificial line-sharpening of the published spectrum results in the resolution of a previously unresolved line at -117.6 ppm (Fig. 1a).

An even better resolved spectrum is generated after heat treatment at 900°C (see Fig. 1b).

Koninklijke/Shell-Laboratorium, Amsterdam

d.d. 30th May 1984

This increased resolution is not caused by line-narrowing due to loss of aluminium (as was observed for zeolite ZSM-5, see for example C.A. Fyfe et al., Chem. Lett. 1983, 1547) but rather by a shift of the resonances to higher frequency.

In the case of the lowest intensity peak (due to the T_3 sites in our nomenclature) this shift is almost 1.2 ppm.

It is now accepted that the silicon solid-state chemical shift can be a function of the Si-O bond lengths and Si-O-Si bond angles in silicate structures.

Calcination of these cage-like synthetic clathrasils at very high temperature causes the organic template to leave the framework.

Once the physical interactions of the organic template with the silicate framework are removed, the structure can adopt the most "comfortable" configuration in this new situation, resulting in changes of chemical shift in the solid-state silicon spectrum.

Yours sincerely,

KONINKLIJKE/SHELL-LABORATORIUM, AMSTERDAM

G.R. Hays

N.C.M. Alma

R. Huis

A.E. Wilson

Encl.

GRH/NCMA/RH/AEW/DdG
AG - IS

PB.84.042.

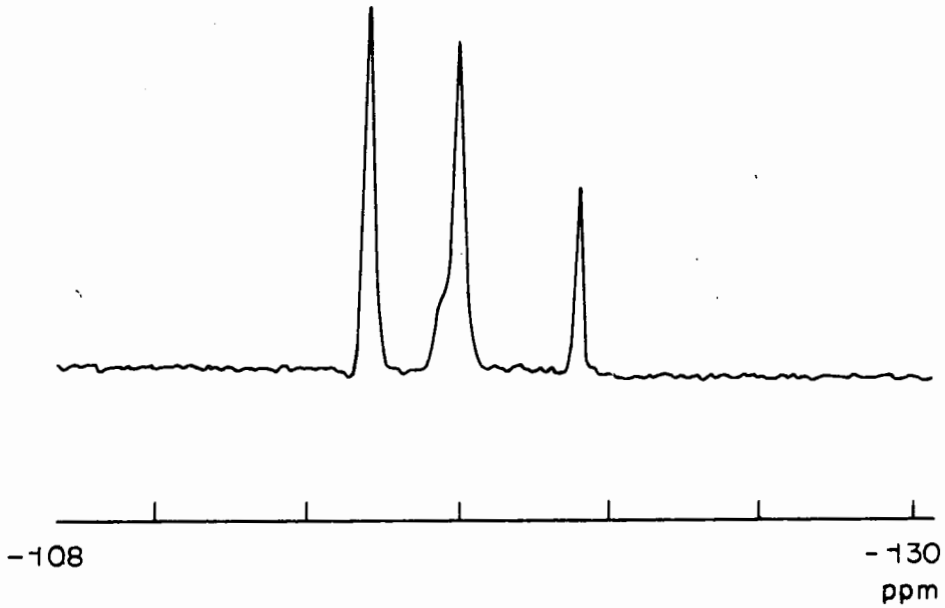
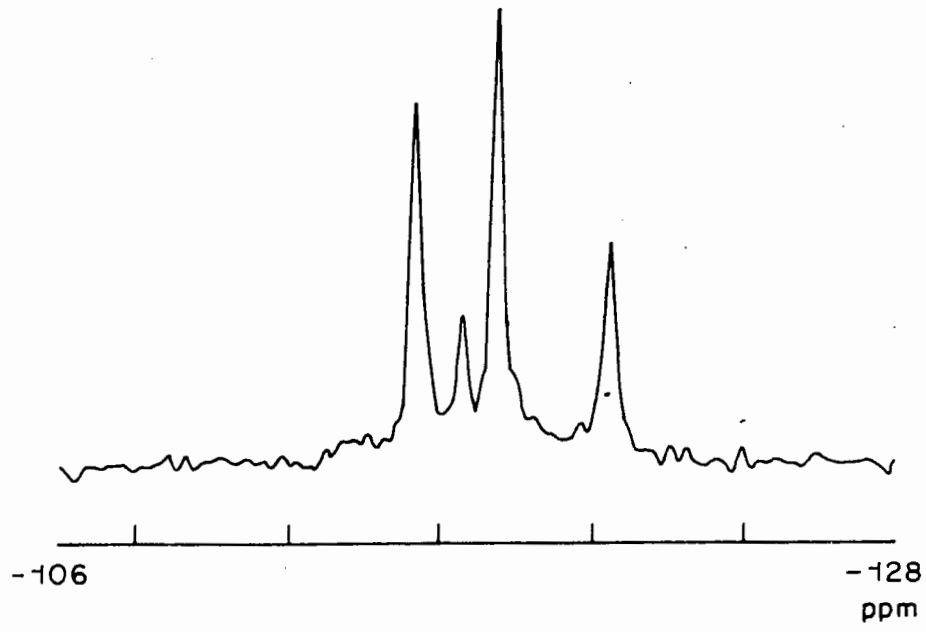


