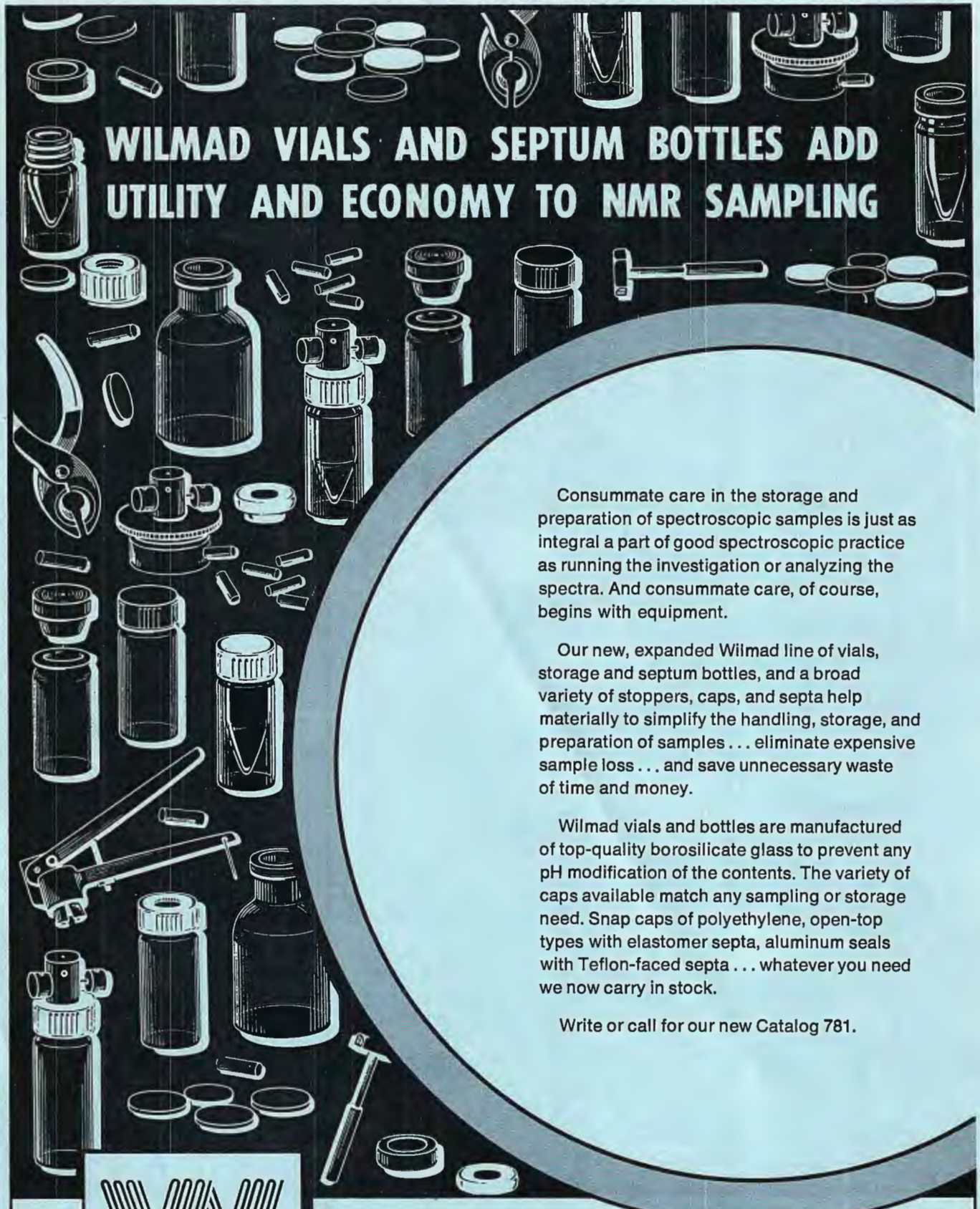
Texas A&M University N-M-R Newsletter

No. 268

January, 1981



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

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

AUTHOR INDEX - TAMU NMR NEWSLETTER NO. 268

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School of Studies in Chemistry

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

19th November, 1980.

Dear Professor Shapiro,

TEMPERATURE AND LSR EFFECTS ON ^{1}H SHIFTS IN DIBENZOCYCLOHEPTATRIENES

We have referred previously to ^{1}H solvent shifts (TAMUNMR 217/17) and J errors (TAMUNMR 237/19) in dibenz[b,f]oxepin(I)[1] and its nitrogen analogue. Dr. J.A.G. Drake's LAOCOON III analyses of the 220 MHz ^{1}H spectra of the related compounds 5H-dibenzo[a,d]cyclohepten-5-one (or dibenzo[b,f]tropone)(II) and 5H-dibenzo[a,d]cyclohepten-5-ol(III) show that the chemical shifts have linear solvent and temperature dependences over the ranges studied (Table). At low concentrations of III in chloroform-d_1 solution, two peaks emerge for each of the H(5) and OH(a and b), possibly indicative of the presence of a protonated species (slow decomposition occurs). The predominantly negative θ (Table) may be due, as suggested for I[1], to the predominance of (reaction-field) deshielding, arising from changes in dielectric constant of the solvent, over the increased shielding to be expected, as temperature is decreased, from closer association between solute molecules (analogous to the effect of increased concentration). Addition of Eu(fod)$_3$ to II in chloroform-d_1 induces ^{1}H paramagnetic shifts, H(4)>>>H(11)>>H(1)>>H(3)~H(2), directly proportional to the metal/substrate ratio over the range studied and consistent both with the ^{1}H chemical-shift assignment and with complexing of the reagent metal ion to the carbonyl oxygen in II. H(4) in II is distinctive in having the largest LIS shift ratio and also in having a positive θ coefficient.

Incidentally, measurements on fluoren-9-one[2], briefly reported in TAMNUNMR 256/11, have been extended to 1-methylfluorene and truxene[3].

Yours sincerely,

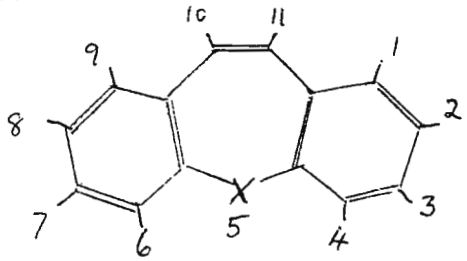
Denry Jones

D. W. Jones

1. J.A.G. Drake and D.W. Jones in B.Pullman (ed) "Nuclear Magnetic Resonance Spectroscopy in Molecular Biology", Reidel, 1978, p.493
2. J.A.G. Drake and D.W. Jones, *Spectrochim. Acta, 36A*, 23 (1980).
3. J.A.G. Drake and D.W. Jones, *Org. Magn. Resonance, 14*, 272 (1980).

Table ^1H shifts extrapolated to 273 K (δ, ppm) and temperature shifts (θ, 10^3 ppm K^{-1}) from 220 MHz spectra of (i) II in 0.50 mol dm^{-3} $CDCl_3$ (224-322 K); (ii) II in 0.08 mol dm^{-3} CS_2 (247-294 K); and (iii) III in 0.13 mol dm^{-3} $CDCl_3$ (269-320 K).

Proton	(i) II in $CDCl_3$		(ii) II in CS_2		(iii) III in $CDCl_3$	
	δ	θ	δ	θ	δ	θ
H(1)	7.482	-0.38	7.420	-0.12	7.368	-0.66
H(2)	7.585	-0.59	7.530	-0.26	7.285	-0.69
H(3)	7.512	-0.57	7.440	-0.18	7.413	-0.62
H(4)	8.196	0.21	8.060	0.89	7.654	-0.26
H(5)	-	-	-	-	5.384	+0.17
OHa	-	-	-	-	2.541	-3.21
b	-	-	-	-	2.521	-3.18
H(11)	6.994	-0.34	6.945	-0.14	7.104	-0.53
$CHCl_3$	7.245	-0.30	-	-	7.246	-0.42



I X = O
II X = C=O
III X = CHOH

268-3

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TEL. : (91) 62.15.54

Professeur P. J. COZZONE

Marseille, le November 24, 1980

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

Dear Professor Shapiro :

We are currently involved in a large project on COLIPASE, a small protein (93 residues) which, as a cofactor of pancreatic lipase, plays a key role in the intraduodenal lipolysis of dietary triglycerides. Besides its biological importance, the lipase/colipase/substrate interface system constitutes an attractive model to study protein-protein and protein-lipid interactions. We have recently identified a particular hydrophobic aromatic domain on colipase as being the Lipid Binding Site (P. Canioni and P. Cozzone, Biochimie 1979, 61, 343-354 and FEBS Lett. 1979, 97, 353-357 - P. Canioni, P. Cozzone and L. Sarda, Biochim. Biophys. Acta 1980, 621, 29-42). Two tyrosine residues located in the segment 50-57 of this domain are involved in the binding process and give a strong photo-CIDNP effect in presence of a lumiflavin dye (P. Canioni, P. Cozzone and R. Kaptein, FEBS Lett. 1980, 11, 219-221). In collaboration with Paul Canioni and Louis Sarda in this laboratory and Robert Kaptein at the University of Groningen we have conducted in Groningen a series of laser photo-CIDNP experiments at 360 MHz on several colipase-micelle complexes to document the influence of charge effects on the association.

In presence of sodium taurodeoxycholate (TDC) micelles, the CIDNP effect on both Tyr residues is totally suppressed at neutral pH (Fig. 1 and 2a). Under these conditions, the 2 aromatic surface residues are no longer accessible to the dye due to the protection by the TDC-micelle. Electrostatic repulsions between phenolate and TDC negative charges at high pH induce the disruption of the complex which is accompanied by a gradual restoration of the polarization of the 2 tyrosines (Fig. 2).

We have subsequently interacted colipase with chenocholamine, a detergent which bears a positive charge up to around pH 11. As expected, the association remains tight even at pH 11.2 as shown by the almost complete absence of CIDNP effect on the tyrosines at all pH values (Fig. 3). In both cases, the emission line of the C(2)-H proton of His I (His 30) remains present in the complex, illustrating the fact that this residue does not belong to the Lipid Binding Site.

