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NOTE!

Deadline Dates: No. 100 - 12 January 1967
No. 101 - 15 February 1967

ILLINOIS INSTITUTE OF TECHNOLOGY
TECHNOLOGY CENTER
CHICAGO ~~16~~ 60616

Department of Chemistry

TO ALL IIT NMR NEWSLETTER SUBSCRIBER/CONTRIBUTORS :

NMR men of the world - arise ! Revolt against your secretaries who are threatening the Newsletter's very existence. They do this by double spacing, leaving wide margins, stopping far from the bottom of the page, etc., etc. All of these deplorable practices are making your otherwise excellent contributions much longer than they need to be, thus making the Newsletter much costlier than it needs to be; this clearly cannot continue without limit.

Therefore, please keep a careful eye on the physical format of your contributions - including the figures !!!, so that the goldenness of brevity is maximized. Thanks!

BLS

B. L. Shapiro*

12-15-66

* who had to type this notice himself since all the secretaries wouldn't touch it. Oh well.

JET PROPULSION LABORATORY *California Institute of Technology • 4800 Oak Grove Drive, Pasadena, California 91103*

18 November 1966

Refer to: 328-SLM/DDE:cls

Professor Barry L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry:

Nuclear stabilized NMR spectrometers for the direct observation of nuclei other than H^1 and F^{19} are still rather few in number and require important instrumentation modifications and expensive additions over the spectrometer components available with most commercial NMR spectrometer installations. Some time ago we considered the various aspects of the techniques for achieving nuclear stabilization of NMR spectrometers for nuclei other than H^1 and F^{19}. Nuclear stabilized spectrometers can be divided into two types: 1) those with external control samples and 2) those with internal control samples. Systems of the former type have two receiver coils, two receivers, and generally two transmitters; one channel is the control channel and the other is the sample channel. For observation of H^1 it is common to use a water control sample. For such a system one only needs one r.f. frequency. For observation of nuclei other than H^1 with this type of system it is common to use a water control sample and r.f. frequency synthesizer techniques to lock the H^1 control sample frequency to any other frequencies desired.

The second method of achieving nuclear stabilization utilizes a resonance line in the spectrum to derive a control signal. If the control nuclei and sample nuclei are the same kind then only one receiver coil and r.f. source is needed.* In this case a substance having a sharp single line is usually added to the mixture or substance one wishes to study. This technique has been used for some time for H^1 and we and several others have found it quite successful for recording F^{19} spectra. There are certain problems which can arise with this technique.

In some cases one is hard-put to select an appropriate internal reference because of chemical incompatibilities; however, sometimes with reference samples in sealed capillaries inserted in the sample to be studied one can

* If the control nuclei and the sample nuclei are different then in general more than one receiver coil and more than one transmitter coil may be needed.

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get around this problem. In the NMR studies of gases the selection of a reference compound may be difficult. Aqueous solutions also present problems, but we have successfully recorded spectra using a reference sample in a coaxial tube or internal capillary. However, for nuclei with low sensitivities or abundancies, any reference sample added to a sample or inserted in a sealed container will significantly dilute the number of nuclei of interest in the volume seen by the receiver coil of conventional Varian inserts. Another possible solution to this difficult problem which we considered was addition of a second receiver coil in the transmitter field in a single receiver circuit. This coil would be wound about a sample of a reference material which would be placed very close to the sample to be studied. This system has been successfully tested for H^1 by John Baldeschwieler and co-workers and should work well for F^{19} too where a very small sample suffices and signal to noise is not a problem. With the nuclei N^{14}, C^{13}, and P^{31}, our tests with an internal lock system, using available 5 mm Varian inserts and 5 mm sample tubes with common Varian r.f. units and aluminum probes, suggested to us that the size and disposition of the reference sample required for an extra receiver coil would introduce numerous problems of placement, phase adjustment, homogeneity, tuning and loss of signal. For these reasons and because we did not have a set of frequency synthesizers some time ago we investigated another approach.

We would like to describe a rather simple addition to common Varian 5 mm NMR inserts which makes achievement of nuclear stabilization rather simple for nuclei other than H^1 and F^{19} (provided one has the appropriate V-4311 unit and V-4331 probe). We have constructed cylindrical sample containers which can be placed coaxially about 5 mm inserts as shown in Fig. 1a. We have found that a reference sample in such a container has a homogeneous enough field (frequency sweep line width for C^{13} about 8-9 cps) to give a signal suitable for locking the field and frequency of an NMR spectrometer with a simple audio modulation system of the type we have been using for some time. We have successfully recorded frequency sweep N^{14}, C^{13}, B^{11}, P^{31}, F^{19}, and H^1 spectra when the spectrometer was stabilized on a sample of the corresponding nucleus in such a sample container external to the receiver coil. Resolution of the order of 0.3-0.5 cps was achieved for N^{14}, C^{13} and P^{31}. The ultimate in design which would increase the field homogeneity at the sample and reference would be the construction of a one-piece insert with the receiver coil wound inside as shown in Fig. 1b. We have built several of these one-piece inserts and hope to test them shortly.

See Example Spectra in Figs. 2+3

The placement of a control sample external to the receiver coil which we have described for nuclear stabilization with nuclei other than H^1 and F^{19} offers a number of advantages available only with a more complicated NMR system utilizing frequency synthesis. The spectrometer stays locked when samples are replaced, the study of aqueous solutions and gases is facilitated, the study of chemically reactive systems is made more convenient, the use of time-averaging techniques is feasible, the study of certain relaxation effects

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should be less complicated, the possibility of using NMR for stream analyses appears feasible, and the use of high-resolution NMR in biological studies or kinetic studies requiring a flow system appears practical.

In a future letter we will present something on what we have done since we joined the club of frequency snythesizer owners.

Possible Titles: "Poor Man's Field-Lock for Many Nuclei" or "What We Tried to Do Before The Hewlett-Packard Salesman Came Along".

With best regards,

Stan Manatt

Stanley L. Manatt

Don Elleman

Daniel D. Elleman

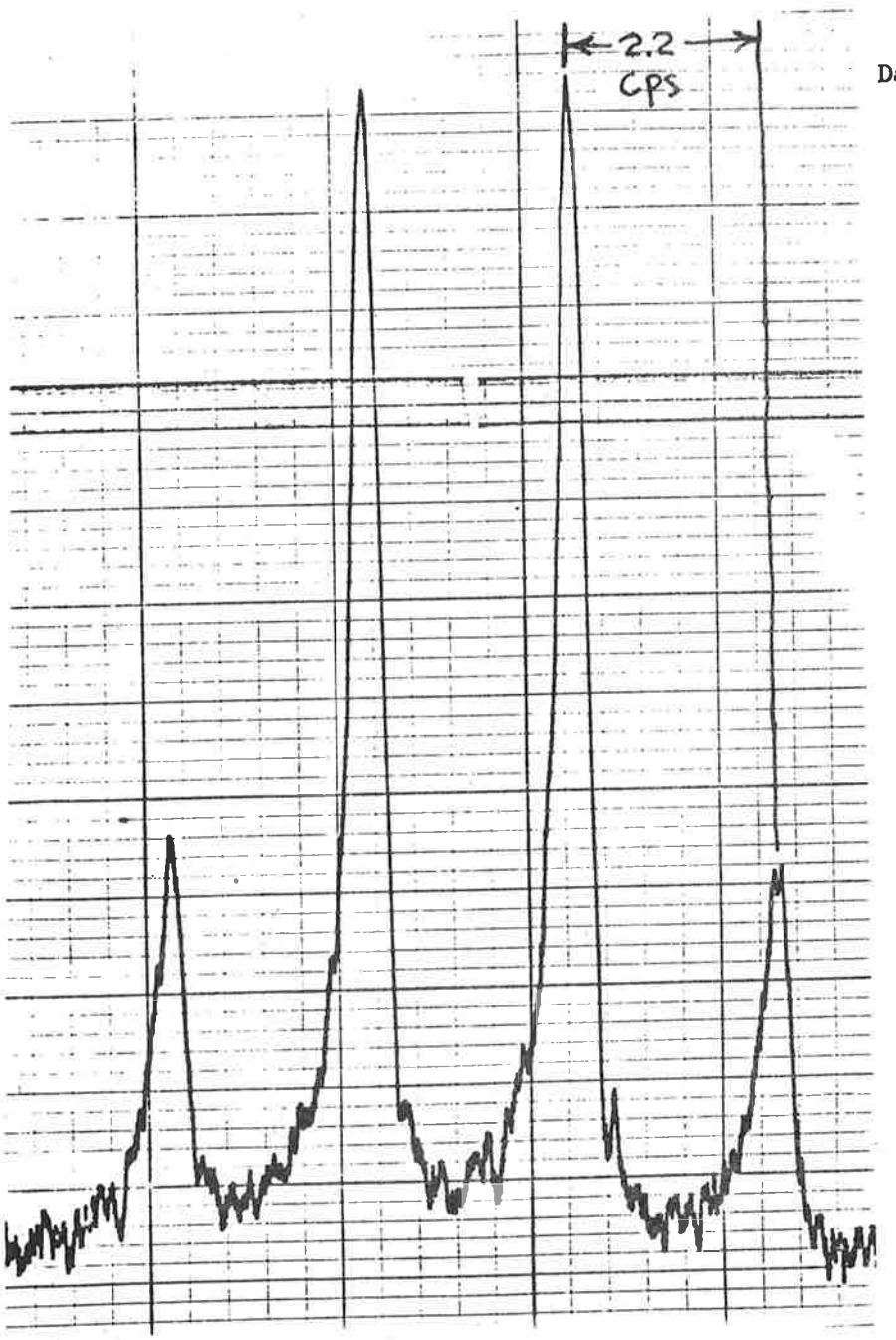


Fig. 2. Frequency Sweep N^{14} Spectrum of $CH_3N{\equiv}C$ at 4.3 Mc. Spectrometer Locked on $C(NO_2)_4$.