FIG. 1a: ^{29}Si NMR SPECTRUM OF DODECASIL–1H CRYSTALLIZED IN THE PRESENCE OF 1–AMINO–ADAMANTANE. THE POSITIONS OF THE LINES ARE –115.7 (T_1),–117.7 (T_3), –118.1 (T_2) AND –121.3 (T_4) ppm. THE SPECTRUM WAS ACQUIRED USING A BRUKER CXP–300 SPECTROMETER AND HAS BEEN SUBJECTED TO SLIGHT GAUSSIAN RESOLUTION ENHANCEMENT



DRAWN Dr
DWG 84.05.1131

FIG. 1b: DODECASIL–1H AFTER CALCINATION AT 900°C POSITIONS OF THE LINES ARE –115.3 (T_1), –116.5 (T_3), –117.5 (T_2) AND –120.5 ppm (T_4). THE RELATIVE INTENSITY RATIO'S ARE 100: 29: 97: 50

Standard Oil Company (Indiana)
Amoco Research Center
Post Office Box 400
Naperville, Illinois 60566
312-420-5111

May 28, 1984

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

Dear Barry:

^{2}H NMR of Hydrocarbons

Recently we obtained natural abundance ^{2}H NMR spectra on a series of hydrocarbons at 46 MHz. We prepared 50% (v/v) solutions of these hydrocarbons in $CHCl_3$ to which a few drops of $CDCl_3$ were added as a chemical shift reference (δ = 7.26 ppm). Spectra were obtained in 80 to 160 scans using NOE suppression and an 8 s delay between pulses. Because we found a maximum T_1 of 1.5 s, these conditions should yield quantitative data.

It is known that ^{2}H distribution is not uniform[1] and that individual sites show an enhancement or depletion of ^{2}H. The ^{2}H spectra of pentane through decane are shown in Figure 1. As one goes from pentane to octane, up to three methylene peaks are resolved but in the spectra of the longer alkanes resolution in this region is lost. We set the integral of the methyl group to 6 and measured the integral of the combined methylene resonances relative to this value. We found minor variations between the measured and expected methylene to methyl ratios as is shown in Table I.

We also examined trans nonenes where the double bond was in the 2, 3 and 4 positions and spectra of these are shown in Figure 2. In these samples we were surprised to find that the olefin contained only about 15% of the expected level of ^{2}H. Furthermore, the CH_2 group α to the olefin only contained 75% of the expected ^{2}H level.

Sincerely,

Joe Ray

G. J. Ray, B-5
X5217

C. T. Price, F-9
X5154

JH16:GJR/59-9907-04
Attachment

[1] David M. Grant, et al., J. Am. Chem. Soc. 104, 4492 (1982).

Figure 1. Alkane Series

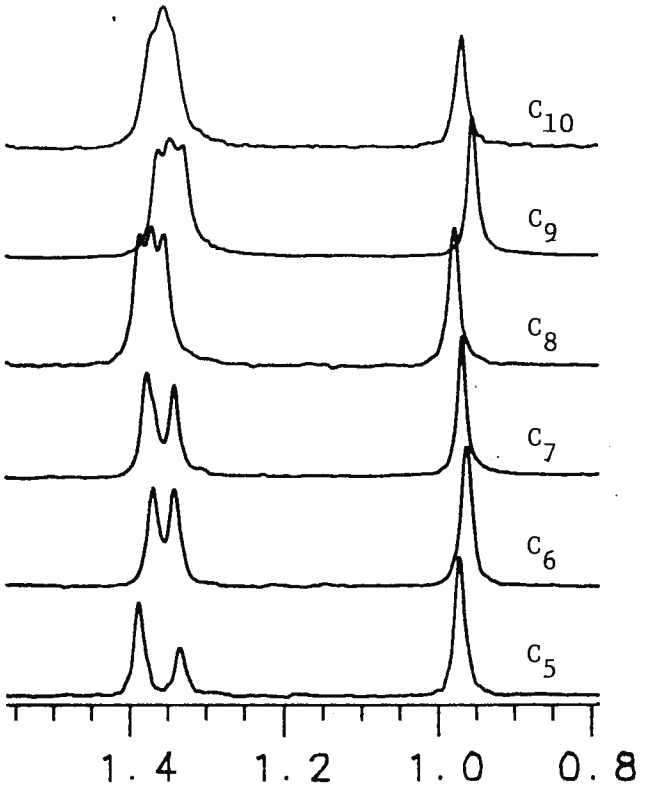


Figure 2. Trans-Nonenes

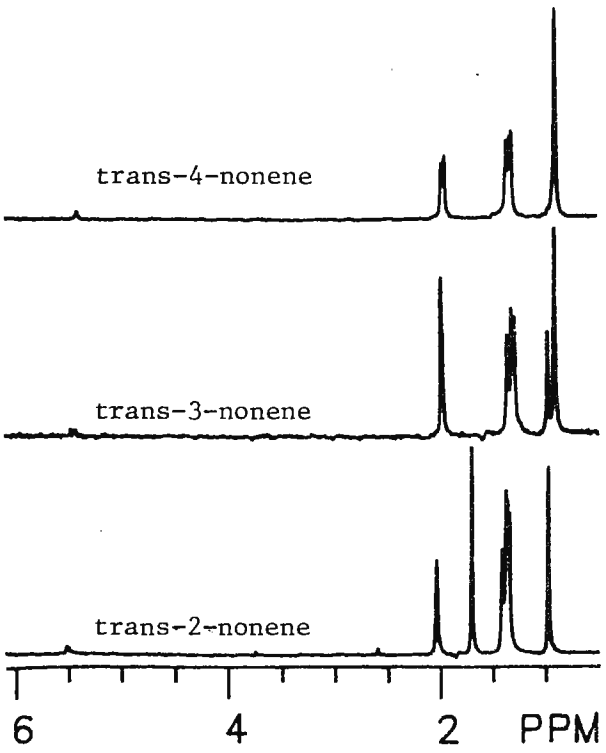


Table I

Observed (and Expected) Intensities of Various ^2H Peaks in Spectra of Hydrocarbons