We think that this series of straight forward experiments illustrate very well the potentials of the laser photo-CIDNP technique to study binding processes.

Sincerely yours,

Patrick J. Cozzone

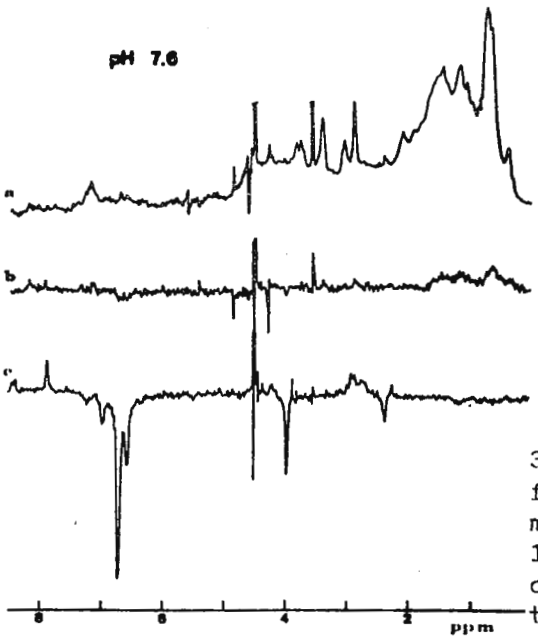
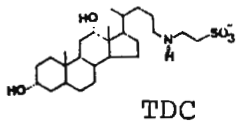


Fig. 1

360 MHz proton NMR spectrum and photo-CIDNP difference spectrum of porcine colipase A. Experiments were performed at 35°C in 12 mM TDC with 150 mM NaCl and 0.4 mM flavin at pH 7.65. Concentration of colipase : 1.5 mM (a) dark spectrum, (b) photo-CIDNP difference spectrum. Light and dark FID's were taken alternately (20 scans); (c) photo-CIDNP difference spectrum of colipase in the absence of TDC (pH 7.2).



TDC

Fig. 2

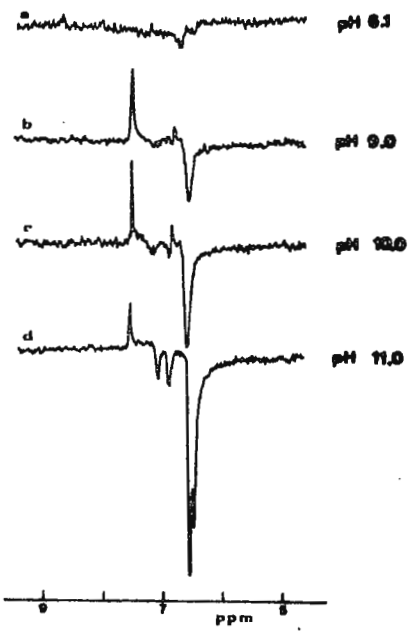
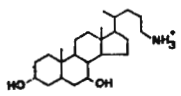
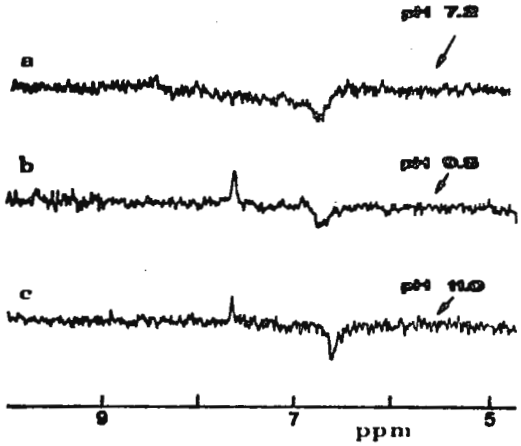


Fig. 3



chenocholamine



268-5 - Dr. Bremser c/o -

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Professor B.L. Shapiro
Department of Chemistry
Texas A & M University

College Station, TX 77843
U S A

26.11.1980 WBr-V
WHE - B 9
Tel. 0621/60-8401

CNMR-Interpretator

Dear Barry,

threatened by your green reminder I am forced to reveal a first glimpse of our new baby, a program for "interpretation of CNMR spectra".
In contrast to conventional search programs scanning large data bases for similar or identical reference spectra, this program relies solely on a description of the expectation ranges of chemical shifts in characteristic substructures. On the other hand only large structure linked data collections allow a realistic estimate on the influence of structural changes on spectral features and thereby the prediction of possible substructural units. The enclosed example shows a similarity search (SAHO) and an interpretation for the spectrum of 3'-methylacetophenone. The suggested structural units leave only one solution, namely the correct answer.

That's all for this year. Best regards,

Wolfgang

Enclosure

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   3.       137.3 PPM       S                1
   4.       133.8 PPM       D                1
   5.       128.8 PPM       D                1
   6.       128.5 PPM       D                1
   7.       125.6 PPM       D                1
   8.        26.5 PPM       Q                1
   9.        21.2 PPM       Q                1

EINGABEOPTION ? (N;K;+;-;E)
=E

SUCHLAUF STARTEN ?
=J


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    S P E K T R E N  -  S U C H E
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MIT  30757 SPEKTREN


ERGEBNIS:  10 SPEKTREN

SPEKTRUM      N A M E

CNMR5764      4'-METHYL-ACETOPHENONE
CNMR6327      3'-METHYL-ACETOPHENONE
CNMR7344      2'-METHYL-ACETOPHENONE
    ZUSAETZLICH   7 TOECHTER



INTERPRETATION ?
=J

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   SPEKTRENINTERPRETATION
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 4.168   ( 13)   -   ASYM. DISUBST. BENZENE
 1.821   ( 54)   -   ACETYL (AROM) OHNE ORTHO-SUBST.
 1.625   (  7)   -   METHYL (AROM) OHNE ORTHO-SUBST.

 KEINE WEITEREN LINIEN


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Professor Oleg Jardetzky

(415) 497-6153
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December 1, 1980

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

Dear ~~Professor Shapiro~~ Barry:

I would like to announce the 6th AMPERE Summer School on Biological Applications of NMR to be held September 8-18, 1981, in Schloss Seggau bei Leibnitz, Steiermark, Austria. The school will consist of lectures, group discussions and problem sessions and is intended both for spectroscopists and for biochemists. Topics to be covered include: Basic principles of NMR spectroscopy; New techniques: 2DFT, relaxation analysis, photo-CIDNP; NMR of macromolecules--methodology for resolution and assignment of signals; Proteins: structure, dynamics and interactions with small molecules; Nucleic acids: structure, dynamics and interactions with small molecules; NMR studies of enzyme mechanisms; Membranes: molecular motion, protein-lipid interactions; NMR of whole cells and organs: ^{13}C and ^{31}P studies of metabolism and metabolic regulation, NMR imaging in vivo.

Following is a tentative list of the faculty: E.R. Andrew (Nottingham), I.D. Campbell (Oxford), P. Cozzone (Marseilles), R.A. Dwek (Oxford), R.R. Ernst (Zurich), K. Hausser (Heidelberg), R. Kaptein (Groningen), M.P. Klein (Berkeley), P.C. Lauterbur (Stony Brook), J.L. Markley (Purdue), E. Oldfield (Urbana), D. Patel (Murray Hill), W.D. Phillips (St. Louis), J.H. Prestegard (Yale), G.K. Radda (Oxford), B.R. Reid (Seattle), J. Seelig (Basel), R.G. Shulman (Yale), I.C.P. Smith (Ottawa), A. Xavier (Lisboa) and members of the Organizing Committee.

Members of the International Organizing Committee are O. Jardetzky (Stanford, USA) Chairman, V. Bystrov (Moscow, USSR), S. Forsen (Lund, Sweden), G.C.K. Roberts (London, UK), K. Wüthrich (Zurich, Switzerland).

Attendance is limited to 120 participants and the cost is 3000 Austrian Schillings, including room and board. Applications should include a brief curriculum vitae and a letter of recommendation from a university faculty member. DEADLINE FOR APPLICATION is April 1, 1981. Applicants will be notified of the decision on their application by May 1, 1981. Letters of application and all correspondence should be addressed to: Dr. G.C.K. Roberts, National Institute for Medical Research, The Ridgeway, Mill Hill, London NW7 1AA, England. Telegrams: Natinmed London NW7. Telex: 922666 (Natinmed London). Telephone: 01-959 3666.

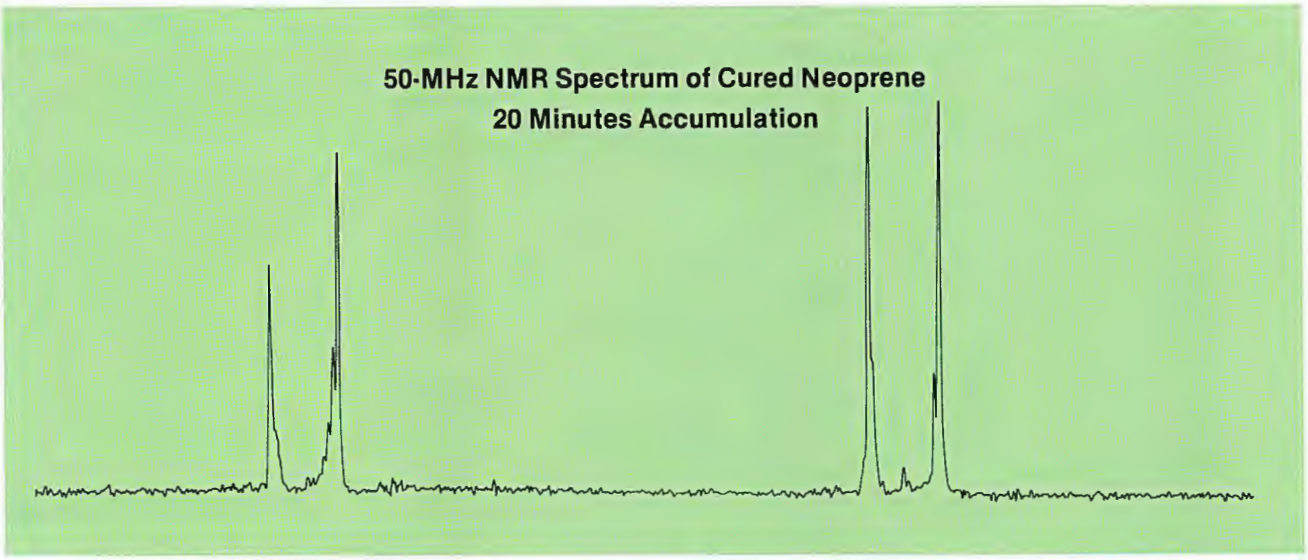
Yours sincerely,

Oleg Jardetzky

Oleg Jardetzky

If you can't observe solids as readily as liquids on your superconducting FT NMR...

