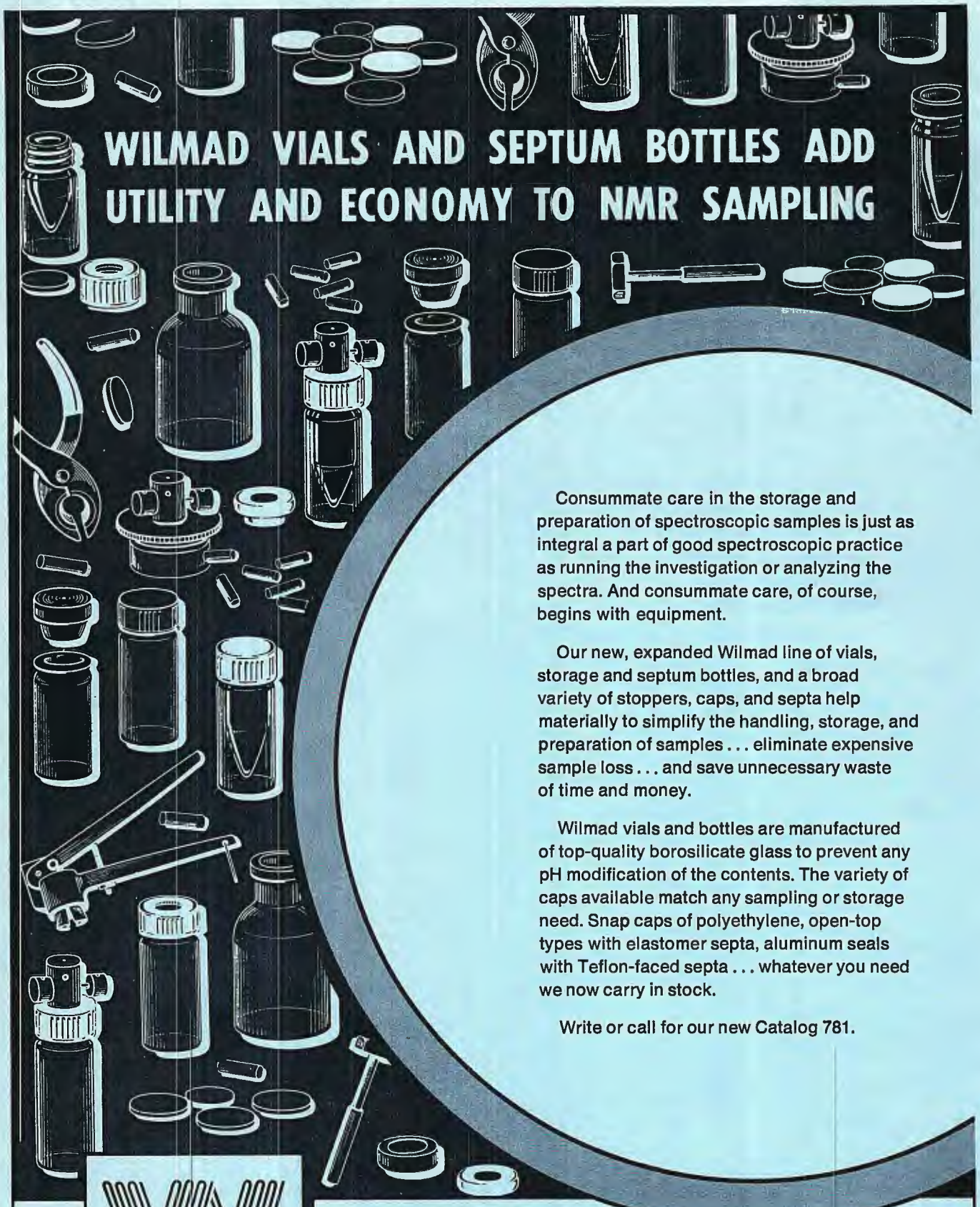
Texas A&M University N-M-R Newsletter

No. 267

December, 1980



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DEADLINE DATES: No. 268 5 January 1981
No. 269 2 February 1981

All Newsletter Correspondence, Etc., Should be Addressed To:

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

AUTHOR INDEX - TAMU NMR NEWSLETTER NO. 267

Prof. Dr. Gerhard Hägele
Institut für Anorganische
und Strukturchemie

4000 Düsseldorf, 25.10.1981
Universitätsstraße 1
Tel. 0211-311-2288

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

Dear Professor Shapiro!

Our recent interests were concentrated towards NMR properties of chiral organo phosphorus compounds. Comparing derivatives of the type

we found, that chirality strongly influences resonancefrequencies and less the coupling constants of $-CH_2-CH_2-P-$ units. e.g.:

We took 400 MHz ^1H NMR spectra of excellent resolution using the new Superconspectrometer WM400 for a test on the ABCDX system of $ClCOCH_2CH_2P(O)(CH_3)Cl$, 30% v/v in $CDCl_3$. This 400 MHz ABCDX analysis led into a perfect trap of ambiguity. Both data sets (1) and (2) from table 1 give rise to practically identical proton and phosphorus NMR simulations! Only by comparison with 90 MHz ^1H and $^1H\{^{31}P\}$ NMR spectra, data sets (3) and (4), the most likely solution to that ABCDX problem could be achieved.

So let us conclude "it is good to have an excellent NMR spectrometer but it is better to have two"!

Michael Engelhardt Gerhard Hägele Horst Schneiders

Michael Engelhardt Gerhard Hägele Horst Schneiders

Key word: Spectral ambiguities in High Field NMR Spectra

Table 1

^{1}H NMR Parameters for Cl-C(=O)-CH_2-CH_2-P(=O)(-CH_3)-Cl 30% v/v in $CDCl_3$

Data set:		(1)	(2)	(3)	(4)
δ_H	A	3.4205	3.4205	3.4157	3.4146
	B	3.3579	3.3578	3.3539	3.3523
	C	2.6198	2.6198	2.6171	2.6168
	D	2.5143	2.5142	2.5154	2.5156
ν_H	A	1368.210	1368.214	307.413	307.312
	B	1343.140	1343.134	301.805	301.711
	C	1047,917	1047.925	235.536	235.508
	D	1005,702	1005.696	226.384	226.408
$^2J_{HH}$	AB	-18,698	-18.699	-18.578	-18.603
	CD	-15.299	-15.287	-15.239	-15.313
$^2J_{PH}$	CX	-10.389	-10.389	-10.273	-------
	DX	-12.200	-12.225	-12.424	-------
$^3J_{HH}$	AC	9.873	5.493	5.766	5.788
	AD	5.394	9.868	10.024	9.974
	BC	5.471	9.931	9.562	9.732
	BD	9.885	5.332	5.364	5.367
$^3J_{PH}$	AX	11.990	11.989	12.338	-------
	BX	13.012	13.013	12.869	-------
rms		0.082	0.063	0.048	0.058
		400 MHz ^{1}H	400 MHz ^{1}H	90 MHz ^{1}H	90 MHz ^{1}H$\{^{31}P\}$

(δ_H [ppm] and ν_H [Hz] vs. TMS, J given in Hz)

BLUE HEN NMR COMPLEX

CHEMISTRY DEPARTMENT
UNIVERSITY OF DELAWARE
NEWARK, DELAWARE 19711
(302) 738-1150

October 30, 1980

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

Title: Calculation of powder pattern lineshapes

Dear Barry:

Siderer and Luz (J. Mag. Res. 37 499 (1980)) have published a useful formula for the calculation of axially symmetric powder patterns with a single Lorentzian broadening factor. We have programmed this calculation for a TI 59 Programmable Calculator and will provide copies of the program upon request. It will calculate shift anisotropy patterns (8 sec. per point), and plot them if the PC 100 printer is used.

For estimating the parameters to be used the following rules of thumb are helpful:

(1) The maximum intensity point $\sigma_{max} \simeq \sigma_{\perp} \pm \Delta/2$ where Δ is the line-broadening factor.

(2) If $\sigma_{1/2}$ is the position of 1/2 maximum height on the sharp edge, then $\Delta \simeq |\sigma_{max} - \sigma_{1/2}| \times 1.3$.

(3) The intensity at $\sigma_{\perp}$ is at 88% maximum on the sharp edge of the peak.

These rules are empirical and have been tested for $\Delta/|\sigma_{//} - \sigma_{\perp}|$ between 0.4% and 10%. They have an obvious redundancy which is useful as a check.

Our program will also calculate intensities of Pake doublets due to dipolar or quadrupolar (I=1) couplings; these take 16 sec./point.

We have found this program to be most useful for "quick fits" to observed spectra and as a source of input data to a more sophisticated computer line-fitting program.

Sincerely,

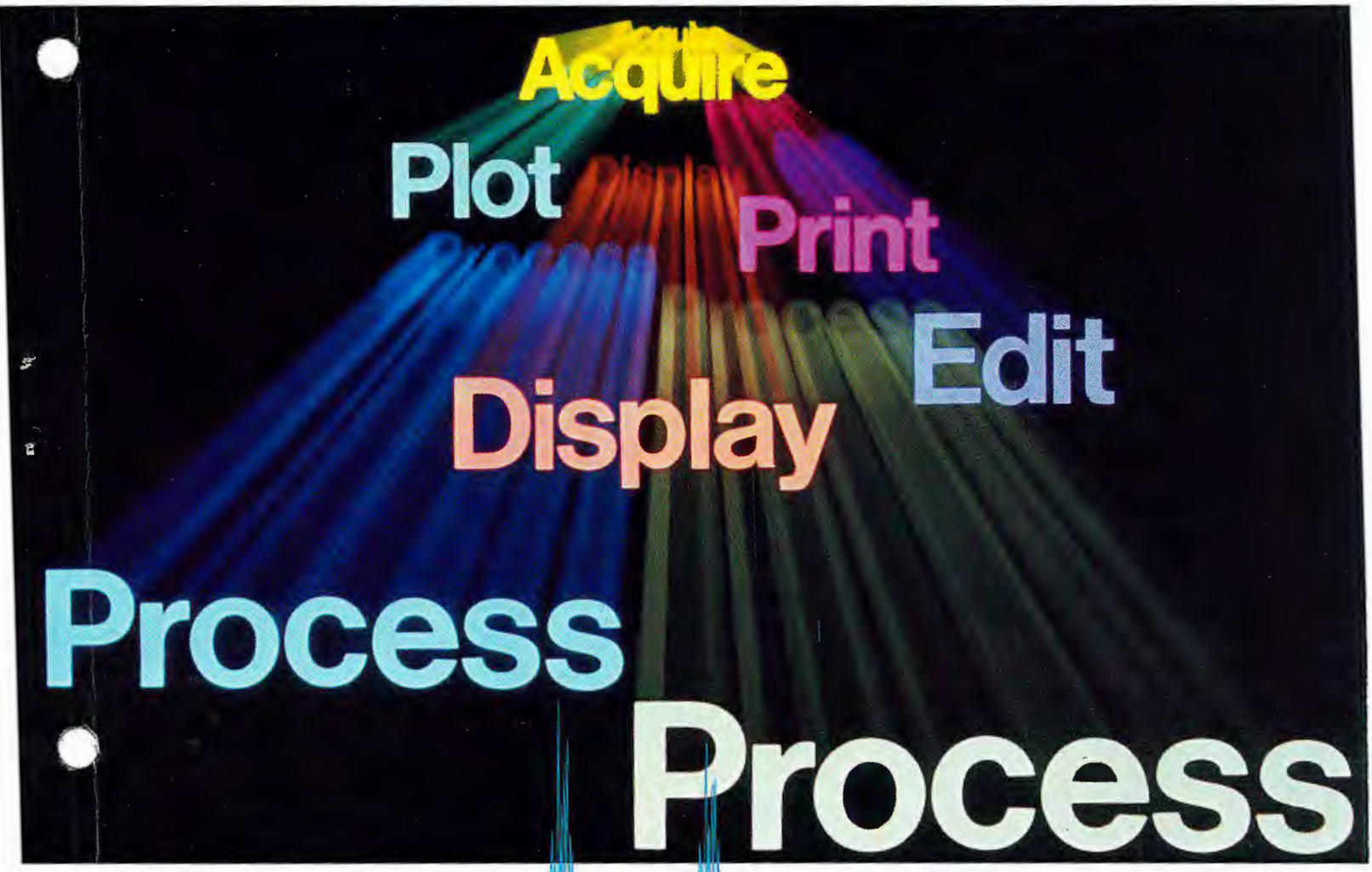
Joe

Joseph H. Noggle

Cecil

Cecil R. Dybowski

Bruker NMR



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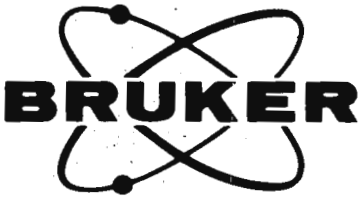

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November 5, 1980

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

Proton NMR at 300 MHz for Characterization of Multicomponent Phospholipid Vesicles

Dear Dr. Shapiro:

Since acquiring the CXP-300 spectrometer, we have been taking advantage of its high-resolution and high-field capabilities. One area of interest to us is the study of single and multicomponent phospholipid vesicles.

H-1, C-13 and P-31 NMR, in conjunction with lanthanide shift reagents, have been extensively used in determining the physical properties of phospholipid vesicles. For binary mixtures, the C-13 NMR of specifically labelled lipids has proven to be extremely useful; however, in order to obtain information about both components of a mixture, each phospholipid species has had to be alternately C-13 labelled.

We have taken a different approach: only one of the two phospholipid species is C-13 labelled and the proton NMR spectrum of the mixed dispersion is recorded. Both species can then be monitored simultaneously and under identical experimental conditions.

The accompanying figure shows the 300 HMz H-1 NMR spectrum of a 25/75 mole ratio mixture of (N-Me-^{13}C) 1,2-distearoyl-sn-glycero-3-phosphorylcholine (α-DSL) and unenriched 1,3-distearoyl-sn-glycero-2-phosphorylcholine (β-DSL) in the presence (top) and absence (bottom) of $PrCl_3$. We have been interested in the non-naturally occurring β-lecithins for some time, since they have been found to be less susceptible to lipolytic enzymes than α-lecithins.[1] Furthermore, β-DSL, when present in trace amounts, enhances the stability of small α-DSL vesicles.[2]

The choline N-methyl signals from both the α-and β-DSL are well-resolved in the H-1 NMR spectrum. Each resonance is split into two components of unequal intensities due to molecules residing on the outer (O) and inner (I) surfaces of the vesicles. The $\overset{+}{N}(CH_3)_3$ signal of the α-DSL is additionally split

by 150 Hz due to coupling to the directly bonded C-13 nucleus. Inside-outside distributions of the α- and β-DSL, as shown in the figure, are identical. The $\overset{+}{N}(CH_3)_3$ linewidths exhibit similar temperature behavior (not shown), indicating complete miscibility of the two components and the absence of a phase separation.

Please credit this letter to Dr. J. P. Yesinowski's account.