	I_{CH_3}	I_{CH_2}
Pentane	6.0	6.6 (6.0)
Hexane	6.0	7.9 (8.0)
Heptane	6.0	10.2 (10.0)
Octane	6.0	15.0 (17.0)
Nonane	6.0	13.5 (14.0)
Decane	6.0	18.8 (16.0)

	I_{CH_3}	$I_{\alpha CH_3}$	I_{CH_2}	$I_{\alpha CH_2}$	I_{Olefin}
t-2-Nonene	3.0	2.7 (3.0)	8.3 (8.0)	2.0 (2.0)	0.3 (2.0)
t-3-Nonene	6.0	---	7.5 (6.0)	3.6 (4.0)	0.3 (2.0)
t-4-Nonene	6.0	---	6.1 (6.0)	3.1 (4.0)	0.3 (2.0)

ROSKILDE UNIVERSITETSCENTER
MARBJERGVEJ 35

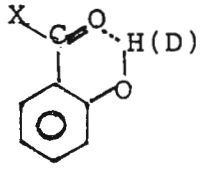
Poul Erik Hansen, Institut I
POSTBOX 260 DK-4000 ROSKILDE TELEFON 02 - 75 77 11

Prof. B. L. Shapiro
TEXAS A&M UNIVERSITY
Department of Chemistry
College station
TEXAS 77843-3255
U S A

DATO/REFERENCE 19840517/kt | JOURNALNUMMER | DERES REFERENCE/JOURNALNUMMER

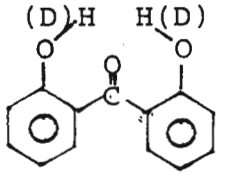
Dear Prof. Shapiro,

Your green reminder hit me in my preparations to visit Edmonton. We have for some time pursued deuterium isotope effects on ^{13}C nuclear shielding of intramolecularly hydrogenbonded phenols

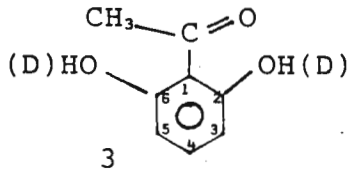


1

and found a correlation between hydrogenbondstrenght and $^{2}\Delta COD^{1}$. To investigate whether the hydrogen-bond would be weaker in compounds such as 2 and 3



2



3

in which two hydrogen-bonds are to the same carbonyl groups (2) or the carbonyl group is divided in its ability to form hydrogen-bonds with both OH-groups (3), a new perturbation of equilibrium became obvious in 3. The effect is very clear in the case in which <u>one</u> OH group is deuterated. The resonances of C-2,C-6 show an extra splitting as shown in Fig. 1A.

The reason is a preferential binding of the carbonyl group to the OH-group rather than to the OD-group. The difference in

nuclear shielding between C-2 and C-6 is caused by the carbonyl group. Assuming that the difference between C-2 and C-6 observed by Drakenberg et al.[2] for aldehydes at low temperature, ΔCO trans - CO cis $\sim$ 9 ppm can be applied to our case, the ratio of populations is 45 : 55. This leads to an approximate barrier to rotation of 8 KJ.

As C-3 and C-5 show a similar pattern, Fig. 1B and as this difference does not originate directly from the carbonyl group this difference must be caused by the preferred conformation of the OH group.

An estimate can be made of Δ(OH trans - OH cis) = |2.8| ppm.

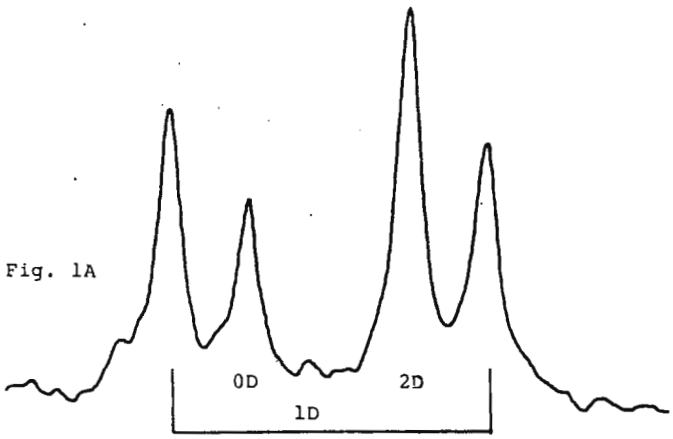
A full report of these findings and of hydrogen-bonding in general will appear shortly.