$J_{H-C-N} = -2.2$ cps

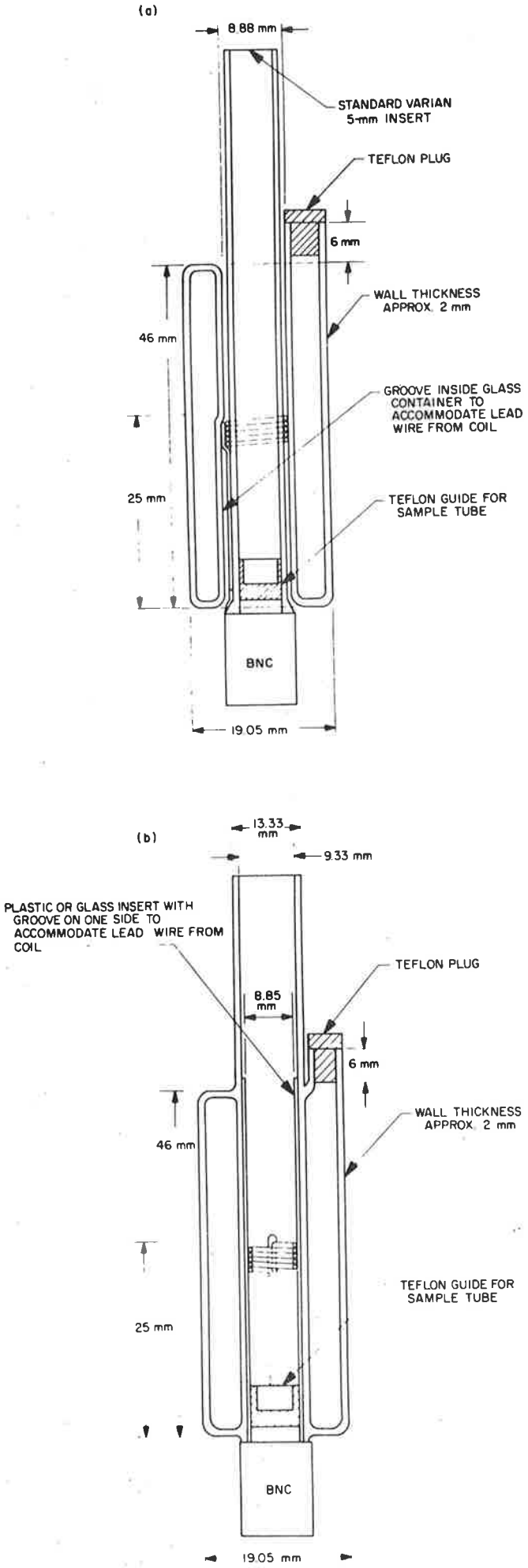

(a)
8.88 mm
STANDARD VARIAN
5-mm INSERT
TEFLON PLUG
6 mm
WALL THICKNESS
APPROX. 2 mm
46 mm
GROOVE INSIDE GLASS
CONTAINER TO
ACCOMMODATE LEAD
WIRE FROM COIL
25 mm
TEFLON GUIDE FOR
SAMPLE TUBE
BNC
19.05 mm
(b)
13.33
mm
9.33 mm
PLASTIC OR GLASS INSERT WITH
GROOVE ON ONE SIDE TO
ACCOMMODATE LEAD WIRE FROM
COIL
TEFLON PLUG
8.85
mm
6 mm
WALL THICKNESS
APPROX. 2 mm
46 mm
TEFLON GUIDE FOR
SAMPLE TUBE
25 mm
BNC
19.05 mm

FIGURE 1

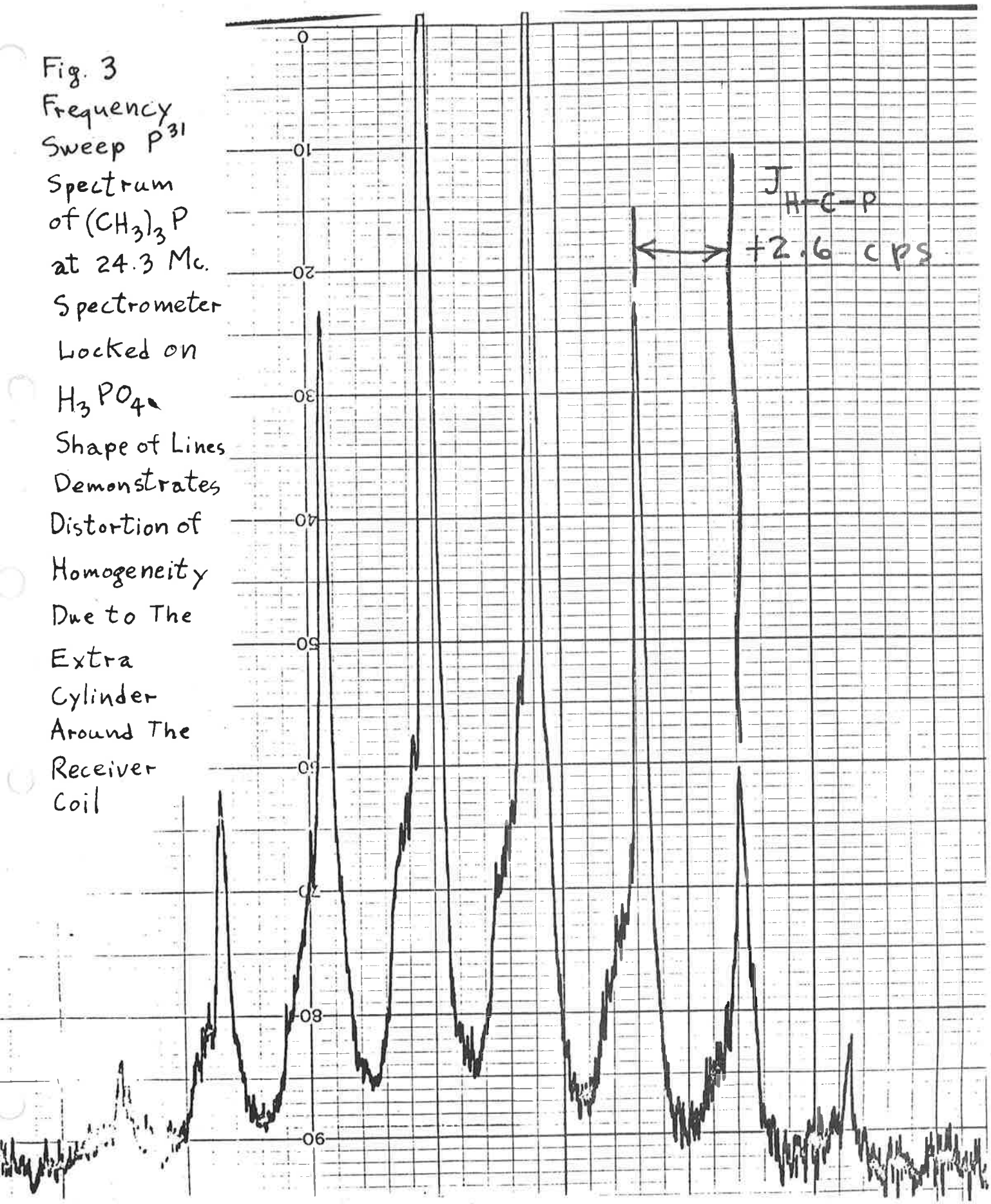


Fig. 3
Frequency Sweep P^{31} Spectrum of $(CH_3)_3P$ at 24.3 Mc. Spectrometer Locked on H_3PO_4. Shape of Lines Demonstrates Distortion of Homogeneity Due to The Extra Cylinder Around The Receiver Coil

99-6

Dr. G. Englert and

Mr. J.-P. Weber

c/o

F. HOFFMANN-LA ROCHE & CO.

AKTIENGESELLSCHAFT

ABTEILUNG Phy. A.
Eng/hb

Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology

C h i c a g o , 60616

Re: Negative sign of $J(H,CH_3)$ in Propyne

Dear Professor Shapiro,

We have previously reported on the n.m.r spectra of some acetylenic compounds oriented in a nematic liquid (1). The signals of the acetylenic protons of several substituted acetylenes showed a considerable upfield shift in the nematic solution because the long molecular axes (H-C≡C-R) were preferably oriented parallel to the magnetic field.

From the known molecular orientation the absolute sign of the direct magnetic couplings can be calculated. In such cases the absolute sign of the indirect J-coupling is also obtained, since its relative sign with respect to the direct couplings can be derived from an analysis of the spectrum.

With propyne (methyl acetylene, $H-C\equiv C-CH_3$) we found a negative sign for $J(H,CH_3)$.

Five spectra of propyne were carefully measured (solvent: 4,4-di-n-hexyloxy-azoxybenzene; 75° C) and the line positions averaged. From suitable frequency differences a zero order set of parameters (chemical shift $\Delta\nu$, direct couplings $A(H,CH_3)$ and $B(CH_3)$) for both possible values of $J = \pm 2.7$ Hz was obtained.

Since we have no available iterative computer programme, we have chosen a more sophisticated way to optimize these parameters, viz. the Box-Wilson method of exploration of response surfaces (2).

A criterion for the agreement between experimental and theoretical line positions ν_i is given by the expression:

./.

$$\delta(\Delta\nu, A, B) = \sqrt{\sum_{i=1}^{n} (\nu_{i\,obs.} - \nu_{i\,calc.})^2 / n}$$

n = number of lines

Here n = 13, since we used only 13 of the 16 lines of the spectrum, which were easily recognized and assigned. Seeking for the optimal set of parameters which minimizes δ, we approximated the function δ by a polynom of second order. The coefficients were determined by a least square method using 15 values of δ obtained from 15 theoretical spectra (15 sets of parameters). The coordinates of the stationary point were calculated afterwards. These coordinates are the optimal values of $\Delta\nu$, A and B, since one can show that they correspond to a minimum of the function δ.

We obtained for the two alternatives:

a) J = - 2.7 Hz:	$\Delta\nu$ = - 65.9Hz A = - 122.7 Hz B = + 879.2 Hz	δ = 1.60 Hz
b) J = + 2.7 Hz:	$\Delta\nu$ = - 65.4 Hz A = - 122.6 Hz B = + 879.3 Hz	δ = 4.36 Hz

This clearly shows that the absolute sign of $J(H,CH_3)$ in propyne is negative. A still more convincing proof can be obtained from a comparison of the experimental and theoretical line positions. In case b) only 3 calculated frequencies were found within the standard deviations of the experimental line positions, the other lines deviating up to 7 Hz. In case a) eight lines were within the experimental standard deviation, the deviation of the rest being only slightly larger.

Our finding is in accordance with the work of Sackmann and Dreeskamp (3), who measured the sign of J by double resonance experiments with respect to $J(^{13}CH_3)$ of the methyl group. The latter sign is generally assumed to be positive, which has been experimentally proved in the case of acetonitrile (1).

With best regards,

K. Englert

F. Weber

(1) G. Englert and A. Saupe, Molecular Crystals 1, 503 (1966)
(2) G. E. P. Box and K. Wilson, J. Roy. Stat. Soc. 8, 1 (1951)
(3) E. Sackmann and H. Dreeskamp, Spectrochimica Acta 21, 2005 (1965)

Southern Illinois
University

CARBONDALE, ILLINOIS 62901

Department of Chemistry

November 15, 1966

Prof. B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, IL 60616

Dear Professor Shapiro:

Conformations of Pent-2-enopyranosyl Derivatives and Relative signs of Allylic & Bi-allylic Proton Coupling Constants

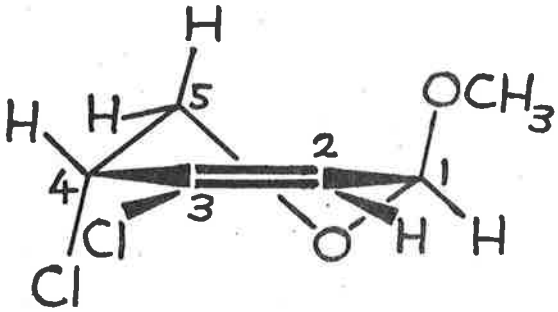
The factors governing the conformational preference of pyranose rings are currently of considerable interest, and to obtain further information in this field we have recently analyzed the proton spectra of some D-glycero-pent-2-enopyranosyl derivatives(I-IV) measured at 60 & 100 Mc/s. Spectra of the ring protons of methyl 3,4-dichloro-4-deoxy α- & β-D-glycero-pent-2-enopyranosides(IV & I) are shown in Figs 1 & 2, respectively. The H_4, H_{5e}, & H_{5a} protons in I-IV were analyzed as ABX sub-systems, and for I & IV the parameters so obtained were combined with first order splittings and weighted mean chemical shifts for the remaining ring protons, and theoretical spectra were computed for the five spin systems using the Frequint IV program. The parameters were then adjusted to give a better fit, as shown in Figs 1 & 2.