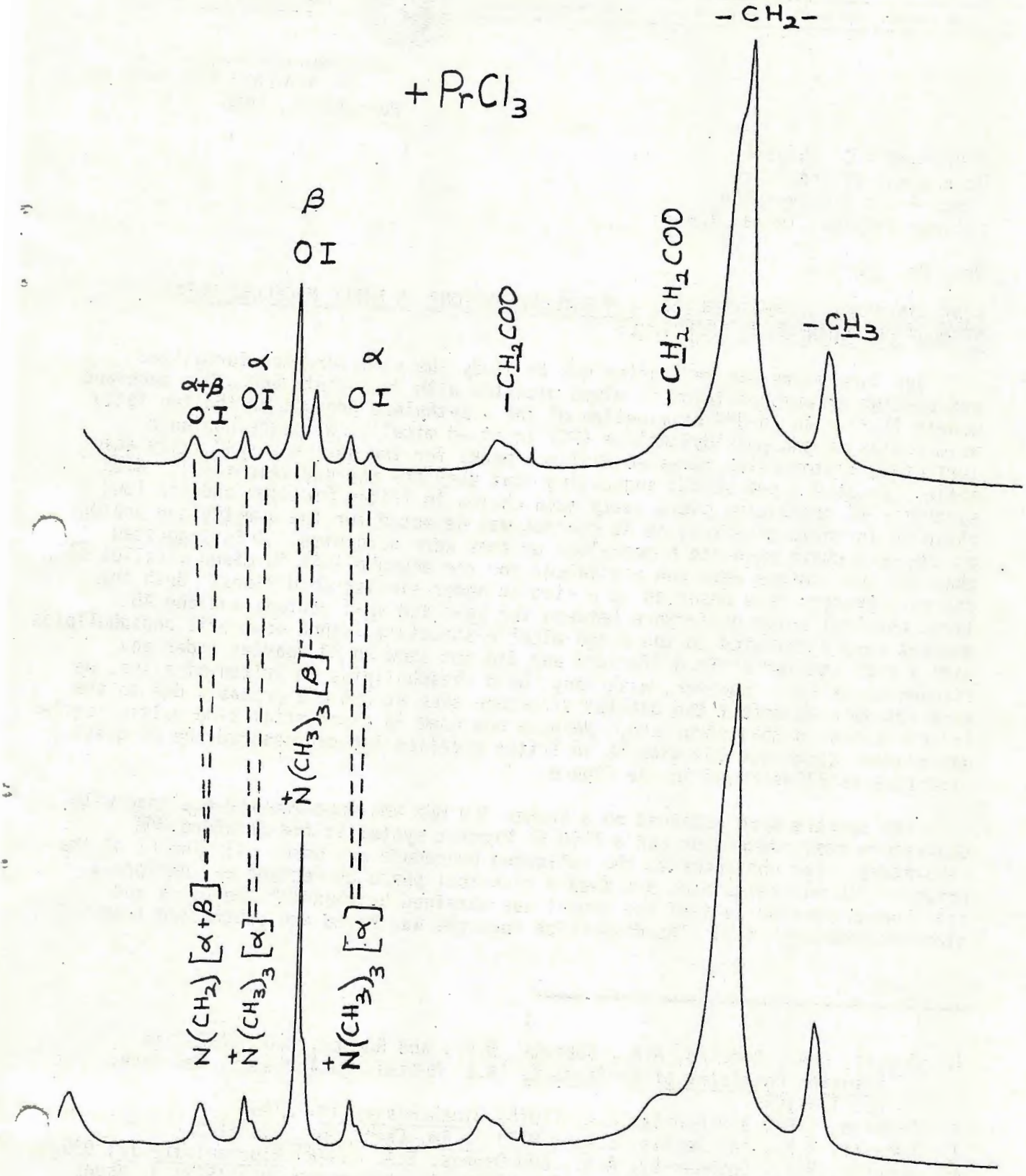
Sincerely,

THE PROCTER & GAMBLE COMPANY
Research & Development Department

Fouad Ezra
513-977-2485

1 A. J. Slotboom, H. M. Verkleij and G. H. DeHaas, Chem. Phys. Lipids, 11, 295 (1973).

2 A. L. Larrabee, Biochemistry, 18, 3321 (1979).


+ PrCl3
- CH2 -
- CH3
- CH2 CH2 COO
- CH2 COO
α+β
α
β
α
N(CH3)3 [α+β]
+N(CH3)3 [α]
+N(CH3)3 [β]
+N(CH3)3 [α]

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DEPARTMENT OF CHEMISTRY M-001
LA JOLLA, CALIFORNIA 92093

November 1, 1980

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

Dear Dr. Shapiro:

FINE STRUCTURE OF PHOSPHOLIPID α-METHYLENE PROTONS IN MIXED MICELLES USING RESOLUTION ENHANCEMENT TECHNIQUES

Our laboratory has been using NMR to study the structure, conformation, and packing of phospholipids in mixed micelles with detergents and other membrane models (1,2). An ^{1}H-NMR examination of the α-methylene protons on the two fatty acid chains of phosphatidylcholine (PC) in mixed micelles with the nonionic surfactant Triton X-100 revealed distinct peaks for the sn-1 and sn-2 fatty acid chains (about 0.1 ppm shift) suggesting that they are non-equivalent (3). With synthetic PC containing short fatty acid chains in Triton micelles and for long chain PC in ionic micelles, an AB quartet was detected for the α-methylene protons of the sn-2 chain when the β-methylene protons were decoupled. This suggested that the two protons were non-equivalent for the phospholipid in mixed micelles (4). The sn-1 protons were observed as a singlet under similar conditions. Both the large chemical shift difference between the sn-1 and sn-2 protons and the AB quartet were attributed to the mixed micelle structure, since monomeric phospholipids gave a much smaller shift difference and did not show an AB quartet under any circumstances (4). However, with long chain phospholipids in Triton micelles, we were not able to detect the quartet structure even at 360 MHz probably due to the broader lines in that structure. We have now used an exponential sine multiplication enhancement technique (5) with PC in Triton micelles and can observe the AB quartet structure as illustrated in the Figure.

The spectra were obtained on a Bruker 360 MHz NMR spectrometer equipped with quadrature phase detection and a 1180 FT Nicolet system at the Stanford NMR Laboratory. For enhancement, the following procedure was used: i) The FT of the original FID was calculated and then a classical phase correction was performed. ii) The dispersion part of the signal was obtained by changing the block and starting address. iii) The dispersion spectrum was saved and subtracted from

---

1. Dennis, E.A., Ribeiro, A.A., Roberts, M.F., and Robson, R.J. (1979) in Solution Chemistry of Surfactants (K.L. Mittal, Ed.) Plenum, New York, pp. 175-194.
2. Ribeiro, A.A., and Dennis, E.A. (1975) Biochemistry 14, 3746.
3. Roberts, M.F., and Dennis, E.A. (1977) J. Am. Chem. Soc. 99, 6142.
4. Roberts, M.F., Bothner-By, A.A., and Dennis, E.A. (1978) Biochemistry 17, 935.
5. Clin, B., de Bony, J., Lalanne, P., Biais, J., Lemanceau, B. (1979) J. Magn. Res. 33, 457.

itself by going through the addition spectra routine with K = -1. One of the parameter input knobs was used to control the shift of the two spectra with respect to one another and the corresponding enhanced spectrum was displayed on the screen. If the noise was too great, the line broadening of the FID was increased. For the spectrum shown, the line broadening was 1 Hz and there was a difference of 5 points between the two subtracted spectra. The advantages of this technique for this system were: i) No special routine is needed for the mathematical transformation. ii) Unlike some other common methods, the operation was performed in the frequency domain saving the time of the Fourier transformation for all of the unsuccessful attempts required to obtain a satisfactory spectrum. iii) With the Nicolet computer, a front panel knob can be employed to adjust the variable parameter and the enhanced spectrum is directly visualized on the scope. Other programs are also suitable if they have an adjustable parameter for the addition routine. The experimentor can quickly obtain a satisfactory spectrum. However, one has to insure that the chemical shift between the two dispersion spectra is smaller than the linewidth of the peaks of interest. The α-methylene protons' natural linewidth is about 15 Hz and it was broadened by 1 Hz before performing the enhancement with a separation of about 2 Hz between the two dispersion spectra.

Sincerely yours,

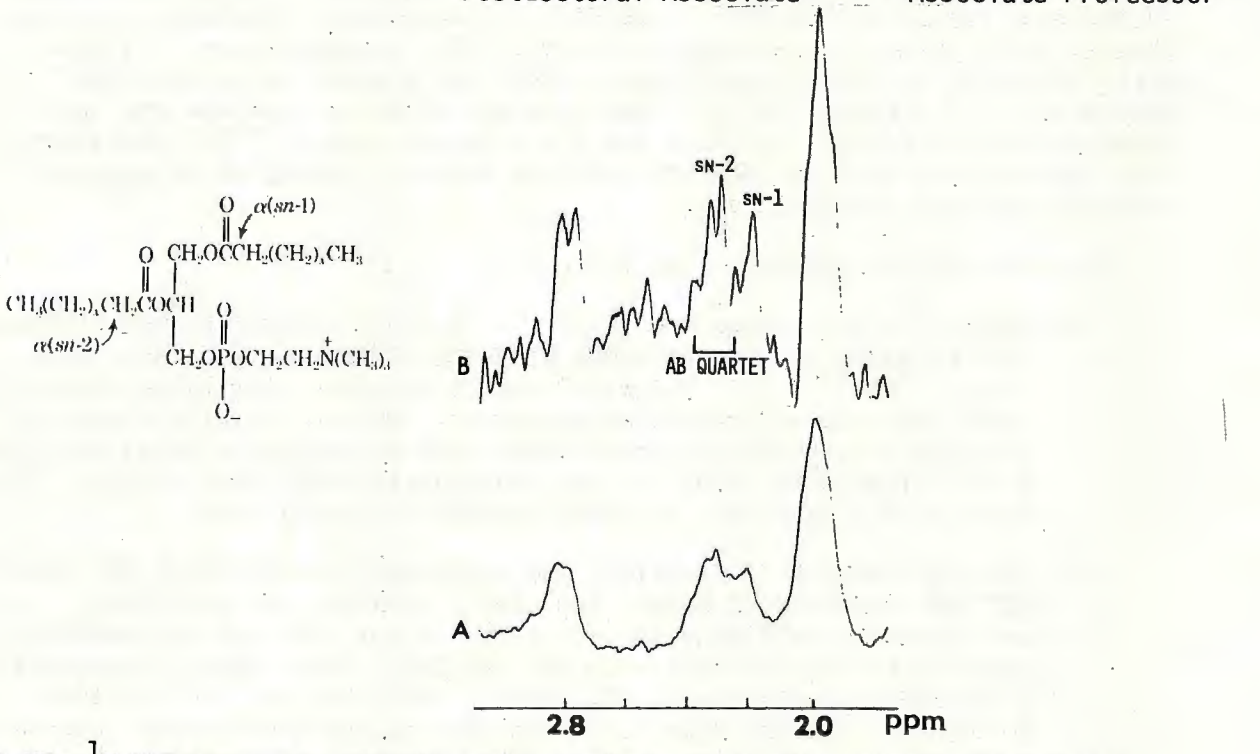
Jacqueline de Bony

Edward A. Dennis

Jacqueline de Bony
Postdoctoral Associate

and

Edward A. Dennis
Associate Profeessor



360 MHz ^{1}H-NMR spectrum of Triton/PC mixed micelles at a molar ratio of 4:1 Triton:phospholipid. Spectrum A is the expanded part of the spectrum between 2.0 and 2.8 ppm showing the α-methylene peaks (2.2 - 2.3 ppm) with the β-methylene protons decoupled. Spectrum B is the same A with resolution enhancement.

267-11

Department of Chemistry

The Florida State University
Tallahassee, Florida 32306

November 12, 1980

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

POSTDOCTORAL OPENINGS IN PHYSICAL CHEMISTRY-NMR, ORGANIC CHEMISTRY-NMR

Dear Barry:

I expect to have two postdoctoral research positions (salaries $12,000 to 14,000 plus extensive benefits, depending on experience) available in my group shortly after my move to Syracuse University this coming summer. (A start after September 1, 1981 is preferred.) The new Syracuse University NMR Laboratory will operate two fully multinuclear widebore supercon nmr spectrometer systems as well as three low field spectrometers. The Laboratory will also operate with our WARPATH computer network, giving us unique and powerful software capabilities.

The two position descriptions follow:

(1) ORGANIC or BIOINORGANIC CHEMIST, "^{15}N AND MULTI-NUCLEI NMR ELUCIDATION OF METAL ION INTERACTIONS WITH NUCLEOTIDES". Some synthesis, mostly ^{15}N, ^{31}P, ^{13}C, and metal ion nmr studies, including chemical shift and spin relaxation measurements. We are learning about the specific base-binding process using both paramagnetic metal ions such as Mn^{2+} (nmr spin labeling) and diamagnetic metal ions such as ^{113}Cd, other spin ½ nuclides, and also quadrupolar metal ions.

(2) PHYSICAL CHEMIST, "THEORETICAL AND EXPERIMENTAL STUDIES OF THE CONFORMATIONAL DYNAMICS OF DOUBLE AND SINGLE STRANDED DNA MOLECULES". We are currently obtaining an extensive ^{13}C nmr data set on carefully prepared native and denatured DNA samples. These data, obtained at dispersed magnetic fields (37.7 MHz - 100.6 MHz for ^{13}C) include linewidths, T_1s and NOEs, all functions of the overall and internal conformational dynamics of the DNA molecule. This project is a

collaboration with Randolph L. Rill (biochemist, Florida State University) and Bob London (physical chemist, Los Alamos National Laboratory). The postdoctoral researcher will direct primary attention to the theoretical side of the project, but experimental work can be included. Programming experience in Fortran would be asset.

I anticipate filling these two positions before spring and urge candidates to write to me and have two letters of recommendation forwarded to me separately. I will be pleased to provide detailed information on these and other projects under way in my group. Incidentally, I should point out that Syracuse is a city of ca. 300,000 in central New York state. Living costs in the community are quite moderate and while I cannot promise as much sun as in Florida, winter skiing is very convenient.

Yours sincerely,

George C. Levy
Professor

GCL/lh

P.S. The blue note arrived this morning. Substantive letter follows.

FACULTÉ DES SCIENCES
Université de Nantes - U E R DE CHIMIE

CHIMIE ORGANIQUE PHYSIQUE
E.R.A. nº 315 - C.N.R.S.

NANTES, le 17.11.80

Dr. B.L. SHAPIRO
TEXAS A.M. UNIVERSITY
College of Science
College Station

TEXAS 77843 USA

WANTED : Ancient Probe for HA 100 or XL 100

Dear Barry,

We are interested in buying a second hand probe from a Varian narrow gap HA 100 or XL 100-12 spectrometer. We should like to have a V4405 probe for ^{1}H observation, ^{1}H decoupling, deuterium lock and variable temperature experiment. Any equivalent probe or insert would be suitable. Anyone interested in selling such equipment is requested to contact :

Dr. G.J. MARTIN
Faculté des Sciences
Université de NANTES
(F) 44072 NANTES CEDEX

With best regards,

Yours sincerely.

G.J. MARTIN

Téléphone (40) 74.50.70 - 2, rue de la Houssinière - 44072 NANTES-CEDEX (FRANCE)

Prof. Dr. R. Kosfeld
UNIVERSITÄT DUISBURG (GH)
Fachbereich 6 - Chemie
Fachgebiet Physikalische Chemie
Bismarckstraße 90
D-4100 Duisburg 1

Duisburg, 21.10.1980

Prof. Bernard L. Shapiro
Department of Chemistry
Texas A & M University

College Station, TX 77843
USA

Subject: Separation of absorption / dispersion mixed NMR spectra into pure absorption mode and pure dispersion mode

Dear Barry,

we have tried to separate mixed NMR spectra into pure absorption mode and pure dispersion mode without using Hilbert Transformation. The separation is based upon the symmetry properties of the pure modes. The even part of the mixed spectrum is related to absorption and the odd one to dispersion. We have tested the separation in the case of simulated spectra (one example, see figure 1). Then we have applied the method to real spectra (see figure 2).

