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DEADLINE DATES: No. 287 2 August 1982
 No. 288 6 September 1982

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

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

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USE OF ALUMINIUM-27 NMR IN SOLID STATE CHEMISTRY

Recently we took delivery of a JEOL solid state accessory for our FX200 spectrometer. Included in the package were probes for carbon-13 and silicon-29 nuclei. The latter has been found to be particularly useful in examining silicates of all types, but often it is aluminium-27 that can give important information on the system under examination. To our delight we found that the carbon-13 probe could be retuned to the aluminium frequency, so we can examine two important nuclei with one probe (two for the price of one)! Aluminium-27 NMR is good at distinguishing tetrahedral from octahedral states and as a consequence it can be used to monitor the presence of various aluminium species in solid state reactions. One quite nice example we have come across recently concerned the dealumination of a zeolite. This is done by reacting the zeolite with silicon tetrachloride to form aluminium chloride which is subsequently removed by washing. We have found that aluminium-27 NMR can follow this reaction. Figure 1A shows the aluminium spectrum of a zeolite that has been treated with silicon tetrachloride and three species AlCl_4^- , tetrahedral 'lattice' and octahedral aluminium, probably $\text{Al}(\text{OH})_6^{3+}$ can be identified. Figure 1B shows the aluminium spectrum of the material after washing - all the AlCl_4^- and probably most of the $\text{Al}(\text{OH})_6^{3+}$ has gone. The ability of aluminium NMR to follow solid state reactions of this type, assures the technique a very wide application not only in zeolite chemistry but in many others as well.

We should like to thank Dr Mike Barlow of our Research Centre for providing the samples and much interesting discussion on the aluminium chemistry of aluminosilicates.

I. Poplett

S. Knight

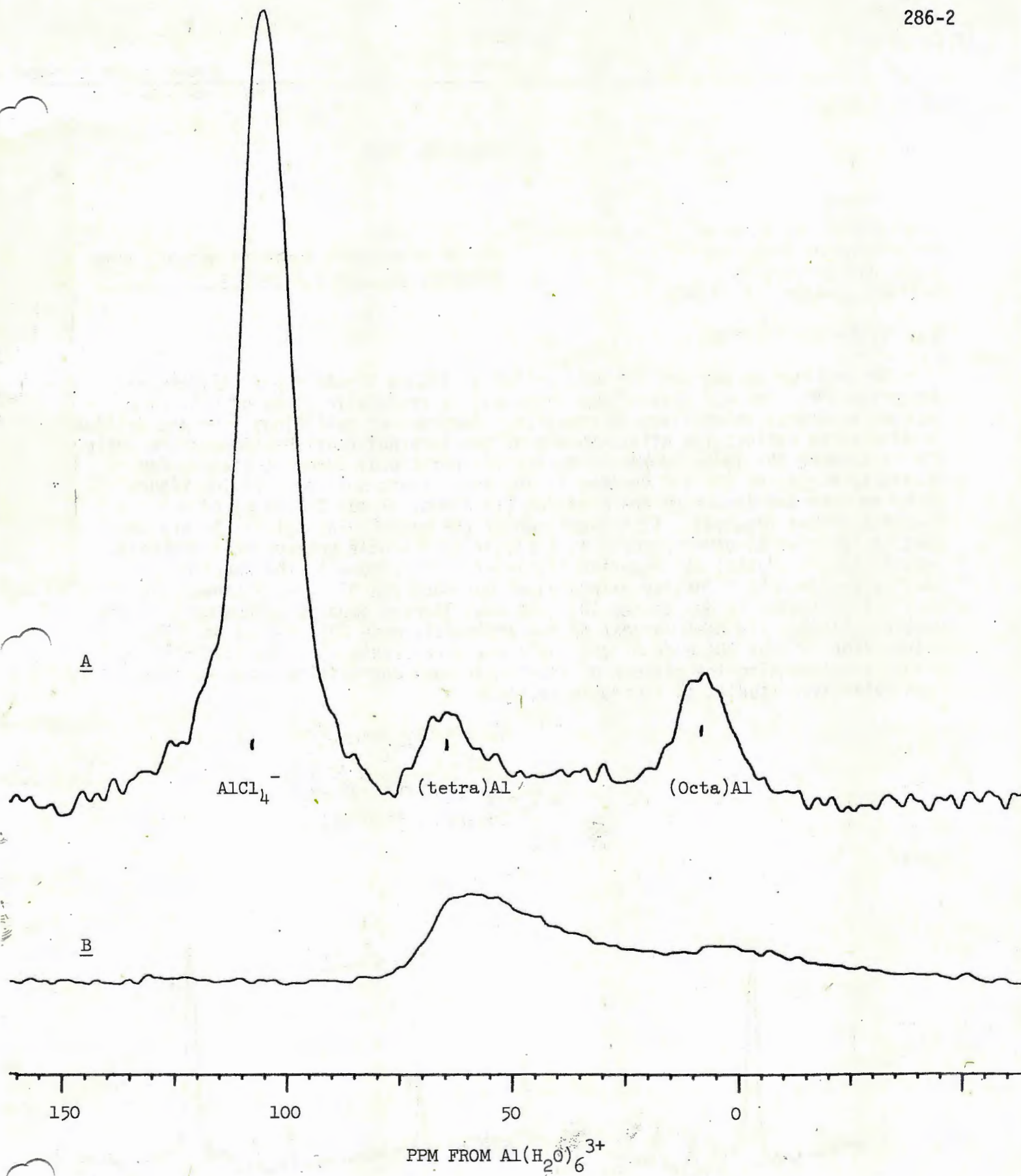


FIGURE 1

June 18, 1982

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

On the Asymptotic Route to Solubilizing
Polymers in Liquid Crystals

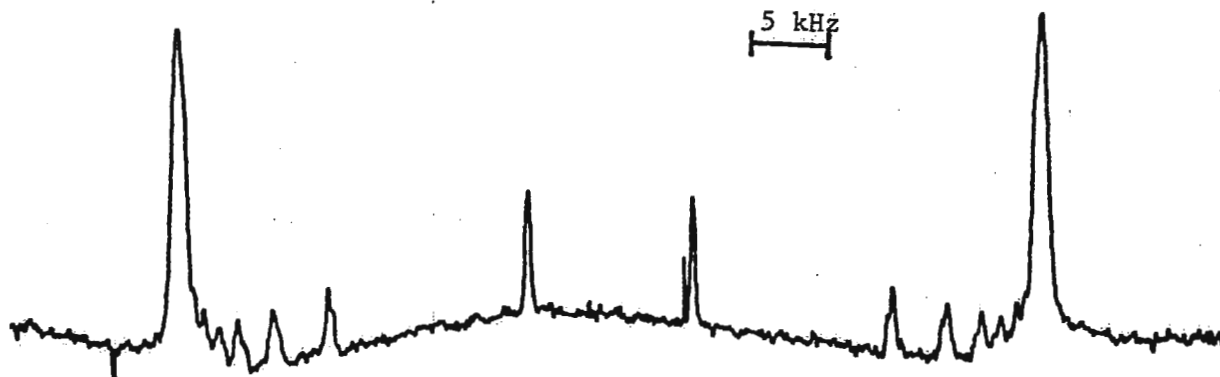
Dear Professor Shapiro:

We continue to explore the conjunction of liquid crystals and polymers via deuterium NMR. Part of this effort deals with a systematic study of deuterium labeled n-alkanes solubilized in nematics. Quadrupolar splittings from the various labeled sites reflect the effectiveness of the internal reorientation of the chain (as it samples the large number of accessible rotational isomeric states) for spatially averaging the C-D vectors in the anisotropic solvent. In the figure below we show the deuterium NMR spectrum (13.8 MHz; ~ 1 mol % alkane) of n-hexatriacontane ($C_{36}D_{74}$). (The magnitude of the quadrupolar splittings are decreased relative to other reports by a factor of one-half because this aliphatic nematic liquid crystal has negative diamagnetic anisotropy.) The spectrum is readily assigned by inductive extension of our modeling of lower alkanes; the smallest splitting is due to the CD_3 ; the next largest doublet corresponds to the penultimate CD_2 ; the next largest to the antipenultimate CD_2 , and so on. The bottom line is that the ends of the chain wag more freely than the internal segments, complementing the pattern of reorientational correlation times derived from relaxation studies in isotropic solvents.

Very truly yours,

E. Samulski
Edward T. Samulski

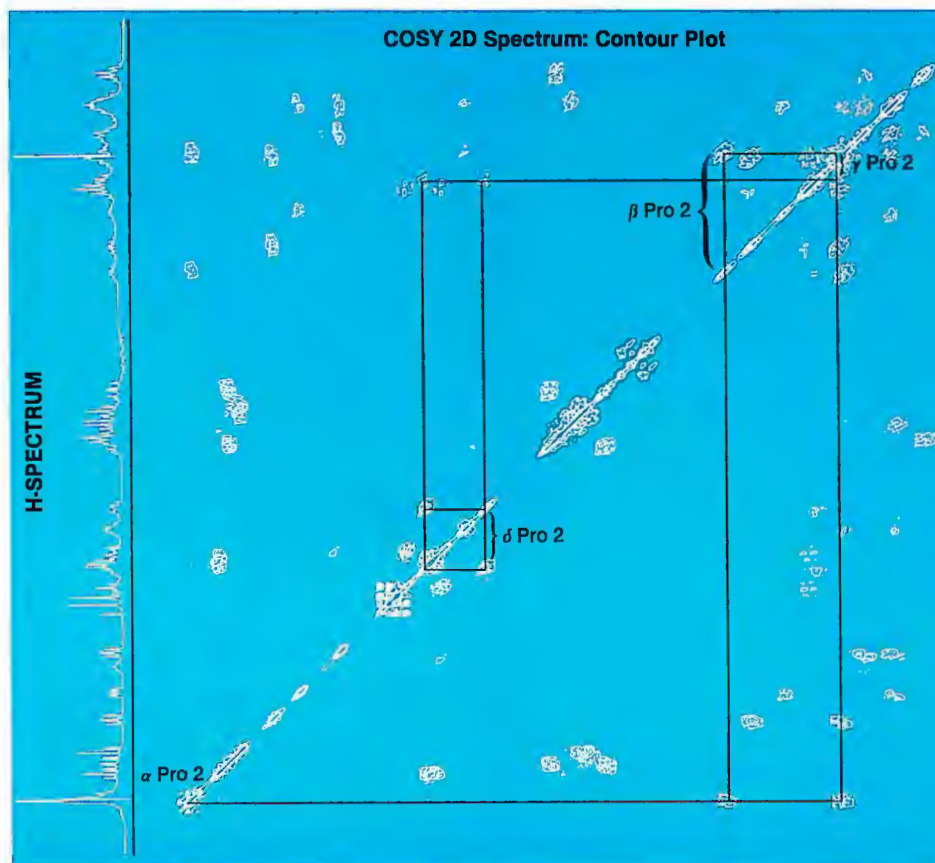
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n-hexatriacontane- d_{74} in a nematic solvent (ZLI1167). 50° C.

C₅₀H₇₃N₁₅O₁₁ by 2-D NMR

When complex molecules are submitted to analysis—e.g. the oligopeptide Bradykinin sample in this experiment—two-dimensional spectra such as proton correlated spectroscopy (COSY) can often produce dramatic simplification of seemingly intractable spectra, even at high magnetic field. Here is evidence:



Chemical Shift Assignments Using 2D Proton Correlated Spectroscopy (COSY).

The pulse sequence for COSY is given by:

$$90^\circ_x - t_1 - 90^\circ \pm x - \text{Acq}(t_2) \pm y \quad (1)$$

The chemical shifts of mutually coupled protons can be extracted from over-lapping multiplets by means of a contour plot obtained from the COSY Experiment Data. The normal Proton Spectrum is represented along the diagonal axis, and the symmetrical off-diagonal cross peaks provide the clue to any coupled protons.

The COSY Contour Plot of Bradykinin triacetate (Arg-Pro*-Pro-Gly-Phe-Ser-Pro-Phe-Arg), illustrates the technique on the aliphatic moiety. With the known position of the H α -Pro 2 (*), the chemical shifts of all the six remaining protons (2 β ; 2 γ ; 2 δ) can easily be located as shown by the connecting lines.

(1) Ad Bax and Ray Freeman, JMR 44, p. 542-561 (1981)

Q.E.D. Both spectrum and contour plot of this COSY experiment were produced on a WM 400 at the Bruker Applications Laboratory. The WM Series of high-field NMR spectrometer systems comes with an extensive software system, including programs for 2-D processing display and plotting. A full-color graphic display processor further facilitates speed and clarity of stack and contour plots.

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New Literature Available from BRUKER

NMR-Tomography

— A simple introduction into a fascinating NMR technique —

The "NMR-Imaging" technique is without any doubt a revolutionary new method for obtaining pictorial information about internal structures e.g. of the human body. The evolution of this method has now reached the state where non-specialists have recognized the extraordinary power of this technique and consequently BRUKER has now available an introductory six-page brochure for those not familiar with this new method. In order to facilitate the understanding of the physical background to this method the basic principles are given in a simplified manner and are illustrated by a large number of figures.

In a short survey it is shown that for the last twenty years the instrumental development in the pulsed NMR field has been synonymous with the name of BRUKER and it is pointed out that the first commercially available Fourier Transformation (FT) spectrometers were developed by BRUKER in 1969. Since NMR tomography is based on both "pulsed" and "FT"-NMR, the unique experience of BRUKER in these fields represents the ideal basis for the recently developed imaging systems.

After a short introduction, the principles of NMR are described in the brochure followed by a short representation of the "Projection-Reconstruction-Technique". Due to the expected extraordinary importance of NMR tomography in the field of diagnostic medicine a comparison of the average X-ray tissue contrast with NMR data is given, as well as some remarks about theoretically possible risks for patients. At the end of this brochure an "outlook" is given into new applications and of the expected development of NMR tomography.



The three new BRUKER brochures.

With the general title "BRUKER Info", periodically illustrations of BRUKER's latest results are added to the NMR Tomography brochure.

If you wish to obtain the new brochure containing two "BRUKER Info" illustrations please return the reply card.

Two-Dimensional NMR aspect 2000

A practical introduction into this new technique by an experienced spectroscopist.