The β-D-derivatives(I-III) displayed small values of $J_{4,5e}$ and $J_{4,5a}$ (TABLE), no measurable allylic ($J_{2,4}$) or bi-allylic ($J_{1,4}$) couplings, and hence were assigned the half-chair conformations I-III as shown, in which H_4 lies inside the dihedral angle subtended by the methylene protons at C_5, and in which the allylic dihedral angles are ≈40° for both H_1 and H_4. The methyl β-D-glycoside(I) (Fig. 2) also showed a small coupling (0.4 c/s) between H_1 and one of the H_5 protons (tentatively assigned as H_{5a}. On the other hand, the methyl α-D-glycoside(IV) showed values of $J_{4,5e}$ and $J_{4,5a}$ greater than 5 c/s, and allylic coupling $|J_{2,4}|$ = 1.2 c/s, and a bi-allylic coupling $|J_{1,4}|$ = 1.3 c/s. This derivative was, therefore, assigned the alternative half-chair conformation (IV as shown) in which H_4 lies outside the methylene dihedral angle, and in which the allylic dihedral angle of H_4 is ≈80°.

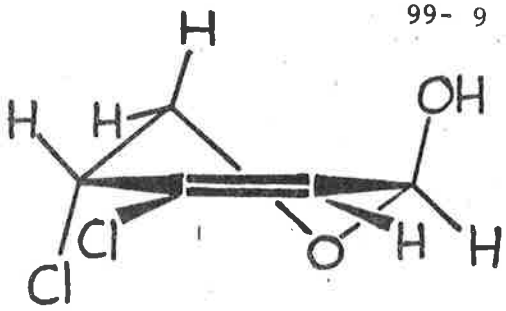
TABLE. Coupling constants (c/s) in 3,4-Dichloro-4-deoxy-D-glycero-pent-2-enopyranosyl Derivatives.

Compound	$J_{1,2}$	$J_{1,4}$	$J_{1,5a}$	$J_{2,4}$	$J_{4,5e}$	$J_{4,5a}$	$J_{5e,5a}$
I[a]	3.3	----	-0.4[d]	----	1.04	2.70	-12.49[d]
II[b]	3.4	----	----	----	1.45	2.67	-12.7[d]
III[c]	3.4	----	----	----	0.91	2.71	-12.79[d]
IV[a]	3.1	1.3	----	-1.2	5.56	7.87	-11.61[d]

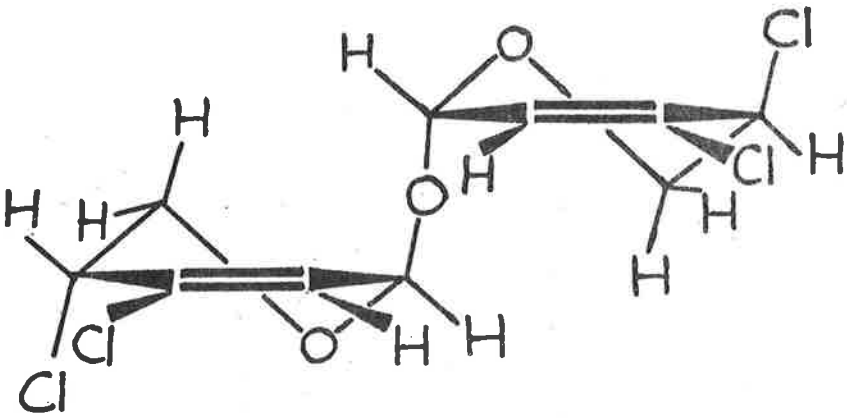
a. In $CDCl_3$ at 100 Mc/s, b. In $CDCl_3$:pyridine (2:1) at 60 Mc/s
c. In pyridine at 100 Mc/s, d. Assumed negative.



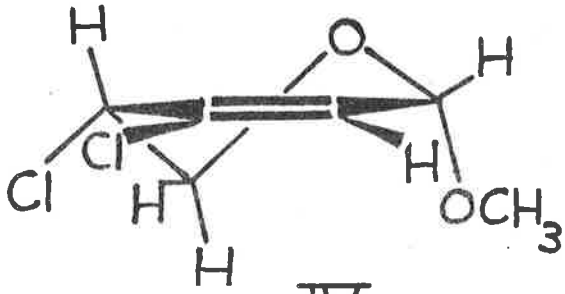
I



II



III



IV

Frequency-swept spin decoupling experiments (Fig. 3) performed at 100 Mc/s by Dr. K. A. McLauchlan showed that $J_{1,4}$ and $J_{1,2}$ are of like sign, and that $J_{2,4}$ and $J_{1,2}$ are of opposite sign.

The preference of the α and β derivatives for different half-chair conformations each containing the substituent at C_1 in quasi-axial orientation, we must again attribute to the dominating presence of an anomeric effect[1]. We have interpreted the difference in stability of the anomeric methyl glycosides I & IV revealed by GLC analyses of their acid catalyzed equilibration reactions in methanol in terms of the differing orientations of the chlorine and hydrogen atoms at C_4 with respect to other atoms, in the two half-chair conformations.

Sincerely yours,

B. Coxon

B. Coxon with H. J. Jennings & K. A. McLauchlan

1. B. Coxon, Tetrahedron, 22, 2281 (1966).

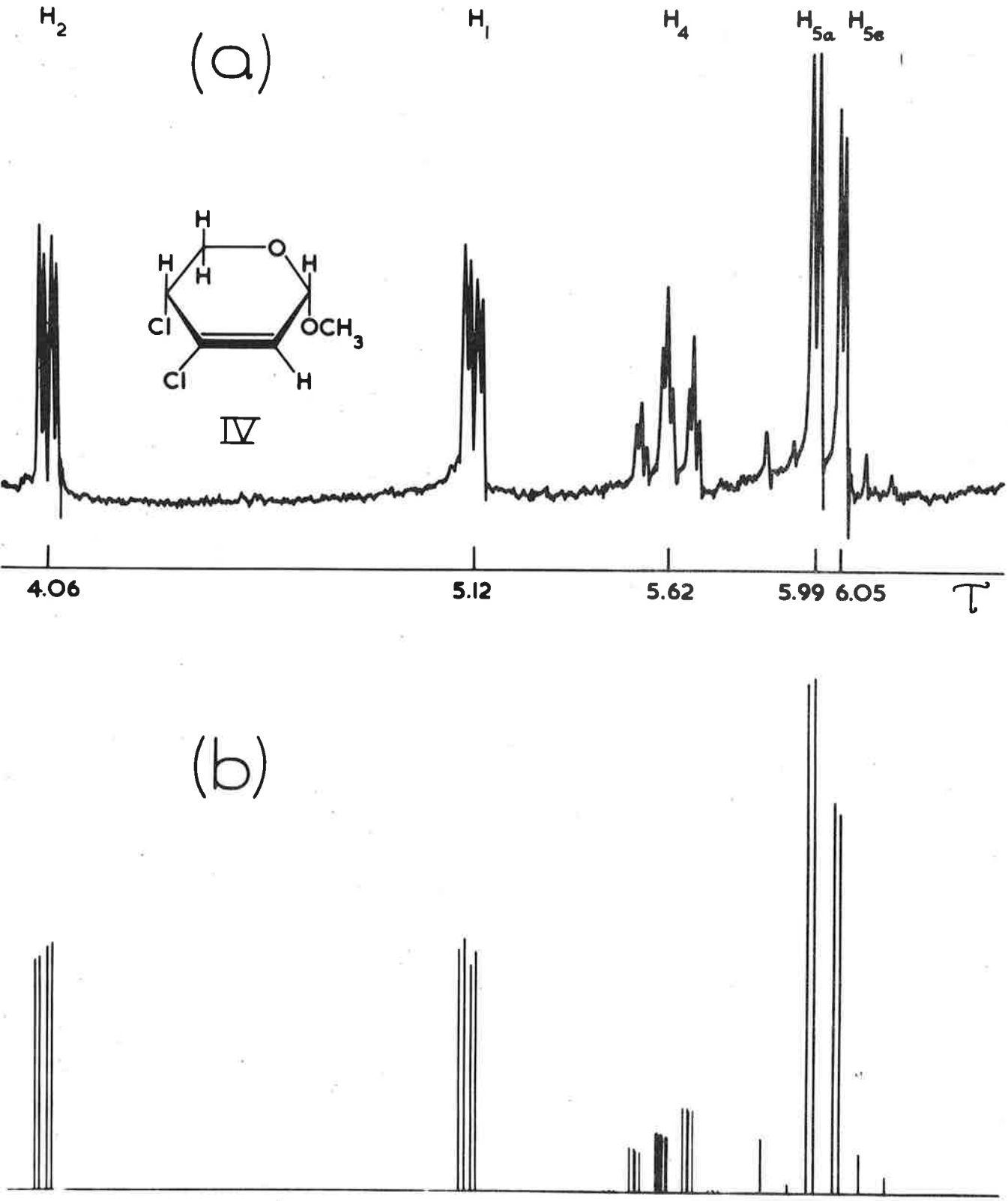


Fig. 1. Spectra of the ring protons of methyl 3,4-dichloro-4-deoxy-α-D-glycero-pent-2-enopyranoside(IV), (a) in $CDCl_3$ at 100Mc/s, (b) computed.