You can see, that one advantage of this procedure is to get both pure modes of an NMR signal by measuring only one spectrum (one can use it for "DISPA" plots). Another application is an "off-line phase correction" of spectra with small phase errors. We have done it for NMR-Wideline spectra. You can see the result in figure 3. The "Second Moment" versus temperature shows a steadier behaviour in the case with additional separation than in the case without separation.

Diffuculties arise from finding the point of symmetry. There are two possible ways.

The first one is to mark the spectrum by some point of reference during the time of recording (marking a defined point of frequency or field). The point of symmetry can also be determined by computations (certain conditions have to be satisfied by the choice of this point). We have chosen this possibility. It works very well for simulated spectra, but we have not completely solved the problems in connection with real spectra (baseline problems).

Sincerely

Robert Kosfeld (Robert Kosfeld)

Ulrich Matuschek (Ulrich Matuschek)

MERCK INSTITUTE / FOR THERAPEUTIC RESEARCH

RAHWAY, NEW JERSEY 07065

BYRON H. ARISON, PH.D.
SENIOR INVESTIGATOR
DEPARTMENT OF BIOPHYSICS

TELEPHONE (201) 574-6746
(201) 574-5394

October 30, 1980

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

Re: Water in Deuterated Dimethyl Sulfoxide

Dear Barry:

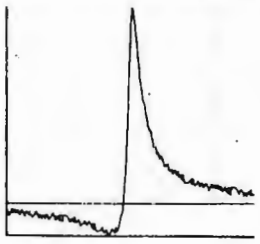
The "two kinds of water" observed by Martinelli and Ripamonti in DMSO-d_6 batches (September 1980 issue) almost certainly reflects the presence of H_2O and HDO, the individual signals resulting from a slow exchange condition. The separation of the peaks looks to be close to what one would expect for the isotope shift at 270 MHz. The varying ratio, furthermore, can be accounted for in terms of different degrees of exchange of the solvent methyls. We have seen a similar effect in some acetone-d_6 preparations.

Sincerely yours,

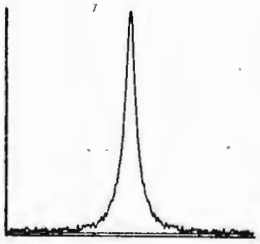
Byron

Byron H. Arison

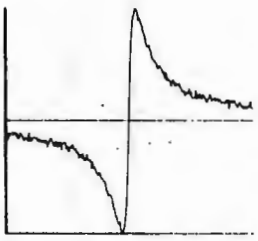
/oah



mixed (simulated)



separated



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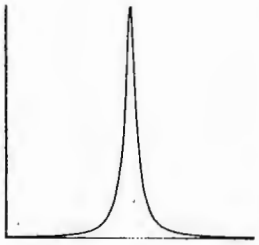
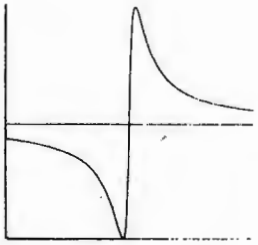
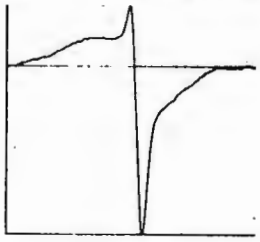


pure (simulated)

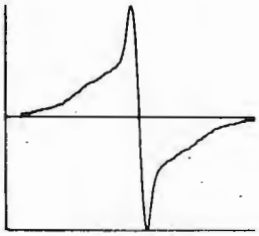


pure (simulated)

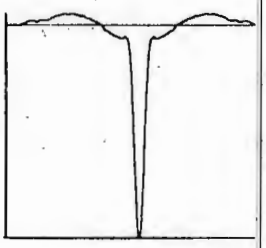
Figure 1



mixed (experimental)

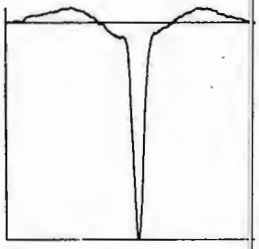


separated



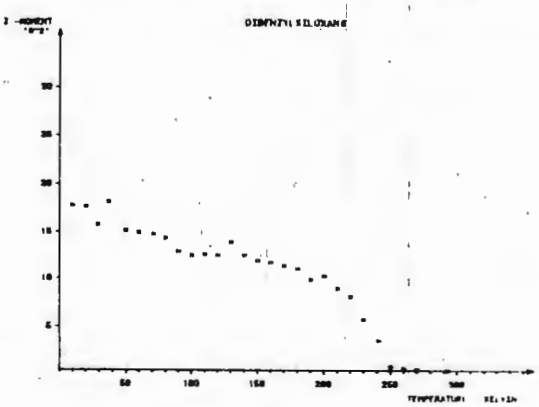
separated

pure (experimental)

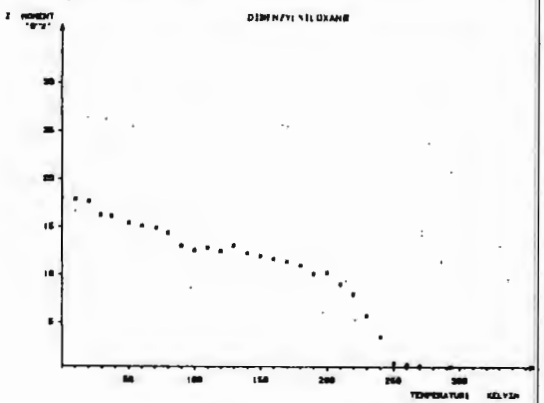


pure (experimental)

Figure 2



without Separation



with Separation

Figure 3

INSTITUTE OF CHEMICAL PROCESS FUNDAMENTALS
CZECHOSLOVAK ACADEMY OF SCIENCE
165 02 PRAHA 6 - SUCHDOL

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

November 10, 1980
3428/80

re.: Silicon-29 Sensitivity to Ring Size in Trimethylsiloxy Substituted Cycloalkenes

Dear Barry,

After too many (though fruitful) years in which we could do our ^{29}Si work only through collaboration with better equipped laboratories, we have finally got our share of a modern spectrometer. While getting acquainted with a XL - 200 we investigated a series of cis-1,2-bis(trimethylsiloxy)cycloalkenes-1 prepared by Prof. Hrnčiar´s group at Bratislava. The results (see Fig.) show again a considerable sensitivity of $\delta(^{29}Si)$ in Me_3SiO- to substituent effects, this time to its ring size.

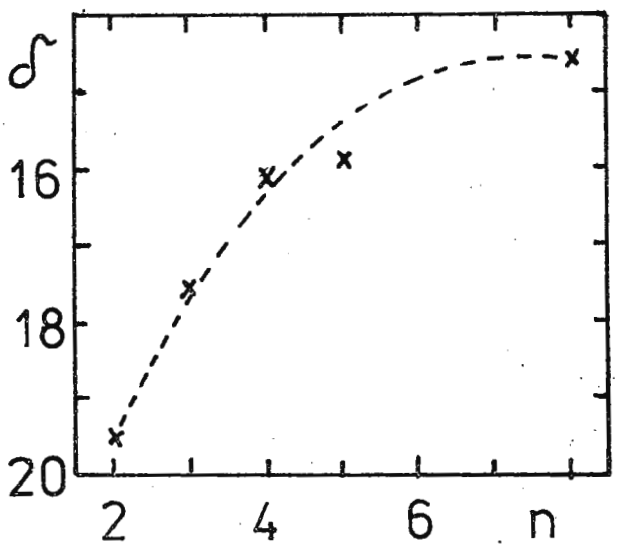
I hope that this contribution will reinstate our lab on your mailing list.

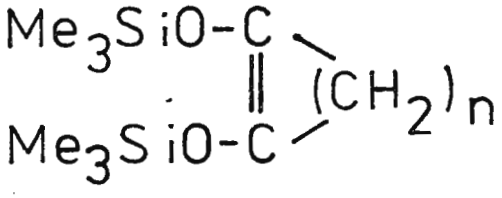
With the best regards,

Sincerely yours,

Jan

J. Schraml





VRIJE UNIVERSITEIT

VAKGROEP FYSISCHE CHEMIE
SUBFACULTEIT DER SCHEIKUNDE

1081 hv amsterdam
de boelelaan 1083
telefoon 020 - 548

Amsterdam, 1 november 1980.

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

Dear Professor Shapiro,

The ^{14}N quadrupolar coupling constant of nitrobenzene-d5 in mixtures with benzene and acetonitrile.

Quadrupolar electric field effects in NMR spectra have been studied in our laboratory recently by using a WH-180 WB Bruker NMR spectrometer, operating at an ^{14}N frequency of 12.98 MHz and a ^{2}H frequency of 27.64 MHz (H_0 = 4.2 Tesla). The electric field cell which contains the sample is described elsewhere [1]. The applied electric fields range up to 10^7 V m^{-1}.

The electric field causes polar molecules in the liquid sample to align. Quadrupolar interactions then become visible as splittings of the lines in the NMR spectrum. The magnitude of the line splitting for the ^{14}N and $^{2}H_{para}$ nuclei in nitrobenzene-d5 is

$$\Delta\nu = \frac{3}{2}(e^2qQ/h) < \frac{3}{2}\cos^2\theta - \frac{1}{2} >_E \qquad (1)$$

in which e^2qQ/h is the quadrupolar coupling constant (q.c.c.) and θ is the angle between the dipole moment μ of the molecule and the applied electric field E. The term $< \frac{3}{2}\cos^2\theta - \frac{1}{2} >_E$ is the alignment of the molecules; the brackets denote an averaging over the molecular tumbling.

At a given field strength E, and consequently at a given molecular alignment, it follows from (1) that

$$\Delta\nu\ (^{14}N)/\ \Delta\nu\ (^{2}H_{para}) = \text{q.c.c.}\ (^{14}N)/\text{q.c.c.}\ (^{2}H) \qquad (2)$$

As the ^{2}H q.c.c. is known to be 180 $\pm$ 10 kHz [2,3] one is able to deduce the ^{14}N q.c.c. from the ratio of the ^{14}N and $^{2}H_{para}$ line splittings measured at one and the same electric field.

Measurements were carried out on mixtures of nitrobenzene-d5 with benzene and acetonitrile at concentrations ranging from 100 to 23 mole % nitrobenzene. As far as can be concluded now, the ^{14}N q.c.c. of nitrobenzene-d5 appears to be approximately constant, within experimental error, over the whole concentration range in both solvents. For pure nitrobenzene the q.c.c. (^{14}N) / q.c.c. (^{2}H) ratio equals 7.9 $\pm$ 0.2; for nitrobenzene/benzene mixtures this value is 7.94 $\pm$ 0.08 and for nitrobenzene/ acetonitrile mixtures 7.99 $\pm$ 0.07. So the ^{14}N q.c.c. is found to be respectively 1.42 $\pm$ 0.12, 1.43 $\pm$ 0.09 and 1.44 $\pm$ 0.09 MHz.

We conclude that the electric field gradient of an ^{14}N nucleus in nitrobenzene is not affected when surrounding nitrobenzene molecules are replaced by benzene or acetonitrile.

Further measurements will be carried out to determine all tensor components of the ^{14}N q.c.c. of the nitro-group by comparison of ^{14}N and ^{2}H line splitting data of nitrobenzene-d5 and meta-dinitrobenzene-d4 dissolved in benzene.

References:

[1] Plantenga T.M., Ruessink B.H. and Maclean C., Chem. Phys. 48 (1980) 359

[2] Wei I.Y. and Fung B.M., J. Chem. Phys., 52 (1970) 4917

[3] Jacobsen, J.P. and Schaumberg, K., J. Magn. Reson., 28 (1977) 1

yours sincerely

MacLean, C., Plantenga, T.M., Bulsink, H.

University of Durham Department of Chemistry

Science Laboratories, South Road, Durham, DH1 3LE
Telephone: Durham 64971 (STD code 0385)

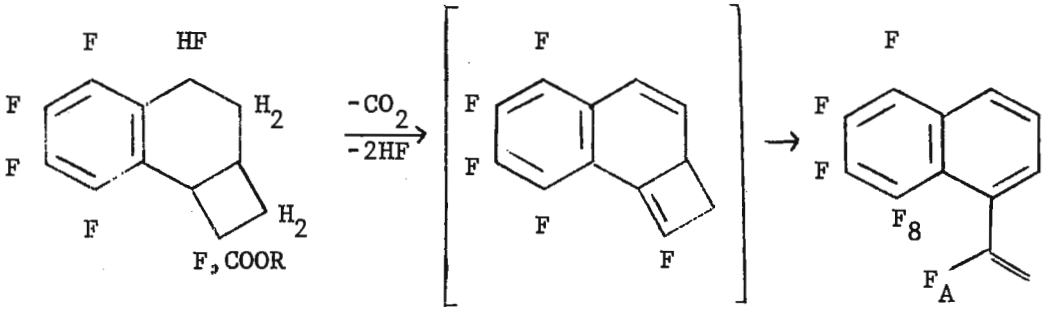
3rd November, 1980.

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

Dear Barry,

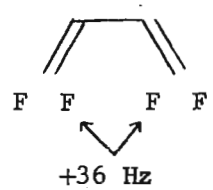
Unexpected $^5J_{F,F}$ coupling

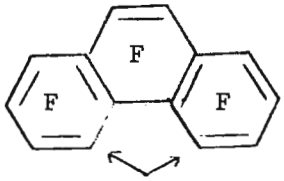
We have studied through-space F,F coupling constants for many years and recently came across another situation in which $^5J_{F,F}$ was evident. In collaboration with our colleague, Gerald Brooke we attempted to confirm the tricyclic structure of 1 by a decarboxylation. Unexpectedly two HF units were lost yielding $C_{12}F_5H_5$ (3), a 1-substituted naphthalene:



1 3

A large 45 Hz F,F coupling shown by 3 can only be attributed to the F8,FA interaction and the molecule can be drawn with F8 and FA close together. The α-fluoro vinyl group in 3 is constrained by conjugation to a position of approximate planarity and, so far, only one rotational isomer has been found. We can compare our $^5J_{F,F}$ with literature values for related situations.





167 Hz

Interestingly, there are very small downfield shifts of the fluorine nuclei associated with these steric effects and this appears to be typical

of these fluorocarbons. Sometimes the steric interactions cause large downfield shifts, or large through-space coupling constants, but rarely do the averaged effects lead to both observations.

Yours sincerely,

Ray Matthews A. Royston

Ray Matthews A. Royston

Hunter College

OF THE CITY UNIVERSITY OF NEW YORK | 695 PARK AVENUE, NEW YORK, N.Y. 10021 | DEPARTMENT OF CHEMISTRY

November 18, 1980.

(212) 570-5666

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

Dear Barry:

Title: Perkin-Elmer R-24A NMR spectrometer for sale.

We wish to sell the above instrument, which is equipped with a scope monitor and a lock-decoupler accessory. The spectrometer is about six years old and needs some minor servicing. Its sensitivity exceeds specification, and it is useful for instruction and for routine proton nmr determination.