The common 2-D experiments are described, measuring conditions and microprograms are given. Application examples on various spectrometers demonstrate the capabilities of the method and naturally the outstanding performance of BRUKER spectrometers in 2-D spectroscopy.

DEPT

Distortionless Enhancement by Polarization Transfer

A new method with significant advantages over other polarization transfer techniques is described in a new brochure.

This method developed at the Griffith University by Drs. Bendall, Doddrell and Pegg can be performed on any BRUKER Spectrometer equipped with a CXP or high speed pulse programmer. Using this sequence the sensitivity in coupled spectra can be significantly increased or the multiplicity selection in ^{13}C spectra can be performed without the critical adjustments required for other polarization transfer pulse sequences.

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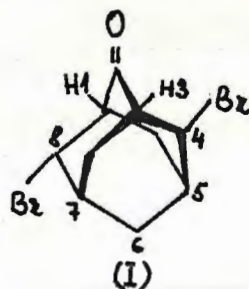
3.6.1982
č. j. : 681/82

Dear Prof. Shapiro,

recently we have studied

"Limiting Induced Shifts of 4^a, 8^e-dibromadamantanone(I) "

We found that the LIS /Eu(FOD)₃/ of hydrogen atoms H1 and H3 are different and similarly as in the case of 4-brom¹ and 4-methyl² adamantanones (II,III) this difference lies out off experimental errors.



	comp.	(I)	Table 1 (II ^a)	(II ^b)	(III)
LISH1/LISH3		1.08	1.17	1.06	1.12

Our friends in Belgium studied these compounds by single crystal X-ray analysis. For us the interesting results were that the distances H1-O and H3-O are same in the range of experimental error so that the different LIS can not be explained by the distortion of adamantanone skeleton. The calculation of the position of Eu in complex adduct(pseudocontact model) shows nonsymmetric coordination to the oxygen atom(with respect to H1 an H3). The calculation was fixed with coordinates obtained as from X-ray analysis as with the ideal adamantanone ones.

The best explanation of nonsymmetric coordination is likely due to the bulky substituent at carbon C4(C9). This assumption can be demonstrated on the 4^a - and 4^e - bromadamantanones(II^a and II^b) ,Table 1, where the higher value of the ratio LIS H1/H3 was measured for Br in axial position than for Br in the equatorial one.

Milan Hájek
Milan Hájek

Sincerely

Alena Horská
Alena Horská

Prague Institute of Chemical Technology, Czechoslovakia

- 1.L.Vodička,J.Tříška,M.Hájek,Coll.Czech.Chem.Commun.45,2670(1980)
- 2.D.J.Raber,C.M.Janks,M.D.Johnston,N.K.Raber,J.Amer.Chem.Soc.102,6591(1980)



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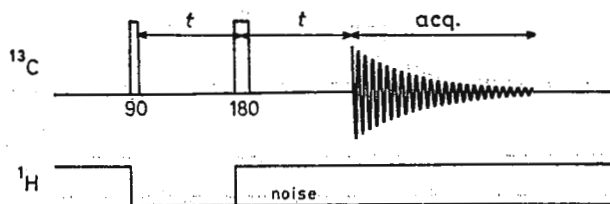
82/06/03133

AMSTERDAM, June 3rd, 1982

Dear Professor Shapiro,

^{13}C NMR MULTIPLICITY DETERMINATION USING BRUKER BNC-28 SOFTWARE

The recently described pulse sequence ¹ (see figure) for recognition



$$t = 1/J(^1\text{H}, ^{13}\text{C}) = 8 \text{ msec.}$$

of spin multiplicity (e.g. CH_3 and CH +ve, CH_2 and C_{quat} -ve) can be adapted for use with BRUKER BNC 28 software (S00500 UR) the appropriate annotated pulse program being:-

T1 = 30 000 = R2 (decoupler off after $\sim 5T_1$)
T2 = 0 = P3 (90° ^{13}C pulse)
T3 = 8 = S2 (decoupler on after t sec.)
T4 = 0 = P2 (180° ^{13}C pulse)
T5 = 8 = SC (pulse 1 + acquisition after t sec.)
T6 = -1

The following parameters are also necessary;

Machine mode (MM) = QD = 11 = CP

Decoupler mode (DM) = GD

Delay before acquisition (DE) = 0

../cont'd...

Pulsewidth 1 = 0.1 μ sec.

" " 2 = 180^0 (24.0 μ sec. on our machine)

" " 3 = 90^0 (12.0 μ sec. on our machine)

Pulse 1, necessary to start acquisition, must not disturb the spin system so its width is less than the 0.5 μ sec. necessary to activate the pulse generator. If the 90^0 pulse has been accurately determined only minimal phase correction will be necessary though in some cases a strong unambiguous peak e.g. TMS is helpful.

A shorter sequence:-

T1 = 30 000 = R2

T2 = 0 = P2 (90^0)

T3 = 8 = S2

T4 = 0 = SC (180^0)

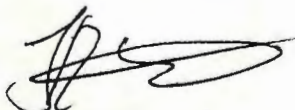
T5 = -1

can also be used but then it is necessary to represent the second t of 8 msec. by the corresponding number of left shifts in the FID [N = Data points (FID). 8 (t) / Acquisition time (AQT)].

1. C. Le Cocq and J-Y. Lallemand, J.C.S. Chem. Comm., 150 (1981).

Sincerely yours,

D.M. Grove



Dr. D.M. Grove Ing. J.M. Ernsting

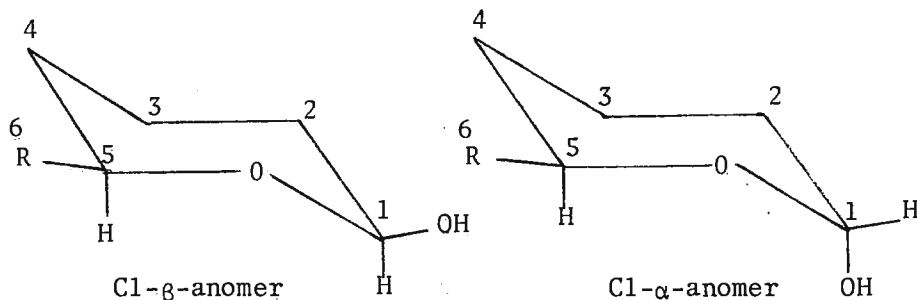
Prof. B. L. Shapiro
 Dept. of Chemistry
 Texas A & M University
 College Station
 TX 77843

June 7, 1982

TRITIUM NMR STUDIES ON ^3H -LABELLED L-FUCOSES

Dear Prof. Shapiro:

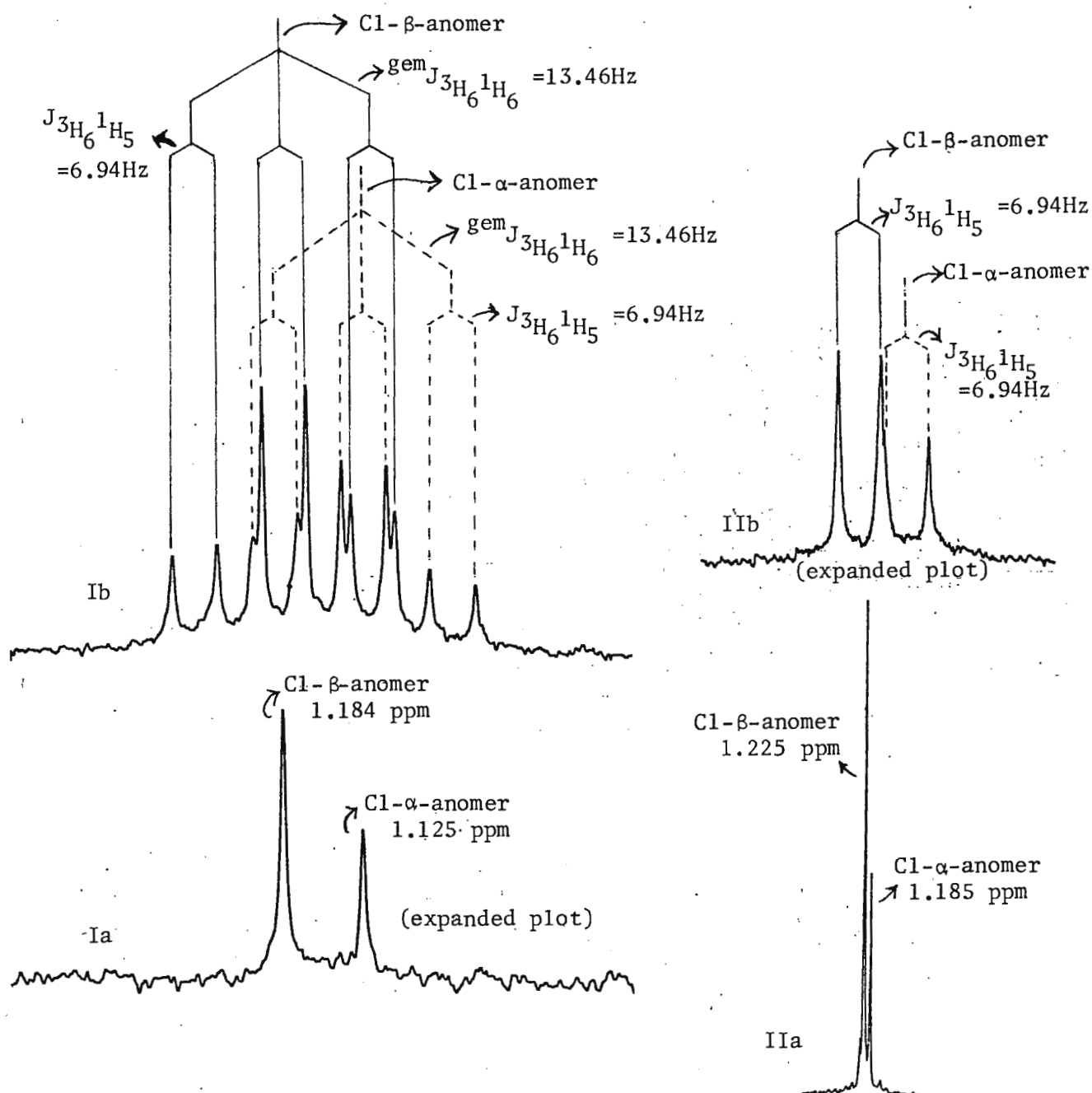
Our efforts are constantly directed towards adding new tritiated compounds to our catalog and improving the specific activities of existing products. All such labelled compounds are routinely analysed by ^3H nmr spectroscopy at 213.47 MHz on our Bruker WP 200 nmr spectrometer equipped with the ASPECT 2000 Computer. While most of them are run with proton noise-decoupling to eliminate ^3H - ^1H couplings resulting in simplified tritium spectra, occasionally ^1H -coupled spectra are run (by just turning the decoupler off) to get detailed information about ^3H -labelling patterns. Sometime ago we had run the tritium spectra of 6-mono- and 6-tri-tritiated L-Fucoses. While the ^1H -decoupled spectra essentially look alike for the two compounds (Figures Ia and IIa; sorry for the different plotting scales!), the ^1H -coupled spectra clearly bring out the difference: the 6-mono-tritiated compound shows a multiplet including geminal $^3\text{H}_6$ - $^1\text{H}_6$ couplings (Fig.Ib) which are absent in the ^1H -coupled spectrum of the 6-tri-tritiated compound wherein only the vicinal $^3\text{H}_6$ - $^1\text{H}_5$ couplings are seen (Fig.IIb). (All spectra were run in CD_3OD employing the "ghost referencing" procedure).



I, low specific activity L-Fucose[6- ^3H] ($\text{R}=\text{CH}_2$ ^3H)

II, high specific activity L-Fucose[6- ^3H] ($\text{R}=\text{C}^3\text{H}_3$)

(hydroxyl group on positions 2, 3 and 4 are omitted)



213.47 MHz ^3H nmr spectra:

Ia: ^1H -decoupled spectrum of low specific activity $[6\text{-CH}_2^3\text{H}]\text{-L-Fucose}$;

Ib: ^1H -coupled spectrum of above; IIa: ^1H -decoupled spectrum of high specific activity $[6\text{-C}^3\text{H}_3]\text{-L-Fucose}$; IIb: ^1H -coupled spectrum of above.

With best regards,

Sincerely,

Douglas Moakley Lawrence Thomas Srinivasan

Douglas Moakley

Lawrence P. Thomas

P. R. Srinivasan



UNIVERSITY OF UMEA
Department of Organic Chemistry

Umeå 1982-06-08

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

Dear Barry,

Avoid Carbon Tetrachloride as Solvent in PARR Experiments.

Several applications have recently appeared using paramagnetic relaxation reagents to obtain structural information of organic molecules in solution¹. Carbon tetrachloride has by far been the most used solvent in these studies partly because of a suitable PARR solubility in this solvent. The main reason however for this choice has been the expected inertness of this medium.

First of all, the most commonly used PARR, $\text{Cr}(\text{acac})_3$, is far from shift inert in carbon-13. We have observed intramolecular changes up to .5 ppm using .05 M $\text{Cr}(\text{acac})_3$ and internal referencing (cyclohexane). Secondly, using various weakly interacting substrates in CCl_4 solution we failed to observe any substrate concentration dependence on T_1^e . Hence, this last observation made us reconsider if carbon tetrachloride really is non-interacting.

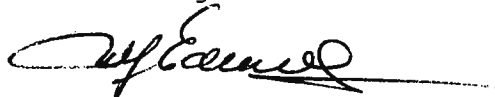
First we found that the effective equilibrium constant for chloroform: $\text{Cr}(\text{acac})_3$ complexation increased by going from CCl_4 to a pure hydrocarbon solvent. Moreover, one can, by assuming a small step diffusion mechanism, calculate a C-13 T_1^e of 1.23 s for CCl_4 (.05 M $\text{Cr}(\text{acac})_3$, room temp., 5.9 T)². This value is significantly larger than the experimental value, .8 s, but close to the experimental value for the central carbon in neopentane (1.27 s).