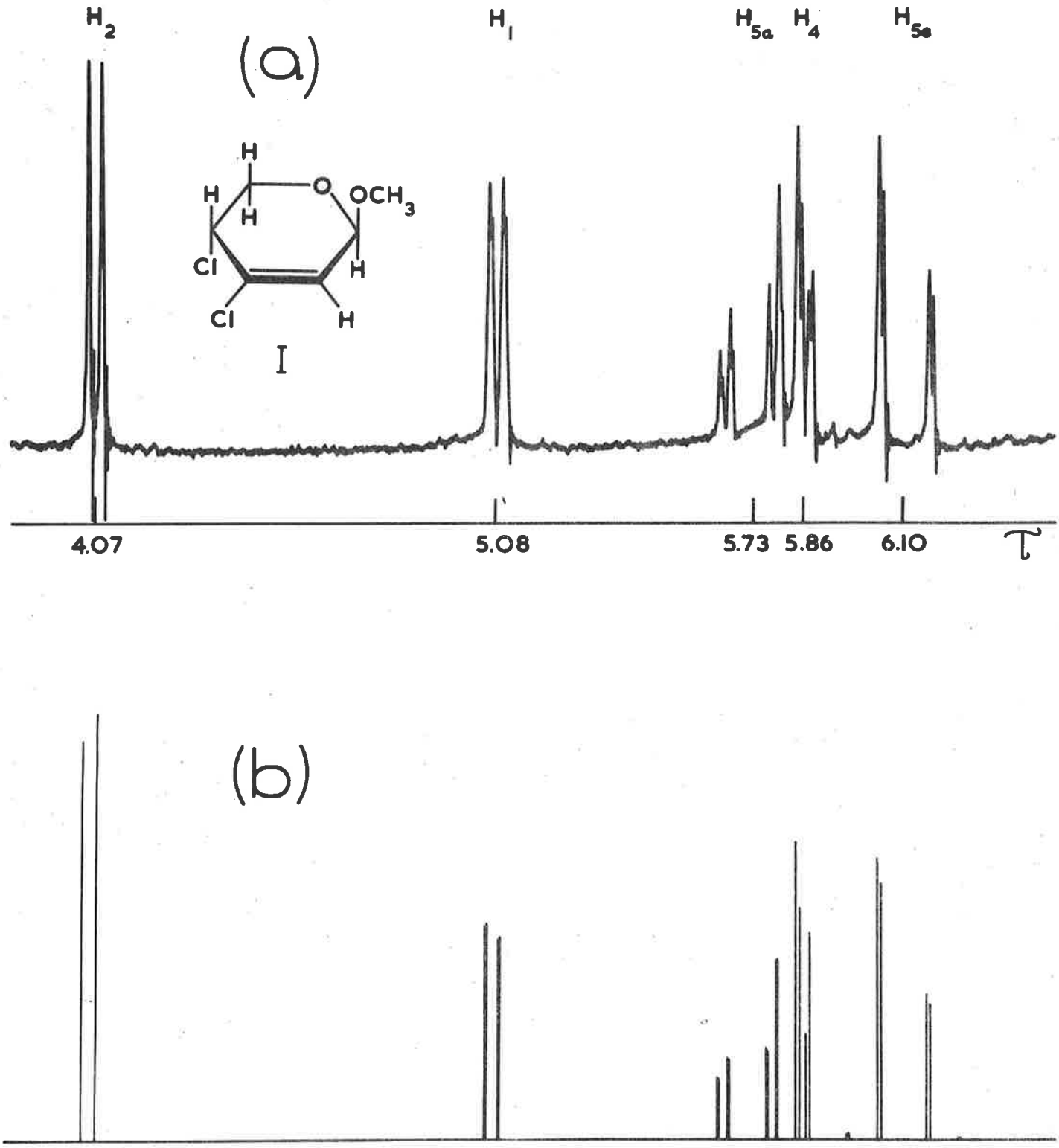


Fig. 2. Spectra of the ring protons of methyl 3,4-dichloro-4-deoxy -β-D-glycero-pent-2-enopyranoside(I), (a) in $CDCl_3$ at 100Mc/s, (b) computed.

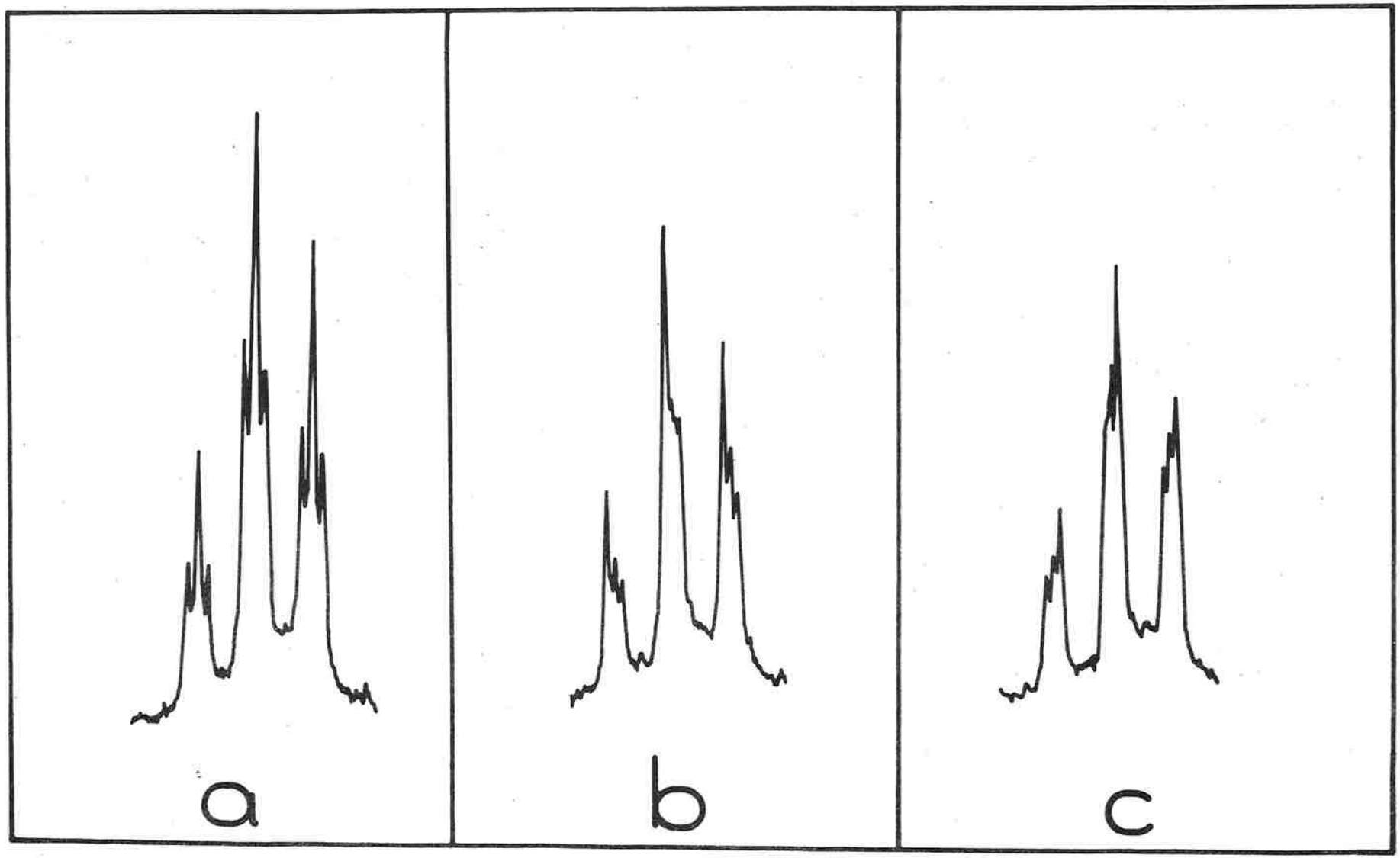


Fig. 3. Frequency swept spin decoupling experiments with methyl 3,4-dichloro-4-deoxy-α-D-glycero-pent-2-enopyranoside(IV) in $CDCl_3$ at 100 Mc/s. Resonance due to H_4 (a) unperturbed, (b) with irradiation of the low field pair of lines of the H_2 quartet, and (c) with irradiation of the low field pair of lines of the H_1 quartet.

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15. 11. 66

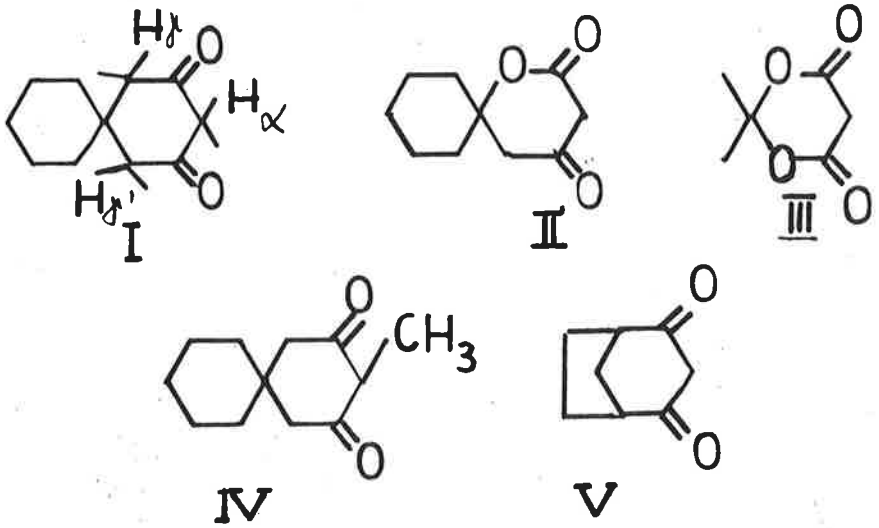
Prof. B.L. Shapiro
Dept. of Chemistry
Illinois Institute of Technology
Technology Center
Chicago/Illinois 60616/USA

Effect of Electronic Factors on the Keto-Enol-Equilibrium of 6-Membered ß-Dicarbonyl-Compounds

Dear Dr. Shapiro,

Nmr-studies on the effect of ring size in cyclic ß-diketones [1]) led us to an nmr-investigation of the keto-enol-equilibrium of 6-membered ß-dicarbonyl-compounds I - VI [2,3,4]). The nmr-spectra were recorded in two solvents:

a) an aprotic solvent (CCl_4, $CDCl_3$),
b) a hydrophilic solvent (CD_3OD).



As examples two typical spectra are reported in detail: (δ in ppm)

I($CDCl_3$)			I(CD_3OD)[a]	
diketone	enol	pentamethylene-protons	diketone	enol
1,44	1,44		-	1,47
2,58	2,33	γ,γ'-protons	-	2,32
3,34	5,47	α -protons	-	5,32[b]

III($CDCl_3$)		III(CD_3OD)
diketone		diketone
1,76	methyl-protons	1,76
3,56	α-protons	3,85[b]

a) the solution contained a trace of acid to prevent dissociation

b) partially deuterated signal

The result of these measurements are compiled in Table 1. In the case of aprotic solvents a calculation of the percentage of enol is possible, whereas deuteration in CD_3OD allows no exact evaluation of these data. Despite this fact the following conclusions could be drawn: in the series I to III the +M-effect produced by the oxygen steadily increases, thus diminuishing the "carbonyl"-character of the attached C=O-group. This is obvious from the recorded spectra which show a decreasing "enolisation"-tendency going from I to III. A methyl group in the 2-position does not drammatically change these findings. A comparison of I with cyclohexanedione-(1.3)(A) demonstrates also the great effect of the pentamethylene-group on the "enolisation"-tendency (A: 86 %, I: 16 % enol) [1]. The reason for the decrease of the enol form of I compared to A results from the greater solubility of I in aprotic solvents. This was originally intended by introducing the spiro-group [2,3,4].

In CD_3OD the enol concentration decreases from I (~ 8o-1oo % enol) to III (≪ 5 % enol). From I to III the enol-form is less favored. This is also due to the +M-effect of the oxygen atoms. V shows a similiar behaviour to I.

Table 1: Percentage of enol in the 6-membered ß-dicarbonyl-compounds I-V

compound	$CCl_4(CDCl_3)$ [%]	CD_3OD [c)] [%]
I	23 [a)], 16 [b)]	~ 8o-1oo [d)]
II	≪ 5	~ 2o-3o [d)]
III	≪ 5	≪ 5 [d)]
IV	~ 25	-
V	≪ 5	~ 8o-1oo [d)]

a) after 15 min., b) after 76 h, c) because of the low solubility of I-V in methanol it was impossible to prepare highly concentrated solutions to avoid deuteration
d) no exact figures because of deuteration

These nmr-data are consistent with ir-measurements of I-V[2,3,4]. I is completely enolized in the solid state (ir) but rapidly gives the ketoform in aprotic solvents (this is also evident from Table 1). II in aprotic solvents (ir) is transformed more rapidly into the ketoform than I; but shows less enol form in methanol than I. III is not enolised at all. The α-protons are highly acidic as in I and II, therefore the protons are immediately deuterated in CD_3OD.