Any reasonable offer will be considered. Interested parties may contact me at the above number, or Mr. John Potter at 212-570-5832.

Sincerely yours,

Bob

Robert L. Lichter
Professor & Chairman

RLL/ra

267-21

Maria Jablonowski c/o
GESELLSCHAFT ZUR FÖRDERUNG DER SPEKTROCHEMIE
UND ANGEWANDTEN SPEKTROSKOPIE E. V.
INSTITUT FÜR SPEKTROCHEMIE

Postanschrift: Institut für Spektrochemie, Postfach 778, 4600 Dortmund 1

To
Professor B.L. Shapiro
Chemistry Department
Texas A & M University
College Station

Texas 77843 - USA

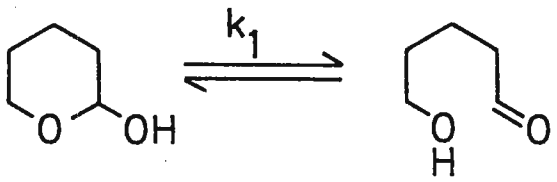
Bunsen-Kirchhoff-Straße 11
(Abzweig Ardeystraße)
Fernruf (0231) 129001-04
4600 DORTMUND 1,

Ihre Zeichen	Ihre Nachricht vom	Unsere Zeichen	
		Jab/Mas.	30.10. 1980

Betreff: Re: Kinetics of Ring-Chain Tautomerism of Tetrahydropyranole

Dear Professor Shapiro,

The cyclic half-acetale tetrahydropyranole exists in equilibrium with the open-chain aldehyde 5-hydroxypentanale [1].



The position of the equilibrium depends on the temperature. The mutual exchange - a pseudo-first order reaction - is catalysed by protons and the rate of exchange is pH- and temperature-dependent. The kinetics of this process can be studied using DNMR.

In aqueous solutions the cyclic form predominates, (at 25° C : 96.3%; at 80° C : 81.1%). The exchange at 25° C and pH 7 is slow in relation to the NMR time scale, and consequently, the noise-decoupled ^{13}C-NMR-spectrum shows sharp signals not only of tetrahydropyranole but also of 5-hydroxypentanale. Increasing the H^+-concentration causes fastening of the exchange rate and broadening of the NMR-signals.

To determine the rate-constants and activation parameters of this exchange process, the ^{13}C-NMR spectra of tetrahydropyranole dissolved in a mixture of H_2O and HCl at pH 1.0 were recorded at various temperatures between 30° C and 80° C. (External lock was used. The temperature was measured simultaneously by the 'chemical thermometer' cyclooctane-methylene iodide [2]).

A line-shape analysis of these spectra was carried out using the DNMR program by Binsch and Kleier [3]. The dependence on the temperature of the so obtained rate constants was evaluated according to the Eyring equation and gives a free enthalphy of activation of 16.4 ± 0.2 kcal/mol for the exchange at pH 1.0.

The results of a similar ^{1}H-NMR investigation are in good agreement.

Please credit this contribution to the subscription of Dr. R. Gerhards.

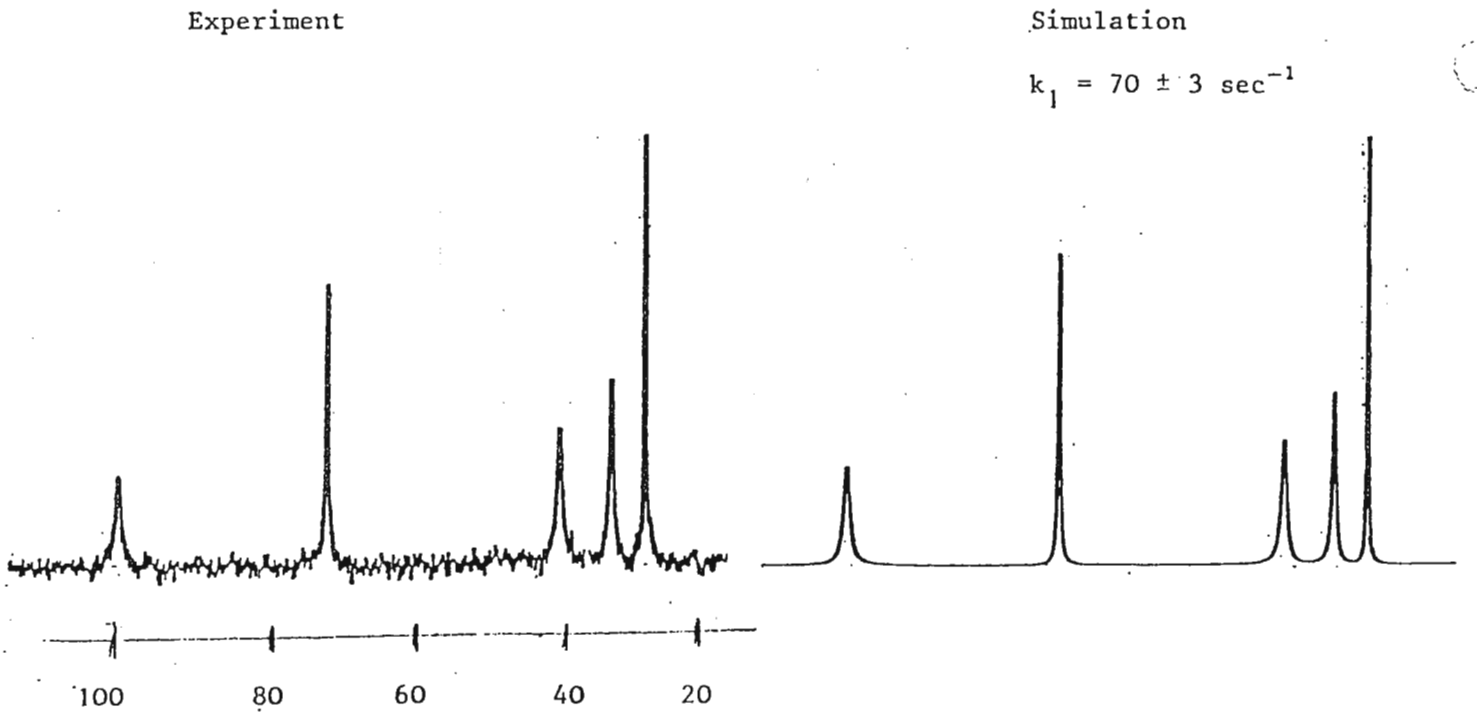
Sincerely yours,

Hans Jablonowski

1) C.D. Hurd, W.H. Saunders, J. Amer. Chem. Soc. 74, 5324 (1952)

2) D.W. Vidrine, P.E. Peterson, Analyt. Chem. 48, 1301 (1976)

3) G. Binsch, J. Amer. Chem. Soc. 91, 1304 (1969).



^{13}C-NMR-spectrum of tetrahydropyranol in H_2O at 47.4° C and pH 1.0

T in °C	K by experiment	k_1 in sec^{-1} by simulation
32.6	23.9	20 ± 5
38.7	18.9	30 ± 5
47.4	13.2	70 ± 3
57.5	9.2	115 ± 3
64.7	7.0	200 ± 10
78.8	4.3	400 ± 20

EIDG. TECHNISCHE HOCHSCHULE
ZÜRICH
Laboratorium
für Physikalische Chemie
Prof. Dr. R. R. Ernst
RIER/mü

CH-8006 Zürich,
Universitätstrasse 22
Tel. (01) 32 62 11

Oct.30, 1980

Prof. B.L. S h a p i r o
Department of Chemistry
Texas A & M University
C o l l e g e S t a t i o n
Texas 77843 U S A

POSTDOCTORAL POSITION AVAILABLE FOR NMR SPECTROSCOPIST

Dear Barry,

For the continuation of an interdisciplinary research project on the application of novel NMR techniques to biomolecules, including two-dimensional spectroscopy and Overhauser effects, we are looking for a

NMR SPECTROSCOPIST

with a broad background and interest in NMR methodology and biological applications of magnetic resonance.

The desired starting date is August to September 1981. The initial employment would last for one full year, but it is renewable. The salary will be equivalent to that of an Assistant I according to ETH regulations.

Inquiries should be sent to

Prof. R.R. Ernst
Laboratorium für
Physikalische Chemie
ETH-Zentrum
8092 Zürich, Switzerland

Prof. Kurt Wüthrich
Institut für Molekularbiologie
und Biophysik
ETH-Hönggerberg
8093 Zürich, Switzerland

Sincerely yours,

Richard R. Ernst

Kurt Wüthrich

267-25

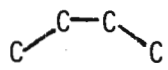
TITLE: THE EFFECT OF THE ORIENTATION OF THE TERMINAL SUBSTITUENT UPON VICINAL CARBON-CARBON COUPLINGS

North Texas State University
Denton, Texas 76203
Department of Chemistry

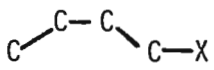
November 18, 1980

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

Some years ago the dihedral angle relationship was established for carbon-carbon couplings, whereby vicinal carbon-carbon couplings depended upon the dihedral angle in a fashion analogous to that for proton-proton couplings (i.e., maximum J(CC) at $\phi = 0^0, 180^0$). However, carbon-carbon couplings have complicating features not encountered with proton-proton couplings. One such feature is the effect of the orientation of a terminal substituent on J(CC)). Accordingly, in the linkage C-C-C-C-X, the value of the vicinal J(CC) may vary depending upon the geometry of the substituent X with respect to the carbon framework. Theoretical calculations[1] suggest that the effect of the substituent is greatest with the carbon framework cis (1), which might be expected intuitively since the substituent in this conformation may be close to the coupling carbon. This calculations further suggest that in this conformation of C-C-C-C the value of J(CC) should be much greater when the substituent is transoid (2) than when it is cisoid (3).



1



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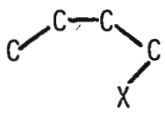
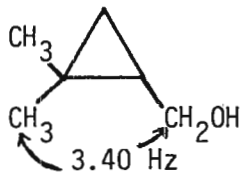


3

We have found some examples which may bear out this prediction. In the cyclopropane series 4-6, the cis vicinal coupling steadily decreases as the carbinol carbon becomes more highly substituted (with methyl groups), thereby populating more heavily those conformations wherein the hydroxyl group is directed more nearly towards the coupling carbon. The data of compounds 4-6 were obtained by the gracious assistance of Mike Barfield and Steve Walter at the University of Arizona.



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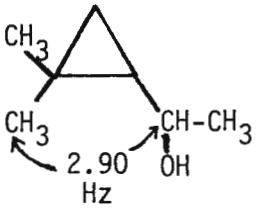

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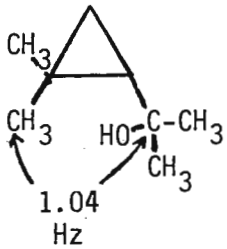


6

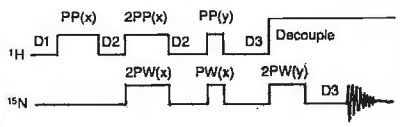
Sincerely,

J. L. Marshall

[1]M. Barfield et al., J. Amer. Chem. Soc., 98, 6253 (1976).

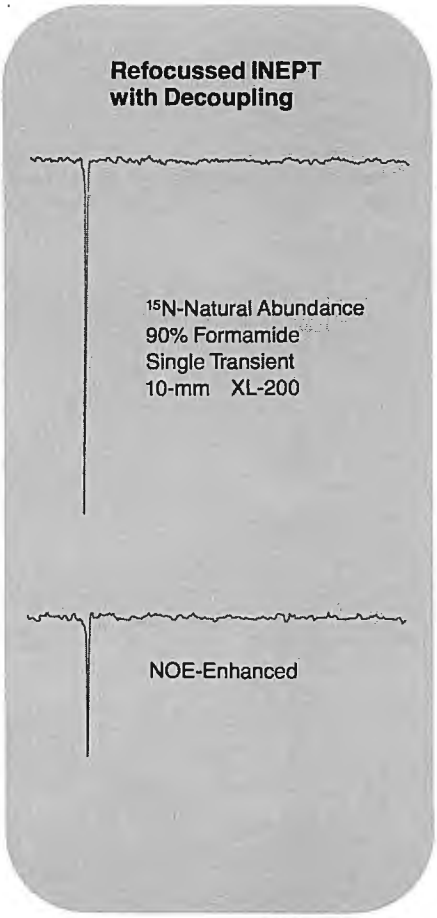
How much do you know about the Varian XL-200?

Pulse Diagram



PSG Code

```
"INEPT WITH REFOCUSSING
   AND DECOUPLING
%I(SIMPUL)
PROCEDURE PULSESEQUENCE;
    VAR PP,J,MULT,D3:REAL
BEGIN
    GETVAL(J,'J   ');
    GETVAL(MULT,'MULT   ');
    GETVAL(PP,'PP   ');
    D2:=1.0/(4.0*J);
    PUTVAL(D2,'D2   ');
    IF MULT=3.0
      THEN D3:=1.0/(4.0*J);
      ELSE D3:=1.0/(2.0*J);
    PUTVAL(D3,'D3   ');
    ASSIGN(OPH,V1);
    INCR(V1);
    "EQUILIBRATION"
      STATUS(A);
      DELAY(D1);
    "TRANSFER POLARIZATION"
      STATUS(B)
      DECPULSE(PP,OPH);
      DELAY(D2);
      SIMPULSE(2.0*PW,2.0*PP,
        OPH,OPH,0.0,0.0);
      DELAY(D2);
      SIMPULSE(PW,PP,OPH,V1,0.0,0.0);
      DELAY(D3);
    "START DECOUPLING"
      STATUS(C);
      MOD2(OPH,V2);
      INCR(V2);
      PULSE(2.0*PW,V2);
      DELAY(D3);
    "OBSERVE FID"
      STATUS (D);
END;
```



Almost everyone knows about the XL-200's reliability and ease of operation. But are you aware of its power, flexibility and sophisticated research capabilities?

Beneath its basic exterior, the XL-200 offers you true research power to perform complex experiments.

For example, you can frequently obtain enhanced sensitivity from low-γ nuclei through INEPT sequences on the XL-200. See: Freeman and Morris, *J. Amer. Chem. Soc.*, **102**, 72 (1979); and, Morris, *J. Amer. Chem. Soc.*, **102**, 428 (1980).

Illustrated here is a simple implementation of these ideas.

The XL-200's Pulse Sequence Generation capabilities were used to perform the enhanced sensitivity experiment above. Acquisition Processor features are another important benefit for XL-200 owners.