Yours sincerely

Poul Erik Hansen

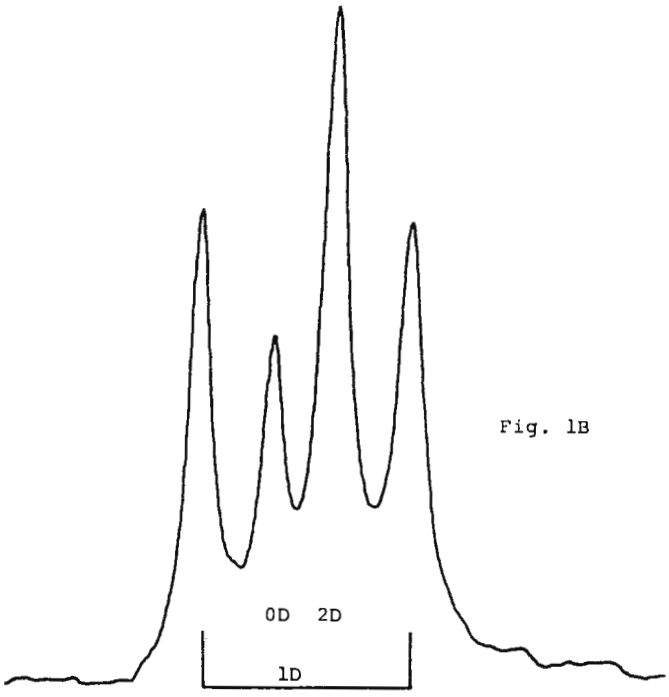
1. P. E. Hansen, Ann. Reports on NMR Spectrosc. 15, 105 (1983).
2. T. Drakenberg, R. Jost and J. M. Sommer, J. C. S. Perkin I, 1682 (1975).

Fig. 1A



C-2,C-6

Fig. 1B



TEL AVIV UNIVERSITY
RAYMOND AND BEVERLY SACKLER
FACULTY OF EXACT SCIENCES
DEPARTMENT OF CHEMISTRY

אוניברסיטת תל-אביב
הפקולטה למדעים מדוייקים
ע"ש ריימונד ובברלי סאקלר
החוג לכימיה

May 10, 1984
Ref.: 5368

The effect of radiation damping on T_1 measurements

Dear Dr. Shapiro,

Since we received our new Widebore AM-360 from BRUKER last October, we no longer have to go to the Weizmann Institute to run high-field spectra, which also means losing the opportunity to peek into their copy of your Newsletter. Therefore, the time seems ripe to join the community of your subscribers and get a copy of our own.

In this letter we have a bit of advice for those who, strangely enough, need to measure T_1 values of water at 360 MHz. The high sensitivity and the high Q-value of the ^{1}H-coil make it of course impossible to detect the solvent signal (following a 90^o pulse) from a regular sized 5 mm H_2O sample, without saturating the receiver. A quick solution (besides using a smaller sample) is to somehow attenuate the received signal, most conveniently achieved on our system by replacing the regular ^{1}H-preamplifier by the ^{19}F preamp. Now an inversion-recovery T_1 measurement on water becomes possible, but the results (shown in the insert and upper curve of the accompanying figure) are somewhat puzzling, indicating an apparently biexponential recovery. Since the sample in this case is a solution of sonicated calf-thymus DNA, one might jump to conclusions such as cross-relaxation or slowly exchanging "bound" water. However, a closer inspection of the initial part of the semi-logarithmic $M_\infty - M_z$ vs. time plot (see insert on top of the figure) reveals a behaviour which is incompatible with biexponential decay.

The explanation suggest itself when the measurement is repeated in two different ways:

a) after detuning the ^{1}H-coil

or

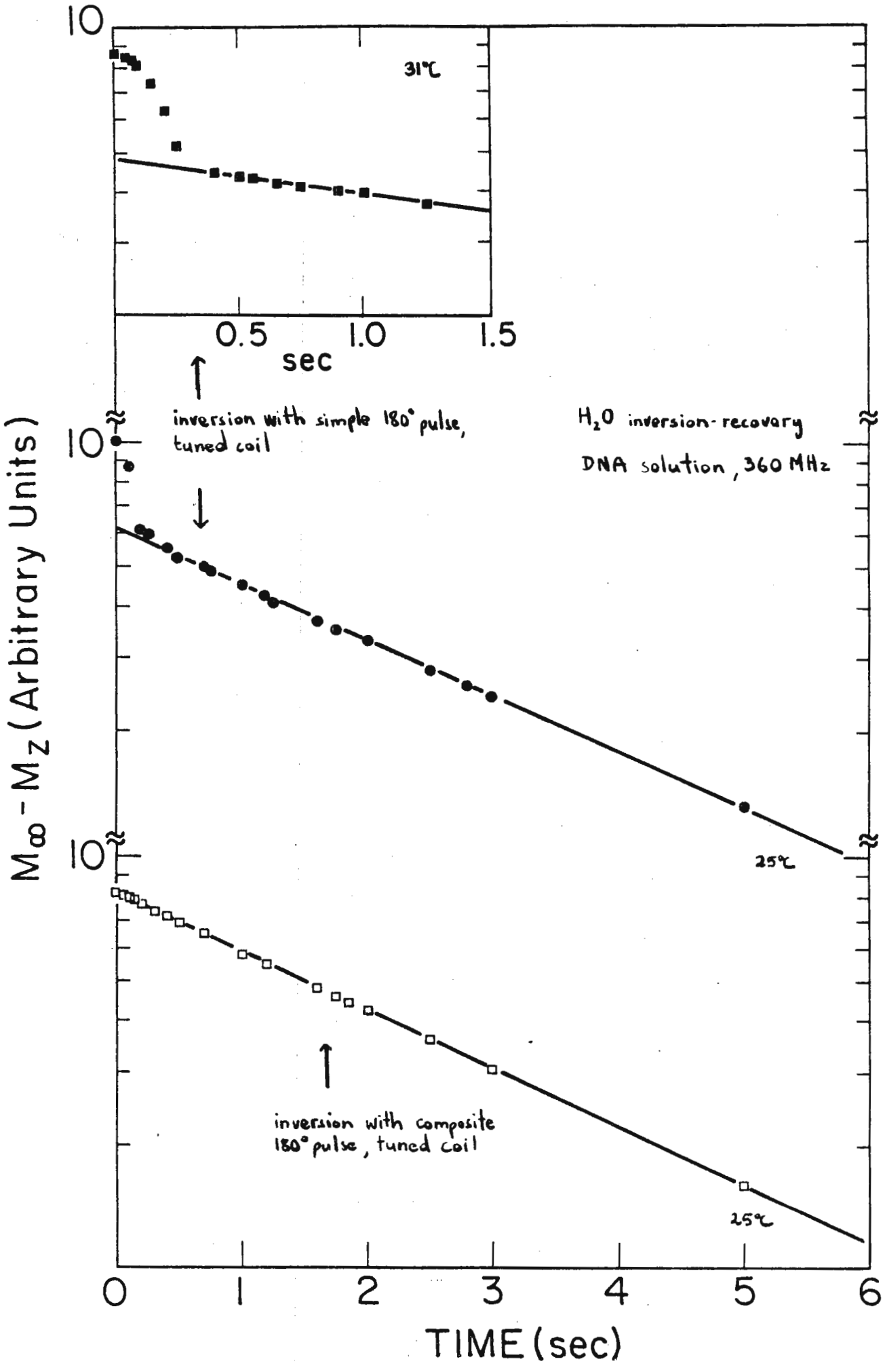
b) using an optimally tuned coil, but applying a composite pulse for the 180° inversion.