...you just don't have an XL-200!



^{13}C spectrum of cured neoprene with carbon black in a Kel-F rotor using high-power gated decoupling (400 transients at 3-second intervals). The resolution has been enhanced by a Lorentzian-to-Gaussian transformation to bring out the fine structure. The width of the plot is 10 KHz. *Sample courtesy of E. I. Du Pont de Nemours and Company*

With the new ^{13}C solid-state accessory for the XL-200, you can spin solid or powdered samples at the magic angle, increase sensitivity using cross-polarization, and achieve efficient line narrowing with strong dipolar decoupling. Yet operation is surprisingly simple! You can introduce and eject the rotor pneumatically without disturbing the probe or the spinning axis adjustment. You monitor the spin rate on the spectrometer's built-in tachometer, just as in liquid-sample experiments. Front panel controls let you adjust optimal cross-polarization and decoupling conditions independently and conveniently.

There are other unique aspects to the XL-200 superconducting FT NMR Spectrometer, such as the data handling and spectrometer control system: a 13-bit ADC, which accommodates stronger signals on each transient; a standard 32K CPU, independent of the acquisition processor and programmed in PASCAL, a high-level, structured language; a built-in interactive 5M-word disk with dual platters; a large, flicker-free raster scan display.

The software, too, is exceptionally sophisticated. It permits multitasking (simultaneous acquisition, processing, printing, etc.) and queuing (automatic sequential execution of requested tasks) on the same or on different NMR experiments. You can also array parameters (up to three variables, including temperature) within a given experiment; generate your own convenient macro-commands; create your own special or general-purpose pulse sequences in a simple, English-like code; even do your own computer programming in PASCAL.

Then there's the matter of the XL-200's broadband accessory which, with only a single probe for liquid samples, enables you to observe a host of nuclei (including ^{13}C) between 20 and 81 MHz. And there's the remarkable low-loss dewar system, which operates over three months on only 25 liters of liquid helium.

The XL-200 is in a class by itself—with a price tag and an operating economy that belie its advanced design.

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Ref :

Date : December 12, 1980

Prof. C. L. Khetrapal
Dr. A. C. Kunwar

Raman Research Institute
BANGALORE 560 080

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

Title: Molecular order in Liquid Crystals with opposite diamagnetic anisotropies

Dear Prof. Shapiro,

We just received your "dangerous Yellow weapon!" Before it really 'hurts' us, we submit the following contribution.

Recently, we have studied the variation of the orientation of molecules dissolved in liquid crystals with positive and negative diamagnetic anisotropies and in the mixture of the two types of the solvents, in connection with our search for finding out convenient ways and means for changing the molecular order at 'will'. We studied the proton NMR spectra of benzene oriented in the nematic phases of Merck ZLI-1167 and N-(p-ethoxybenzylidene)-p-n-butylaniline (EBBA) and in the mixtures of the two. The PMR experiments were conducted in 3 weight per cent solutions of benzene in ZLI-1167 (solution I) and EBBA (solution II) separately. Then known amounts of solution (II) were gradually added to solution (I) and the spectra were recorded at each concentration. At a critical concentration, we found that the dipolar couplings change to twice their values and have opposite signs. The results can be interpreted in terms of the change of the angle between the directions of the optic axis of the liquid crystal and the applied magnetic field from 90° to 0°. Above the critical concentration, the molecules orient like in solution (II), i.e., with the optic axis along the direction of the magnetic field. Below the critical concentration, the preferred orientation is like that in solution (I). Further studies and possible applications of this observation are in progress.

With regards,

Yours sincerely,

C.L.Khetrapal A.C.Kunwar

C.L.Khetrapal A.C.Kunwar

268-11

National Chemical Research Laboratory

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1 DEC 1986

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

Dear Professor Shapiro

CONFIGURATIONS DETERMINED FROM THE SIGNS OF J(HH) AND J(CH)

Photodimerisation of 2-chloro-1,4-naphthoquinone can lead to four possible symmetrical dimers (1-4). Dimers (3) and (1) were isolated after sunlight irradiation of an acetic anhydride solution and crystalline 2-chloro-1,4-naphthoquinone, respectively, whereas dimer (4) was synthesized from 1,2-phthaloyl-2a-chloro-2a,3,8,8a-tetrahydro-3,8-dioxonaphtho[b]cyclobutadiene[1]. The structures of these dimers were elucidated from extensive ^{1}H and ^{13}C n.m.r. studies. In determining the configuration of the cyclobutane rings a knowledge of the signs of the (H,H) and (C,H) couplings in these moieties have been especially useful.

The two protons (A and M) and a ^{13}C methine carbon atom (X) of the cyclobutane ring of the dimers formed an AMX spin system. The relative signs of the (H,H) [J(AM)], directly-bonded (C,H) [^{1}J(AX)] and over more than one bond (C,H) [$^{>1}J$(MX)] coupling constants were determined with ^{13}C-{^{1}H} SPI experiments[2]. The magnitude of the (H,H) coupling constants were obtained from ^{13}C-satellite ^{1}H n.m.r. spectra. The experimental values for the three dimers are given in the Table.

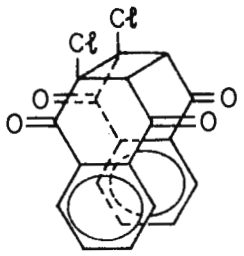
TABLE (C,H) and (H,H) coupling constants

Dimer:	1	2†	3	4
^{1}J(CH)*	151.2		148.6	148.6
$^{>1}J$(CH)	-6.3	+	-4.9	+2.8
J(HH)	+11.2	+	+4.0	-1.0

* Assumed positive. † Expected signs

A cross-ring ^{4}J(HH) in cyclobutane is small and positive when the interacting protons are *cis* and negative when they are *trans*[3]. ^{3}J(HH) and ^{3}J(CH) are normally assumed positive, whereas ^{2}J(CH) can be negative or positive[4]. ^{2}J(CH) is negative in the cyclobutane moiety of the dimers.

HEAD-TO-HEAD



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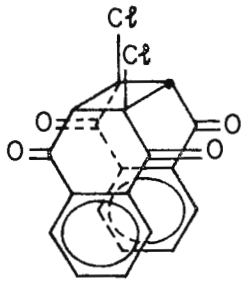

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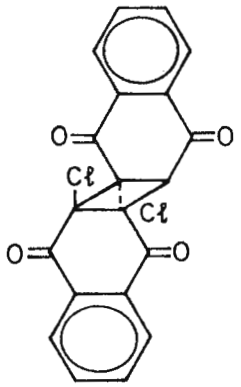

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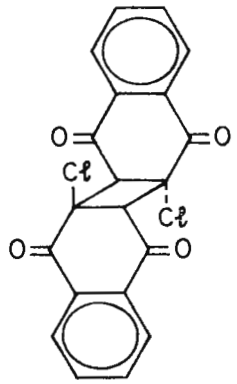

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anti

Only the signs of the (H,H) and (C,H) couplings, between the protons and a methine carbon atom of the cyclobutane ring of the dimers, are required to distinguish between dimers (1,3), (2) and (4). One could differentiate between dimers with configurations (1) and (3) from the magnitude of these coupling constants.

Yours sincerely

P.L. Wessels

P.L. Wessels
SENIOR CHIEF RESEARCH OFFICER

1. F.J.C. Martins, J. Dekker and A.M. Viljoen, *S. Afr. J. Chem.*, 31, 29 (1978).
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4. D.F. Ewing, *Annu. Rep. NMR Spectrosc.*, 6A, 389 (1975).

New England Nuclear

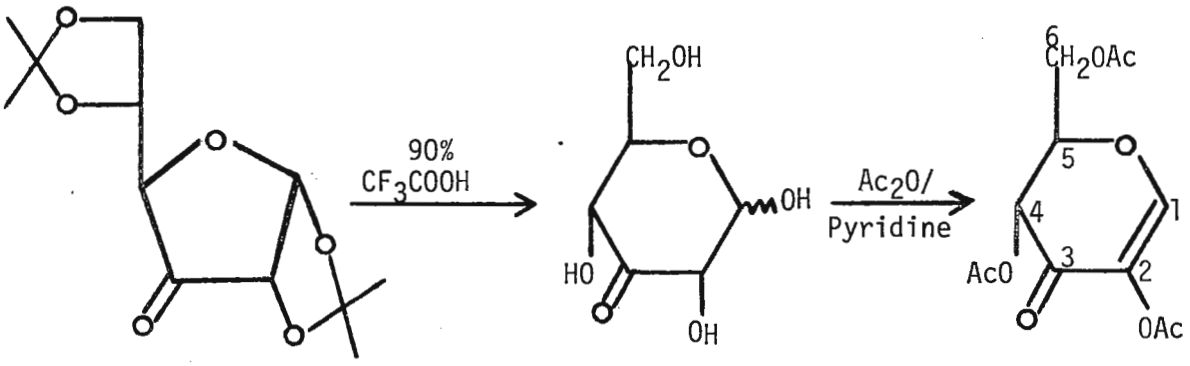
Prof. B. L. Shapiro
Dept. of Chemistry
Texas A & M University
College Station
TX 77843

December 2, 1980

^{1}H and ^{13}C NMR Spectral Analysis of a Carbohydrate Transformation Product

Dear Prof. Shapiro:

In connection with some of our synthetic projects in carbohydrate chemistry, we achieved the synthesis of the dihydro-4-pyrone II, namely 2,4,6-tri-O-acetyl-1-deoxy-D-erythrohex-1-enopyranos-3-ulose, in only two steps from the commercially available 1,2:5,6-di-O-isopropylidine-3-ketoglucofuranose (I) as shown below:



I II

II had been prepared earlier by much longer routes starting from different precursors and its characterization by proton nmr had been only partial[1-3]. We present here a complete analysis of the same by proton (at 200 MHz) and C-13 nmr spectroscopy.