Pulse Sequence Generation

- PASCAL language-based code with resident compiler
- English-like sequence code
- Error checking compiler
- Large text library for source code storage
- Sophisticated editor for convenient programming in PASCAL
- Use of PASCAL statements within sequence code
- Simple PSG components such as:

PULSE	OFFSET	SPAREON	HLV
OBSPULSE	DELAY	SPAREOFF	DBL
DECPULSE	IFZERO	DECPHASE	ADD
SIMPULSE	LOOP	RCVRON	SUB
STATUS	DECR	RCVROFF	MOD2
ASSIGN	INCR	RND	MOD4

- Ability to specify and vary phase and receiver off-times dynamically
- Use of indirect variables for phase control
- Up to three nested loops for repetitive action
- Ability to execute simultaneous observe and decoupler pulses
- External device control under sequence control
- Use of floating-point parameter format
- User-creation of new delay, pulse, frequency, integer and flag parameters
- Flexible branching within sequences
- Ability to phase-shift within a pulse with no dead times
- Use of math statements for sequence timing calculations
- Complete separation of sequence code from parameter sets
- Dynamic variable calculations
- Use of indirect parameter labels in sequence code
- User control of parameter display characteristics
- **Example sequences**
  Standard two-pulse
  Carr-Purcell-Meiboom-Gill T2
  Quadrupole echo
  Cross-polarization
  Miltiple-contact cross-polarization
  Selective excitation
  Quadrature selective excitation
  INEPT
  INEPT with refocussing and decoupling
  PREP
  J-Cross polarization
  Refocussed J-cross polarization
  Noise off-resonance spin echo
  Inversion-recovery spin echo
  Multiple quantum 2D
  Proton-carbon correlated 2D
  Heteronuclear enhanced 2D
  Double quantum ^{13}C-^{13}C spectroscopy

Acquisition Processor

- Independent 32-bit arithmetic bit-slice 32K CPU
- 50-nanosecond hardware timing
- Software-programmed for highest flexibility
- FIFO architecture for event streaming at 50-ns resolution
- State-of-the-art LSI construction
- 50-kHz spectral widths standard
- Pulse timing to 0.1 microsecond
- Automatic filter selection
- Four observe phases under CPU control
- Four decoupler phases under CPU control
- Explicit and relative mode phase selection
- Quadrature detection
- Single or double precision acquisition with 32-bit data path
- Direct periodic data save to non-volatile memory
- Transmitter and decoupler frequencies under CPU control
- Decoupler gating under CPU control
- Decoupler modulation under CPU control—CW, noise, square wave and external
- Decoupler modulation frequency under CPU control
- Decoupler high/low power switch under CPU control
- Precision decoupling power in 60 one-dB steps below one watt under CPU control
- Computer controlled VT
- Low/High and High/Low VT mode switch under CPU control
- Up to three simultaneously arrayable acquisition parameters
- Dynamic phase selection in multi-transient data collection
- 48-bit microprogram specialized instruction set
- Noise amplitude and scaling limit checking
- Lock/VT/high noise interlocks

In NMR research the XL-200 is first among first-class spectrometers. Following are lists of other features that make the XL-200 your logical choice for complex and sophisticated research NMR.

Standard Features

- Computer controlled VT
- $^{19}F/^{1}H$ 5-mm VT probe
- ^{15}N-^{31}P 10-mm VT broadband probe
- ^{1}H universal transmitter cards for observe, decouple and lock
- Built-in printer, plotter, keyboard, TV display and 5M-word dual disk
- 32K acquisition processor memory
- 32K main CPU memory
- Large, calibrated chart paper
- Interactive display knobs
- Autolock for automatic locking, even after sample change
- Pulsed/timeshared lock modes
- Universal fixed and broadband rf transmitters with interchangeable functions
- 3-month helium hold-time with only 25 liters needed for refill, *including* transfer loss
- 14-day nitrogen hold-time—45 days with optional refrigerator
- Welded dewar
- 25-watt rf transmitter output—200-watt pulse amplifier
- 10-μsec ^{1}H 90° pulse/15-μsec ^{13}C 90° pulse
- Internal ^{2}H lock
- Pushbutton PROM-based program loading
- Disk-based data system
- Flicker-free TV display with graphics capability
- Simplified 1-meter probe tuning
- 0.4 to 1.6 MHz offset synthesizer
- 13-bit ADC

Accessories

- ^{19}F transmitter
- Large sample and 5-mm broadband probes
- Nitrogen refrigerator
- Magnet power supply
- Maintenance kit
- Magic-angle/cross-polarization solids probes

Data System

- PASCAL language
- State-of-the-art operating system
- Disk-based using modular design software concept
- Concurrent and sequential PASCAL
- Floating-point data and math format
- Multitasking-simultaneous acquire, plot, print, display, parameter entry
- Queuing of acquisitions, plots, prints and calculations
- Spooling of plots and prints
- Disk-resident data tables
- Separate FID and spectral storage
- System resident PASCAL compiler for user programming
- User access to data files
- Expandable user-defined command and parameter architecture
- Floating-point or integer transform
- Convolution difference/gaussian apodization functions
- Parameter set libraries
- 2D transform
- Plot graphics
- T1, T2, 3-parameter least-squares-fit analysis programs
- Spin simulation
- LAOCOON with magnetic equivalence
- User-definable disk libraries
- PASCAL system source code availability
- NOE calculation
- Add-subtract-convolution spectral manipulation

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reference

13th November 1980

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

Dear Professor Shapiro,

^{31}P nmr detection of two conformations of an enzyme-ligand complex

In our work on dihydrofolate reductase, ^{31}P nmr has proved most useful for studying coenzyme binding (1,2). The ^{31}P resonances of the two pyrophosphate phosphorus nuclei have very similar chemical shifts in the free coenzyme, but the shift difference is increased on binding so that two doublets ($^{2}J_{pp} \sim 20$ Hz) can be clearly resolved, as shown in the Figure for the enzyme-NADP^{+}-methotrexate complex.

However, if the antibacterial drug trimethoprim is used in place of methotrexate the ^{31}P spectrum becomes more complicated (see Figure). It consists of four doublets, one at -12.9 ppm (doublet splitting ill-resolved in this spectrum), two at -14.9 ppm and one at -16.4 ppm. This complex appears to exist in two slowly interconverting conformations present in approximately equal amounts. One corresponds to that seen for the enzyme-methotrexate-NADP^{+} complex (conformation I) and the other to that seen for the enzyme-trimethoprim-thioNADP^{+} complex (conformation II). The two conformations differ in the conformation of the bound coenzyme, as indicated by differences in $^{3}J_{PH}$ ($\Sigma J^{31}P\text{-}O\text{-}C_{5'}\text{-}H_{A,B}$ = 13 Hz and < 5 Hz for the two signals of conformation I (cf. ref.1), and < 5 Hz, < 5 Hz for conformation II) and $^{2}J_{pp}$ ($\sim$ 20 Hz for I, $\sim$ 11 Hz for II). In addition there are large differences in the nicotinamide proton chemical shifts, and smaller differences in the resonances of two histidine and two tryptophan residues of the protein for the two conformations.

To understand the structure-activity relationships of trimethoprim analogues, it is important to know which of these two conformations is preferred in each case, and ^{31}P nmr appears to provide a particularly simple method of obtaining this information.

Yours sincerely,

G.C.K. Roberts J. Feeney

E.I. Hyde B. Birdsall A. Gronenborn.

References:

(1) Feeney, Birdsall, Roberts & Burgen (1975) Nature, 257, 564.
(2) Hyde, Birdsall, Roberts, Feeney & Burgen (1980) Biochemistry, 19

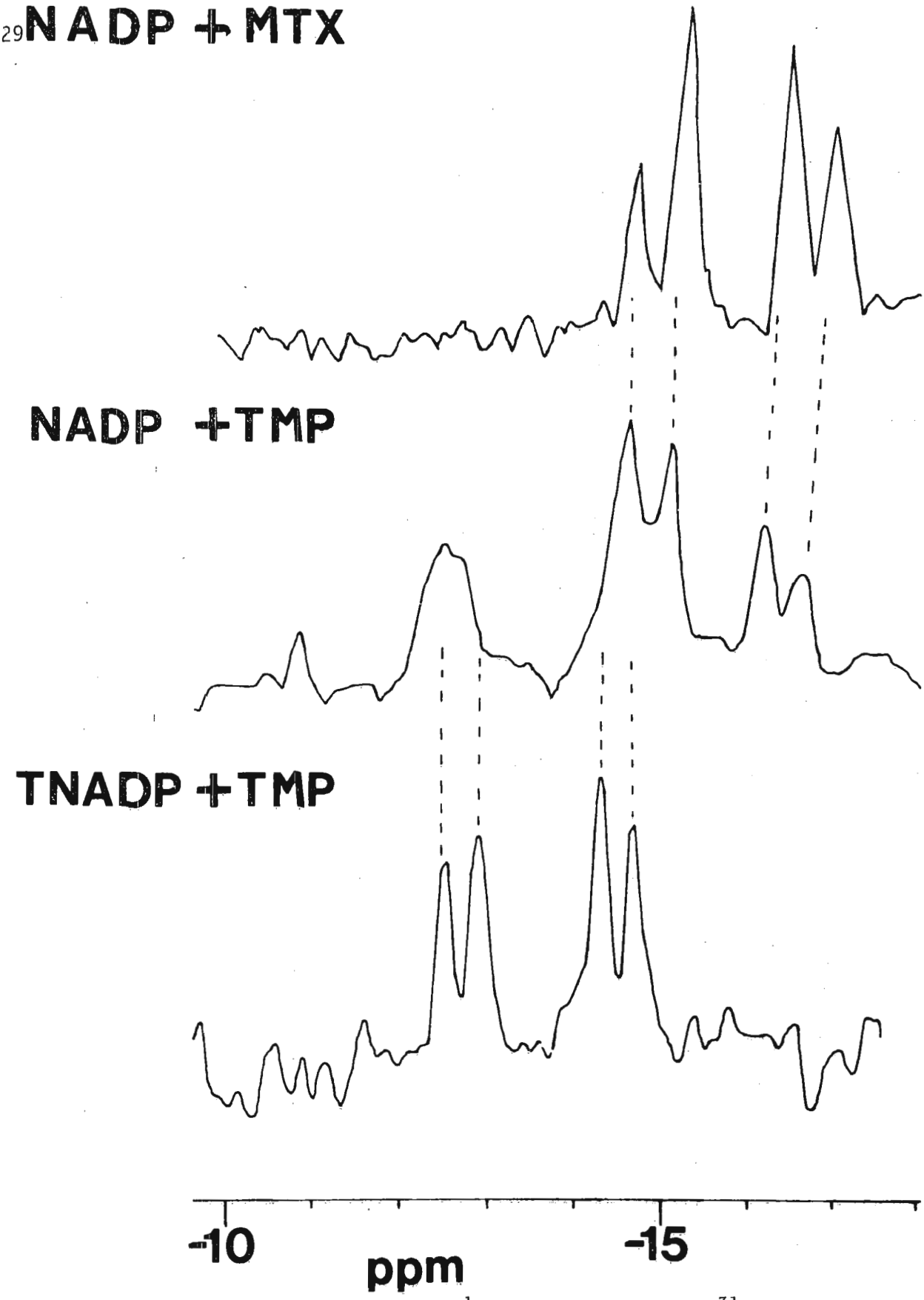


Figure. Pyrophosphate region of the ^{1}H-decoupled 40.5 MHz ^{31}P spectra of the complexes of dihydrofolate reductase with $NADP^+$ and methotrexate (top), $NADP^+$ and trimethoprim (centre) and thio$NADP^+$ and trimethoprim (bottom). Samples consisted of 1.4 ml of approximately 1 mM enzyme. Spectra were obtained on a Varian XL-100 in the block averaging mode, averaging 300-500 blocks of 200 transients. Chemical shifts are relative to external inoragnic phosphate, pH 8, upfield shifts negative.

UNIVERSITÉ RENÉ DESCARTES (PARIS V)

U E R DES SCIENCES PHARMACEUTIQUES ET BIOLOGIQUES

DÉPARTEMENT DE CHIMIE ORGANIQUE

(CNRS) ERA 07.613

PROFESSEUR B. P. ROQUES

S.C.N. n° 21 de l'INSERM

November 20th, 1980

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

Fit of self-association data of DNA intercalating drug determined by ^1H NMR using pocket-calculator HP 67 or desk-calculator HP 85.

Dear Professor Shapiro,

Numerous aromatic drugs (acridine, actinomycin, ethidium bromide, ellipticine...) are DNA intercalating agents. Due to the large shielding of aromatic protons resulting from the intercalation of the drug between adjacent base pairs, NMR is well suited to study the geometry and thermodynamic parameters of such complexes in aqueous solution. However the chemical shifts of the protons of the free intercalating agent are usually concentration dependent. Therefore a detailed analysis of drug-DNA interactions requires a preliminary self-association study of the drug.

Assuming the formation of stacked n-mers, informations can be obtained about the equilibrium constant and the geometry of the self-association complex with the following simplifications, i) no cooperativity, ii) additivity of the magnetic anisotropy, iii) shielding effect only from the nearest neighbours (1). Straighforward calculations lead to :

$$\delta = \delta_m + (\delta_d - \delta_m)\ (2KB + 1 - \sqrt{4KB+1})\ /\ KB \qquad \text{I}$$

where : K is the self-association constant
B is the total drug concentration
δ_m and δ_d are respectively the monomer and the dimer chemical shifts.

.../..

K, δ_d and δ_m are unknown. Linearization of I can be achieved giving II .

$$\sqrt{\frac{\delta_m - \delta}{B}} = \sqrt{\frac{K}{2(\delta_m-\delta_d)}} \quad (2(\delta_m-\delta_d) - (\delta_m-\delta)) \qquad \text{II}$$

When δ_m can be accurately determined ($B \longrightarrow 0$) II can be graphically solved. Unfortunately the sensitivity range of ^1H NMR in water (minimal concentration $\sim 5.10^{-5}M$) prevents such an extrapolation of δ_m for $K > 500\ 1M^{-1}$. In this case two different ways for numerically solving of I or II have been developped in our laboratory.

The first one uses a trial and reject method associated with a linear least square regression of II. This method considers an initial approximation δ_m as a starting base and makes an explorating upward move from it. Then it chooses the value which leads to the greatest correlation coefficient and determines the other parameters by linear regression. This approach has been developped on a HP 67 calculator (140 steps used allowing a maximum of 8 data points). This program has been translated for HP 85 calculator allowing fit on 20 data points and direct plot of experimental and theoretical curves.

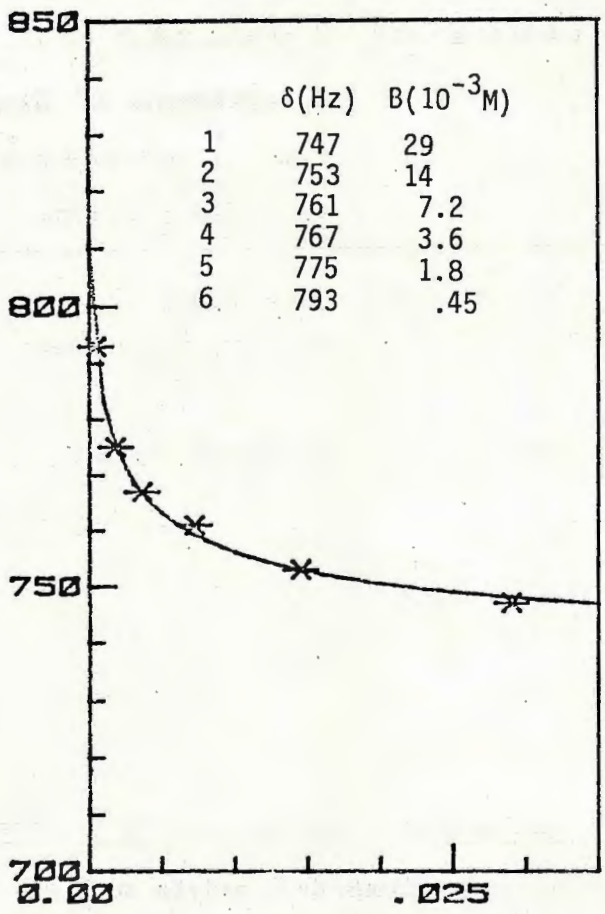
The second one uses a general non linear optimization program (Newton method) already developped for other NMR applications (T_1 fitting) (2,3). This program developped on HP 85 desk calculator (16 K, matrix rom) uses 3 different sets of initial values (in order to prevent local minima of the least-squares objective function) and a maximum of 20 data points. Approximate estimation of standard deviation and of confidence intervals of the parameters are given on the analogy of linear models.