In both cases the resulting T_1 plot is perfectly single exponential e.g., (bottom part of the figure). Thus, the problem in the first measurement appears to be caused by radiation damping (Abragam, page 73). Due to H_1 inhomogenity, inverting the intense water magnetization with a simple 180° pulse leaves a transverse magnetization which is strong enough to induce the radiation damping effect. This causes a reorientation of the magnetization vector towards the z-axis and therefore directly affects M_z, which is detected in the initial part of the T_1 plot. For long enough delays between the 180° and 90° pulses, the initially created transverse magnetization has sufficiently decayed to make the radiation damping negligible, and the normal spin-lattice relaxation determines M_z.

Sincerely,

Peter Bendel Gil Navon

Peter Bendel Gil Navon


31°C
0.5
1.0
1.5
sec
inversion with simple 180° pulse, tuned coil
H2O inversion-recovery
DNA solution, 360 MHz
25°C
inversion with composite 180° pulse, tuned coil
25°C
M∞ - Mz (Arbitrary Units)
TIME (sec)
0
1
2
3
4
5
6

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Switching: 5-20μs
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: −75dB

Phase Noise: −70dBc, (0-15KHz)
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Switching: 5-20μs
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: −70dB

Phase Noise: −70dBc, (0-15KHz)
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Resolution: 0.2Hz-100KHz (opt.)
Switching: 5-20μs
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: −70dB

Phase Noise: −63dBc, (0-15KHz)
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DEPARTMENT OF CHEMISTRY, B-014
LA JOLLA, CALIFORNIA 92093

May 26, 1984

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

RE: Grandson of Amazing Gate

Dear Barry,

Several years ago the late Bob Vaughan developed an amazingly effective rf gate based on inexpensive double balanced mixers. Bob's student Albert Highe improved the design by placing two such mixers back-to-back with a copper shield between them, and the circuit became known locally as "son of amazing gate".

During recent spectrometer modifications in our laboratory, Alan Ronemus replaced our SOAGs with the enclosed circuit. Fig. 1 is a full scale copy of a printed circuit board (foil side) which holds two SRA-1H mixers from Minicircuits, Inc. (electrical equivalents from manufacturers such as Merrimac have different pinouts). The use of shielded double sided circuit board (relieved around active pins) with a ground plane instead of point to point wiring improved the on/off ratio to better than 85 dB. The "high" power mixers can take +10 dBm rf input and when switched on with 40 ma drive current applied to pins 3&4, give +4 dBm output. The gate board fits conveniently in a Pomona Box #2428.

Fig. 2 shows a circuit for producing the required drive starting with a TTL pulse. The hot carrier diode between base and collector of the 2N4401 was recommended to us by John Wright (UCSD NMR Facility) as an effective remedy for a delayed response of the driver to TTL input. The circuit shown has a delay of ca. 30 nanoseconds. One such driver is needed for each mixer in the GSOAG. 1 watt collector resistors are needed to accomodate the drive current.

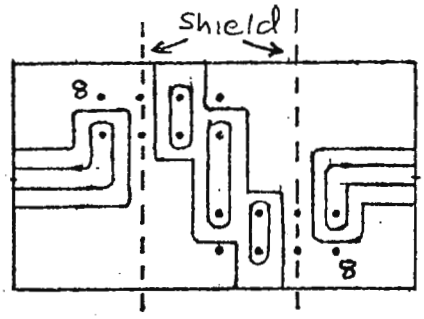
We trust that this communication will stave off further multicolored notices.