The final indication of a specific PARR: CCl_4 interaction comes from experiments, where the size of the chelate ligands was increased³. By going through the series, propanedionato, 2,4-

pentanedionato and 3-methyl-2,4-pentanedionato, we observed a significant decrease of the T_1^e (CCl_4) : T_1^e (cyclohexane) ratio. The T_1^e of the CCl_4 is also shortest having the most substituted chelate, $\text{Cr}(\text{3-Meacac})_3$. This trend is opposite to what could be expected assuming a translational diffusion relaxation mechanism.

Thus the lack of substrate concentration dependence on T_1^e in a carbon tetrachloride solution should not be taken as the sole evidence that the relaxation of the substrate nuclei is governed by translational diffusion. Carbon tetrachloride can compete effectively with the substrate in the second coordination sphere.

Best regards



Ulf Edlund

1. T. A. Holak and D. W. Aksnes, J. Magn. Reson., 45, 1 (1981) and references therein.
2. J.G. Hexem, U. Edlund and G. C. Levy, J. Chem. Phys., 64, 936 (1976).
3. U. Edlund and H. Grahn, Org. Magn. Reson., 18, 207 (1982).

Berne, june 6,1982

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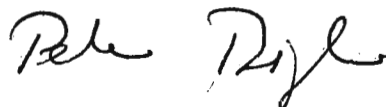
Dr. Bernard L. Shapiro
Department of Chemistry
Texas A&M University
College Station, TX, 77843

FID-Difference Techniques for XL-100 Users

Dear Dr. Shapiro,

there exist very useful NMR experiments for structure elucidation based on FID-Difference techniques as for instance NOE-Difference or Decoupling-Difference. With slight modifications of our homebuilt accessory for switching the ^1H -Decoupler (offset, mode) on an XL-100 in unattended operation (see VARIAN INTERFACE Vol 7, 1980) and the implementation of the saleable CAPS-Kit for rf-phase shifts, the FID-Difference experiments are accessible. In the case of homonuclear NOE experiments the decoupler offset, which is on only during a suitable pulsedelay, is switched alternately on/off resonance with a frequency selected for irradiation. The difference spectrum is built up simply by adding/subtracting alternately the FIDs- the sum must be an even number- without shifting the phase of the observe transmitter. This means, that only the software for changing the sign of alternately incoming FIDs is used, while the phase shifting properties of the CAPS unit are suppressed. The experiments are done with the standard disk based SYMON-program. For anyone interested, we provide further details.

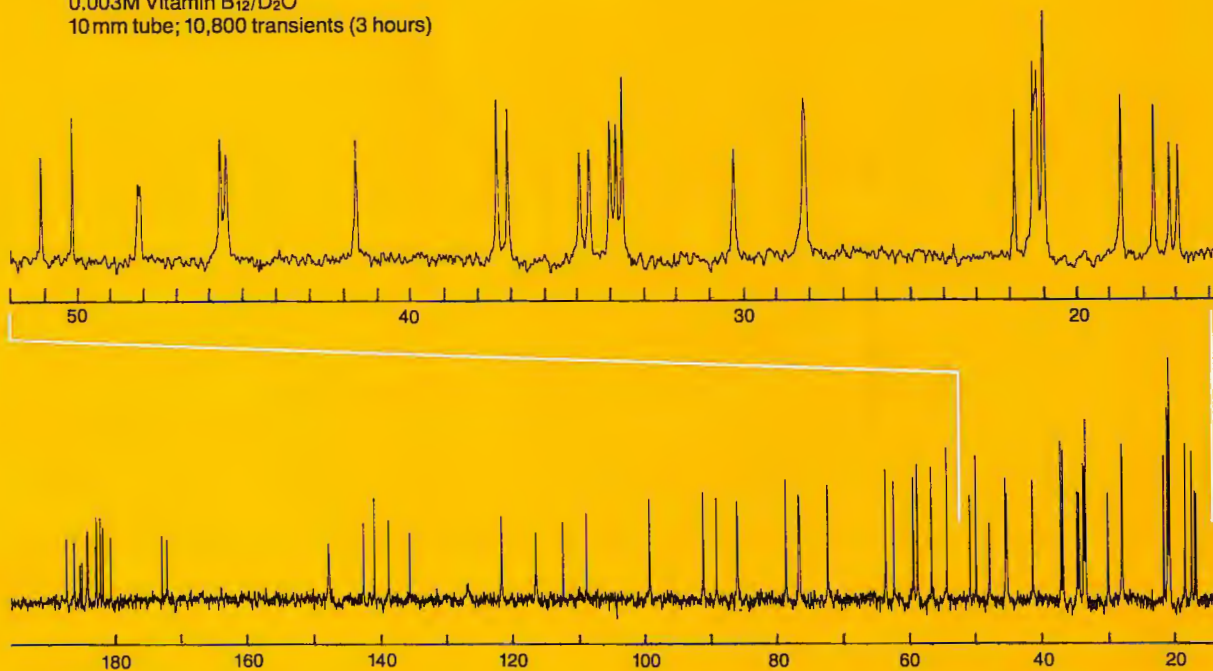
Yours



Peter Bigler

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Département de Recherche Fondamentale de Grenoble

Grenoble, le 18 juin 1982

N/référence : DRF/CH/82-251/mjc
your letter June 7 1982

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

Dear Professor Shapiro,

Data Transfer from an Aspect 2000 to a larger computer

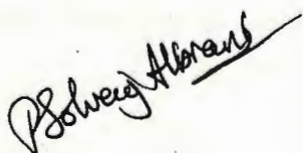
Some of your readers might be interested by our local solution to the problem of transferring NMR spectra from an Aspect 2000 Bruker minicomputer to a larger institutional computer. This is achieved here by sending the data, converted into ASCII code, via the RS232 CH-B output connector at the back of the Aspect 2000, unto a TECHTRAN 950 minidisk unit. These minidisks can be eventually read on another TECHTRAN unit interfaced to the PRIME computer of our Department. The writing of the data on the TECHTRAN unit is done by a program which was written in assembly language as an overlay to the Bruker FC.810515 NMR program. This overlay can be called by the U1 command which has been implemented for users' written programs. We are using a transmission rate of 4,800 bauds and for our particular needs, the writing of a 4k spectrum takes about 1 mn. More information about this program will be sent upon request.

Yours sincerely,

P.S. ALBRAND

J.P. ALBRAND

C. TAIEB



286-17
USSR Academy of Sciences
Shemyakin Institute
of Bioorganic Chemistry

Ul. Vavilova, 32
117988 Moscow, B-334
USSR

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

May 25, 1982

Title: Exposed Groups
Detection in Protein.

Dear Barry,

In order to delineate the exposed and buried groups of polypeptide neurotoxin II isolated from the Central Asian cobra venom we have added a paramagnetic probe - nitroxyl radical TEMPONE in the solution. The aim of the experiment was to create an environment for the neurotoxin II molecule that would induce paramagnetic relaxation of those protons located on its periphery. EPR spectra showed that the spin probe does not bind specifically to neurotoxin II so that its interaction with the molecule must be mainly by collision. This is also confirmed by the linear dependence of the proton signal broadening on the TEMPONE concentration.

From the aromatic part of the spectrum (Figure) follows that the Trp-28 signals are the most influenced by the spin probe addition whereas Tyr-25 - the least. Anomalously low field backbone C^αH proton signals in the region of 5,4-5,3 ppm didn't broaden obviously due to shielding by the side chain groups. Among the methyl groups one could detect the most exposed nature of the Thr-35, Ile-37 and Val-50 side chains as well as that Leu-1, Leu-52 and yet unidentified Thr-f are buried.

The results thus obtained are in full accordance with the spatial structure of the neurotoxin II molecule in solution (1) and corresponding erabutoxin b X-ray structure (2). The detailed account of this work will appear in the Bioorganicheskaya Khimia journal (in Russian first).

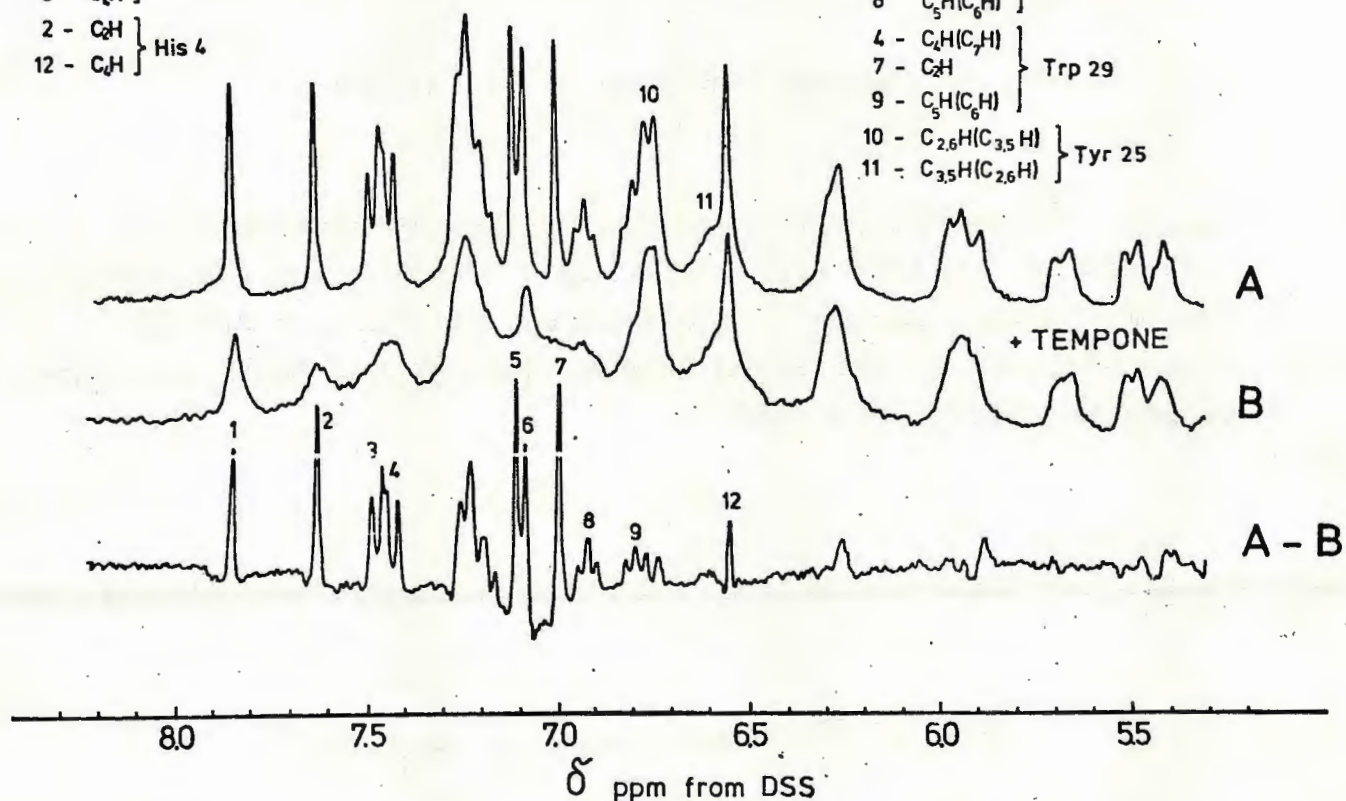
Sincerely yours,



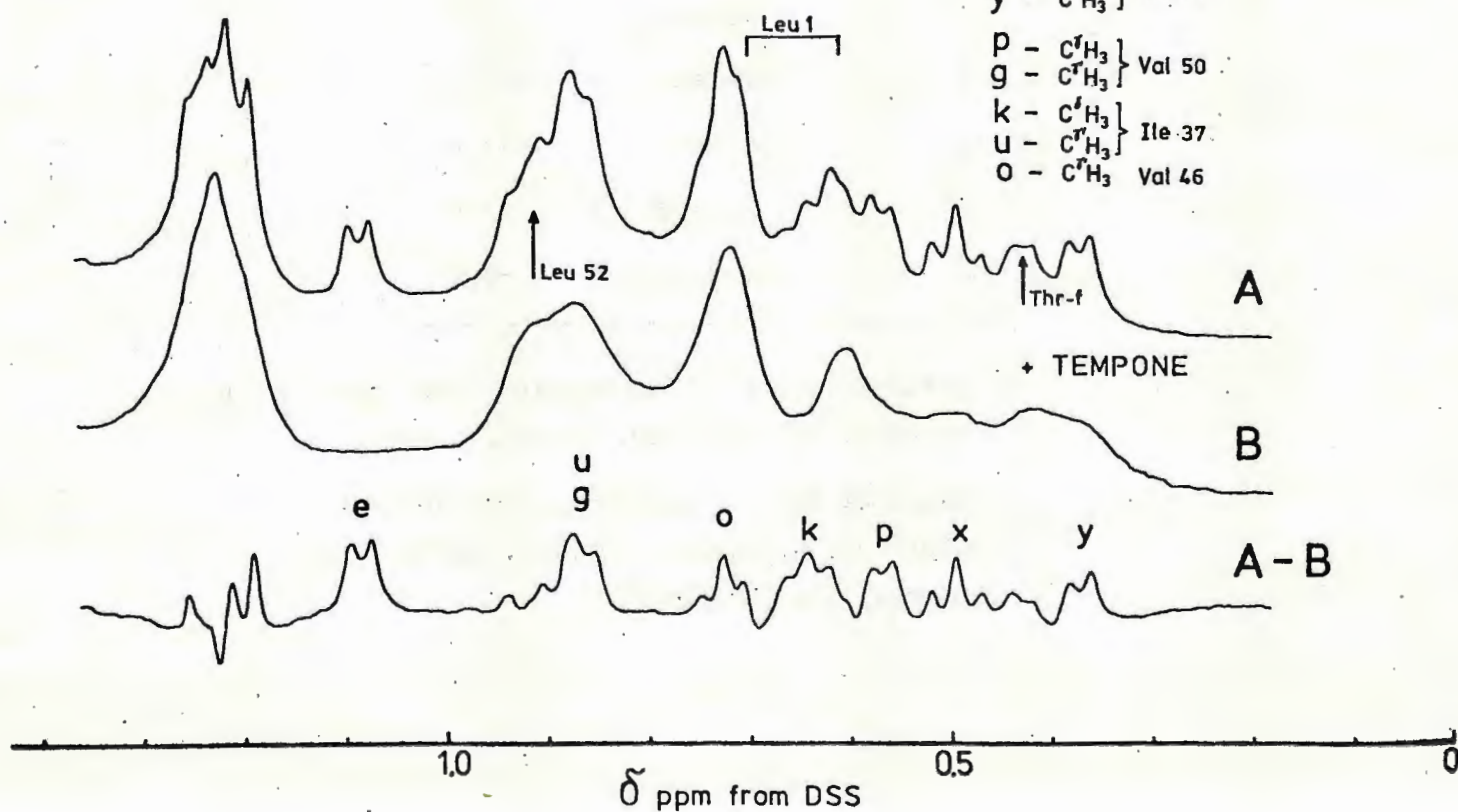
Vladimir Bystrov

1 - C_2H } His 37
 6 - C_4H }
 2 - C_2H } His 4
 12 - C_4H }

3 - $C_4H(C_7H)$ }
 5 - C_2H } Trp 29
 8 - $C_5H(C_6H)$ }
 4 - $C_4H(C_7H)$ } Trp 29
 7 - C_2H }
 9 - $C_5H(C_6H)$ }
 10 - $C_{2,6}H(C_{3,5}H)$ } Tyr 25
 11 - $C_{3,5}H(C_{2,6}H)$ }



e - C^rH_3 Thr 35
 x - C^rH_3 } Ile 36
 y - C^rH_3 }
 p - C^rH_3 } Val 50
 g - C^rH_3 }
 k - C^rH_3 } Ile 37
 u - C^rH_3 }
 o - C^rH_3 Val 46



- (1) V.F.Bystrov et al., in Advances in Solution Chemistry
(Eds. I.Bertini, L.Lunazzi, A.Dei) Plenum Press, 1981,
pp. 231-235.
- (2) B.L.Low, Adv.Cytopharmacology 3, 141 (1979).