Sincerely yours ;

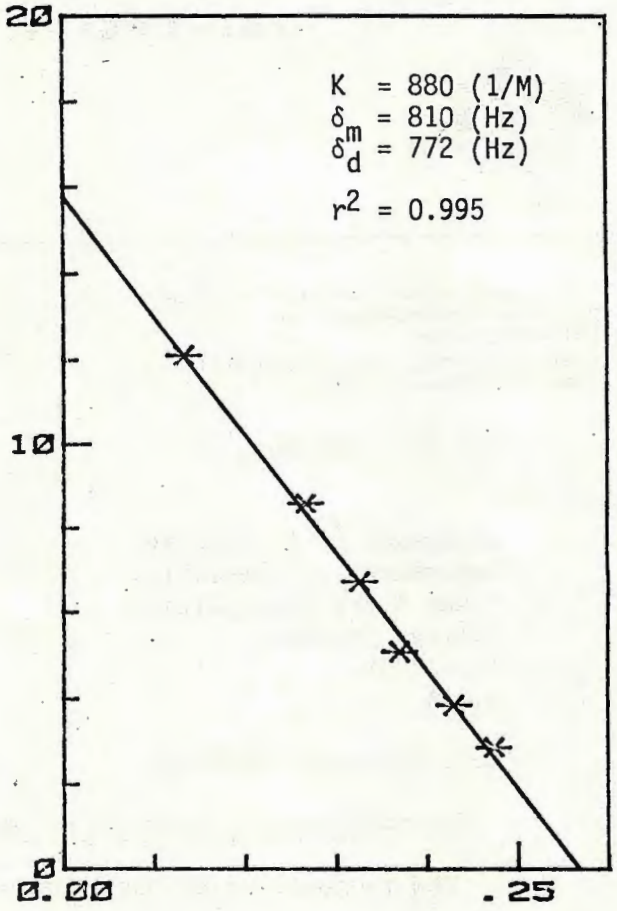
A. Delbarre P. Laugaa D. Marion B.P. Roques

References

1. J.L. Dimicoli and C. Helene, J. Amer. Chem. Soc. 95 (1973), 1036.
2. D.W. Marquardt, Chem. Eng. Progr. 55 (1959), 65.
3. W.H. Swann, FEBS Lett. 2 (1969) S.39.
4. A. Delbarre, B.P. Roques, J.B. Le Pecq, J.Y. Lallemand and N. Dat Xuong, Biophys. Chem. 4 (1976), 275.



Chemical shifts (Hz) of H_1 of 9.OH ellipticine at 90 MHz in aqueous solution versus concentration B (Mole) (4).



Linearization of experimental data with equation II and computed parameters by the first method.

Table : Parameters computed by non linear least square fitting for H_1 of 9.OH ellipticine.

δ_m : chemical shift of monomer (Hz)
δ_d : chemical shift of dimer (Hz)
K : self-association constant (l/M)

Set	Initial values	Fitted values	Standard deviation
	δ_m = 800	δ_m = 809	4
1	δ_d = 700	δ_d = 771	30
	K = 800	K = 751	215
	δ_m = 850	δ_m = 809	4
2	δ_d = 650	δ_d = 771	30
	K = 950	K = 751	215
	δ_m = 900	δ_m = 809	4
3	δ_d = 650	δ_d = 771	30
	K = 500	K = 751	215

UNIVERSITY COLLEGE OF SWANSEA

Department of Chemistry

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UNITED KINGDOM
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J. H. Purnell M.A., Ph.D., Sc.D., C.Chem., F.R.I.C.
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Head of Department.

A. Pelter Ph.D., D.Sc., D.C.C., C.Chem., F.R.I.C.
Professor of Organic Chemistry.

J. H. Beynon, D.Sc., C. Chem., F.Inst.P., F.R.I.C.
F.R.S.,
Royal Society Research Professor.

Our Ref. JMW/bei.

5th November, 1980.

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

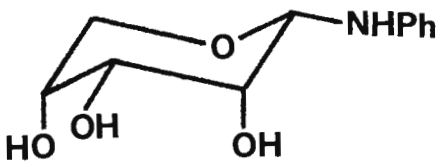
Dear Professor Shapiro,

Tautomerism in ribose anilides, and maintenance of magnet cooling systems.

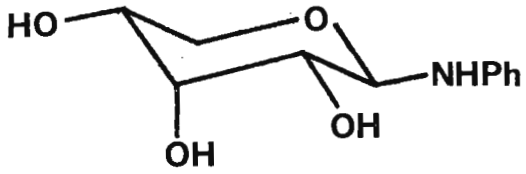
The tautomerism of the monosaccharides and various derivatives such as oximes, hydrazones and glycosylamines is a fascinating subject. Consider for example the fact that crystalline D-glucose oxime has the β-pyranose structure but in an aqueous solution the acyclic syn form predominates. In contrast D-arabinose oxime exists in the acyclic anti form in the solid state while the acyclic syn form predominates in solution (ref.1). Glycosylamines are well known for their tendency to isomerise in solution and their identification is not always straightforward. We have examined the two "anilides" of D-ribose (N-phenyl-D-ribosylamine); derivative A, m.p. 126-7°, $[\alpha]_D^{20}$ + 178° (pyridine), and the more common derivative B (a hemihydrate), m.p. 113-5° $[\alpha]_D^{20}$ + 60° (pyridine). Derivative A is obtained when anhydrous conditions are used for the preparation (Ref. 2). Pyranose, furanose and acyclic imine structures have been suggested in the past for these derivatives. For those of us who do not have facilities for high resolution n.m.r. of solids, investigating such compounds requires the use of solvents in which isomerisation is very slow. Such solvents include dimethylsulphoxide, NN-dimethylformamide and pyridine, and the data given below refer to pyridine-d_5 solutions.

P.m.r. measurements at 100 MHz show that A is the α-pyranosylamine (1) characterised by a broadened doublet at 5.2 δ for the anomeric proton

(J_{HCNH} = 8.5 Hz, W_h ~ 2 Hz) and a multiplet at 3.7 δ for the shielded axial proton at C-5. B is a mixture of the α- and β-pyranosylamines, the β-anomeric proton resonating at 5.45 δ ($J_{1,2}$ = 8 Hz); the anomeric proton of the α-anomer constituent was a sharp doublet ($J_{1,2}$ = 2.5 Hz) after D_2O exchange. 220 MHz spectra gave the following data; for the α-anomer, $J_{1,2}$ ~ 2.5 Hz (not resolved) $J_{4,5eq}$ < 1 Hz, $J_{4,5ax}$ = 2 Hz, $J_{5ax,5eq}$ = 11 Hz); for the β-anomer, $J_{1,2}$ = 8 Hz; multiplets for the remaining pyranose ring protons were broad and overlapping. The α- and β-pyranosylamines are in the $^1C_4$ and $^4C_1$ conformations respectively (1 and 2).



1



2

The ^{13}C n.m.r. spectra confirmed that 1 and 2 were pyranose and not furanose derivatives. We hope to exclude the possibility that derivative B is a different tautomer which isomerises to a mixture of 1 and 2 very rapidly (in less than 2 minutes) by high resolution n.m.r. of the solid.

Finally I would like to solicit advice concerning the maintenance of the magnet cooling system on our Varian XL 100 spectrometer. We are concerned about an increase in pressure (from 34 to 40 lb/sq.in) which has occurred in the internal water circuit over the last 12 months after only a small increase during the previous five years. We have received conflicting advice on flushing with oakite. Your readers' experiences would be of great interest to us.

May this contribution sustain J.M.W's subscription, recently taken over from Claude Haigh.

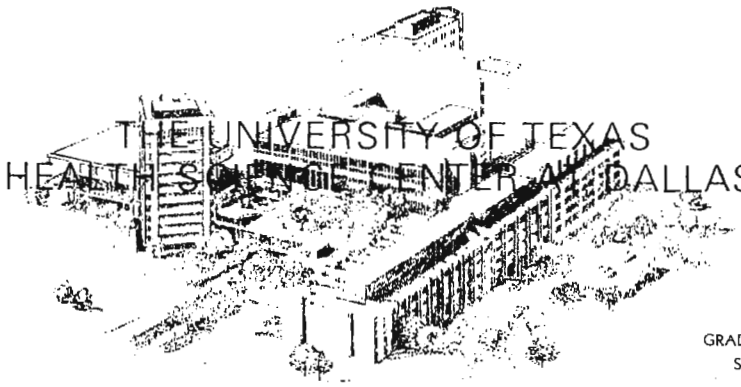
Yours sincerely,

Mike Williams

J. M. Williams. G. P. Ellis (U.W.I.S.T.,Cardiff)

References

1. P. Finch and Z.Merchant, J.C.S. Perkin I, 1975, 1682.
2. G. P. Ellis and J. Honeyman, J.Chem.Soc., 1952, 1940.



DEPARTMENT OF RADIOLOGY

SOUTHWESTERN MEDICAL SCHOOL
GRADUATE SCHOOL OF BIOMEDICAL SCIENCES
SCHOOL OF ALLIED HEALTH SCIENCES

November 14, 1980

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

"Field Shimming on Biological Samples:
A Broadband Lock Accessory"

Dear Dr. Shapiro:

Among the problems one encounters with "high resolution" NMR studies of perfused organs is the very practical one of field shimming. A common technique is to simply shim on a deuterated solvent sample and then try to position the biological sample within a sample tube such that it sits within the region of best homogeneity. This means that in going from one "sample" to the next, positioning the organ in an identical manner is essential. This is not always possible, however.

The ubiquity of water protons in living tissues make the use of a proton shimming signal very attractive. Such a system based upon a broadband lock accessory has been extremely helpful in ^{31}P NMR studies of perfused hearts both for conventional probe work and surface coil experiments. In these cases, the proton signals from the tissue water are used for obtaining the best field homogeneity. Furthermore, because of the strength of the proton signal, it is not necessary to employ a coil system with an impedance match at the proton resonance frequency. Hence, the "x" nucleus observe coils will work in most cases.

The broadband lock circuit shown in Figure 1 has proved an invaluable asset in obtaining optimum results for ^{31}P NMR studies of perfused hearts. The first design for this circuit was given to me by Dr. Craig Bradley. The addition of the tunable bandpass filter on the output of the unit allows the removal of the unwanted upper or lower mixing sideband and improves the performance of the lock unit considerably. Obviously a fixed highpass or bandpass element could be used but will then limit the lock accessory to a single frequency. In its present form, the lock unit can be used to provide a spectrometer lock for any nucleus having sufficient sensitivity. The frequency of the synthesizer is set to produce the desired

Dr. Shapiro November 14, 1980

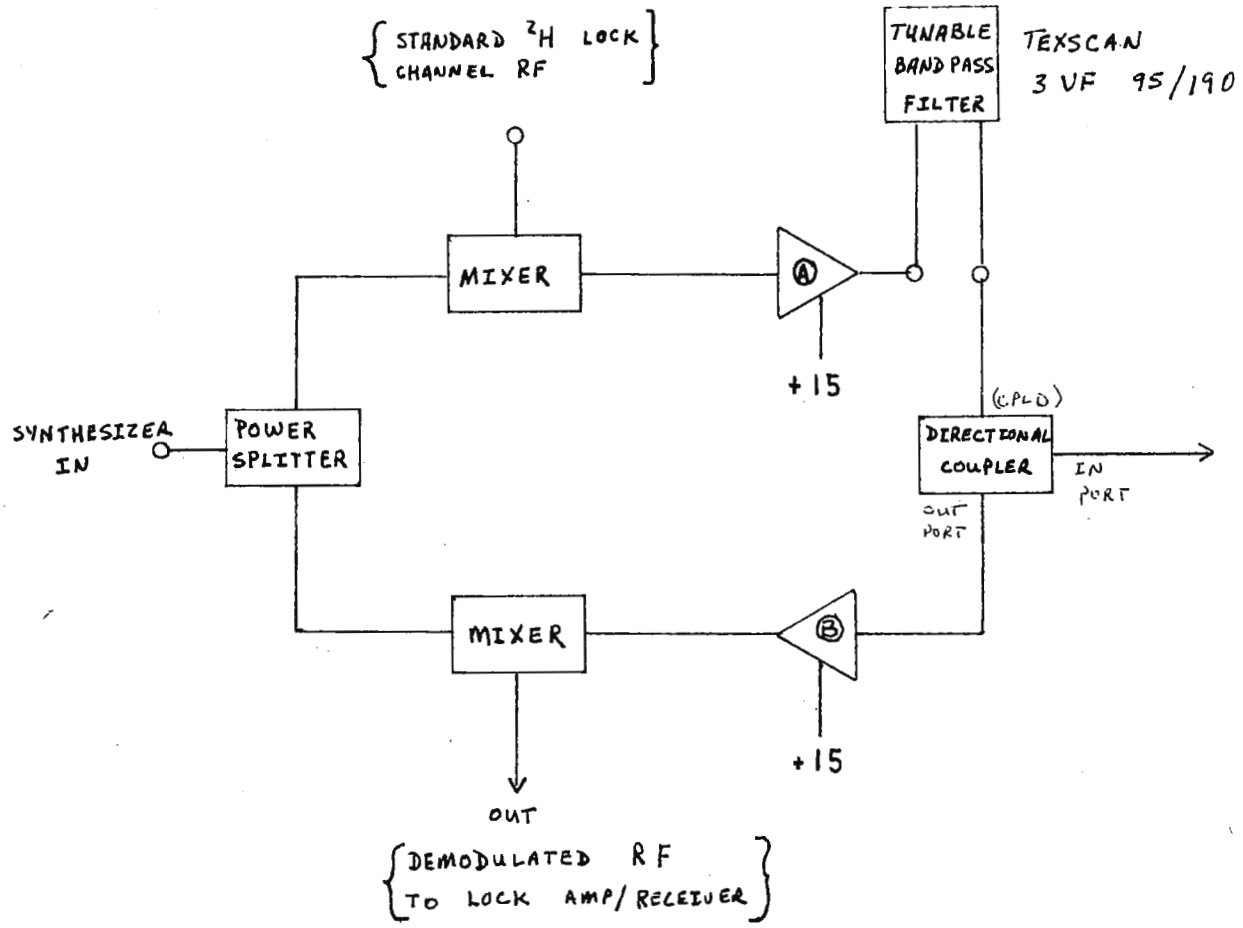
lock frequency upon mixing with the fixed deuterium lock frequency of the spectrometer. The synthesizer should be synchronized to the spectrometer's main LO clock for the best stability.

Any readers wanting further details can contact me.