Sincerely,

Bob

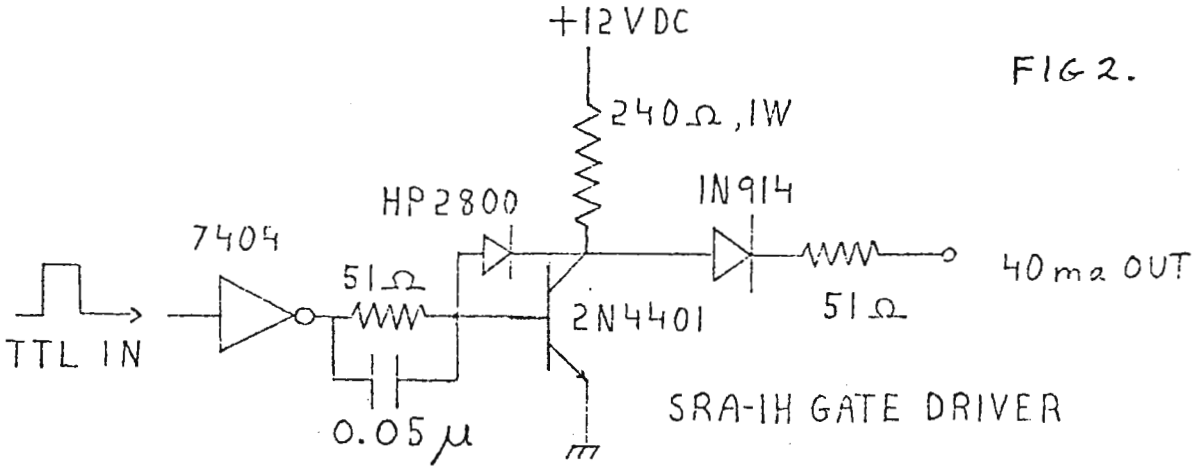
Regitze

Robert L. Vold
Regitze R. Vold
Alan Ronemus



SRA-IH GATE BOARD

FIG.1



SRA-IH GATE DRIVER

FIG 2.

November 13 - 16, 1984
at the New York Penta Hotel
New York City, NY

EASTERN ANALYTICAL SYMPOSIUM, INC.

TWO-DIMENSIONAL NMR FOR THE ORGANIC CHEMIST

Tuesday, November 13th

This short course is designed for organic chemists who already routinely use one-dimensional proton and carbon-13 NMR spectra. The course will start with the assumption that the participants have no prior experience with two dimensional techniques.

The following topics will be discussed: (1) general principles of two-dimensional NMR and the most common types of 2-D spectra, (2) pulse sequences and selection of experimental parameters, (3) interpretation of 2-D spectra, and (4) recent developments in 2-D techniques. A problem solving period will be included. Spectra determined on various instruments will be used as illustrations.

The course will be taught by Dr. Roy Bible, a Research Fellow and Manager of Physical Methodology at the R & D division of G. D. Searle & Co., Skokie, IL, and Leroy F. Johnson, Manager of the Applications Laboratory of the General Electric Co., NMR Instruments, Fremont, CA. Further information on this course may be obtained from Dr. Bible at (312) 982-7787.

Preregistration for these two courses will be limited to 50, on a first-come, first-served basis. The preregistration fee is $150.00 for each one day course. In addition, course registrants **must** be preregistered for EAS. It is anticipated that these two courses will be quickly filled; if you are interested in attending one, or both, early preregistration is urged! Preregistration for these two courses and for EAS, may be accomplished using the attached preregistration card.

WEDNESDAY, NOVEMBER 14, 1984, AM

Advances in 2-Dimensional NMR Spectroscopy
Chairman: G. N. Chmurny, NCI-Frederick Cancer Research Facility, Frederick, MD

1) "2-D NMR and Organic Structure Analysis"
Gwendolyn N. Chmurny, Frederick Cancer Research Facility, Frederick, MD.

2) "One and Two-Dimensional Spin Filtering and Pattern Recognition in NMR"
Richard R. Ernst, Laboratorium für Physikalische Chemie, Eidgenössische Technische Hochschule, Zurich, Switzerland.

3) "Novel NMR Techniques"
Ad Bax, Laboratory of Chemical Physics, National Institutes of Health, Bethesda, MD

4) "Enzyme Catalyzed Reaction Rates Determined by 2-D NMR in Vitro and in Vivo"
Robert S. Balaban, Laboratory of Kidney and Electrolyte Metabolism, National Institutes of Health, Bethesda, MD.

WEDNESDAY, NOVEMBER 14, 1984, PM — High Resolution, High Field, and Multinuclear NMR Spectroscopy
Chairman: R. E. Santini, Purdue Univ., West Lafayette, IN

1) "A Survey of Multinuclear High Field NMR"
Robert E. Santini, Purdue University, West Lafayette, IN

2) "Experiences with ^{15}N NMR"
Roy Bible, G.D. Searle & Company, Skokie, IL

3) "Applications of Solid State NMR to Biological Systems"
R. Andrew Byrd, Office of Biologics, Food and Drug Administration, Bethesda, MD

4) "Biochemical Applications of Modern NMR Methods"
James A. Ferretti, NHLBI, National Institutes of Health, Bethesda, MD

THURSDAY, NOVEMBER 15, 1984, AM — New NMR Methods for the Analysis of Polymers
Chairman: L. W. Jelinski, AT&T Bell Laboratories, Murray Hill, NJ

1) "2-D NMR Spectroscopy of Polymers"
Frank Bovey, Bell Labs, Murray Hill, NJ

2) "Spin Diffusion in Polymers"
David VanderHart, National Bureau of Standards, Washington, D.C.