References

1) H. Dürr, B. Eistert and K. Schank, Liebigs Ann.Chem. to appear.
2) B. Eistert and W.Reiss, Chem.Ber. 87, 92, 1o8 (1954).
3) B.Eistert und F.Geiss, Tetrahedron (London) 7, 1 (1959).
4) B. Eistert und G.Heck, Liebigs Ann.Chem. 681, 123 (1965).
5) H.Dürr, Ber.Bunsenges.phys.Chem. 69, 641 (1965).

Sincerely yours

(Dr. H. Dürr) (Prof. Dr. B. Eistert)

HARVARD MEDICAL SCHOOL
THE DEPARTMENT OF PHARMACOLOGY
25 SHATTUCK ST., BOSTON, MASS. 02115

November 25, 1966

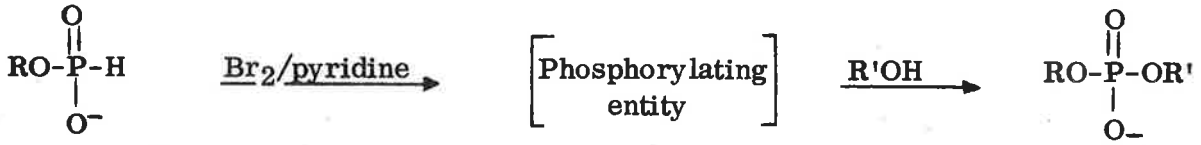
Professor B. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

P^{31} Chemical Shift of Trialkyl Trimetaphosphate in a Phosphorylating System?

Dear Professor Shapiro:

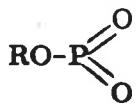
May I make my debut in your illustrious Illinois I.T.N.M.R. Newsletter with the following:

I have attempted to use P^{31} n.m.r. to detect the intermediates in a phosphorylation reaction. Most phosphorylating systems generated from monoalkyl phosphate are inapplicable due to the interaction of the incipient phosphorylating agent with the phosphate anion. For this reason the system generated on bromine oxidation of monoalkyl phosphonate[1] was chosen. This starting material (I, R = Et, $\delta = -3.2$ p.p.m., $J_{PH} = 630$ c.p.s.) is rapidly removed on addition of bromine.[1]

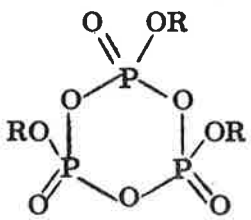


I

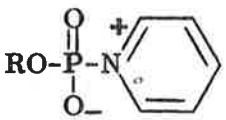
A number of common intermediates have been suggested to occur in phosphorylation reactions, i.e. monomeric metaphosphate II[2], trimeric metaphosphate III[3] and N-phosphoryl pyridine IV[4].



II



III



IV

Professor B. Shapiro November 25, 1966

Chemical shift values which might correspond to II and IV were not observed (as expected !) after the 10 - 15 mins. required for filtering the reaction solution (1 M in 10 ml.) under vacuum from pyridinium bromide, sealing the n.m.r. tube and reaching temperature equilibrium in the spectrometer. However, the values which were obtained were noticeably lower than the chemical shifts quoted by Schwarzmann and Van Wazer[5] and Burckhardt, Klein and Calvin[6] for terminal (P_t) and medial (P_m) polyphosphate groups. As controls I repeated their preparations for which I obtained the same values (± 1 p.p.m.),

		δ p.p.m. (from 85% H_3PO_4)			
phosphorylation system (R = Me, Et)[1]	- A	P_t	+9	P_m	+20
Van Wazer preparation[5]	- B		+13		+30
Calvin preparation[6]	- C		+15		+29

It is unlikely that such differences could be attributed to solvent effects[7]. The difference between the values for P_t in system A and systems B and C presumably results from the difference in degree of esterification and ionization in the group. The production of sym. P^1P^2-dialkyl pyrophosphate is to be expected since the bromine oxidation is extremely sensitive to traces of water (yields of dialkyl phosphate were always moderate, although consistent even on delayed addition of alcohol[1]).

The interpretation of P^{31} n.m.r. spectra of Langheld ester preparations in terms of cyclic metaphosphates[6] has recently been questioned.[8] Values for the linear and cyclic polyphosphates would not be expected to be identical[6]. Thus, there are differences between the chemical shifts for sodium tripolyphosphate (+ 18) and trimetaphosphate (+ 21)[7], and between acyclic (+1), five-membered (- 15), and six-membered (+ 8) cyclic phosphate triesters[9]. It is, therefore, tempting to suggest that the low field value observed for P_m in system A corresponds to the cyclic trialkyl trimetaphosphate IV and that $P_{mlin} - P_{mcyclic} = 10$ p.p.m. Although no concrete evidence has even been presented to support the existence[3] of such structures they would appear to be more plausible in an oxygen-deficient phosphorylating system than in an equilibrated polyphosphate system[8].

Qualitative interpretations of chemical shift data are always dangerous but often instructive.

Professor B. Shapiro

November 25, 1966

I should point out that this work was carried out at the Isotope Department, Weizmann Institute of Science, Rehovoth, Israel, with the technical assistance of Dr. Daniel Fiat.

Yours sincerely,

Jack Cohen

Jack S. Cohen

References:

1. G.M. Blackburn, J.S. Cohen and Lord Todd: J. Chem. Soc., (C) 239, (1966).

2. A.R. Todd: P.N.A.S., 45: 1389, (1959).

3. G. Weimann and H.G. Khorana: J.Am. Chem. Soc. 84: 4329, (1962).

4. N.K. Hamer: J.Chem. Soc. 326, (1964).

5. E. Schwarzmann and J.R. Van Wazer: J.Am. Chem.Soc. 83: 365, (1961).

6. G. Burckhardt, M.P. Klein and M. Calvin: J. Am. Chem. Soc. 87: 591, (1965).

7. R.A.Y. Jones and A.R. Katritzky: Angew. Chem. Int. Ed. 1: 32, (1962).

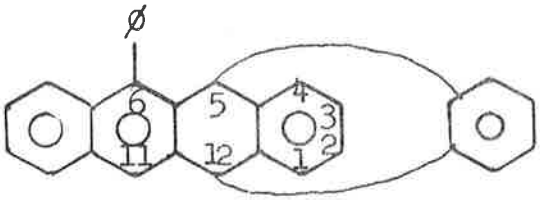
8. J.R. Van Wazer and S. Norval: J. Am. Chem. Soc. 88: 4415, (1966).

9. G.M. Blackburn, J.S. Cohen and Lord Todd: Tetrahedron Letters, 2873, (1964).

Sir,

Wilcox and Roberts[1] have reported that the chemical shift of the bridgehead proton in 2,5-diphenyl triptycene is at lower field than in triptycene itself, whereas ring current anisotropy predicts an upfield shift of 0.5 ppm. They invoked steric compression[2] to explain the shift to lower field. In 2,5-diphenyl triptycene some uncertainty exists about the angle of twist of the pendant phenyls and therefore some doubt as to the significance of the observed paramagnetic shift.

We have prepared 6-phenyl-5,12-o-benzo-5,12-dihydronaphthacene (benzotriptycene) in which the pendant phenyl is forced more completely into a perpendicular relationship to the naphthalene system. The bridgehead proton adjacent to the pendant phenyl is shifted to higher field, but only by 0.18 ppm, compared to the bridgehead proton at C-12. The shift estimated from the Johnson-Bovey diagram,[3] assuming complete perpendicularity of the pendant phenyl is 2 ppm.



We have also prepared 6-methyl-5,12-o-benzo-5,12-dihydronaphthacene in which the bridgehead proton adjacent to methyl falls at 0.38 ppm to lower field than the proton at C-12.

The data provide further examples of the effect of steric compression on chemical shifts, vividly exemplified in the compounds of Winstein, et al.,[2c] and point out the dangers attendant on using ring current anisotropy in structural assignments where the diamagnetic effect of aromatic ring currents is involved. The larger the ring current shift expected, the more likely will it be compensated by an equivalent steric compression shift.

---

[1] C. F. Wilcox, Jr. and F. D. Roberts, J. Org. Chem., 30, 1959 (1965).

[2a] C. Reid, J. Molec. Spect., 1, 18 (1957); [b] T. Schaeffer, W. F. Reynolds and T. Yonemoto, Can. J. Chem., 41, 2969 (1963). [c] S. Winstein, P. Carter, F.A.L. Anet and A.J.R. Bourn, J. Am. Chem. Soc., 87, 5247 (1965).

[3] C. E. Johnson, Jr. and F. A. Bovey, J. Chem. Phys., 29, 1012 (1958).

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99-20

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23rd November, 1966. Your Ref.

Our Ref. JKB/CF

Associate Prof. B. L. Shapiro,
Chemistry Department,
Illinois Institute of Technology,
Chicago, 60616,
U.S.A.

Dear Professor Shapiro,

Thank you for reminding me that a contribution to the Newsletter is due. I should like to offer some thoughts on:

Proton exchange involving more than two sites

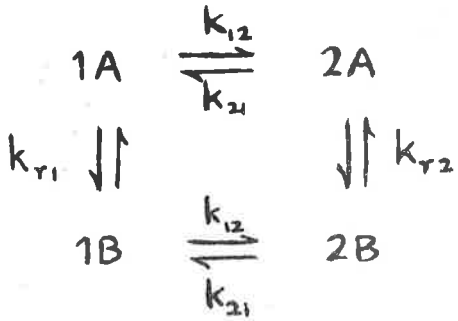
Exchange between two sites when the rate constants are comparable with the difference in resonance frequencies is readily treated using the analysis of Gutowsky and Holm (J. Chem. Phys., 25, 1228 (1956)); measurements on the spectrum over a suitable range of temperatures lead to a straight-line Arrhenius plot which gives the energy of activation for the exchange process. The equations relating the behaviour of the spectrum to the mean lifetimes have been generalised for cases where any number of sites is involved, but so far as I know there exists no general treatment including the reaction kinetics whereby the temperature-dependence of the spectrum can be predicted.

We have been interested in some examples that appear to involve four sites, where hindered internal rotation occurs in molecules that simultaneously are involved in a chemical equilibrium. This type of situation is fairly common; the chemical equilibrium concerned may range from a major tautomerisation or chemical reaction (reversible) to merely the formation and breaking of a hydrogen bond or a loose molecular association. A simple but important case is when a 180° rotation leaves the molecule structurally unchanged but results in exchange of two protons at sites A and B. If such a process is possible in both of the species 1 and 2 in equilibrium, we have the following situation, in which we label the states A or B according to the site occupied by a particular proton.