Sincerely

Ray

Ray L. Nunnally



Ⓐ = Avantek GPD 402

Ⓑ = Avantek GPD 401

Mini-Circuit Labs:
- mixer: SRA-1
- power splitter: PSC-2
- directional coupler : PDC-10-1

FIGURE 1

1880 Kodak 1980

A 100-year start on tomorrow

November 11, 1980

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

Dear Professor Shapiro:

Re: An Eclectic Wide Bore 200 MHz Spectrometer

We have recently completed the construction of a wide-bore 200 MHz spectrometer with multinuclear and solid state capability. The system is built around an Oxford Instruments 98 mm magnet. We are using a Nicolet 1180 data system including a 293B pulse programmer and r.f. electronics of our own design. Shim coils and multinuclear probes were furnished by Dr. Craig Bradley of Cryomagnet Systems. The probes cover octave ranges i.e., 5-10 MHz, 10-20 MHz, 20-40 MHz and 40-80 MHz. The 40-80 MHz probe can accommodate 20 mm tubes, the lower frequency probes can take 25 mm tubes. We have looked at 25 different nuclides to date. Sample spectra for the 5-10 MHz probe representative of the two ends of its range are shown in Fig. 1.

The solid state ^{13}C probe was supplied by Dr. Vic Bartuska of Chemagnetics, Inc. A sample spectrum of solid L-alanine (avg. of 100 FID's; spin rate >4KHz) is shown in Fig. 2. The ^{14}N induced splitting of the methine carbon is less than the previously observed value at lower field as predicted by theory. Surprisingly, adequate decoupling was achieved using ~100 W of proton r.f. power.

Please credit this contribution towards the subscription of the Eastman Kodak Research Laboratories.

Sincerely,

N. Zumbulyadis

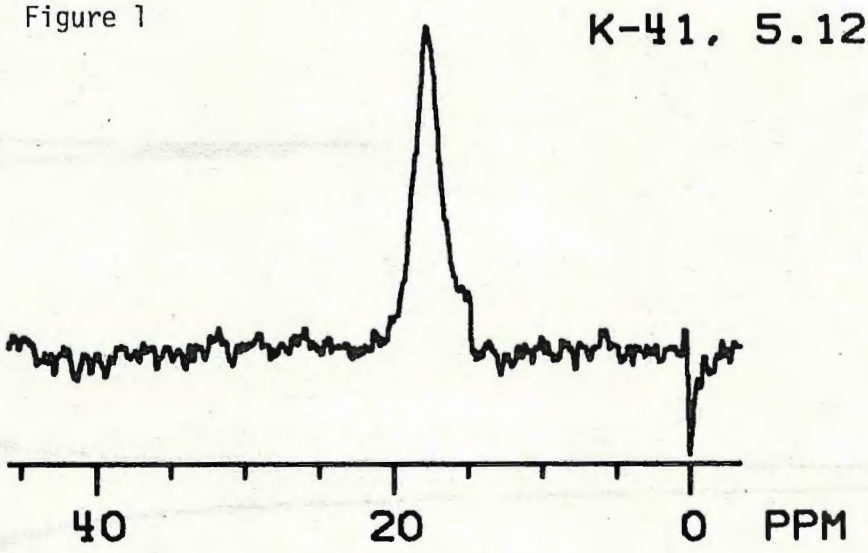
NZ:SG:eca

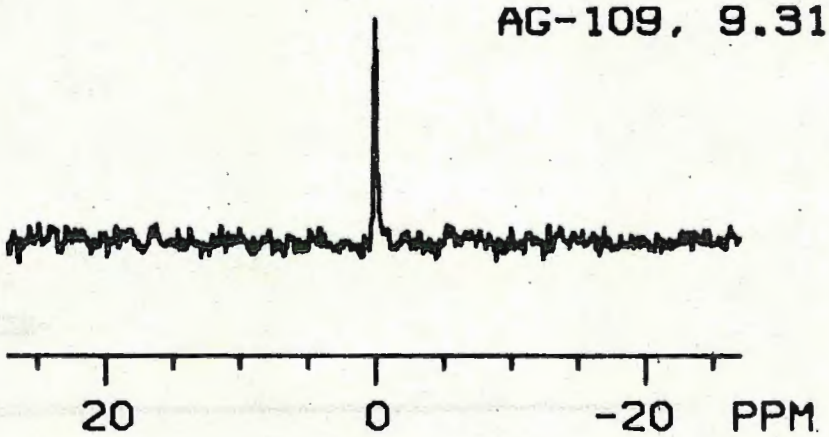
Nicholas Zumbulyadis
Research Laboratories

S. Gross

Stan Gross
Research Laboratories

Figure 1

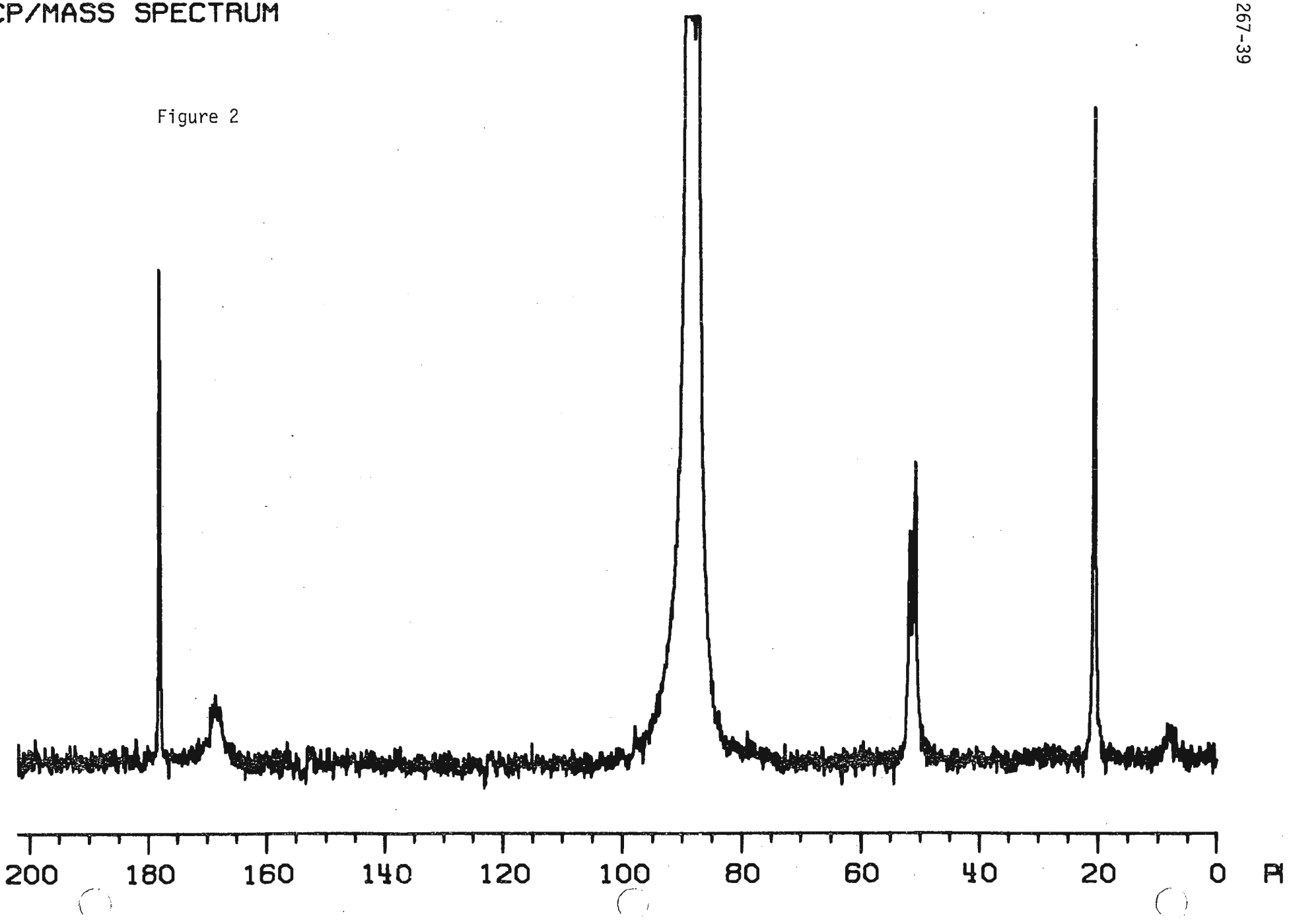




ALANINE
SOLID STATE C-13 NMR
CP/MASS SPECTRUM

267-39

Figure 2



OSU

The Ohio State University

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

Campus Chemical Instrument Center
Alan G. Marshall, Director
(614)-422-3446
26 November, 1980

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

BIOPHYSICAL POSTDOCTORAL POSITIONS

Dear Barry,

Additional postdoctoral positions will be available in my laboratory, beginning in January, 1981. Salary will be $14,000 per annum, plus benefits. The successful candidate(s) will be expected to participate in at least one of the following areas.

(A) Solution structure of ribosomal RNA. See BBRC 96, 805 (1980), and two papers appearing shortly in Biochemistry.

(B) Dispersion versus Absorption (DISPA). See J. Phys. Chem. 84, 1372 (1980) and references therein.

(C) Indium porphyrins. See JACS 102, 1460 (1980).

(D) Fourier transform mass spectroscopy. See J. Chem. Phys. 73, 1581 (1980), and references therein.

Interested candidates should send a curriculum vitae and three recommendation letters as soon as possible.

Sincerely,

Alan G. Marshall
Professor of Chemistry and Biochemistry

AGM:agm

267-41

UNL

The University of Nebraska–Lincoln

Department of Chemistry
Lincoln, NE 68588
402-472-3501

November 19, 1980

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

RE: Conversion of ^{31}P CW mode to FT mode in a XL-100

Dear Dr. Shapiro:

Our XL-100 was originally only a CW instrument which later on was modified by Varian for FT operation in ^{13}C and ^{1}H. Since I am planning a series of ^{31}P experiments in Enzyme-Nucleotide interactions in collaboration with Dr. S.M. Schuster, a biochemist in our department here, I found it necessary to have FT capability in our XL-100. This conversion could easily be accomplished at a very nominal cost making use of the FT logic circuits already available in the system.

Step 1:

The crystal in the transmitter is replaced with one operating at 10•12 MHZ instead at 10•13 MHZ and oscillator is retuned.

Step 2:

A gate is interposed between the Power amplifier and the oscillator in the transmitter brick.

Step 3:

In the ^{31}P nuclear local oscillator, another gate is inserted between the oscillator section and X-4 multiplier; this gate is used in FT mode to turn off the local oscillator 10 μ sec before until 50 μ sec after the transmitted pulse.

Step 4:

One more gate is incorporated in the pre amplifier (Refer schematics 87-126-779 and 87-109-830) and this shunt gate is closed by the FT logic circuits shorting out the input from 10 μ sec before 50 μ sec after the transmission of the FT pulse.

Step 5:

In order to amplify the gated rf from the transmitter, a final rf amplifier operating around 40.5 MHZ is needed; following the design of ^{13}C rf amplifier, this board may be duplicated with few changes in the components and retuned; we bought this board from Varian at a discount price with no guarantee on it; in fact, for trial purposes ^{13}C amplifier itself may be used retuning the capacitors.

This completes the modification procedure. I have enclosed the sample spectrum obtained in the FT mode.

Anyone interested in the exact details may write to me. I hope this note will help keep the newsletter coming.