3) "Deuterium NMR Spectroscopy as a Probe of Motion and Orientation"
Ed Samulski, Institute of Material Sciences, University of Connecticut, Storrs, CT

4) Discussion panel

THURSDAY, NOVEMBER 15, 1984, PM — NMR Imaging
Chairman: S. L. Smith, Univ. of Kentucky, Lexington, KY

1) "Fundamentals of NMR Imaging"
Stanford L. Smith, University of Kentucky, Lexington, KY

2) "Clinical Applications of NMR Imaging"
W. Allen Carr, University of Kentucky, Lexington, KY

3) "NMR Imaging of Polymeric Materials"
Gerald C. Chingas, U.S. Naval Research Laboratories, Washington, D.C.

INSTRUCTIONS

Pre-registration should be made by completing this form and returning it with your payment **by ~~October~~ 29, 1984** to Irene Nurkiewicz, EAS Registration, 16 Fairbanks Lane, Basking Ridge, NJ 07920. Pre-registrations postmarked after October 29th will be charged at the full at-meeting rates.

FEES:	PRE-REGISTRATION	AT-MEETING
Regular	$ 25.00	$35.00
Student	$ 5.00	$10.00
EAS Short Courses*	$150.00	not available
Workshops*	$ 25.00 each	$35.00 each (if still available)

*Fee for workshops and short courses is in addition to the registration fee

PREREGISTRATION 1984 EASTERN ANALYTICAL SYMPOSIUM

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☐ Mr ☐ Mrs
☐ Miss

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- Automated Sample Prep. ..... $ _____
- Near IR Reflectance ..... $ _____

EAS Short Courses:

- HPLC of Biopolymers ..... $ _____
- 2-Dimensional NMR ..... $ _____

Total Amount Enclosed ..... $ _____

Payment must accompany pre-registration card. Make check payable to: "Treasurer, 1984 EAS."
Pre-registration forms received after October 29th will not be acknowledged by mail.
*Workshop dates have not been finalized. You will be informed of your workshop date(s) by mail.

40 West Brokaw Road
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Telephone: 800-538-7792
800-662-6203 (In California)

May 31, 1984

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

Subject: Spectrometer Modifications and Chemical Shift Imaging.

Dear Barry:

We've modified our IBM Instruments WP 200 SY Spectrometer to allow us to perform rudimentary imaging experiments and pulse field gradient diffusion measurements. To control one of the shims, we use the SPF2 (Decoupler) output of the pulse programmer. The SPF2 signal is buffered with a high speed (LM318) op-amp. The buffered signal is connected to the summing point of one of the driver op-amps in the B-SN 14 shim supply. The gradient can then be controlled by the BB and DO commands. Removal of the 22 nf integrating filter capacitor in the shim supply results in gradient rise times of ca. 20 μS.

We've used our modified spectrometer to perform a new experiment which encodes chemical shift information into an NMR image. The chemical shift information was encoded by a technique similar to two dimensional J Spectroscopy. We formed an ^{1}H NMR image of a phantom consisting of tubes containing acetone and ethanol. The peaks are clearly separated in both spatial and chemical shift dimensions.

Preprints will be available soon!

Yours truly,

Joel

Joel F. Martin

Chuck

Chas. G. Wade

P.S. Please credit the account of Chas. G. Wade.

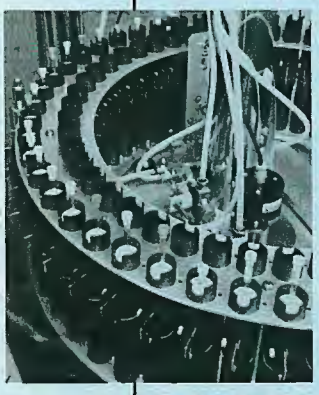
Find out how friendly and versatile NMR can be.

The QE-300 spectrometer was specifically developed for high efficiency, high-throughput carbon and hydrogen NMR. It provides a complete range of routine and high resolution capabilities with a 300 MHz (7 Telsa) superconducting magnet, synthesizer based frequency control, quadrature phase detection, high-speed pulse programming, and a dual $^{13}C/^{1}H$ switchable probe. To make operation dramatically simple, the QE-300 also features auto-locking, auto-shimming, and auto-spectral phasing plus an incredible menu driven software package called CHARM.

And now, to make the QE-300 even more versatile, we've added a number of accessories to make your work faster and more efficient than ever before.

Around-the-clock data acquisition and processing

First, there's the QE-300 Automatic Sample Changer for unattended, sequential analysis of up to 100 samples. It lets you collect spectra continuously—overnight and even over weekends. So, instead of facing a stack of samples when you walk into your lab in the morning, you have a stack of results, completely phased and annotated to your specifications.



Multiple megabyte speed and power

Winchester and other optional hard disk accessories for the QE-300 dramatically expand your data handling capacity. And to speed up Fourier Transforms and spectral phasing, you can add a high-powered array processor as well.