Figure: The 300 MHz proton spectra of the 6mM neurotoxin II solution in D₂O (pH* 7.0 32°C). Top - aromatic and C^αH region, Bottom - methyl region of the spectra. (A) Without and (B) after addition of the 24 mM TEMPONE spin probe. (A-B) The corresponding difference spectra.

²⁹Si chemical shifts of (CH₃)₃SiX^a

N°	X	δ ²⁹ Si
1	CH ₂ =C=CH	-5.9 ₅
2	CH≡C-CH ₂	2.7
3	CH ₃ -C≡C	-19.5 ₅
4	C ₆ H ₅ -C≡C	-17.9 ₅
5 ^b	CH ₂ =CH	-7.6
6 ^b	CH ₂ =CH-CH ₂	-0.4

^a In ppm relative to TMS signal placed in a concentrical capillary. solvent : CDCl₃.

^b Values of Ref. : J.Schraml, V.Chvalovsky, M.Mägi and E.Lippmaa, Collect. Czech. Chem. Commun., 42, 306 (1977).



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E. J. VINCENT

PROFESSEUR

Marseille , 8 june 1982

Professor B . L SHAPIRO
Department of chemistry
Texas A & M University
College Station , Texas 77843

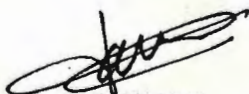
Dear Professor SHAPIRO ,

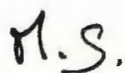
Silicium-29 chemical shifts of allenyl or ethynyl trimethylsilanes have never been reported in the literature as far as we know. This lack of data prompted us to present results concerning this kind of molecules. In the purpose of comparison , ^{29}Si shifts of allyl and vinyl trimethylsilanes are also presented.

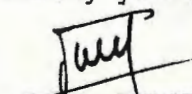
The most striking features of these data are the next :

- δ_{Si} of 1 and 5 are quite similar, although silicium is slightly more shielded for vinyl. This latter effect is induced by the weak χ interaction with vinyl proton for 5 .
- In the case of acetylenic compound 3 and 4 , ^{29}Si is more shielded than for vinyl 5 , due to the strong magnetic anisotropic effect of triple bond.
- Finally, ^{29}Si in propargyl trimethyl silane is more deshielded than in allyl derivative. In 2, according to geometrical aspect, silicium atom, on an average , lies out of shielding cone and, therefore, is deshielded

Sincerely yours


R. FAURE


M. SANTELLI


E. J. VINCENT

University of Illinois at Urbana-Champaign

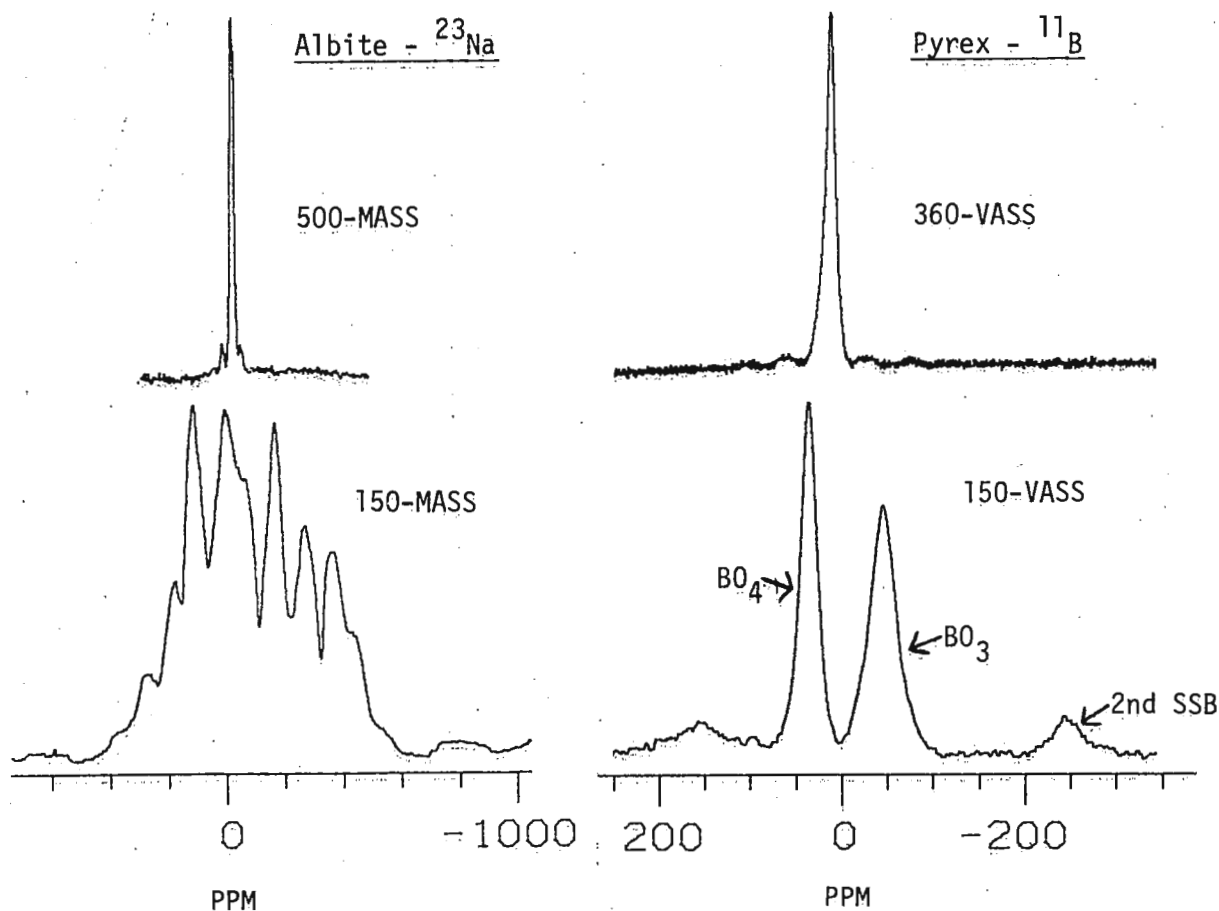
School of Chemical Sciences
505 South Mathews Ave.
Urbana, IL 61801

Dear Barry:

June 14, 1982

QUADRUPOLEAR NUCLEI: THERE ARE NO EASY ANSWERS

How to "do" quadrupolar nuclei is becoming complicated, we find. Are high-fields optimum, or low fields? Should one use conventional "magic-angle" sample spinning, or spin at some other angle to reduce second-order broadening? As the title says, there is no universal panacea. The following shows excellent line narrowing for ^{23}Na in albite (a rock) at 11.7 Tesla, and a very complicated result at 3.52 Tesla, using conventional MASS. This result is expected on the basis of decreased second-order interaction at high-field. On the other hand, high field operation (MASS or VASS) on ^{11}B in a borosilicate glass produces poor results, and only at low-field using VASS does one obtain high-resolution, due to the presence of a quadrupole-induced shift (QIS) effect.



Eric

Eric Oldfield

Suzanne Schramm

Suzanne Schramm

Bob

Robert A. Kinsey

Karen Ann

Karen Ann Smith

National Institutes of Health
Bethesda, Maryland 20205

15 June 1982

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

$^3J_{CH}$ Modulated Spectra on an FX60

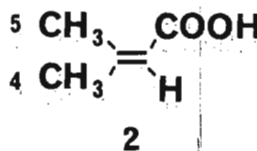
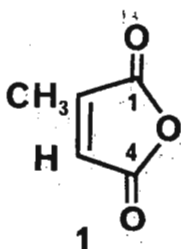
Dear Barry,

One of the challenges facing a group operating a versatile modern spectrometer is that of pushing as much work as possible onto older, more available, machines. Here is a procedure utilizing J-modulation which works on our 6-year-old FX60.

Vicinal CH couplings are a good way to characterize trisubstituted double bonds (trans > cis), but coupled ^{13}C spectra are often unintelligible. Bax and Freeman have described an elegant 2D method providing these valuable figures, (JACS 104, 1099 (1982)) but it requires the same versatile spectrometer which is monopolized by the biophysicists of the Institute. Recent descriptions of $^1J_{CH}$ -modulated spectra suggested a way to substitute the FX60.

Allowing the nuclear precessions to develop for a period, $t, = 1/2J$ after a 90° pulse at the ^{13}C frequency before proton decoupling is started puts the two components of a doublet along the x' axis of the frame rotating at the center frequency. Acquisition with decoupling now provides an FID which FT converts into a spectrum with the appropriate signal nulled. On the FX60 this is accomplished with the T_1 sequence, using 1 usec for the second pulse, as the best approximation possible to no pulse. To maintain the NOE, collection parameters must be set to keep the acquisition time close to a suitable repetition period.

The upper spectra from citraconic anhydride, 1,

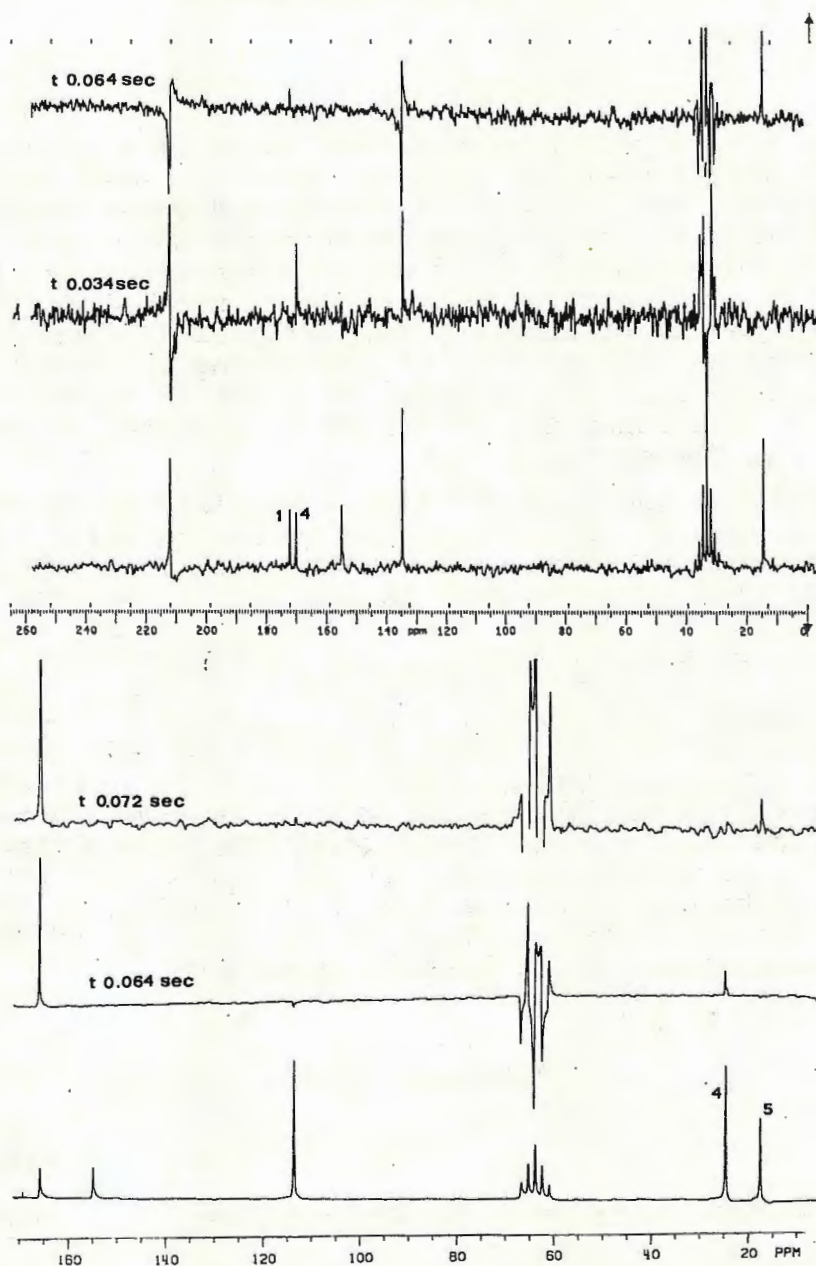


show that the downfield carbonyl, being nulled by a pause of 34 msec., is trans to the olefinic proton, ($^3J_{CH} = \text{ca. } 14 \text{ Hz}$), while the other is nulled by a pause of 64 msec. ($^2J_{CH} = \text{ca. } 8 \text{ Hz}$). The lower spectra are those of dimethylacrylic acid, 2, and show that protonated carbons can be studied, as long as the effects of J_{CH} modulation are considered -- i.e., pauses near $(2n-1)/2 J_{CH}$ are avoided.