^1H and ^{13}C nmr spectral parameters for II[a]

Position	^1H		^{13}C shift
	shift(ppm)	$J_{H,H}$(Hz)	ppm
C-1	7.47s	-	155.38d
C-2	-	-	130.56s
C-3	-	-	181.94s
C-4	5.67d	$J_{4,5}$=13.18	67.77d
C-5	4.70dt	$J_{5,4}$=13.18 $J_{5,6}$=3.78 $J_{5,6'}$=2.57	79.04d
C-6	4.41m	$J_{6,6'}$=12.82 $J_{6,5}$=3.78 $J_{6',5}$=2.57	61.23t
$COCH_3$'s	2.13s 2.19s 2.24s	-	20.08,20.35,20.59q 168.48,168.96,170.32s

a. chemical shifts from internal TMS for $CDCl_3$ solutions; s=singlet, d=doublet, t=triplet, q=quartet and m=multiplet and apply to the ^1H resonances; s,d,t and q apply to proton off-resonance decoupled C-13 spectrum.

With best regards,

Sincerely,

L. Thomas — Srini

Lawrence P. Thomas — Puliyur R. Srinivasan

Surendra Gupta — Douglas Moakley

Surendra Gupta — Douglas Moakley

References:

1. F. W. Lichtenthaler and P. Jarglis, Chem. Ber., 113, 489 (1980).

2. G. O. Aspinall and R. R. King, Can. J. Chem., 51, 394 (1973).

3. J. Weigmüller and H. Kunz, Tetrahedron Lett., 3807 (1978).

UNIVERSITY of PENNSYLVANIA
PHILADELPHIA 19104

School of Medicine G4

DEPARTMENT OF
BIOCHEMISTRY AND BIOPHYSICS

December 2, 1980

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

Dear Barry: Universal Field Lock

The word "universal" has a nice ring to it, and isn't too much of an exaggeration, I trust. In actuality, though, this Field/Frequency lock has so far proved itself on a pulsed NMR machine used to do T_1 and T_2 measurements of protons from 8 to 60 MHz. For high resolution work one would need to phase lock the Observe and Lock Frequency synthesizers. Other Fields (supercon?), absorption mode display etc. are obvious variations on the theme. In fact the theme itself is pretty obvious, although it may never have been played before with this broad band sound.

This is a time-shared lock system, where the transmitter is on 25% of the time and the receiver about 65% of the time. There is a 10% wait after the transmitter pulse so that the receiver can recover. The repetition rate is 1KHz, although 10KHz or 20KHz might be better to prevent possible spikes in high resolution applications. Transmit and Receive is done centerband for simplicity's sake. The transmitter gating has to be done pretty thoroughly, and the synthesizer has to be reasonably well shielded from the outside world. As gates we used two double balanced mixers (Mini-circuits Lab's SRA-1) in series before the power amplifier, and followed the P.A. by series crossed PIN diodes, all three gates being switched by the timer. No transmitter leakage was measurable. The R.F. power is controlled by the turn-on current to the SRA-1's.

The broadband continuously variable phase shifter involved the use of a varactor and AGC to produce four channels of R.F. with 90° shifts between them and applied to four equally spaced taps on a potentiometer. In retrospect I think it would have been easier to employ a broadband quadrature hybrid (Anzac) for the 90° shifts. The input to either of these phase shifters must be a sine wave. Since our synthesizer (Syntest) delivered a square wave, we had to make a broadband square-to-sine converter (not shown). The ultimate system would probably use a more expensive synthesizer delivering a sine wave and phase coherent with the Observe frequency synthesizer.

We chose lithium as a lock substance partly because of its frequency being compatible with a standard Syntest Synthesizer. The sample was concentrated LiCl doped with $MnCl_2$ to give a broader line.

The transmitter drives the probe via a broadband Transmit / Receive circuit (J.L. Engle, TAMU NMR, June? 1979). The probe has a small sample tube close to the Observe sample. Two lock sample coils are used and a two-position range switch. Tuning and a reasonable match are accomplished with one variable capacitor (J.L. Engle TAMU NMR Nov. 1976 p.24). Resonance is indicated by a reflectometer permanently installed in the transmitter line. The preamlifier is broadband, and low noise when it sees an approx. 50 ohm source. We used discrete components, with an NEC's NC921 transistor at the input.

The receiver is gated in two places: The reference gate is another SRA-1. The AF amplifier gate is a junction field effect transistor preceding an integrator at the output to the super stabilizer. The mixer is an SRA-1.

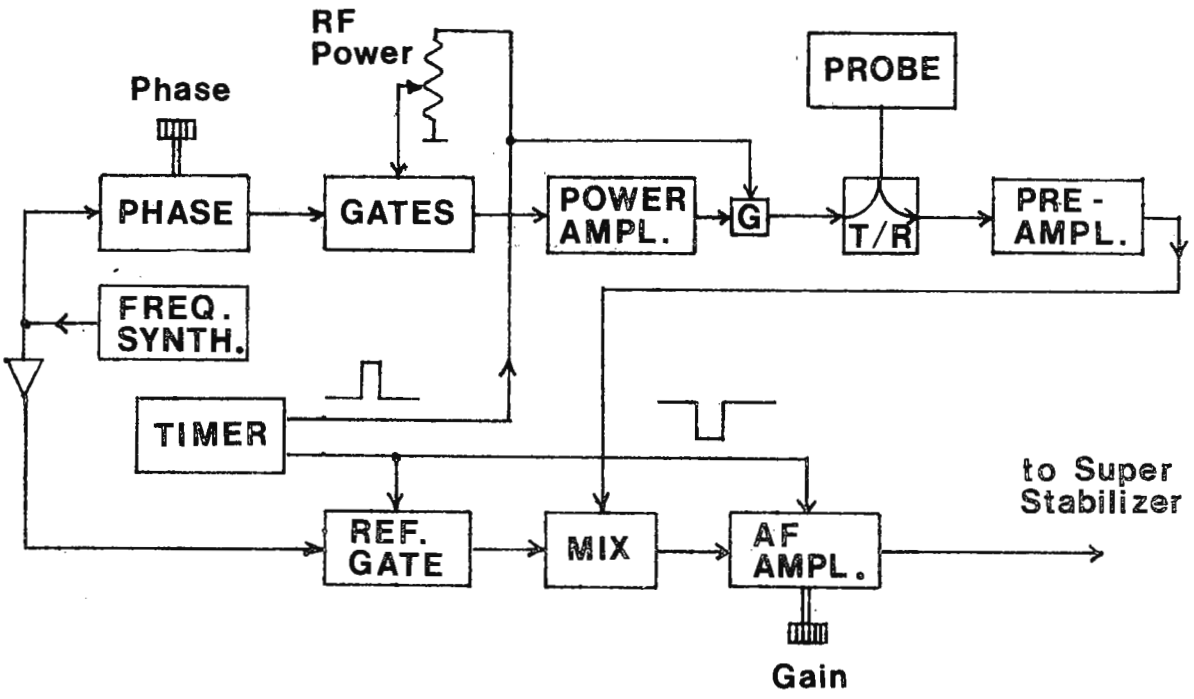
The timer clock is a free running multivibrator with a 25% duty cycle as used by thetransmitter. A pulse stretcher adds the time used for receiver recovery.

Please credit this to Dr. M. Cohn's account.

Sincerely,

Jim Engle

James L. Engle



LOCK SYSTEM BLOCK DIAGRAM

268-17

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

Title: "Quantitative ^{13}C nmr of petroleum products"

Dear Prof. Shapiro,

quantitative ^{13}C nmr has important application in complex samples where other analytical methods fail or are tedious. One favorable case is the characterization of petroleum cuts and coal-derived products. The ^{13}C nmr spectrum enables the distribution of the different carbon types to be determined with a minimum of structural hypothesis. An important information which can be easily derived is the "aromaticity", that is the fraction of aromatic carbon [1] (C_{Ar}/C_{Tot}).

The nmr experiment requires peculiar conditions. The relevant operational parameters which should be optimized in order to obtain the same sensitivity for all carbons and the highest S/N are the flip angle, the acquisition time, the pulse delay (using proton gated decoupling) and the relaxation agent concentration.

A pitfall of the method, however, is that one uses a simple aromatic mixture as standard test. To the best of our knowledge no comparison with an indipendent analysis on a complex mixture has been done.

By the nmr method we have investigated several atmospheric gasoils, and the corresponding hydrotreated products,[2] at different content of aromatic compounds ranging from 7 to 30%.

./..

We have also analyzed these samples by mass spectrometry, according to a well known procedure[3] and we have got an hydrocarbon types distribution[4] which can be translated into the fraction of aromatic carbon. The final results of the two methods are collected in the Table 1 together with the aromatic product percentage determined by liquid-solid chromatography[5]. The two series of data are in good agreement, confirming the reliability of both methods.

The figure shows also the good correlation obtained between the aromatic product concentration in the gasoil and the aromaticity.

Yours sincereley,

E. Santoro, G. Bragato

References

1) C.E. Snape, W.R. Ladner and K.D. Barile, "Anal. Chem." 51, 2109 (1979).
D.M. Cantor, "Anal. Chem." 50, 1185 (1978).
J.N. Shoolery, W.L. Budde, "Anal. Chem." 48, 1458 (1976).

2) Kindly provided by Dr. G. Matarrese (Bollate Research Center).

3) ASTM Method n. D2425.

4) Thanks are due to Dr. L. Motta (Bollate Research Center) for the mass spectral data.

5) ASTM Method n. D2549.

Table 1 - Aromaticity (C_{Ar}/C_{Tot}) of gasoil by ^{13}C nmr and mass spectrometry.