Remote data stations for multi-user productivity

Increased data requires more data processing capability. To meet that need, you can add a remote, interactive data station to the QE-300. It communicates directly with the QE-300 as well as other Nicolet 1280-based NMR spectrometers and enables you to process NMR data off-line while the main spectrometer is being used to acquire spectra.

VT capability for temperature dependent studies

A new microprocessor controlled Variable Temperature accessory for the QE-300 allows acquisition of experimental data at differing sample temperatures. The controller ranges from +160C to −100C with ±0.1C precision.

Enhanced ^{13}C sensitivity

An optional dual 10mm ^{13}C/5mm ^{1}H switchable probe increases ^{13}C sensitivity to more than three times that of the standard $^{13}C/^{1}H$ probe.

Broadband console electronics and X nucleus probes

Broadband console electronics and X nucleus probes for the QE-300 are offered for observing nuclei other than ^{13}C and ^{1}H, including a dual 5mm $^{31}P/^{1}H$ switchable probe for analyzing phosphorous in biological, medical, and related applications.

Call or write today for complete information on the QE-300. We'll help you select the accessories to meet your NMR needs.

The QE-300 with CHARM. Friendly, *versatile* NMR for the laboratory that has alot of work to do.

You've never had NMR like this before.

General Electric Company
NMR Instruments*

255 Fourier Avenue
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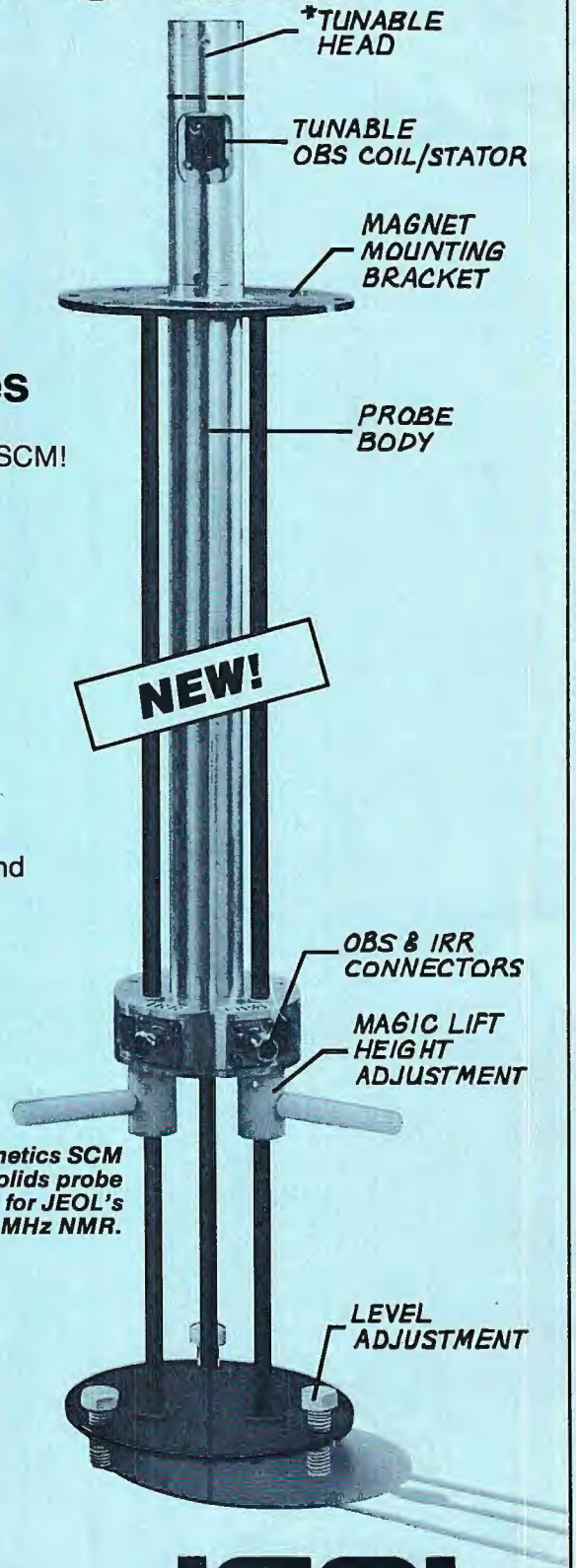
*A business formerly operated by Nicolet Magnetics Corporation.

THE GX SOLID LEADER in NMR

With Multi-Nuclear/ Multi-Field Solid State Probes

- **High field** solid sample probe for JEOL's 270 MHz SCM!
- ***Tunable heads** — interchangeable plug-in matching units for observation of
  ^{13}C (~67.8 MHz)
  ^{31}P (~109.2 MHz)
  ^{29}Si (~53.6 MHz)
  with one probe!
- Self-starting rotor/stator design!
- High Speed magic-angle double air bearing sample spinning **(>4.0 KHz)!**
- **"Magic lift probe"** for quick sample change and probe insertion!
- All this, in addition to a full line of dual and broad-band high resolution liquid sample probes!



Chemagnetics SCM Solids probe for JEOL's 270 MHz NMR.

SOLIDS UPDATE – NOW AVAILABLE

- MULTI-NUCLEAR SOLIDS PROBE FOR THE GX-270
- WIDE BORE MAGNET WITH VARIABLE TEMPERATURE SOLIDS FOR THE GX-270/89

JEOL

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