Yours very truly,

Bob

R. J. Highet



DIVISION OF CHEMISTRY AND PHARMACY
UNIVERSITY OF MUNICH
8000 MUNICH 2

INSTITUTE OF ORGANIC CHEMISTRY
KARLSTRASSE 23

GERHARD BINSCH
PROFESSOR OF THEORETICAL ORGANIC CHEMISTRY

June 25, 1982

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

Computer Program RELAXAN

Dear Barry :

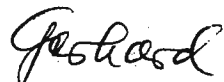
This is a very preliminary announcement of a general NMR relaxation analysis computer program which has been in the works in our laboratory for the past 10 months and whose current working title is RELAXAN. It is designed to be applicable to a variety of selective and nonselective pulse experiments for measuring longitudinal and transverse relaxation effects in coupled spin systems as well as to bandshape studies, including saturation phenomena, and it is based on the strategy of exploiting the total information content (not just line intensities) of a set of spectra for the analysis, i.e. the iterative algorithm is supposed to operate directly on the raw spectral data.

To finish the job we still have a long way to go and it may well turn out that we will eventually decide to split the whole thing into several distinct program versions, in which case there would also be name changes. What is now fairly complete is a common subroutine package for the calculation of WBR relaxation supermatrices, capable of handling any relaxation mechanism (including all cross terms) and applicable to general spin systems composed of nuclei with quantum numbers up to $7/2$ (there are of course dimensional limits). Simplifications arising from symmetry and the weak coupling approximation can be taken into account. We are currently in the process of writing a paper on that aspect of our work, but the package itself will not be published as such. However, if there should be somebody who cannot wait for the final product and if he can argue his case convincingly, we would be prepared to let him have a listing of the thing as it is now (but without documentation, which doesn't exist yet).

Sincerely yours,

Slawomir Szymanski

Adam M. Gryff-Keller


Gerhard Binsch





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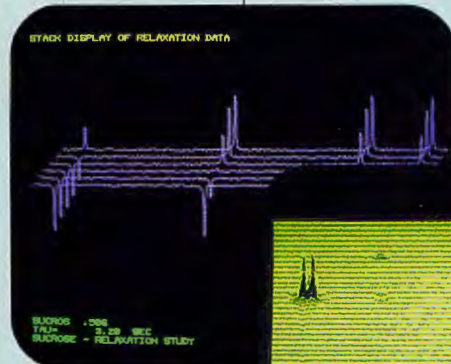
Magnets range from 4.7T to 11.7T (200, 300, 360 and 500 MHz) in both wide and narrow bore. Probes available include fixed-tune, broadband, special cross-polarization/magic angle spinning and the new sideways-spinning solenoidal.

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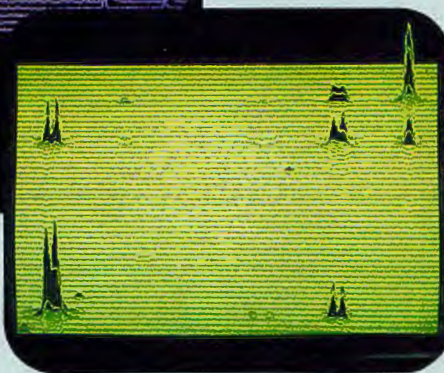
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A NICOLET INSTRUMENT SUBSIDIARY

286-28

June 30, 1982

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

Re: Engineer Positions, NMR

Dear Prof. Shapiro:

Nicolet Magnetics Corporation has two openings for people with Nuclear Magnetic Resonance experience.

The Service Engineer position is responsible for the installation and servicing of Nicolet NMR Systems, and is permanently located in the eastern United States. This position requires NMR or extensive RF and digital experience, as well as availability for 100% travel during the week.

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Would you please post this announcement or pass it along to anyone who might be interested? Candidates should send a letter and resume to:

Toni Wilson
Nicolet Magnetics Corp.
255 Fourier Ave.
Fremont, CA 94539

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Yours truly,

Toni Wilson
Personnel Manager

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University Park, Denver, Colorado 80208

Department of Chemistry / 303-753-2436

June 15, 1982

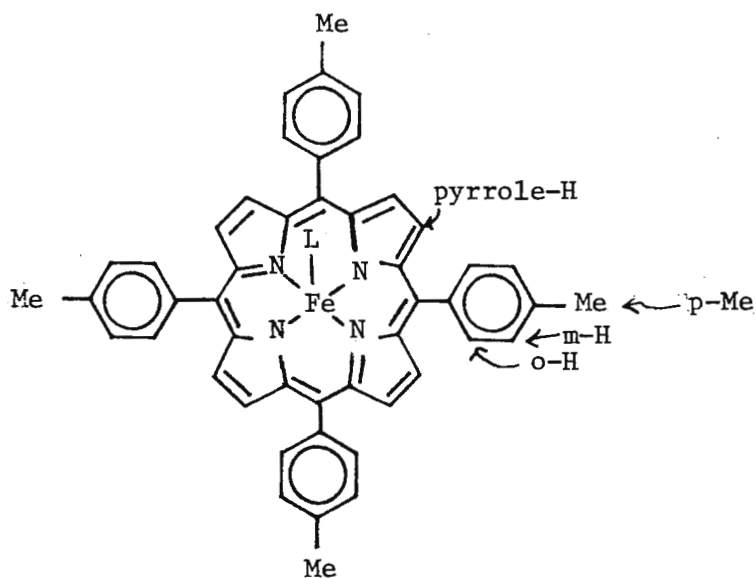
Professor Barry Shapiro
Department of Chemistry
Texas A & M University
College Station, TX 77843

Evidence for existence of FeTTPOH en route from FeTTPCl to (FeTTP)₂O

Dear Barry:

Attempts in the late 60's and 70's to prepare FeTTPOH (iron tetratolylporphyrin hydroxide) by shaking a solution of FeTTPCl with base led to isolation of (FeTTP)₂O, the oxo-bridged dimer. More recently there have been reports of iron porphyrins with axial hydroxides when there are bulky substituents on the phenylrings which prevent dimer formation. In our EPR studies of spin-spin interactions between iron unpaired electrons and nitroxyl radical unpaired electrons we noted that after shaking solutions of spin-labeled iron porphyrin chlorides with base, the EPR spectra and visible spectra of ca. 10⁻³ M solutions were time dependent. To sort out the events we examined the behavior of FeTTPCl. We followed the 250 MHz ¹H NMR spectra (on a Bruker spectrometer at University of Colorado at Boulder) of a ca. 10⁻³ M solution of FeTTPCl for several hours after shaking with 0.1N KOH. The initial spectra showed a species with shifts comparable to those of high-spin FeTTPCl which we assign to FeTTPOH. The splitting of the meta-H signal in FeTTPCl and FeTTPOH is due to the axial ligand on only one side of the porphyrin plane and show rotation of the phenyl rings on the NMR time scale. Similar nonequivalence should exist for (FeTTP)₂O but is not resolved. The broadening of the o-H signal is due to the short distance between the protons and paramagnetic iron center. Within an hour the NMR spectrum changed to about a 1:1 mixture of FeTTPOH and (FeTTP)₂O. Conversion to (FeTTP)₂O was complete within about 12 hours. When higher concentration solutions were used, the rate of conversion from FeTTPOH to (FeTTP)₂O increased. At ca. 0.01M conversion was complete within less than an hour. Fortunately the newer spectrometers can work at lower concentrations than their older counterparts.

Professor Barry Shapiro
June 15, 1982



Chemical shifts in ppm from TMS in CCl_4 solution

	Pyrrole-H	m-H	p-CH ₃	o-H
FeTTPCl	81.8	13.05, 11.87	6.24	~3.5(broad)
FeTTPOH	80.5	10.86, 10.04	4.87	~3.5(broad)
(FeTTP) ₂ O	13.24	7.54	2.81	7.39

Sincerely,

Gareth R. Eaton
Professor

Sandra S. Eaton
Associate Professor

GRE/SSE:jm



The Florida State University
Tallahassee, Florida 32306

Department of Chemistry

June 18, 1982

By-Passing Multipulse Programmers Enroute to MLEV

Dear Barry:

More on MLEV. Having a need to obtain more effective proton decoupling with less power dissipation for our biochemical users, the composite spin inversion sequence (1) and its various permutations into super cycles (2) promised much better performance than our current decoupling schemes. To acquire this Levitt-Freeman decoupling capability on our equipment, we chose the MLEV-16 continuous irradiation sequence:

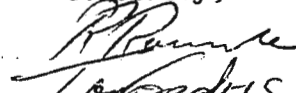
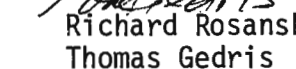
$$(R \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R} \bar{R})_n$$

as used by Levitt, Hill, et. al. in this publication (May '82), but we had additional requirements. We desired a unit which was easy and quick to construct for making copies for each of our instruments. A pulse programmer was considered expensive overkill and not conducive to multiple copies. We needed to keep the cost of each unit low.

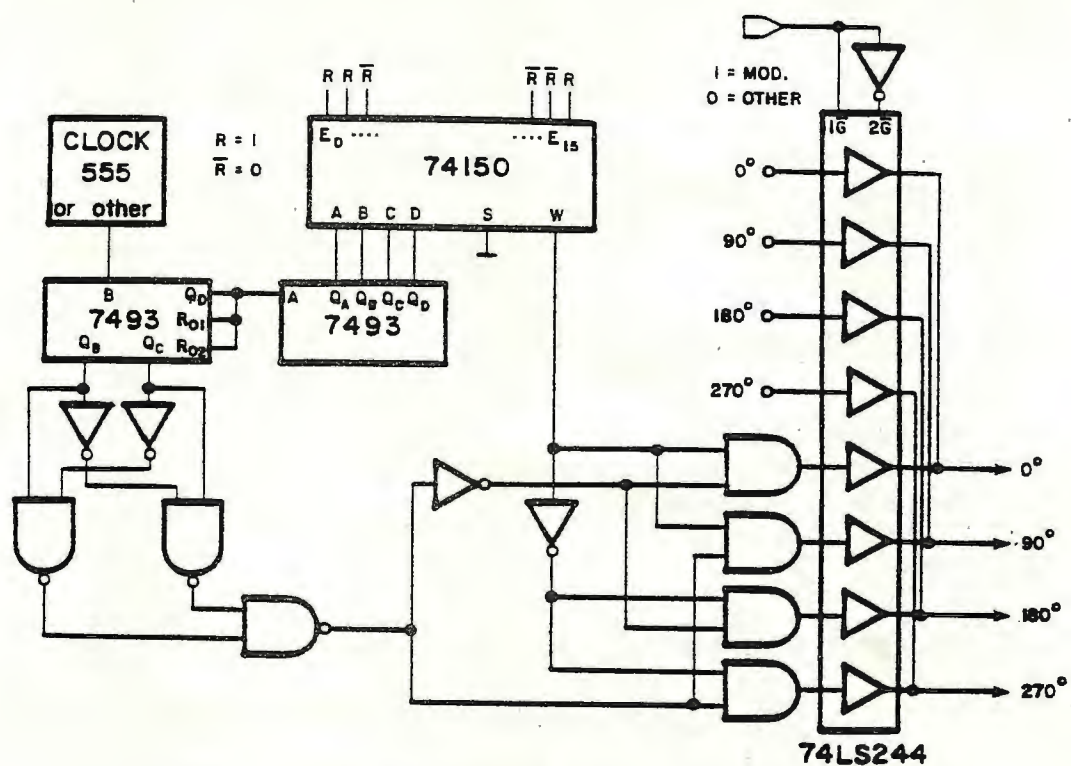
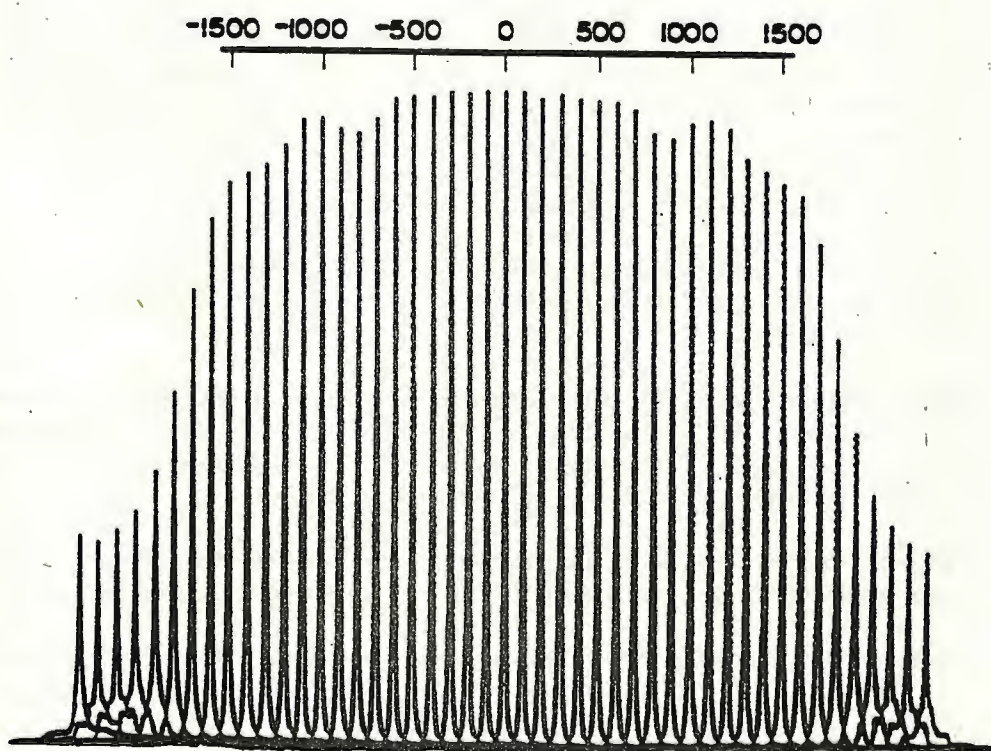
The circuit (Fig. 1) that we derived satisfies the proposed conditions. The basic "R \bar{R} " sequence is hard-wired to the inputs of a sixteen to one data selector and cycled through sequentially by a divide by sixteen counter. Each "R" value is further divided into "X-Y-X" elements to provide proper phase shifting and timing. It can be constructed very quickly and decouples a 3 KHz proton bandwidth (Fig. 2) as measured on our Bruker HX-270. Effective decoupling power was measured at $\gamma H_2/2\pi = 2$ KHz by the off resonance technique with each "R" element requiring 580 microseconds. The phase shifting was done at 30 MHz on the HX-270 system with a 4 phase modulator designed at FSU although quadriphase modulators can be acquired from commercial sources (3).