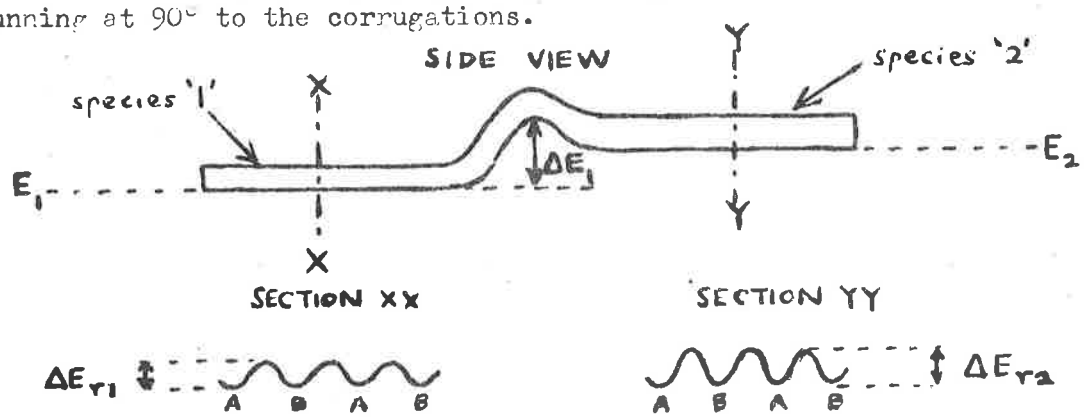
Contd.....

FROM: J. K. Becconsall. CONTINUATION SHEET NO.: 2

TO: Professor B. L. Shapiro. DATE: 23rd November, 1966.



The potential energy function for this system can be represented as a surface which resembles a sheet of corrugated iron with a kink running at 90° to the corrugations.



Rotation corresponds to moving across the corrugations, alternate troughs being labelled 'A' or 'B'.

In equilibrium the numbers of molecules in the four states are related as follows:

$$N_{1A} = N_{1B} \qquad N_{2A} = N_{2B}$$

$$\frac{N_{1A}}{N_{2A}} = \frac{N_{1B}}{N_{2B}} = e^{(E_2 - E_1)/KT}$$

Rate constants are given by:

$$k_{12} = A_0 e^{-\Delta E_1/KT} \qquad k_{21} = A_0 e^{-(\Delta E_1 + E_1 - E_2)/KT}$$

$$k_{r1} = A_1 e^{-\Delta E_{r1}/KT} \qquad k_{r2} = A_2 e^{-\Delta E_{r2}/KT}$$

A particular case of interest is when the 1 <—> 2 equilibration is relatively fast, resulting in a spectrum corresponding to averaged proton sites, while the rotation process A <—> B occurs at a slow or intermediate rate. When rotation is slow so that the A and B environments give distinct nuclear resonances, the temperature dependence of the peak positions is determined by the variation in

Contd.....

the ratio

$$\frac{N_{1A}}{N_{2A}} = \frac{N_{1B}}{N_{2B}}$$

(see above), which affects the weighted-average environment. If E_2-E_1 can be determined from variable temperature measurements under these conditions, the variation in proton resonance frequencies can be extrapolated to higher temperatures where rotation is occurring at an intermediate rate and the observed peaks are starting to coalesce. Comparison of the extrapolated and observed peak separations gives the rate constant for A <—> B interconversion. The theoretical frequency of A —> B processes is

$$N_{1A} k_{r1} + N_{2A} k_{r2}$$

Equating this to

$$(N_{1A} + N_{2A}) k_r (\text{eff})$$

gives us the effective rate constant for internal rotation

$$k_r(\text{eff}) = \frac{N_{1A} k_{r1} + N_{2A} k_{r2}}{N_{1A} + N_{2A}}$$

$$= \frac{A_1 e^{-\Delta E_{r1}/kT}}{1 + e^{-(E_2-E_1)/kT}} + \frac{A_2 e^{-\Delta E_{r2}/kT}}{1 + e^{(E_2-E_1)/kT}}$$

It is seen, therefore, that an Arrhenius plot based on the apparent rotational rate constant for a two-site exchange, even though allowance is made for weighted-average shifts due to changes in the ratio of molecular species, will not in general give a straight line.

A further important special case is when 1 <—> 2 equilibration is again fast, but rotation in one species is virtually forbidden, i.e.

$$k_{r2} \approx 0$$

The frequency of A —> B processes is then

$$N_{2A} k_{r2} = \frac{(N_{1A} + N_{2A}) A_2 e^{-\Delta E_{r2}/kT}}{1 + e^{(E_2-E_1)/kT}}$$

$$\equiv (N_{1A} + N_{2A}) k_r (\text{eff})$$

FROM: J. K. Becconsall.

CONTINUATION SHEET NO.: 4

TO: Professor B. L. Shapiro.

DATE: 23rd November, 1966.

where $$k_r(\text{eff}) = \frac{A_2 e^{-\Delta E_{r2}/kT}}{1 + e^{(E_2-E_1)/kT}}.$$

Again an Arrhenius plot based on the apparent rotational rate constant (with allowance for weighted-average shifts) will not in general give a straight line. However, if

$$e^{(E_2-E_1)/kT} >> 1$$

(i.e. the equilibrium is strongly in favour of species 1), we get

$$k_r(\text{eff}) \approx A_2 e^{-(E_2-E_1+\Delta E_{r2})/kT}$$

and a straight line Arrhenius plot results, with the apparent activation energy

$$\Delta E_r(\text{eff}) = \Delta E_{r2} + (E_2 - E_1).$$

Note that in this case the weighted-average shift correction is also negligible; the behaviour of the n.m.r. spectrum is therefore indistinguishable from that expected for a simple two-site exchange.

Some examples of cases like those mentioned above will be described in forthcoming papers.

Yours sincerely,

J.K. Becconsall

J. K. Becconsall

0030A

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Ref. Vlaardingen, 28-10-1966.

Prof. Dr. B.L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois, 60616.
U.S.A.

Dear Dr. Shapiro,

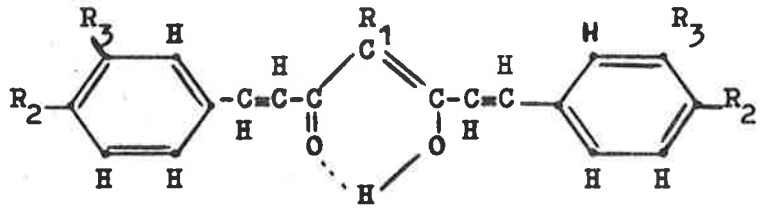
Please, accept this contribution, entitled:

"Keto-enoltautomerism in unsaturated β-dicarbonyls"

as my "renewal" for the NMR-Newsletter.

As part of an investigation by PMR of keto-enoltautomerism in a variety of compounds we ran the 60 Mc spectra (Varian A-60) of a series of unsaturated β-dicarbonyls in several solvents, as well as in the molten state. The table shows the structural formulas and enolcontents of a few compounds.

Table:



Compound:	R_1	R_2	R_3	Solvent	Temp. (°C)	% enol
I	H	H	H	none	150	90
				pyr. or acetone	38	100
II	H	CH_3O-	H	none	170	60
				acetone	40	100
III	CH_3-	HO-	CH_3O-	none	170	50
				d_5-pyridin	40	67
				d_5-pyridin	100	38

Ref.

The high enolcontent of all these unsaturated conjugated derivatives as compared with saturated diketones, such as nonane-2,4-dione (80% enol in CCl_4) is consistent with the observed complete enolization of dibenzoylmethane in CCl_4[1]. The extended conjugated system weakens the C=O bond and strengthens the hydrogen bond by resonance[2] when coplanarity of enolic and aromatic rings and of the trans double bonds is assumed. Molecule models indicate that this is possible.

The undiluted molten substances (synthesized by Pabon[3]) are enolized to a lesser extent (60-90%) than the dissolved substances (100%). The high temperature required to obtain spectra of the melts is the main cause of the observed incomplete enolization. In several solvents with high polarity (pyridin) as well as low polarity (CCl_4) enolization is complete at 40°C. Only when an hydroxy substituent is present in the aromatic ring do the solutions show enolcontents lower than 100%. Most probably in these compounds the carbonyl groups in the ketoisomer are hydrogenbonded intermolecularly to the hydroxy groups.

Incorporation of an α-methylgroup as in methylcurcumin,III, further lowers the enolcontent to 50%. Such a lowering was also observed for acetylacetone and α-methylacetylacetone[4].

In d_5-pyridin at 100°C III exhibits the highest keto percentage in the series studied, viz. 62%.

Interesting in the spectrum of III is the occurrence of a split methoxyresonance. The lower field peak (δ=3.77) is from the methoxyprotons in the enol isomer, for upon raising the temperature its intensity is increased at the expense of the intensity of the higher field signal (δ=3.71). A through space shielding effect of the enolic ring must be operative because in several β-keto esters we observed an unsplit $-OCH_2-$triplet. Here the methylene protons are separated by 3 bonds and in III the methoxy protons are separated by 8 bonds from the enolic ring system.

1) J.L. Burdett, M.T. Rogers, J.Am.Chem.Soc.86,2105(1964)

2) R.L. Lintvedt, H.F. Holtzclaw,Jr., J.Am.Chem.Soc.88,2713(1966)

3) H.J.J. Pabon, Rec. Trav. Chim. 83, 379(1964)

4) H.S. Jarrett, M.S. Sadler,J.N.Shoolery, J.Chem.Phys.21,2092 (1953)

Sincerely Yours,

M. van Gorkom

M. van Gorkom

99-26

varian/611 hansen way/palo alto/california 94303/u.s.a./415/326-4000

November 22, 1966

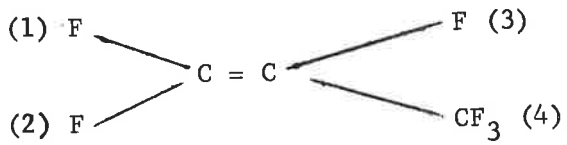
Mrs. B. L. Shapiro
3100 S. Michigan Avenue
Apartment 502
Chicago, Illinois 60616

Dear Lee:

The Dependence of F^{19} Coupling "Constants" on Solvent and Concentration

We wish to report a rather dramatic and convincing demonstration of the dependence of F^{19} spin-spin coupling on concentration and solvent.

Reilly and co-workers (H.M. McConnell, C.A. Reilly, and A.D. McClean, J. Chem. Phys., 24, 479, 1956; J.D. Swalen and C.A. Reilly, ibid, 34, 2122, 1961) first published the F^{19} NMR spectrum of perfluoropropylene



and determined the chemical shifts and coupling constants. It was particularly striking in their work that the two centermost transitions of the CF_3 absorption multiplet were exactly superimposed to yield a single line of double intensity. The latter situation arises from the fortuitous fact that the sum of J_{14} and J_{34} is virtually equal to J_{24}.

We have examined the 56 MHz spectrum of a sealed sample of liquid perfluoropropylene, containing a few per cent of CCl_3F. Although our derived coupling constants differ somewhat from those reported earlier, there is still not the slightest hint of separation in the centermost CF_3 lines (Fig. Ia):

$\emptyset_1$ = 94.1 ppm	J_{12} = 58.1 Hz	J_{23} = 117.7
$\emptyset_2$ = 107.6	J_{13} = 39.8	J_{24} = 21.2
$\emptyset_3$ = 193.1	J_{14} = 8.5	J_{34} = 12.7
$\emptyset_4$ = 69.4		

($\emptyset$ in ppm relative to internal CCl_3F)

It was clear that the aforementioned lines were resolvable if we could induce (non-compensating) changes in the coupling constants by as little as a fraction of a Hz. Such changes could be achieved by exploiting the slight dependence of the coupling constants on solvent, concentration, and temperature. Second-order perturbation effects, of course, are entirely negligible in this example because of the large chemical shifts involved. Furthermore, we are not concerned about effects of magnetic non-equivalence in the rotating CF_3 group —— the latter being an isotropic substituent on the double bond system.