Sample	% by wt of arom.comp.	Aromaticity: C_{Ar}/C_{Tot}		Experimental ^{13}C nmr conditions (Bruker WH 90/DS)
		^{13}C nmr	Mass.Spectr.	
1	29.5	0.16	0.17	flip angle: 90°
2	22.0	0.086	0.108	acquis. time (16 k for data): 1.36 sec
3	17.0	0.075	0.088	pulse delay (prot. gat. dec.): 2 sec
4	16.6	0.087	0.087	$Cr(AcAc)_3$ conc.: 0.05 M
5	11.1	0.055	0.058	Sample volume fraction: 0.5
6	9.6	0.046	0.047	(Solv. $CDCl_3$).
7	8.2	0.041	0.043	
8	7.1	0.026	0.035	

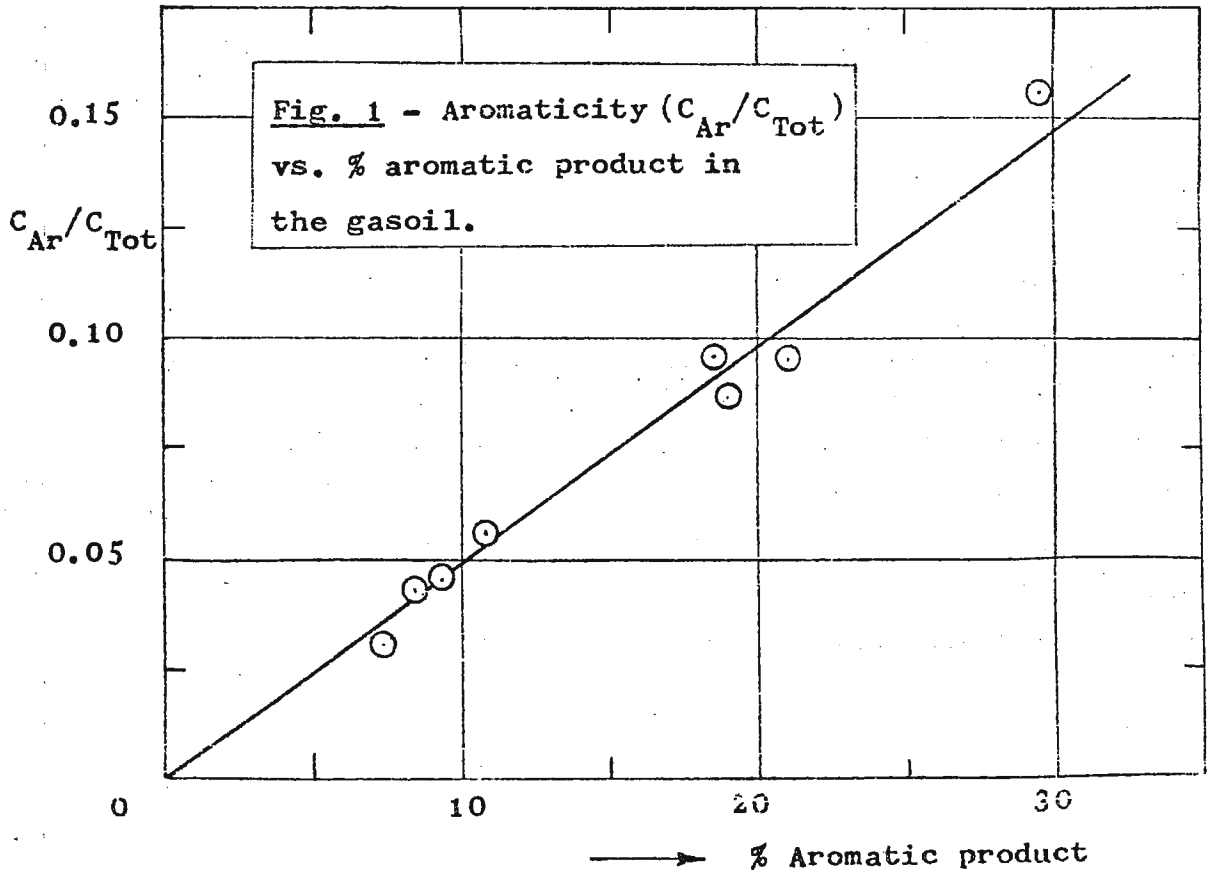

Fig. 1 - Aromaticity (C_{Ar}/C_{Tot}) vs. % aromatic product in the gasoil.

Bruker NMR

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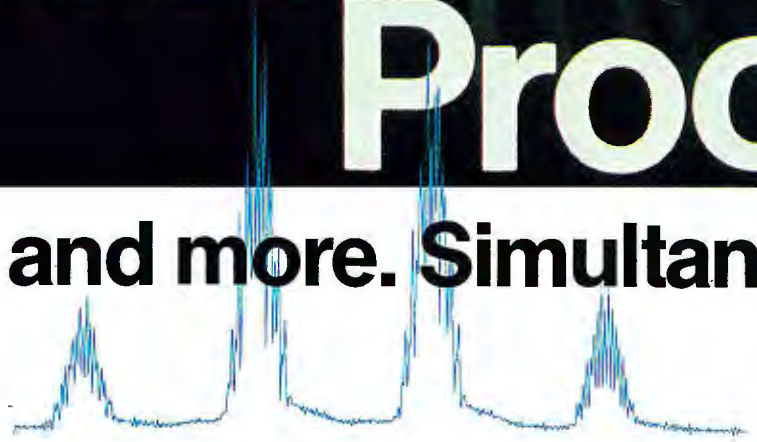

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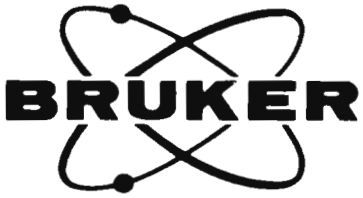

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Prof. Dr. Dieter Ziessow

Berlin, den December 11, 1980
Tel.: (030) 314-4958
Az.:

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Straße des 17. Juni 112
Ernst-Reuter-Haus
~~1 Berlin 10~~
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~~Telefunken-Haus, 15. O. G.~~

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

5th European Experimental NMR Conference

Dear Professor Shapiro:

The next (5th) European Experimental NMR Conference will be held at Königstein/Frankfurt (Main), West Germany. Registration will begin Tuesday, May 12, 1981, and the program will run through noon on Friday, May 15. Sessions are planned in the areas of new techniques in liquids, solid state NMR, 2D NMR, NMR imaging, computer aids, metal nuclei NMR and others. Further information and the Second Circular will be mailed on request. The deadline for contributions will be February 28, 1981.

Sincerely yours,

Dieter Ziessow

268-23

Department of Industry
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Cornwall House Stamford Street London SE1 9NQ

Telegrams Govchem London
Telephone 01-928 7900 ext 649

Professor B L Shapiro
Dept of Chemistry
Texas A & M University
College Station
TEXAS 77845
USA

Your reference

Our reference

Date 2 December 1980

Dear Professor Shapiro,

NMR of Food Additive Polyphosphates

^{31}P FTNMR spectroscopy is a convenient technique for observing the interaction of polyphosphate food additives with a food matrix. We have been studying the behaviour of polyphosphate food additives when injected into enzymatically active chicken meat (1,4). However, the NMR resolution obtained from lumps of meat pushed down into the NMR sample tube is much less than that obtained from solution samples. This is due to difficulties in shimming, inhomogeneous distribution of polyphosphate within the sample, as well as pH and meat structure inhomogeneities. In those instances where a higher resolution is required, the system may be simulated by using the food additive in aqueous solution and the meat sample cut to form the antivortex plug. An example of the method applied to the hydrolysis of tetrapolyphosphate in contact with chicken pectoralis major tissue is shown. The presence of tripolyphosphate signals shows that at least some of the tetrapolyphosphate is hydrolysed via the triphosphate route.