This circuit can easily be treated as a black box and accessed with a simple switch or one bit from a data system. The clock can be either a free-running oscillator or a frequency source from within the spectrometer. It is our wish to point out that those laboratories that lack expensive multipulse programmers still have reasonable access to low-cost Levitt-Freeman decoupling scheme(s). With minor modifications, MLEV-64 and other supercycles can be developed.

Sincerely,



 Richard Rosanske
 Thomas Gedris

1. M.H. Levitt and R. Freeman, J. Magn. Reson., 33, 473 (1979).
2. M.H. Levitt, R. Freeman and T.A. Frenkiel, J. Magn. Reson., 47, 328 (1982).
3. Olektron, 61 Sutton Rd., Webster, MA 01570

Figure 1. (MLEV-16)_n CIRCUITFigure 2. Broadband decoupled ¹³C as a function of proton offset (100 Hz increments) for ethylene glycol.



QUEEN MARY COLLEGE

UNIVERSITY OF LONDON

DEPARTMENT OF CHEMISTRY

Professor D.C. Bradley, FRS. (Head of Department)

Professor R. Bonnett, BSc., PhD., DSc.

Professor K.W. Sykes, MA, BSc., DPhil.

MILE END ROAD

LONDON E1 4NS

Tel. 01-980 4811

ULIRS High Field NMR Service

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

21st June 1982

Dear Professor Shapiro,

Plasma Investigations from Selective Spin Echo Data

Most contributors to this Newsletter are surely aware of the advantages to be gained from the use of the spin-echo technique to observe ^1H spectra of small molecules when they are in the presence of large molecules (e.g. amino acids in whole blood and blood plasma). This WH-400 NMR Service to the University of London has spent considerable time using the spin-echo technique on such systems provided by several bio-medical research groups who obtain samples from hospital patients with a range of metabolic disorders. The accompanying Figure illustrates the sensitivity of the technique. Spectrum A is a ^1H spin echo spectrum obtained on a sample from the starting material (a willing patient) while spectrum B clearly shows the effect of prolonged (lunch-time) exposure of the same starting material to a proprietary hallucinogen marketed by Wm. Teachers and Son, Glasgow, Scotland.

These data have in fact been presented to Her Majesty Queen Elizabeth II, who was amused (there is photographic evidence to substantiate this).

There may be obvious applications for this technique, however the average size of the automobile employed by the Metropolitan Police would seem to preclude the replacement of the famous balloon (orange contents sometimes changing to green) by a WH-400.

In attempting to continue these investigations we have unfortunately been hampered by negative responses from various sources of funding.

Please credit this contribution to the ULIRS/Ed. Randall account.

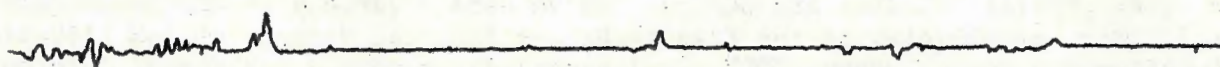
Yours sincerely,

Dr. G.E. Hawkes

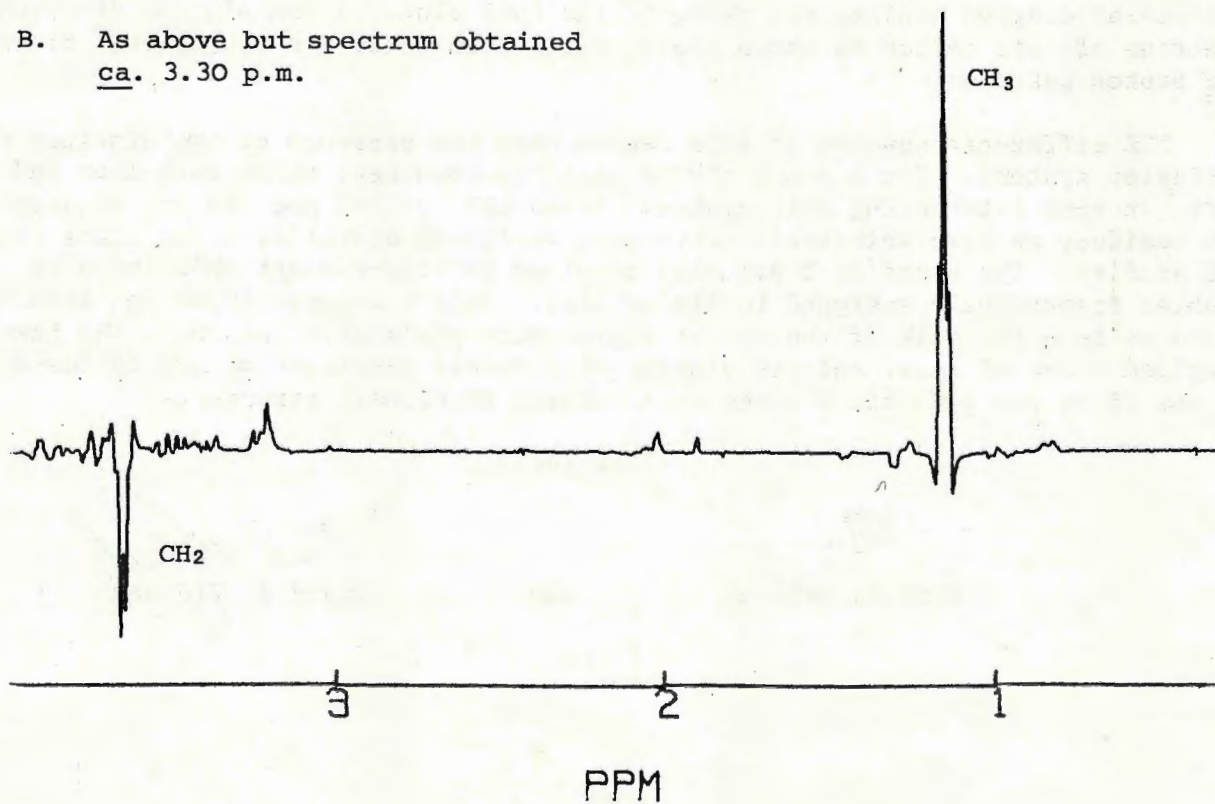
Mr. M.J. Buckingham

Figure

- A. 400 MHz ^1H spin-echo spectrum
of plasma from volunteer - spectrum
obtained ca. 10.30 a.m.



- B. As above but spectrum obtained
ca. 3.30 p.m.



MASSACHUSETTS INSTITUTE OF TECHNOLOGY
DEPARTMENT OF CHEMISTRY
CAMBRIDGE, MASSACHUSETTS 02139

June 22, 1982

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

Dear Barry,

500 MHz ^1H -NMR Games with Ra5

Since it is approaching that time of year when many of us are sneezy and bleary-eyed, it only seems appropriate that we describe some work we've begun on Ra5, a ragweed pollen allergen. Ra5 is produced by the short ragweed 'Ambrosia Elatior' and is a small protein of 45 amino acid residues (MW \sim 5000) cross-linked with 4 disulfides. Its small size and moderate solubility make Ra5 a promising candidate for NMR structural studies. Unfortunately, there is no crystal structure and other physical studies are scarce. We've used a variety of NMR techniques on the 500 MHz spectrometer at the Francis Bitter National Magnet Lab (pH titrations, NOE difference spectroscopy, COSY, 2D-J-resolved) to obtain structural information.

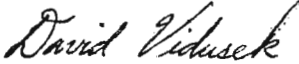
The Ra5 aromatic region (2 Trp and 3 Tyr) can be well resolved and assigned. Titration studies identify Tyr a and b sets of doublets with pKa's \sim 10.5. Tyr c appears to reside in a different type of environment - all its ring protons are submerged in the region \sim 1500 Hz. Two-dimensional correlation spectroscopy of the aromatic region is shown in the accompanying figure and connectivities of some of the scalar-coupled protons are shown on the COSY plot. A convolution difference spectrum of this region is shown above, along with tentative assignments of Trp and Tyr proton patterns.

NOE difference spectra of this region show the presence of two distinct spin diffusion systems. Tyr a and b always light-up together, while both Trps and Tyr c form another interacting spin system. Since all Tyr and one Trp are adjacent to Cys residues we have whimsically attempted assigning disulfide cross-links from the NOE studies. Tyr a and/or b are also involved in ring-current shifting a CH_3 doublet (tentatively assigned to Ile or Leu). This resonance (0.60 ppm from TSP) titrates into the bulk of the methyl region with pKa's of 4 and 10.5. We have compiled a lot of data, and are playing with models generated on a graphics system to see if we can generate a reasonable (albeit fictional) structure.

Sincerely,

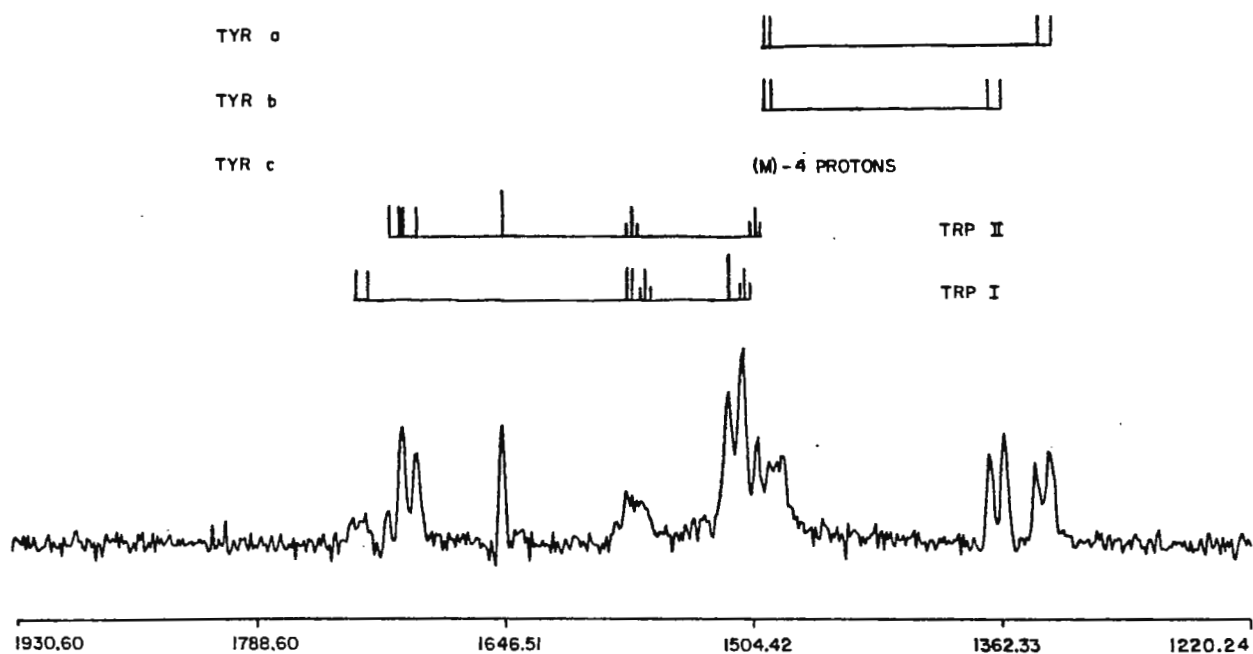

Mary F. Roberts

and

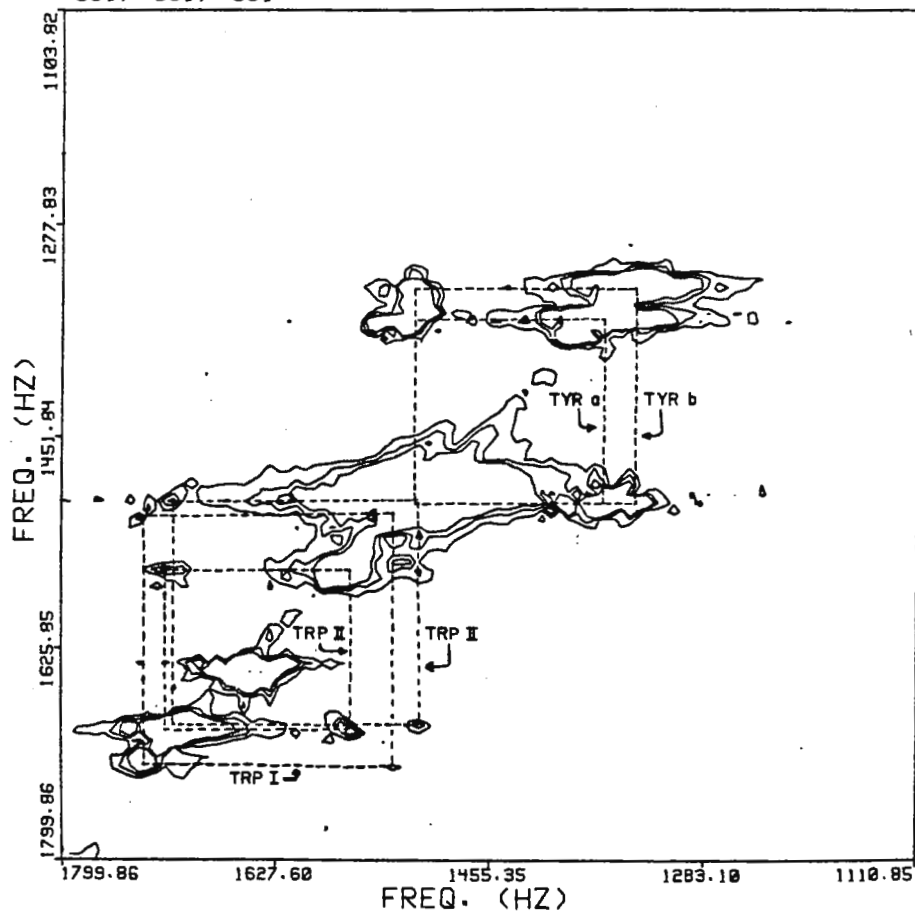

David A. Vidusek

RA5

CONVOLUTION DIFFERENCE



REC. NO. 7 BLKS 1-100 DATE: 05/28/82
RASCOSY LEVEL=.0005
.001, .001, .001



University of Illinois at Urbana-Champaign

School of Chemical Sciences
505 South Mathews Avenue
Urbana, Illinois 61801

June 23, 1982

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

Dear Barry:

POSTDOCTORAL POSITIONS AVAILABLE

I shall have several postdoctoral positions opening up in the near future, for work in the following areas:

- 1) NMR of Inorganic Solids. This person(s) will be involved with our MASS/VASS studies of quadrupolar and rare spin-1/2 nuclei in samples of geologic and industrial (catalytic) significance.
- 2) NMR of Membranes and Proteins. This position is involved with ^2H , ^{13}C , ^{15}N , ^{19}F and ^{31}P studies of membrane proteins, protein crystals, amino acids, and protein-lipid interactions.