For a gross change in concentration, we merely reversed the solute-solvent relationship of our original sample. Fig. Ib depicts the CF_3 absorption in a sample of CCl_3F saturated with C_3F_6 vapors at 1 atm pressure. When we switched to a solution of C_3F_6 vapors in CCl_4 (Fig. Ic), baseline resolution of the center lines could actually be attained. The changes involved are summarized as follows:

	J_{14}	J_{34}	J_{24}	$(J_{14} + J_{34})-J_{24}$
C_3F_6 (g) in CCl_3F	8.5	13.0	21.0	0.5
C_3F_6 (g) in CCl_4	8.3	13.2	20.7	0.8

In the CCl_3F solution, it was further noted that J_{13} decreased to 39.1 Hz while J_{23} remained essentially unchanged. This led to a coincidental merger of lines (the 4th and 5th, and the 12th and 13th lines) in the spectrum of F(3), these lines having been previously separated by 1.7 Hz in liquid C_3F_6 (Figs. IIa, b). The relatively small changes in the individual coupling constants had been amplified, as it were, into readily detectable quantities by the repeated spacings in the multiplets.

The results of our temperature study were not as strikingly successful, because we encountered difficulty maintaining high resolution as the temperature of liquid C_3F_6 was lowered. Yet, it could be determined that J_{14} decreased by a fraction of a Hz, while J_{24} and J_{34} increased as the temperature was dropped from ambient to -50°C.

These experiments were performed about a year ago on a Varian A-56/60 NMR spectrometer at Allied Chemical Corp., Central Research Laboratory, Morristown, New Jersey. This instrument has been superseded by the A-56/60A spectrometer, which has a greater inherent sensitivity.

Regards to Barry.

Sincerely,

Ray E.

Raymond Ettinger
Spectroscopy Applications Laboratories
Analytical Instrument Division
Varian Associates

RE:jc

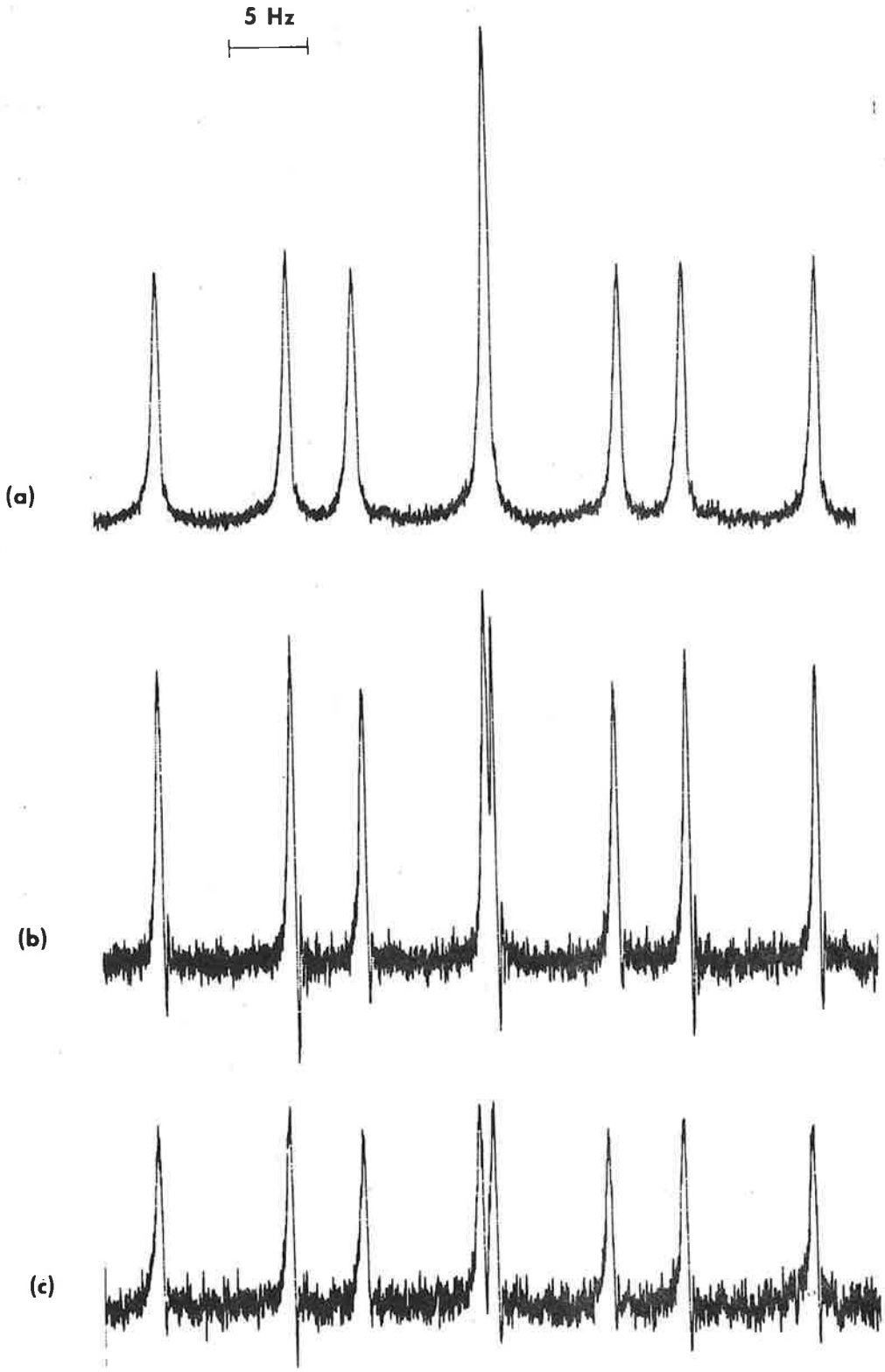


Fig. 1 F^{19} NMR Spectrum of $CF_2=CF(CF_3)^*$ at 56.4 MHz (a) Liquid C_3F_6; (b) C_3F_6 (g) Dissolved in CCl_3F, 1 atm; (c) C_3F_6 (g) Dissolved in CCl_4, 1 atm

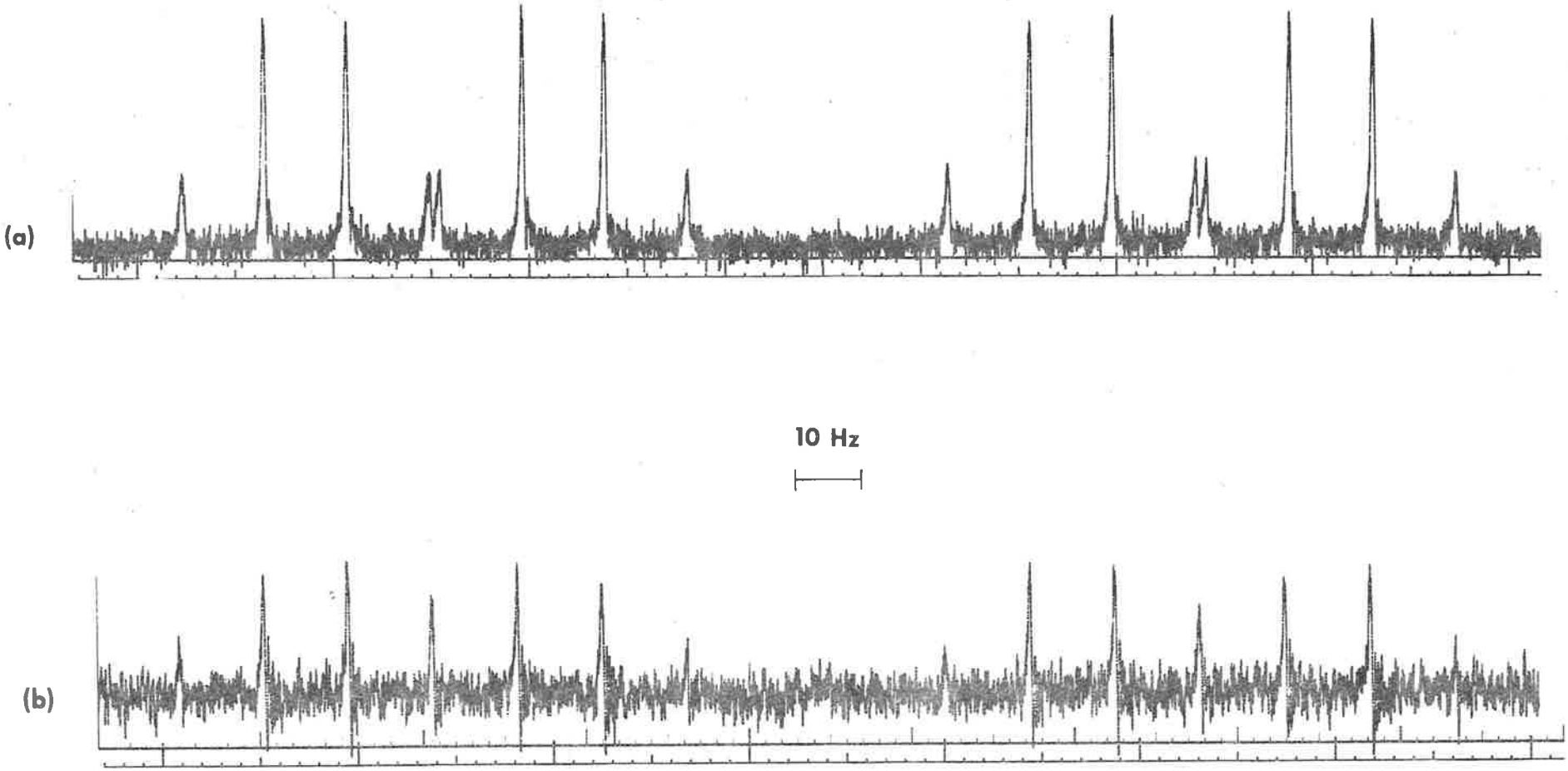


Fig. II F^{19} NMR Spectrum of $CF_2=CF^*(CF_3)$ at 56.4 MHz (a) Liquid C_3F_6; (b) C_3F_6(g) Dissolved in CCl_3F, 1 atm .

99-30

PHYSIKALISCHES INSTITUT
DER UNIVERSITÄT BASEL
KLINGELBERGSTRASSE 82 - TEL. 43 04 22
VORSTEHER: PROF. DR. P. HUBER
Prof. Dr. P. Diehl

Basel, November 23, 1966

Prof. B.L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Ill. 60616

U.S.A.

The limitations of generalisation in subspectral analysis of NMR - spectra.

Dear Barry,

When the method of subspectral analysis was expanded to systems of magnetically non equivalent nuclei as, e.g., AA'XX', ABB'XX' and AA'A''XX'X'' (1) it seemed at first, that there were no limitations to this procedure. The parts of Hamiltonians of a complex system which belong together through selection rules and have the correct dimension for comparison with Hamiltonians of simpler problems were supposed to be transformable into the latter. It turned out, however, in the case of an AA'A''A'''XX'X''X''' (2) system that there arose contradictions in the subspectral transformations indicating the existence of limitations in the method of subspectral analysis.

As a consequence we had to reformulate the method of obtaining sub-spectral transformations, so that the conditions and limitations of transformability became obvious.