M. L. Trimble

C P Richards

M L Trimble

C P Richards

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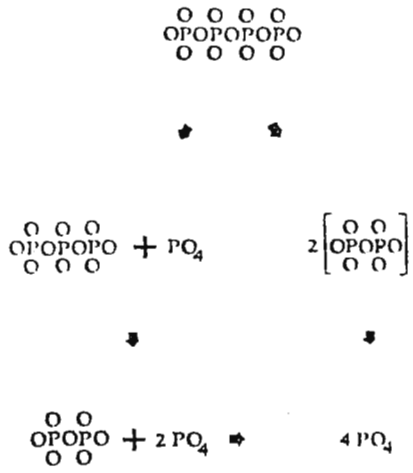
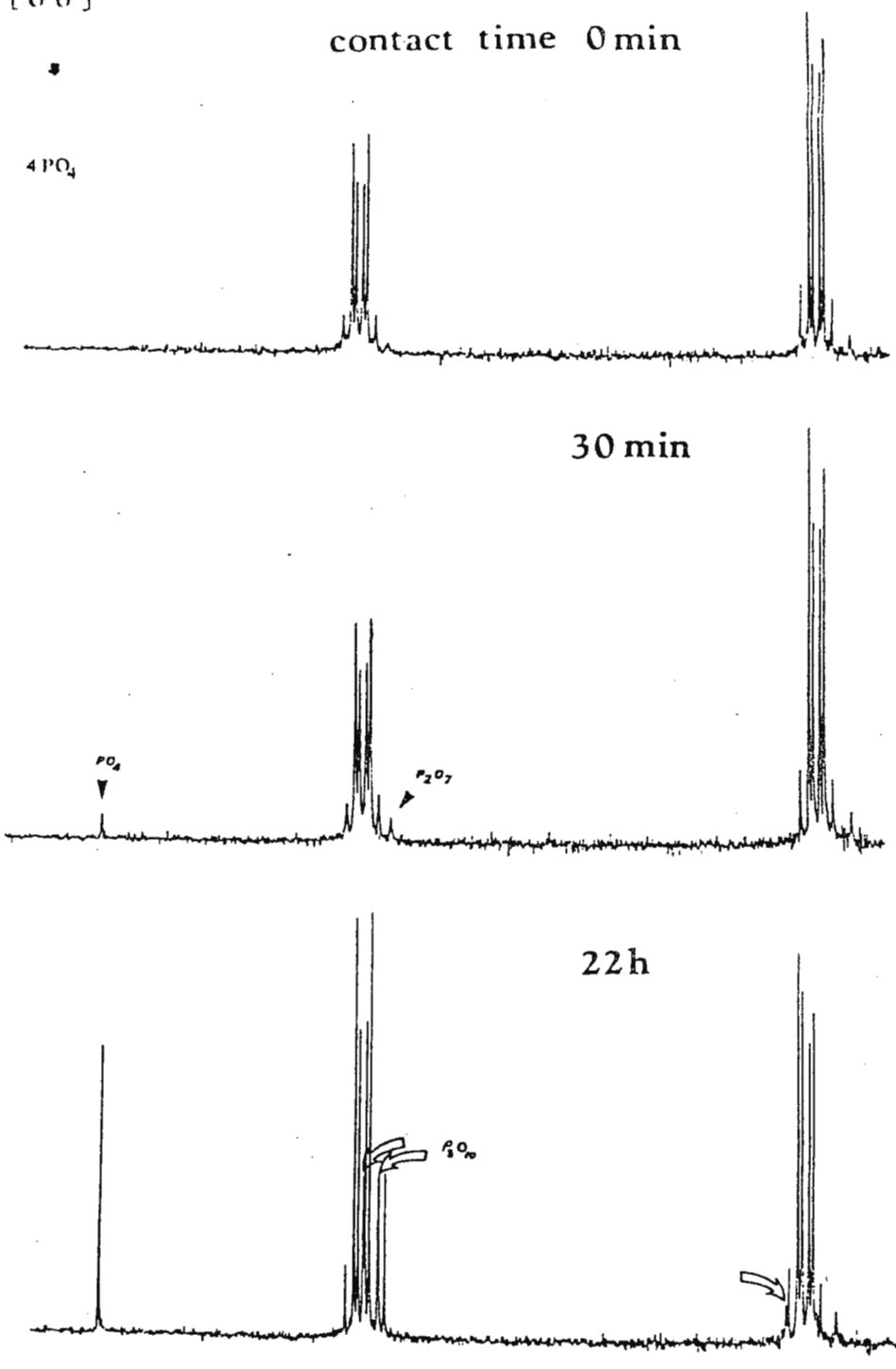
3. M Douglass, M P McDonald, I K O'Neill, R C Osner and C P Richards, J. Fd. Technol. 14, 193 (1979).

4. P J King and C P Richards, Paper given at Joint ISMAR - AMPERE International Conference on Magnetic Resonance, Delft (1980).

Hydrolysis of tetrapolyphosphate





DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
PUBLIC HEALTH SERVICE
NATIONAL INSTITUTES OF HEALTH
BETHESDA, MARYLAND 20205

December 10, 1980

Building 2, Room B2-08

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

Re: Killing 'em softly; ^{31}P NMR studies on microorganisms.

Dear Barry:

Usually when NMR spectra of microorganisms are run the cells are re-suspended in liquid medium after centrifugation. They tend to settle down to the bottom of the NMR tube, where because of the rapid depletion of oxygen and nutrients they begin to die.

One strategem taken to avoid this happening is to re-suspend the cells by bubbling oxygen, sometimes with sophisticated spectrometer interlock (1). This is time-consuming in terms of the acquisition of spectra, and does not prevent intermittent deterioration of the sample.

In search of an alternative approach we attempted to set up a continuous flow-through system, using oxygenated medium, but this "perfusion" is much less successful for cells than for organs. The cells block most filters, and this approach is cumbersome.

A simple but viable alternative which keeps the cells alive over a long period, is to embed them in agarose gel supplemented with desired nutrients. In fact, growth of microorganisms in a gel block is a longstanding microbiological technique. Agarose has the virtue of allowing normal diffusion of oxygen and nutrients because the cells are not close-packed. To ensure continuing availability of nutrients and oxygen, a liquid culture medium above the agarose block is bubbled through with either pure oxygen or air.

An illustration of the use of this approach is the increase in the polyphosphate content of resting E. coli incubated in sulfur-free medium (2) over a 24 hr period (Fig.), indicating the continued viability of the cells. Of course, shorter term experiments can also be done, but with added reproducibility utilizing agarose re-suspension. The motion of the cells is, of course, so slow in packed suspensions that embedding them in agarose leads to no net degradation in the quality of spectra.

Sincerely yours,

Lev Jacobson
Jack S. Cohen
Developmental Pharmacology Branch
National Institute of Child
Health and Human Development

LJ/JSC:ell

Dr. B. Shapiro

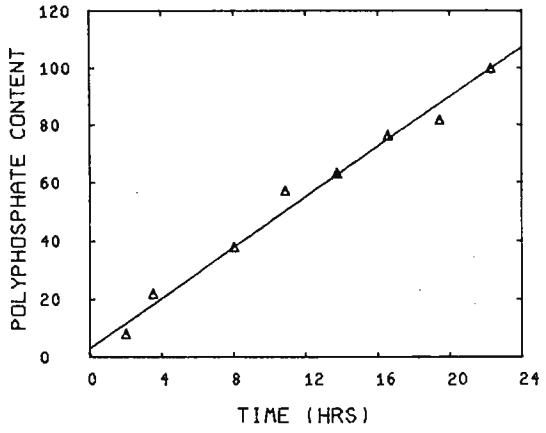
December 10, 1980

(1) G. Navon, S. Ogawa, R. G. Shulman and T. Yamane, Proc. Natl. Acad. Sci. USA 74 888 (1977).

(2) L. Jacobson and J. S. Cohen, unpublished results.

Figure Legend

Polyphosphate content as a function of time of a culture of E. coli grown to mid-exponential phase and re-suspended in sulfur-depleted medium (at ca. 10^8) in agarose (0.25%) at 37°C. Oxygen was bubbled through the medium above the agarose block. The ^{31}P NMR peak at -23 ppm was integrated, relative to the final value after 24 hrs set to 100. Spectra were recorded at 109.3 MHz in 3K scans with 0.5 sec delay time.



Wesleyan UNIVERSITY

HALL-ATWATER LABORATORIES
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DEPARTMENT OF CHEMISTRY

Title: Improved Heteronuclear Two-Dimensional NMR: BICYCLE Phase Cycling

December 12, 1980

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

Dear Barry:

Heteronuclear two-dimensional NMR is now being applied to some samples of real interest. In this sort of experiment one is typically interested in obtaining high resolution in the proton dimension. For many samples a rather wide spectral width in both the observed nucleus and proton dimensions is needed. This problem becomes more acute at high magnetic field strengths. However, there is a restriction on the resolution placed by the use of large spectral widths. The spectral width in the proton dimension is determined by 1/(2x increment of evolution time) and the resolution is given by equation 1 which is an extension of the results of Maudsley,

$$\Delta\nu_{\frac{1}{2}}(\text{proton}_{\text{single slice}}) = \frac{1}{\pi}\left[\frac{\gamma_H}{\gamma_I}\left(\frac{1}{\text{acquisition time}} + \frac{1}{T_{2I}}\right) + \left(\frac{1}{\text{evolution time}} + \frac{1}{T_{2H}}\right)\right] \quad [1]$$

Wokaun and Ernst (1). The problem is that the resolution in a single slice in the proton dimension is partially determined by the total length of the evolution time but a large spectral width requires a small increment of the evolution time and hence a large number of t_1 values. This quickly leads to the storage of more information than the computer can store.

A partial resolution of this problem is to use a phase cycling procedure which mimics quadrature detection. The basic idea is illustrated in Figure 1. In a conventional heteronuclear two-dimensional NMR experiment the cosine of the proton modulation frequencies modulates the observed nucleus signal. The resulting proton spectrum then contains signals which occur at the sum and difference of the proton frequencies relative to the decoupler offset. In the phase cycling approach the experiment alternates between transfering the cosine of the proton frequencies to the real part of the observed signal and the sin to the imaginary part. Some typical data is shown in Figure 1. Figure 1(A) is the proton spectrum obtained from the complex Fourier transform of the cosine modulation, 1(B) is the complex Fourier transform of the sin modulation and 1(C) is their sum. It is seen that there is a good cancellation of images. The important point is that this procedure offers twice the spectral width of the conventional approach for a given increment of the evolution time.

It is important to note that since separate pulse sequences must be used to transfer the cosine and sin modulations the signal to noise gain that occurs in conventional quadrature detection will not be present in this two-dimensional analog. Also, to effectively suppress the presence of a signal at zero frequency to the F_1 dimension an additional phase cycling need be used. The pulse sequence actually used to eliminate the zero frequency signal and to give the quadrature transfer is to cycle both the second proton pulse and the observe nucleus pulse through X, -X, Y, -Y.

This method is rather similar to the EXORCYCLE approach for J spectroscopy (2). Since the method incorporates both quadrature transfer and elimination of zero fre-

quency signals as well as doubling the spectral width by being able to tell the difference between the positive and negative sides of the carrier it is dubbed BICYCLE.