Multinuclear solid and solution FT instruments operating at 500, 360(2), 300, 250, 220(2), 150 and 100(2) MHz will be available for the successful candidates.

Interested persons should submit a letter of application including a curriculum vitae and list of publications, and arrange to have three letters of recommendation forwarded to:

Professor Eric Oldfield
School of Chemical Sciences
University of Illinois
505 South Mathews Avenue
Urbana, Illinois 61801 U.S.A.

The University of Illinois is an Equal Opportunity/Affirmative Action Employer

Yours sincerely,



Eric Oldfield

500 MHz

PTS
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PTS 500 FREQUENCY SYNTHESIZER

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The PTS 500 is a generator of precision frequencies. It transfers the accuracy and stability of a frequency standard (built-in or external) to any output frequency between 1 MHz and 500 MHz. Steps as fine as 0.1 Hz are available and all functions are remotely programmable.

The PTS 500 is a direct frequency synthesizer of unique design providing high performance for many demanding applications. With its low spurious outputs, fast switching, low phase noise and wide choice of resolution (finest step), it is suited for a range of uses such as NMR, communications and ATE.

The PTS system of synthesis has drastically cut complexity and parts count. The attendant reduction of primary power input and dissipation (less than 50% of that of competitive designs) is a major factor in the reliability which is further enhanced by the use of ceramic ICs, all metal-can transistors and a packaging system maximizing mechanical integrity and stability while keeping weight low. For ease of service most modules are identical and of plug-in design, used also in the PTS 160 and PTS 200 with thousands in use.

SPECIFICATIONS

FREQUENCY	Range:	1MHz to 500 MHz						
	Resolution:	0.1Hz to 100KHz steps (optional), 0.2Hz max. res. above 250MHz						
	Control:	Local by 10-position switches. Remote by TTL-BCD, 1248, buffered par. entry or by IEEE 488 BUS. (option)						
	Switching Time:	20 micro-sec. (within 0.1 rad at new frequency)						
OUTPUT	Level:	+ 3 to + 13dBm, (1V) into 50 ohms, metered in dBm and volt						
	Flatness:	± 0.5 dB						
	Impedance:	50 ohms						
	Control:	Manual by F/P-control, remote by voltage, (+ 0.63 to + 2.00V)						
	Settling Time:	20 micro-sec.						
SPURIOUS OUTPUT	Discrete:	—70dB						
	Harmonics:	—30dB at full output						
	Phase Noise:	—63dBc, (0.5Hz to 15KHz),						
	$L_{(1\text{Hz})}$:	100Hz/100dBc; 1KHz/110dBc; 10KHz/120dBc; 100KHz/125dBc.						
	Noise Floor:	—130dBc/Hz						
FREQUENCY STANDARD	Internal:	3×10^{-9} /day or 1×10^{-9} /day (optional)						
	External Drive:	5.000 or 10.000MHz, 0.5V into 300 ohms						
	Aux. Output	10.000MHz, 0.4V into 50 ohms						
GENERAL	Oper. Ambient:	0 to 55°C, 95% R.H.						
	Power:	105-125V, 50-400Hz, 50 Watts						
	Dimensions/Weight:	19 x 5½ x 18" Relay rack or bench cabinet, 35 lbs.						
PRICES (domestic)	Resolution:	100KHz	10KHz	1KHz	100Hz	10Hz	1Hz	0.1/0.2Hz
	\$	5,675.—	5,900.—	6,125.—	6,350.—	6,575.—	6,800.—	6,850.—
	Freq. Standards: (Option)	3×10^{-9} /day, \$450.— (Oven)				1×10^{-9} /day, \$200.— (TCXO)		
	IEEE 488 Interface:	\$650.— (This option replaces the standard parallel entry BCD interface)				Delete Front Panel Controls: —\$200.—		

PROGRAMMED TEST SOURCES, inc.

BOX 617, LITTLETON, MA. 01460 617-486-3008

500-1-82



University of Cincinnati

Cincinnati, Ohio 45221
(513) 475-2263

DEPARTMENT OF CHEMISTRY

June 11, 1982

SILICONE ELASTOMER POSTDOC

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

Dear Barry:

We anticipate a postdoctoral position to become available in about mid-July, 1982. The NSF-funded project involves the study of structure and reinforcement mechanisms in filled and unfilled silicone networks. Co-principal investigators on the project are Professor James E. Mark of the Chemistry Department and Dr. Neal R. Langley of Dow Corning Corporation, Midland, MI.

The successful candidate will be primarily involved in ^{29}Si (and possibly ^{13}C) NMR in fluid and glassy (CP/MAS, etc.) systems, and may become involved in synthesis aspects of the work, if he/she is so inclined. Salary for the first year is \$14,000 plus benefits, with renewal for subsequent years contingent upon mutual agreement by all concerned.

Interested parties should submit their vita and arrange for two letters of recommendation directed to me at the above address.

Of course, the University of Cincinnati is an equal opportunity employer, and especially encourages applications from female and minority candidates. If you are not immediately aware of any interested individuals, I would appreciate your passing this on to colleagues who may know of some. Thanks.

Regards,

A handwritten signature in cursive script, appearing to read "Jerome L. Ackerman".

Jerome L. Ackerman
Associate Professor

dld

Question: Is a Silicone Elastomer Postdoc analogous to a quarter horse or a green thumb? Fowler must be spinning in his grave!

BLS



DEPARTMENT OF ORGANIC CHEMISTRY
ZERNIKELAAN, GRONINGEN
THE NETHERLANDS
TEL.: 050 - 117173

June, 22, 1982

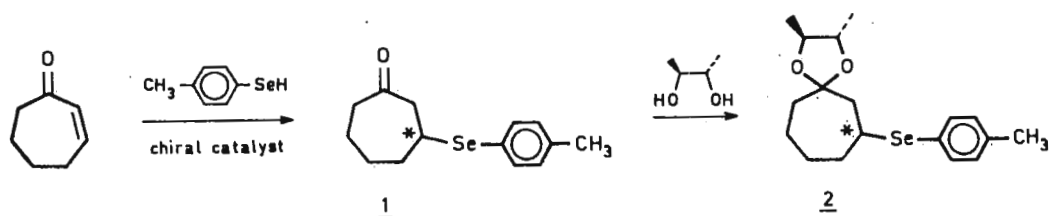
Dr.B.L. Shapiro
Department of Chemistry
Texas A & M University
College Station
Texas 77843
U.S.A.

Dear Professor Shapiro,

Determination of enantiomeric excess by ^{77}Se -NMR

In our laboratory the group of Professor H. Wynberg works on asymmetric synthesis by means of induction by asymmetric catalysts. Our NMR service group is therefore often asked to determine the ratio of enantiomers formed in a reaction. This is done either by means of asymmetric shift reagents, or by conversion into a mixture of diastereomers by reaction with an asymmetric reagent. Usually ^1H or ^{13}C -NMR is used for these determinations but recently we successfully applied ^{77}Se NMR for these investigations. As ^{77}Se has a large chemical shift range (>2000 ppm) and usually gives rather narrow lines, a good separation of the signals of the diastereomeric compounds may be expected.

The ^{77}Se NMR spectrum of a mixture of diastereomers 2, made by the following reaction, is given in the figure.



In this spectrum ditolyldiselenide [3] was used as an internal standard and chemical shifts are denoted in ppm relative to [3] at $\delta = 500.00$.

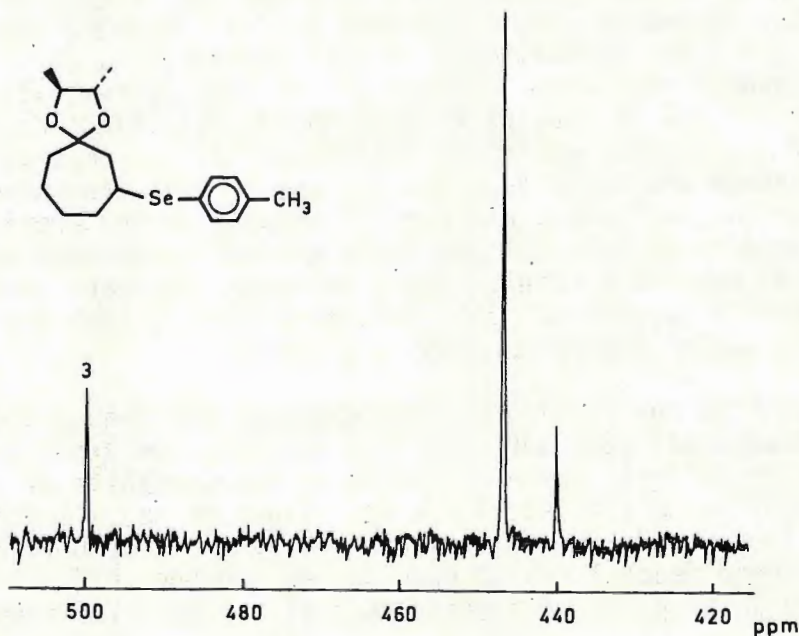
The enantiomeric excess as determined from this ^{77}Se NMR spectrum was in good agreement with the results obtained from ^{13}C NMR. The spectrum was run at 38.17 Mhz on our Nicolet NT 200 instrument.

Please credit this contribution to the subscription of Dr. W.D. Weringa.

Sincerely Yours,

H. Pluim

W.H. Kruizinga





The University of Western Ontario

Department of Chemistry
Chemistry Building
London, Canada
N6A 5B7

June 24, 1982

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

Some Intrinsic $^2\text{H}/^1\text{H}$ Isotope Effects on ^{13}C Shieldings

Dear Barry:

Apparently I have exhausted your series of warning letters and must attempt to get my subscription reinstated. To this end, I want to describe briefly a couple of observations (and the application thereof) of the title shifts, adding to the several recently presented in this newsletter, e.g. 265-53, 276-34, 278-8, 280-3, 283-37.

Part A

In the course of examination of some tricyclo[5.2.1.0^{1,5}]decanes we had prepared the 8-oxo-9,9-dimethyl derivative A and wanted to confirm the ^{13}C assignments. We treated the compound under homoenolization conditions in a deuterated medium ($\text{t-BuO}^-\text{K}^+/\text{t-BuOD}$ at 185°). As expected, ^2H exchange occurred at three sites (C-6, -7 and the exo-9-methyl). However, its ^{13}C spectrum showed that two methylene carbons experience isotope shifts of ~ 0.1 ppm (the size of shift expected for carbons geminal to deuterium in saturated systems), but only one CH_2 is geminal to deuterium. Furthermore the shifted component ($\Delta\delta$, -0.076 ppm) for one of these methylene signals appeared as a triplet, $J \sim 1$ Hz, indicating a vicinal interaction, $J_{\text{CCCD}} > J_{\text{CCP}}$, in general. Since the exo-6-deuterium atom eclipses C-4 in this system, it could be tentatively concluded that vicinal ^2H isotope effects on ^{13}C shieldings depend on the relative orientation of the vicinal nuclei so that these can be comparable to geminal effects. A number of reviews and texts describe the use of deuterium substitution in saturated systems as a useful assignment aid for ^{13}C spectra on the premise that geminal, two-bond effects tend to be near 0.1 ppm while vicinal three-bond shifts are significantly smaller, ~ 0.01 ppm. Our results for A indicate, however, that some caution is required and Anet's suggestion [JACS 101, 5449 (1979)] that the spatial proximity of the interacting nuclei may be an important factor.

To confirm our finding we also examined the 8-endo-alcohol obtained by reducing A (unlabelled) with LAD. In this alcohol, the exo-8-deuterium is eclipsed with C-11, the exo methyl, which exhibited an isotope shift of -0.076 ppm and the shifted component was a triplet, $J \sim 1$ Hz. Thus, we had a second, sizeable vicinal isotope shift for ^2H and ^{13}C having a dihedral angle near 0° . Finally we prepared some exo-2-deuterio-endo fenchol (B) to examine the isotope shifts for the six vicinal carbons, having an array of different dihedral angles. The observed shifts are shown with B, in ppm.

From these results it is clear that the vicinal $^1\text{H}/^2\text{H}$ isotope shift for ^{13}C has a marked dependence on geometry, with maximal effects found for eclipsed nuclei.

This is in direct contrast to the findings for ^{19}F , reported by Lambert some years ago, JACS 95, 6150 (1973); JACS 96, 5120 (1974).