Sincerely,

S. Rajan

ATP IN ACETATE BUFFER (50 mM); pH 4.0; LOCK: D_2O

```
>PA
SPC WDT (HZ)=  2000
TRU SPC WDT=   2048
ACQ  TIME (S)=    2.000
PULS DLY (S) =    1.000
PULS WD (US) =   50
TRANSIENTS   =  500
DATA LENGTH =  8192
SEN ENH (S) =     0.80
F T  LENGTH =  8192
HF  IN

     PLOT
 VERT SCALE =    20
 START (HZ) =  2000.0
 FINISH (HZ)=     0.0
 HZ. / POINT=     0.50
 HORIZ. EXP =     1.0
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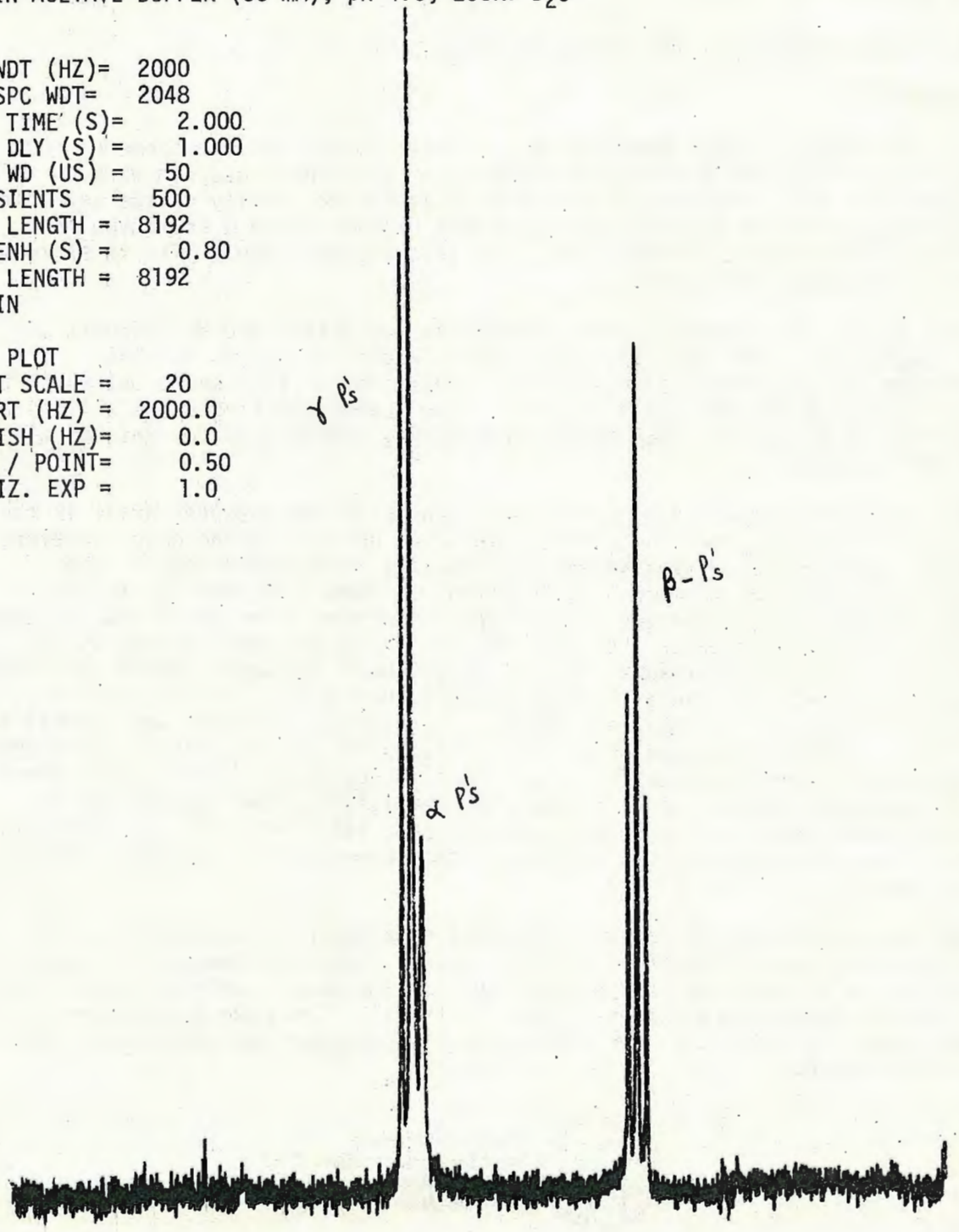


PURDUE UNIVERSITY BIOCHEMICAL MAGNETIC RESONANCE LABORATORY

November 21, 1980

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

Early Observations on NMR Operation at 470 MHz

Dear Barry:

We have recently taken delivery of a Nicolet NT-470 NMR spectrometer and are beginning to use it in PUBMRL operations for practical research work. There have been many inquiries directed to us as to the utility of the solenoid at this field since the Oxford supplied magnet appears to be a prototype for future magnets at fields of 500 + MHz. For this reason I would like to share our experience with the system.

This magnet is externally quite similar to the Oxford 360 MHz magnets of recent manufacture. The main field current is approximately 65 A. This necessitates the use of non-standard power supply for initial energization (the standard Oxford MK III goes only to 60 A). The superconducting shims all operate at less than 10 A, so the usual Oxford shim supply can be used for this portion of the energization.

Our solenoid appears to be quite persistent with the observed drift in the range of 1 to 2 Hz per hour (unlocked). This was not always the case, however. On initial energization of the solenoid by Nicolet in Mountainview, CA the observed drift rate was in excess of 40 Hz/hour. This drift was sufficient to cause induced current changes in the superconducting shims which unacceptably degraded lineshape over about a 48 hour period. A second energization by NTC reduced the drift to less than 20 Hz/hour after about two weeks, which was still unacceptable to us. On site at Purdue, we were very careful to overcycle the magnet by at least 0.5% of the main field. The overcycle current was maintained for 8 hours. Under these conditions the initial drift was found to be less than 15 Hz/hour, and the drift decayed to near persistence over about 3 days. After 60 days the magnet appears to be essentially persistent. The degradation in lineshape noted above is also absent. We can only attribute this experience to the well known "training effect" which has been observed over the years in high field solenoids.

Our operational resolution and lineshape data are now comparable to what we have observed on a narrow bore 360 MHz system. This performance has been achieved with a 17 shim room temperature system (the usual configuration for an Oxford 360 MHz narrow bore system is only 11 shims). The room temperature coil assembly lacks a 5 shim coil. We will seriously consider the addition of this coil in the future.

Chemistry Building
West Lafayette, Indiana 47907
(317) 749-2438
(317) 749-2439

Professor B.L. Shapiro November 20, 1980

We paid particular attention to cleanliness during assembly of the cryostat and used an all steel pumping system for evacuation of the dewar. As a result we appear to have a hold time in excess of 90 days between helium refills (30 L of helium). The initial cool down of the solenoid did require about 100 L of helium. We attribute this to the large thermal mass of the solenoid compared to lower field types.

I will reserve comments on the electrical performance of the console as it was delivered by Nicolet in an inoperable state. We are presently trying to correct most of the console problems.

In summary, the Oxford 470 solenoid appears to be a practical magnet for routine use when it is properly energized. To the extent that the solenoid which I have described is representative of present magnet technology, I would anticipate that solenoids of 500 + MHz field will exhibit similar characteristics.

Please credit this contribution to John Grutzner's subscription.

Yours,

R E Santini

R. E. Santini
Acting Director

JEOL (U.S.A.), INC. • ANALYTICAL INSTRUMENTS DIVISION
235 BIRCHWOOD AVENUE • CRANFORD, NEW JERSEY 07016
TELEX NO. 13-8840 • (201) 272-8820

November 26, 1980

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

HMB Analog for ^{29}Si MAS

Dear Barry:

Since there has been great interest in other nuclei for CP/MAS, we have started to look at ^{29}Si. Besides the regular organo-silicon compounds and the silicon polymers, there exists a whole other area which has been untouched by conventional NMR. This area includes minerals,[1] different glasses (silicon oxides), zeolites and solids labelled with a trimethylsilating reagents. Unfortunately, all of the materials we have looked at have the silicon in a tetrahedral environment and, hence, the anisotropy is quite small. This is convenient since adjustment of the Magic Angle is, therefore, not very critical. On the other hand, precise measurement with a standard is not easy. We have found that Hexamethylcyclotrisiloxane (Petrarch and Alpha catalogs) is very sensitive since the six-membered ring is apparently planar and the silicons are equivalent, at least on an NMR time scale.

In addition, this standard is very desirable since Hexamethylcyclotrisiloxane can be abbreviated HMCTS or even HMC. In this fashion, all NMR standards can remain three letters with those for liquids ending in "S" and those for solids beginning with "H" (i.e. TMS, DSS, HMB and now HMC). The only minor infraction that we have discovered is that DSS also provides a very good chemical shift reference for solid MAS ^{29}Si.

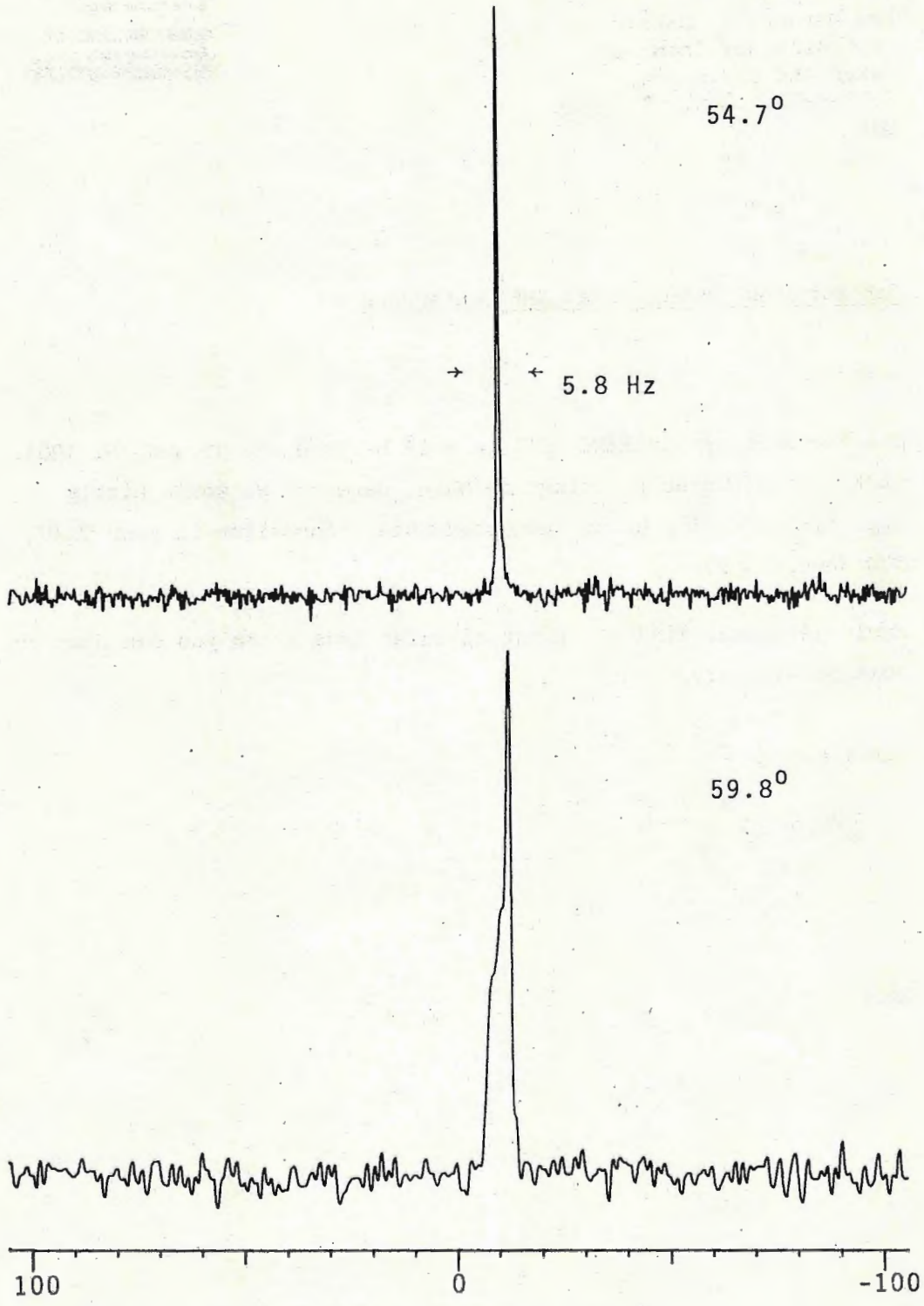
Sincerely,

Michael J. Albright

MJA/mjd

1. E. Lippmaa, M. Magi, A. Samosan, G. Englehardt, and A.-R Grimmer, J. Am. Chem. Soc., 102, 4889 (1980).

FX 60Q
SOLIDS DATA



^{29}Si MAS of HMCTS (or HMC) Relative to DSS

267-47

IWAN N. STRANSKI-INSTITUT
für Physikalische und Theoretische Chemie
der Technischen Universität Berlin
Prof. Dr. Dieter Ziessow

Berlin, den November 10, 1980
Tel.: (030) 314-4958
Az.:

1 Berlin 12
Straße des 17. Juni 112
Ernst-Reuter-Haus
Ernst-Reuter-Platz 7

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

5th European Experimental NMR Conference

Gentlemen:

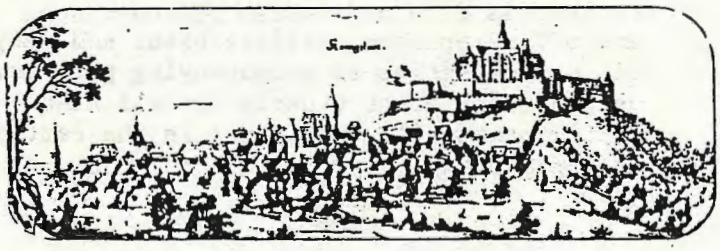
The forthcoming 5th EENC will be held between May 12 and 15, 1981, in Königstein near Frankfurt am Main, Germany. We would highly appreciate it if you had published this information in your TAMU NMR Newsletter.

/ Enclosed please find our first circular from which you can observe some particulars.

Sincerely yours,

Dieter Ziessow

/ Encl.



5th EENC

5TH EUROPEAN EXPERIMENTAL

N.M.R. CONFERENCE

KÖNIGSTEIN/FRANKFURT

MAY 12-15, 1981

FIRST CIRCULAR

DATE AND LOCATION

The 5th EENC will be held in the HAUS DER BEGEGNUNG in Königstein which is situated 20 km north of Frankfurt am Main.

SCOPE OF THE CONFERENCE

This 5th conference follows previous meetings in Canterbury(1974), Enschede (1975), Elsinore(1977), and Autrans(1979).

The general scope of this conference comprises the experimental and instrumental aspects of NMR spectroscopy. The topics will include:

- New Developments in High Resolution NMR for Liquids and Liquid Crystals
- 2D NMR Methods
- High Field Instrumentation
- X Nuclei NMR
- Relaxation
- NMR in Solids and Polymers
- Spin Imaging
- Techniques in Biochemical or Biomedical Applications

Papers on NMR applications will in general not be accepted and the scientists concerned are advised to present these results at the "5th International Meeting on NMR" in Exeter, July 12-17, 1981.

CONFERENCE PROGRAM

The scientific program comprises invited lectures and poster sessions. The contributed papers will primarily be presented as posters and to a small extent as invited short lectures. The selection will be made after the abstract deadline date. The conference language will be English.

The conference begins with a registration mixer on Tuesday evening, 7.oo-10.oo p.m. Lectures start on Wednesday morning and finish Friday noon.

An exhibition of NMR instruments and books will be held during the whole period of the conference.

Lectures, posters and the exhibition take place in the main lecture hall building of the HAUS DER BEGEGNUNG.

ACCOMODATION

The HAUS DER BEGEGNUNG provides accomodation with full board at rates between DM 48 and 55 - depending on the chosen room. The facilities include 15 single rooms with shower, 107 single rooms, 18 two-bed rooms and 11 three-bed rooms without shower (total capacity 191 persons). Further rooms are available in boarding-houses and hotels close to the HAUS DER BEGEGNUNG. The rates for rooms and breakfast vary from DM 20 - 40, to which app. DM 20 must be added for lunch and dinner at the HAUS DER BEGEGNUNG. Participants are asked to indicate a preference for either accomodation on the enclosed pre-registration form (HdB or off-HdB).

SOCIAL PROGRAM

Königstein is a well-known health-resort and offers springs, medical baths and many social activities to accompanying persons. On Wednesday night a party for all EENC participants will take place in the castle of Königstein.

INTERNATIONAL ORGANIZING COMMITTEE

M.J.A. de Bie	Utrecht
P. Diehl	Basel
R. R. Ernst	Zürich
S. Forsen	Lund
R. Freeman	Oxford
A. Loewenstein	Haifa
K. Schaumburg	Copenhagen
P. Servoz-Gavin	Grenoble
D. Ziessow	Berlin

CONFERENCE SECRETARY

5th EENC
H. Caspari
Iwan N. Stranski-Institut der
Technischen Universität Berlin
Straße des 17. Juni 112
D-1000 Berlin 12 (West)
Tel.: (030)3143932

SECOND CIRCULAR

The second circular with the scientific program and abstract deadline will be distributed at the end of 1980. Those persons who have returned the pre-registration form will automatically be supplied.

NT-Series Fourier Transform Superconductive Magnet NMR Spectrometers

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

Outstanding NT-Series features include these:

- **A full range of superconductive magnets from 3.5T to 11.7T in both wide-bore and narrow-bore configurations.**
- **Multinuclear observation with a wide variety of fixed-tune and broadband probes.**
- **Simultaneous acquisition, processing, and plotting for greater sample throughput.**

- **Simplified control of spectrometer operations and parameters by using easy keyboard commands.**
- **Advanced Nicolet-1180 Data System with the most comprehensive FT-NMR software package available.**
- **Extended dynamic range performance with 40-bit acquisition and floating-point processing.**
- **An expandable pulse-sequence library, including T_1, T_2, Redfield, 2D-FT, etc.**
- **Convenient computer-control of field shimming, observe and decoupling frequencies, sample temperature.**
- **Precise digital plotting with full annotation of spectral parameters and flexibility of hard-copy format.**

The multiple-technique NT-Series spectrometers provide the user with the ability to easily adapt to the newest techniques and experimental configurations.

Some of these are:

- **High-resolution studies of solids with Waugh-Pines cross-polarization and magic-angle spinning**
- **High-sensitivity wide-bore ^{13}C studies of high molecular weight polymers.**
- **Automated T_1 and T_2 measurements.**
- **Chemical dynamics studies.**
- **Temperature-programmed experiments.**
- **^{31}P experiments on living organs.**

NICOLET TECHNOLOGY CORPORATION
145 East Dana Street
Mountain View, California 94041
Telephone: 415/969-2076

FX SERIES
OF
FT NMR SYSTEMS

FX Features

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- Bilevel Software Package
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- FX Series Work Station
- Digital Quadrature Detection
- Oxford SCM Systems
- Programmable Variable Temperature
- Double Precision (32 bit word length)
- Floppy; Moving Head Disc Systems
- Programmable Multi-Pulser: INEPT, Selective Excitation, Cross Polarization, Bilevel Decoupling, etc.

FX-60QS:

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

FX-270:

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

FX-90Q:

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

FX-200:

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

JEOL

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