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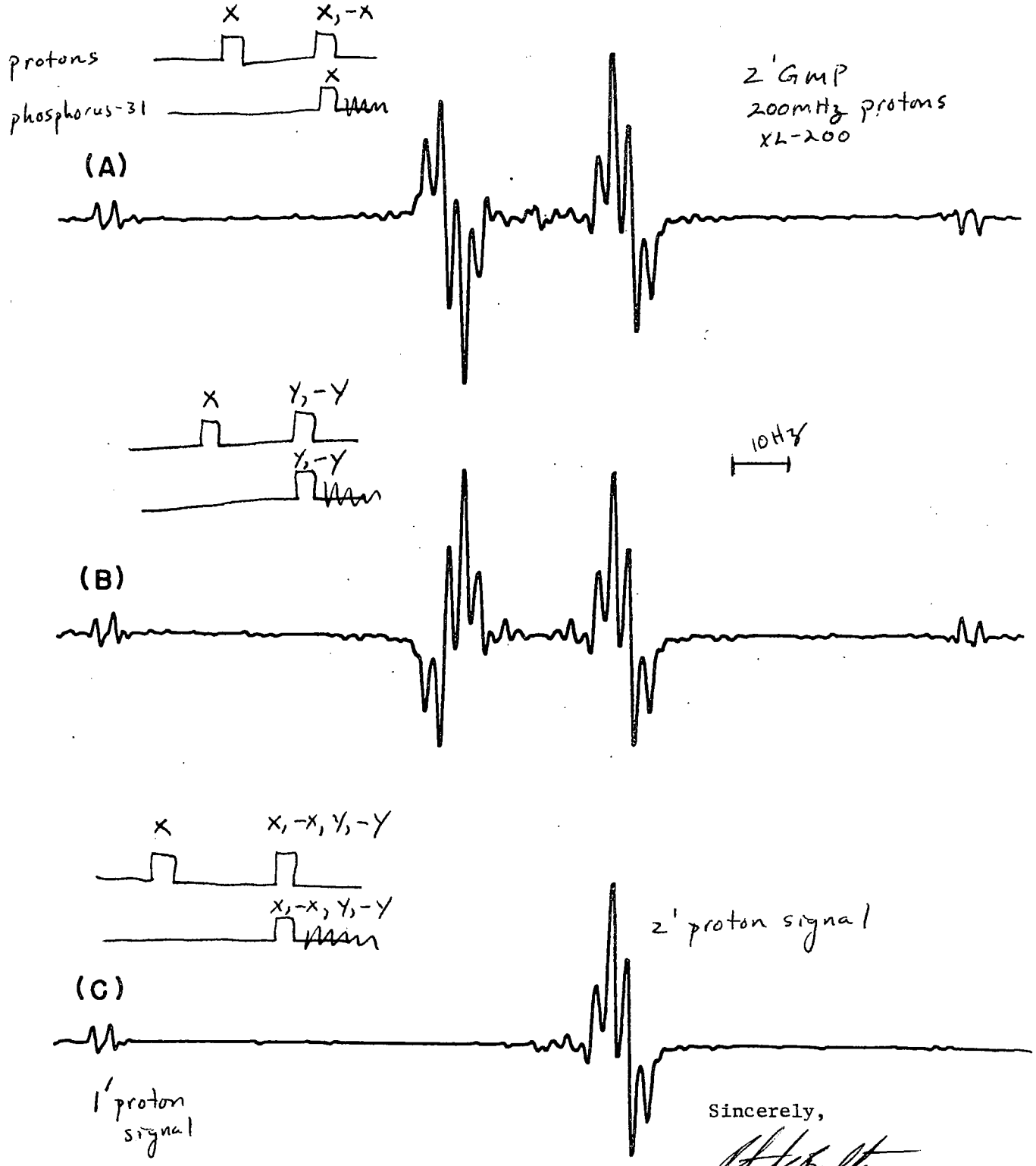
(2) G.Bodenhausen, R.Freeman and D.L.Turner, J.Magn.Reson. 27, 511 (1977).



Sincerely,

Philip H. Bolton
Assistant Professor

PHB:lb

268-29

National Research Council Canada
Conseil national de recherches Canada

Division of Chemistry
Division de chimie

File Référence

December 16, 1980

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

Exchange by Dissociation and Displacement

Dear Barry,

The relationships between reaction rates, rate constants and lifetimes via magnetic resonance have been known for many years.[1,2] These have been used to calculate rates and determine mechanisms of exchange between a Lewis acid-base complex and one of its components. The exchange can occur either by dissociation of the complex, in which case the activation enthalpy for exchange equals the heat of dissociation, or by displacement of one species in the complex by excess of free species.

$$A^* + A - B \rightleftharpoons A^* - B + A$$

In the latter case the activation enthalpy for exchange will be less than the heat of dissociation. Both types of exchange mechanisms have been observed in various systems. To the best of my knowledge no case has yet been reported where both exchange mechanisms can operate in the same system.

We have investigated the exchange of an excess of either dimethyl sulfide or boron trichloride with boron trichloride-dimethylsulfide complex in methylene chloride and sulfur dioxide solutions via proton, ^{13}C and ^{11}B resonance spectroscopy. From the concentration dependence of the exchange rates it was found that dimethyl sulfide exchanges by a dissociation mechanism while boron trichloride exchanges by a displacement mechanism. The rate constants as a function of temperature are shown on the accompanying graph. The enthalpies of activation are 5.0 ± 0.5 kcal/mole for BCl_3 exchange and 20 ± 1 kcal/mole for dimethyl sulfide exchange.

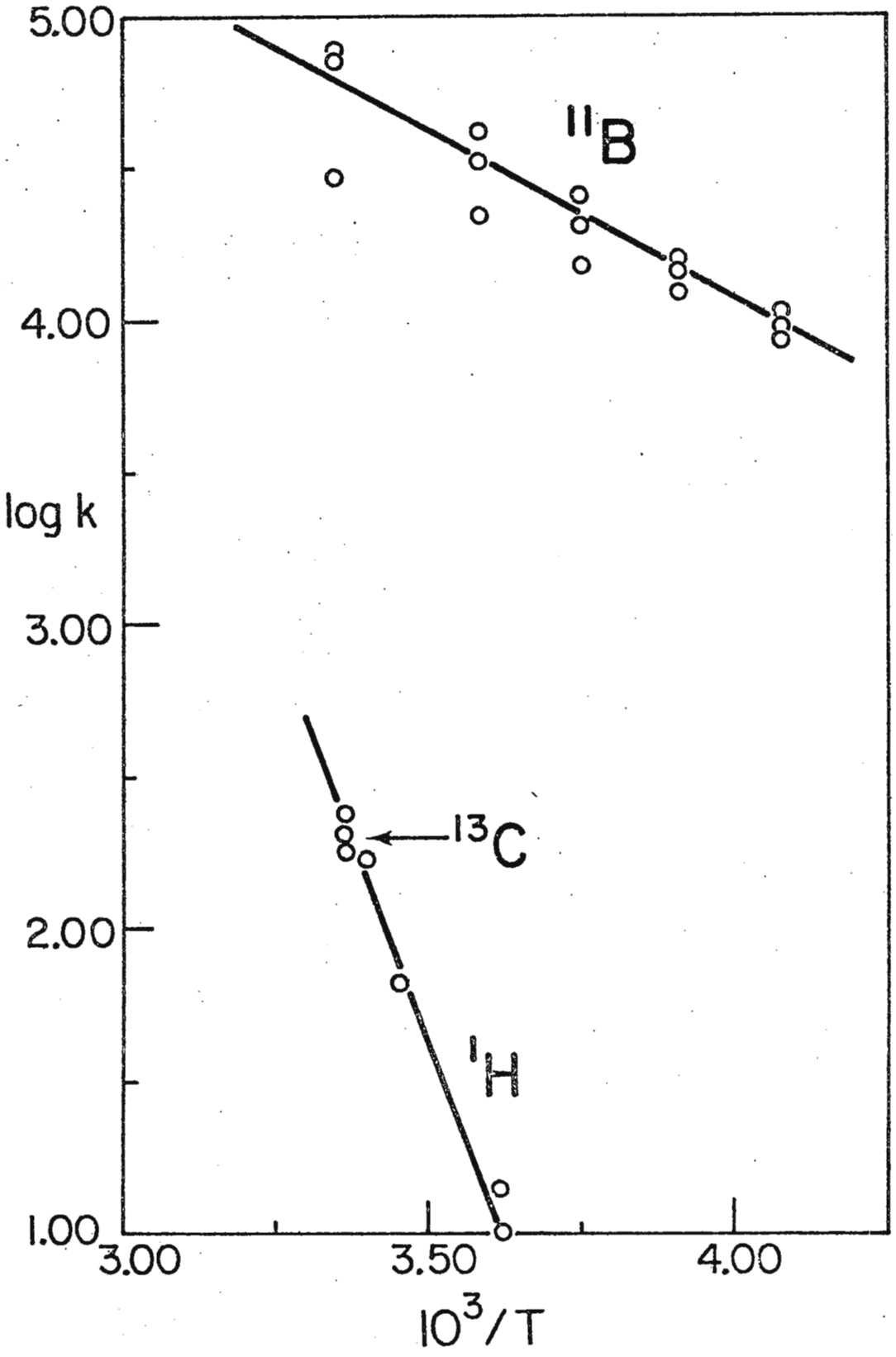
(1) S. Brownstein, A.M. Eastham and G.A. Latremouille, J. Phys. Chem., 67, 1028 (1963).

(2) J.B. de Roos and J.P. Oliver, Inorg. Chem., 4, 1741 (1965).

Yours truly,

B. Glavincevski
S. Brownstein

Ottawa, Canada
K1A 0R9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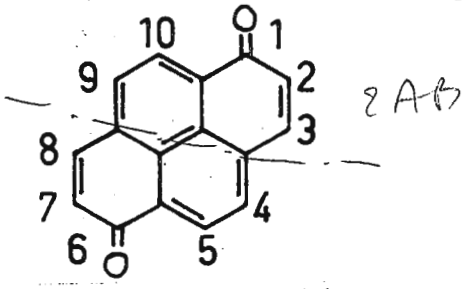
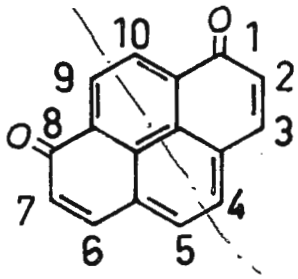
LEIDEN, dec. 19th 1980
Dr. Bernard L. Shapiro
Department of Chemistry
Texas A & M University
College Station, Texas 77843

Dear professor Shapiro: PYRENE QUINONES

We are interested in the chemistry of 1,6-pyrenequinone (I) and 1,8-pyrenequinone (II). Their preparation by CrO_3 oxidation of pyrene and the separation of the resulting 1:1 mixture of I and II has been described.[1]
I and II have been characterized thusfar by IR-and electronic spectra. The ^{1}H-NMR spectra of I and II have not been published. We like to report the ^{1}H-NMR data of I and II (JEOL PFT100, $CDCl_3$). The ^{1}H-NMR patterns are fully in agreement with the structures. ^{1}H-NMR spectroscopy can more elegantly differentiate between I and II than other analytical techniques used thusfar.