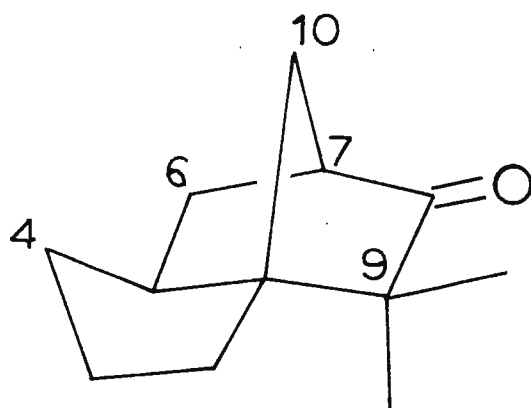
Part B

In another project we have utilized incorporation experiments with $^{13}\text{CD}_3\text{COO}^-\text{Na}^+$, to test for hydride shifts in the biosynthesis of some sesquiterpenes. As an example, I will give the results for rishitin (C). Having earlier established the pattern for acetate incorporation in C [Tett. Lett. 3271 (1976); Can. J. Chem. 56, 645 (1978)], using $^{13}\text{CH}_3^{13}\text{COO}^-\text{Na}^+$, we knew that the carbons indicated by dark circles arise from the methyl carbon of acetate. The point of interest was the possibility of a hydride shift from C-5 to C-4 during the genesis of C. If this indeed were true, the absorption for C-5 should exhibit a geminal isotope shift since deuterium at C-4 would have been its original bonded partner in the labelled precursor. In the event, C, isolated after the incorporation experiment, gave rise to the expected ^{13}C spectrum in which the C-5 signal at 129.0 ppm appeared as a doublet with the higher field component shielded by 0.045 ppm, confirming the proposed hydride (deuteride) shift. My colleague, Albert Stoessl, calls this a "beta-hop" since the observed β -isotope effect is caused by the hydride "hop". This test is exactly analogous to experiments using doubly labelled mevalonate (MVA) such as 4,4-dideuterio-4- ^{13}C -MVA utilized by Barton et al, Chem. Comm. 843 (1978) in which ^2H migration occurred within the same MVA moiety. [In some cases, of course, the simpler 4,4-dideuterio MVA has been utilized, e.g. Can. J. Chem. 58, 1894 (1980).] Doubly labelled $^{13}\text{CD}_3\text{COO}^-\text{Na}^+$ is an even simpler, commercially available precursor and acetate incorporation is generally higher than the levels attained with MVA experiments.

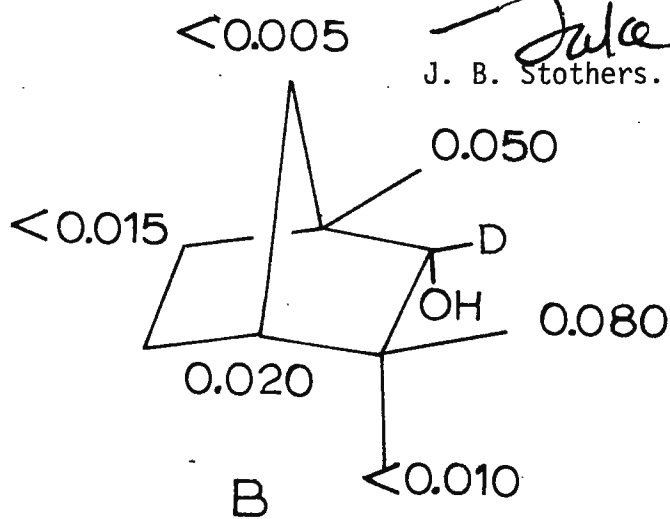
I trust that my subscription to the TAMU newsletter will now resume and I look forward to receiving the missing issues. With best wishes,

Sincerely,

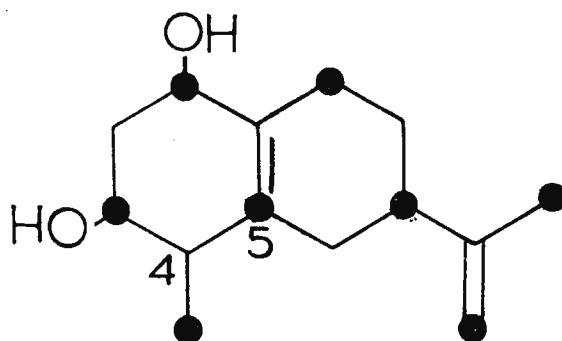
Julia
J. B. Stothers.



A



B



C

University of Illinois at Urbana-Champaign

School of Chemical Sciences
505 South Mathews Avenue
Urbana, Illinois 61801

June 24, 1982

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

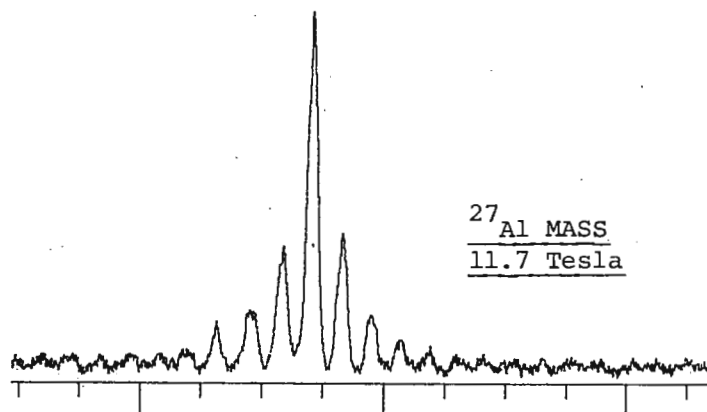
Dear Barry:

Very Attractive Samples?

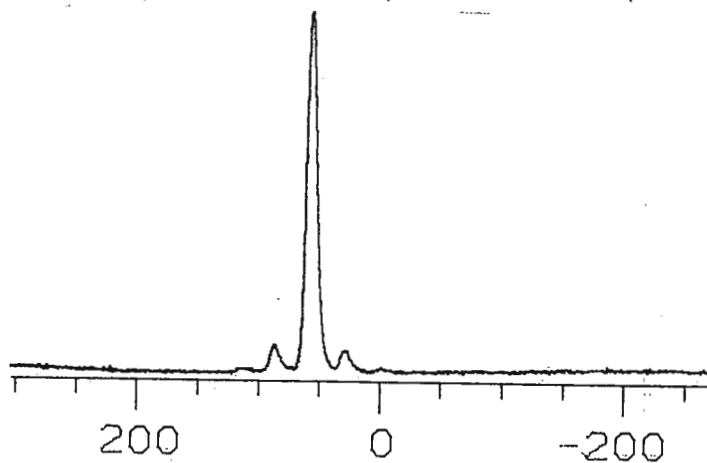
We have recently been looking at a variety of minerals using MASS and VASS techniques. One rather surprising result was obtained with some naturally occurring sanidines ($K,Na-AlSi_3O_8$), a highly disordered form of feldspar. Some samples gave apparently large CSAs as determined by MASS (figure), while others gave very small CSAs. The actual powder pattern envelopes appeared rather too symmetric however, so we investigated further.

Sanidine,
Sample A

^{27}Al MASS
11.7 Tesla



Sanidine,
Sample B



PPM from $Al(H_2O)_6^{+++}$

Using that "other" form of resonance (at 9 GHz) we found large ferromagnetic (or superparamagnetic) signals from the large CSA sample, but only a little Fe^{3+} signal from the small CSA sample.

Clearly, our results indicate that large magnetic field inhomogeneities are present in the sample with numerous sidebands, due presumably to Fe-contamination.

In other minerals, we've even found it necessary to use magnetic separation to clean-up our materials.

Yours sincerely,

				
Eric Oldfield	Robert A. Kinsey	Jim Kirkpatrick	Karen Ann Smith	Bernard Montez

Princeton University

DEPARTMENT OF CHEMISTRY

PRINCETON, NEW JERSEY 08544

June 2, 1982

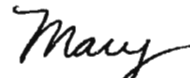
Professor B. L. Shapiro
Department of Chemistry
Texas A & M University
College Station, Texas 77843

Title: XL-100 on sale!

Dear Professor Shapiro:

We have an operating Varian XL-100 NMR spectrometer for sale, with the following bells and whistles: 12" magnet, V-4412 probehead, ^1H , ^{19}F , ^{31}P and ^{13}C FT-observe, ^2H pulsed lock, ^{19}F external lock, high power pulse amplifier, variable temperature unit, Diabolo disk drive, 620/L computer, and Princeton University (i.e. home-built and better than new!) heat exchanger. Anyone interested in purchasing all (preferably) or parts of this workhorse should contact me at the above address, or by phoning 609-452-3928.

Sincerely,



Mary W. Baum

UNIVERSITY OF CALIFORNIA, SAN FRANCISCO

BERKELEY • DAVIS • IRVINE • LOS ANGELES • RIVERSIDE • SAN DIEGO • SAN FRANCISCO



SANTA BARBARA • SANTA CRUZ

SCHOOL OF PHARMACY
DEPARTMENT OF PHARMACEUTICAL CHEMISTRY

SAN FRANCISCO, CALIFORNIA 94143

June 18, 1982

Re: Varian XL-100-15 for Sale

Dear Barry:

We have a Varian XL-100 spectrometer with a 15 inch magnet which a TAMUNMR Newsletter reader may be interested in purchasing. This spectrometer was set-up for doing Fourier Transform NMR (described in Rev. Sci. Instr. 45, 1095 (1974)), but the computer system is unavailable. The following is a partial list of included components:

- 15" magnet with V-7800 power supply and heat exchanger
- Deuterium lock
- Gyrocode spin decoupler
- Frequency counter
- Variable temperature controller
- Components for observation of ^1H (5 and 12 mm), ^{13}C (5, 8, and 12 mm)

Anyone interested in this system (or parts) may contact me.

Sincerely,

Thomas L. James
Associate Professor of Chemistry
and Pharmaceutical Chemistry

TLJ:gdc

(415) 666-1569

450:1

THE INTERNATIONAL SOCIETY OF MAGNETIC RESONANCE - AUGUST 1983 MEETING

The International Society of Magnetic Resonance will hold its 8th triannual meeting in Chicago, Illinois, August 22-26, 1983. The meeting will cover the broad field of magnetic resonance, including the theory and practice of nuclear magnetic resonance, electron paramagnetic resonance and nuclear quadrupole resonance spectroscopy. Included will be applications in physics, chemistry, biology and medicine. The meeting will strive to foster interaction among scientists in different fields of magnetic resonance and to encourage interdisciplinary exploration.

For information write: University of Illinois at the Medical Center, Conferences and Institutes, 912 South Wood Street, Second Floor North, Chicago, Illinois 60612, U.S.A., (312) 996-8025.

D. Fiat



Apples-to-apples comparison of NMR sensitivity

To make a long story short, Nicolet's 300 MHz spectrometer offers the industry's highest ^{13}C sensitivity by a significant margin.

Nicolet Model 300WB: *Guaranteed 500:1 signal-to-noise ratio.*
Nearest competitor's 300 MHz model: *Guaranteed 300:1 signal-to-noise ratio.*

This is an apples-to-apples comparison, since both of the S/N specifications above are based on ASTM samples; and as you can see, the Nicolet 300WB delivers 67% more sensitivity.

How do we *know* our 500:1 S/N is for real? We do it by computer determination. Computer calculated values are inherently more precise, because RMS noise is calculated directly from the digitized data. In fact, Nicolet's computerized S/N ratio calculation is the accepted industry standard. With the Nicolet 300WB you can reduce run time, and you'll still be able to identify smaller peaks.

Now, sensitivity is one thing but ease of operation is another. As the world's acknowledged leader in NMR software, you can be sure that even inexperienced operators will appreciate the Nicolet 300WB's simplicity. And for the more sophisticated

NMR spectroscopist the Nicolet 1280 Data System offers enough computing power to handle the toughest NMR determinations.

So, if you've heard someone else claim the "world's highest sensitivity" for NMR spectrometry, remember this apples-to-apples comparison.

There's a lot more you should know about the Nicolet NMR... examine its capabilities... get in touch with the Nicolet Magnetics salesman in your area or call Rich Bohn at (415) 490-8300.



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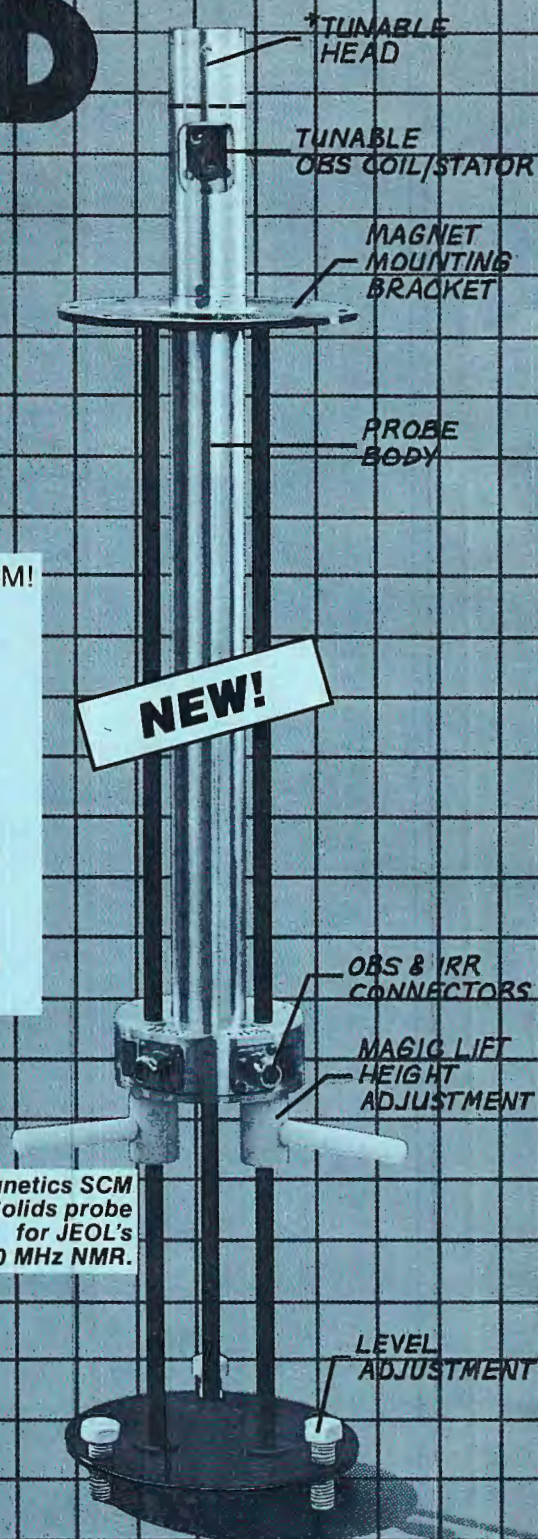
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