1,6-pyrenequinone (I)			1,8-pyrenequinone (II)		
δ (ppm)		assignment	δ (ppm)		assignment
6.71 }	AB	H 2,7	6.68 }	AB	H 2,7
7.68 }	J=10.0 Hz	H 3,8	7.68 }	J=9.7 Hz	H 3,6
7.84 }	AB	H 4,9	7.67 }	S	H 4,5
8.49 }	J=7.6 Hz	H 5,10	8.64 }	S	H 9,10

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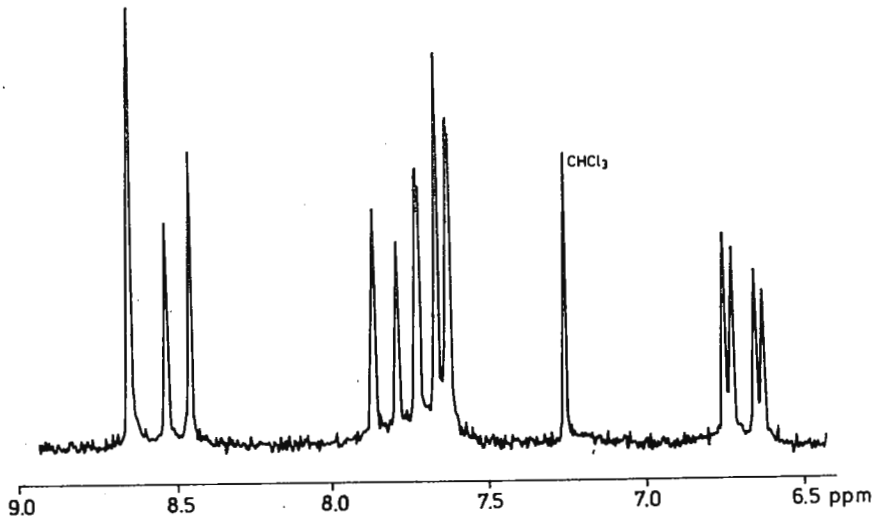
vervolgblad



^{1}H NMR spectrum of I and II, ca. 1:1 mixture

1. Reference: A.J. Fatiadi, J. Chromatog, 20, 319 (1965)

Sincerely yours,

J. Terheyden, C. Tintel, J. Cornelisse, C. Erkelens, J. Lugtenburg.

CONTINUING EDUCATION PROGRAM ANNOUNCEMENT

TITLE:	WORKSHOP ON NUCLEAR MAGNETIC RESONANCE IMAGING
DATE:	February 19 & 20, 1981
PLACE:	The University of hexas Medical School at Houston Department of Radiology and The American Association of Physicists in Medicine (AAPM)
CREDIT:	12 Hours - AMA Category I
FEE:	$225.00

For further information contact the Office of Continuing Education, The University of Texas Medical School at Houston, P. O. Box 20708, Houston Texas 77025, Phone (713) 792-5346.

UNIVERSITÉ PARIS VII

ITODYS **INSTITUT DE TOPOLOGIE ET DE DYNAMIQUE DES SYSTEMES**
1, rue Guy de la Brosse - 75005 PARIS - Tél. : 336 25.25, poste 36-15

LABORATOIRE DE CHIMIE
ORGANIQUE PHYSIQUE

J.-E. DUBOIS, *Directeur*

PARIS, December 24th, 1980.

N/Réf. BT/BA/CV/80.77

Title : ^{33}S linewidth measurement at 15 MHZ.

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

Dear Dr. Shapiro,

Thank you for your yellow reminder. Last year we published a study on molecular reorientation of CS_2 diluted in alkanes[1], ^{33}S linewidths were used to determine the reorientation correlation time of CS_2 and were measured at 30 MHZ on a Bruker WH400 spectrometer.

Recently we ran some further experiments at 15 MHZ on our Bruker WP200 spectrometer fitted with a broadband probe. Unfortunately the well-known acoustic ringing occurs at this low frequency, leading to a rolling base-line and precluding accurate linewidth measurement. The ringing persists for approximately 1.5 ms, regardless of the spectral width.

As the ^{33}S linewidth of CS_2 lies in the 300 $\sim$ 600 HZ range (i.e. T_2 = 1 $\sim$ 0.5 ms) depending upon medium viscosity, most of the ^{33}S FID occurs during probe ringing. Consequently if acquisition is delayed 1.5 ms to eliminate the ringing (succesfully used for ^{14}N at 14 MHZ with the same probe) the sensitivity of ^{33}S measurements falls so much that experiments on dilute solutions is impossible. We also tried to acquire the second half of the echo in a $90°-\tau-180°-\tau-AQ$ sequence, but if τ is long enough to eliminate the ringing following the 180° pulse, there is no more ^{33}S NMR signal ! For the same reason, the sequence proposed by Brooke, Openshaw and Cushley[2] cannot help when life-times of the ringing and the NMR signal are comparable.

...

We think that only a 15 MHz ^{33}S probe specially designed to eliminate acoustic ringing at the relevant frequency can solve this problem.

Sincerely yours,

B. TIFFON B. ANCIAN.

1 B. Ancian, B. Tiffon and J.E. Dubois
Chem. Phys. Letters, 65, 281, (1979).

2 A. Brooke, T.R. Openshaw and R.J. Cushley
TAMU Newletters, 254, 57, (1979).

268-35

The University of Connecticut

STORRS, CONNECTICUT 06268

THE COLLEGE OF
LIBERAL ARTS AND SCIENCES
Department of Chemistry

Announcement of an International Conference on Conformational Analysis

I am writing to call your attention to an International Conference on Conformational Analysis which will be held at the New England Center on the campus of the University of New Hampshire from June 29 to July 2, 1981.

The Conference is intended to bring together interested scientists from around the world to share information and exchange views. The format of the Conference is such that it should encourage informal discussion. A total of twelve plenary lectures are planned: three each morning for the 3½ days of the Conference. Evenings will be devoted to poster-session presentations by conference participants and afternoons will be free for recreation or informal discussion.

Plenary lectures by the following internationally known authorities have been scheduled and several additional speakers have been contacted.

Prof. F. A. L. Anet, Univ. of California at Los Angeles

Dr. F. A. Bovey, Bell Laboratories

Prof. E. L. Eliel, Univ. of North Carolina

Prof. K. Mislow, Princeton Univ.

Prof. S. Nelsen, Univ. of Wisconsin

Prof. M. Oki, Univ. of Tokyo

Prof. H. Schaeraga, Cornell Univ.

Prof. F. Vogtle, Freidrich-Wilhelms Univ.

If anyone should like to receive complete information and registration forms for the Conference, they should contact:

Prof. Gary R. Weisman
University of New Hampshire
Dept. of Chemistry
Durham, NH 03824

Unilever Research

Port Sunlight Laboratory
Quarry Road East
Bebington Wirral
Merseyside L63 3JW

Telephone 051-645 2000
Telex 627578

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

9 DEC 80

Dear Professor Shapiro,

EQUIPMENT FOR SALE

We have the following spectrometers for sale. They have both been maintained by the manufacturers and are in excellent working condition.

Bruker WH 90, purchased 1973

5mm ^{1}H, ^{19}F probes;
10 mm and 15 mm ^{13}C probes;
BNC 12 computer, 12K core, teletype, hardware pulse generator.
Available immediately.
Price expected in the region of £5,000.

Jeol FX 100, purchased 1976

1mm, 5mm dual $^{1}H/^{13}C$ probes;
5mm ^{19}F probe;
10mm ^{13}C, ^{31}P, ^{27}Al probes;
Variable temperature controller;
Internal and external ^{2}D lock;
24 K computer with cartridge disc-based foreground/background system (added 1980), PG 200 pulse programmer.
Available April/May 1981.
Price expected in the region of £35,000.

We would prefer to sell the spectrometers as complete systems, but would consider offers for parts if necessary.

Pricing enquiries should be addressed to Mr S Dwight, Supplies Controller, Unilever Research, Port Sunlight Laboratory, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW, UK. Tel. 051 645 2000 Ext. 681.

Any technical enquiries can be directed to myself at the above address or by telephone to extension 660.

Yours sincerely,

C J CLEMETT

PURDUE UNIVERSITY

SCHOOL OF SCIENCE
at INDIANAPOLIS

PHYSICS DEPARTMENT
1201 East 38th Street
Indianapolis, Indiana 46205
(317) 923-1321

December 17, 1980

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

TITLE: NMR Faculty Position

Dear Professor Shapiro:

Our department has a faculty position available for an NMR spectroscopist. The advertisment that is scheduled to appear in the December, 1980 issue of Physics Today is reproduced below:

> Applications are invited for a position at the Associate or Assistant Professor level in the Department of Physics, Indiana University-Purdue University at Indianapolis (IUPUI). The applicant should have experience in Nuclear Magnetic Resonance at the Assistant Professor level or equivalent and an interest in biological or medical applications of NMR. It is expected that he or she would initially collaborate with existing groups currently working on high-resolution NMR or imaging. Options exist to interact with ESR groups or the Biophysics division. Preference will be given to persons who can help develop an undergraduate or masters level laboratory in biological physics and are willing to interact with the Indiana University School of Medicine component of IUPUI. Submit applications to Professor B. D. Nageswara Rao, Department of Physics, IUPUI, 1125 E. 38th Street, Indianapolis, Indiana 46205. IUPUI is an Equal Opportunity Employer.

We would appreciate your bringing this to the attention of TAMU readers. The recruitment process is expected to begin by February 1981.

Sincerely yours,

B. D. Nageswara Rao

Yes, Brünnhilde, there really is a high-field NMR alternative:

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Uncompromising performance, limitless adaptability.

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

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

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

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- **High resolution studies of solids with Waugh-Pines cross-polarization and magic-angle spinning.**
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- **Temperature-programmed experiments.**
- **^{31}p experiments on living organs.**

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- Broad-Band Probes
- "Tilt" Micro Probe

FX-90Q:

- OMNI Probe™ System
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FX-200:

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- Broad-Band Probes
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