The result of this work was, that sub-patterns of energy levels of type (1 : 2 : 1) =  and (1 : 3 : 3 : 1) can always be transformed into ab- and abc-subspectra, whereas the pattern (1 : 2 : 4 : 2 : 1) e.g. does not necessarily correspond to the symmetrical part of an aa'bb' subspectrum. In spectra of the type AA'A''A'''XX' the failure of the transformation method can be shown to be due to the mixture of symmetry species products in the 4 x 4- matrices.

We will discuss these matters in more detail in a forthcoming paper.

Yours sincerely,

Peter D. Trautmann

P. Diehl and D. Trautmann

(1) P. Diehl, Helv.Chim.Acta 48, 567, 1965
(2) R.G. Jones and S.M. Walker, Molec.Phys. 10, 363, 1966.

99-32

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TEL. 292.109 - 292.110

Milan, November 15th, 1966

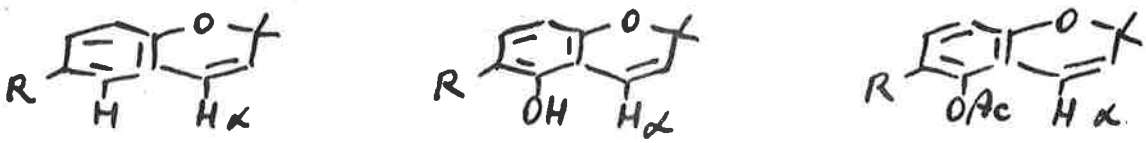
Professor B.L.Shapiro
Department of Chemistry
Illinois Institute of Technology
Technology Center
CHICAGO, Illinois 60616

NMR Study of some 5-hydroxychromenes

Dear Professor Shapiro,

I am very sorry you had to remind me. I hope the following will put me back in your mailing list.

Since a number of 5-hydroxychromenes were available during the study of some natural substances, we have found, with acetylation of the hydroxyl, an interesting effect on the chemical shift of the α proton. The acetyl group causes a marked diamagnetic shift (0.3-0.4 δ) of the hydrogen in peri position (H-α) and a small paramagnetic shift (~0.1 δ) of H-β (Tab. 1).
If we compare the chemical shift of H-α in compounds :

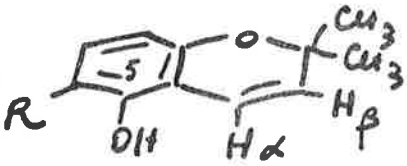


		I	II	III
a	R = H	6.23	6.56	6.26
b	R = $COCH_3$	6.31	6.66	6.36

we see that acetylation compensates the downfield shift induced by the OH.

The deshielding of H-α due to the hydroxyl (peri) is about of the same order (0.3-0.4 δ) of the "peri effect" found by Dudek in naphtalenes (1) (~ 0.4 δ). This effect may be explained, as in α-naphtol, by an interaction of H-α with the electron pair of the oxygen in the preferred trans configuration (electron pair cis to H-α) :

II a II b



Tab. 1

R		CCl_4 OH	CCl_4 OAc	$CDCl_3$ OH	$CDCl_3$ OAc	acetone OH	acetone OAc	DMSO OH	DMSO OAc	CCl_4	$CDCl_3$	acet.	DMSO
H	α	6.56	6.26	6.62	6.33	6.66	6.39	6.57	6.37	+0.30	+0.29	+0.25	+0.19
	β	5.47	5.55	5.57	5.64	5.58	5.75	5.62	5.81	−0.08	−0.07	−0.17	−0.19
$COCH_3$	α	6.66	6.36	6.71	6.38	6.67	6.48	6.60	6.45	+0.30	+0.33	+0.21	+0.16
	β	5.47	5.64	5.57	5.71	5.73	5.88	5.75	5.90	−0.17	−0.14	−0.16	−0.17
$COOCH_3$	α	6.68	6.36	6.71	6.39	6.70	6.52	6.61	6.48	+0.32	+0.32	+0.18	+0.13
	β	5.48	5.60	5.57	5.68	5.74	5.87	5.74	5.91	−0.12	−0.11	−0.13	−0.17
$CO-CH=CH-Ph$	α	6.79	6.34	6.76	6.38	6.72	6.50	6.66	6.49	+0.45	+0.38	+0.22	+0.16
	β	5.52	5.64	5.60	5.72	5.75	5.90	5.77	5.93	−0.12	−0.12	−0.15	−0.17

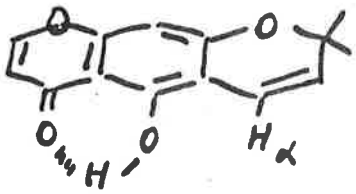
The similar shift of H-α in IIa (6.56 δ) and in IIb (6.66 δ), where intramolecular H-bond locks the OH in the trans configuration, can prove this. The small difference ($\Delta\delta = +0.10$) is attributed to the mesomeric or inductive effect of the substituent $-COCH_3$ experienced also by H-α and H-β . About the same value ($\Delta\delta = +0.08$) is thus found if we compare Ib (R = $COCH_3$) and Ia (R = H).

Now with acetylation :

i) or a trans configuration is still preferred, and the electron pair is delocalised on the ester group, consequently the deshielding of the hydroxyl is destroyed ,

ii) or the cis and trans configurations in the acetates are equally probable, and the deshielding effect of the oxygen is compensated by the shielding of the carbonyl.

This second hypothesis is more probable if we consider a series of compounds of the type : (see tab. 2)



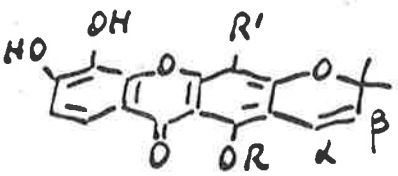
where chelation occurs as in IIb .

With acetylation we get for H-α about the same $\Delta\delta$ (0.25–0.30) as in IIb (0.33) ($CDCl_3$). Since in the former acetates there are two peri interactions, a fixed trans configuration is unlikely. Consequently the preferred position of the acetyl group seems intermediate, with the plane of the C=O perpendicular to the plane of the aromatic system, in such a way that the shielding conical region of the carbonyl comprises also H-α .

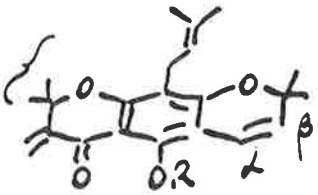
However it cannot be excluded that a certain delocalisation of the electron

Tab. II

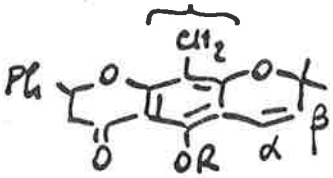
$CDCl_3$



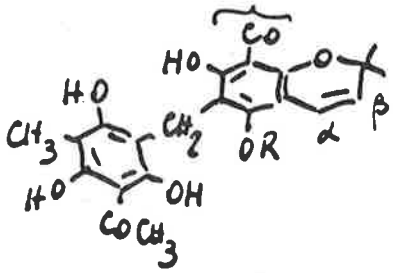
		H-α	Δδ
Jacareubin R' = H			
diacetate	R = H	6.75	+0.25
triacetate	R = Ac	6.50	
Macluraxanthone R' = $C(CH_3)_2CH=CH_2$			
diacetate	R = H	6.75	+0.26
triacetate	R = Ac	6.50	



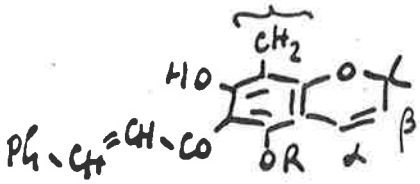
Deoxymorellin (3)	R = H	6.63	+0.28
	R = Ac	6.35	



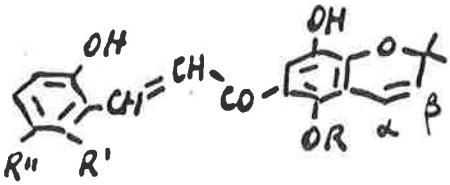
Isoallorotlerin			
triacetate	R = H	6.65	+0.30
tetracetate	R = Ac	6.35	



Rottlerin	R = H	6.66	+0.50
pentacetate	R = Ac	6.16	



Allorottlerin			
tetracetate	R = H	6.68	+0.53
pentacetate	R = Ac	6.15	



B) triacetate	R = H	6.78	+0.37
tetracetate	R = Ac	6.41	
C) triacetate	R = H	6.78	+0.36
tetracetate	R = Ac	6.42	

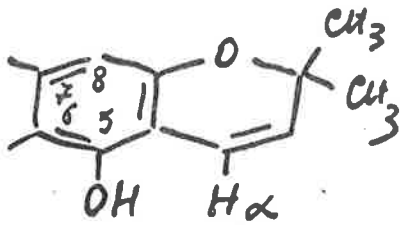
Flemingin B R' = OH R'' = H
" C R' = H R'' = OH

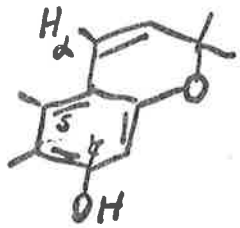
pair on the ester group may contribute to this effect.

When R is a bulky substituent (Lonchocarpin , Rottlerin) one may suggest that the acetyl rotates a little toward the chromene double bond; as a consequence H-α becomes nearer to the C=O and experiences a stronger shielding (due to the anisotropic magnetic susceptibility and to the electric dipole moment of the substituent). An increased value of the shift is thus found ($\Delta\delta$ = +0.45 and resp. 0.50-0.53).

We have extended this investigation to some known natural chromenes (tab. 2) and applied the results to structure elucidation of the new Flemingins (2).

The presence of a chromene ring system in a natural substance can now be easily recognized with NMR spectroscopy. Because, however, most natural chromenes show at least a resorcinol or a more complicated pattern, difficulties are still encountered in the establishment of the ring closure, i.e. in the distinction between the "angular" and "linear" structure in polycyclic compounds :





The acetylation results may be useful, the only method available being the repeatedly questioned Gibbs reaction.

The acetylation of hydroxyls in 6 , 7 or 8 positions of model chromenes does not show any effect on H-α .

The comparison is best made in $CDCl_3$ or CCl_4 because $\Delta\delta$ decreases in solvents such acetone and especially DMSO (see tab. 1) : they give association with the polar groups of the molecule and thus H-α becomes protected.

Yours sincerely

R.Mondelli

R.Mondelli

(1) G.O.Dudek, Spectrochimica Acta, 19, 691 (1963)
(2) presented at the IUPAC Symposium on "The Chemistry of Natural Products" Stockholm, June 1966 , G.Cardillo, L.Merlini, R.Mondelli.
(3) K. Venkataraman, private communication

99-36

THE PENNSYLVANIA STATE UNIVERSITY

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Materials Research Laboratory

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November 14, 1966

Professor Barry Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry:

I thank you for your recent letter reminding me of "overdue" contribution. I received your letter a couple of days ago and I trust that this contribution will reach you in time.

At the present time, my research group is working on two different aspects of N.M.R.:

1) We are doing some work on the broad-line N.M.R. of"low molecular weight" polymers (polystyrene). We expect to get a better understanding of the nature of the molecular motion and the "glass transition" in these polymers. We expect to have a note ready soon and will send this to you in a couple of months.

2) Another research involves explaining the anomalous high resolution chemical shifts in metallocene compounds. See figure on attached sheet.

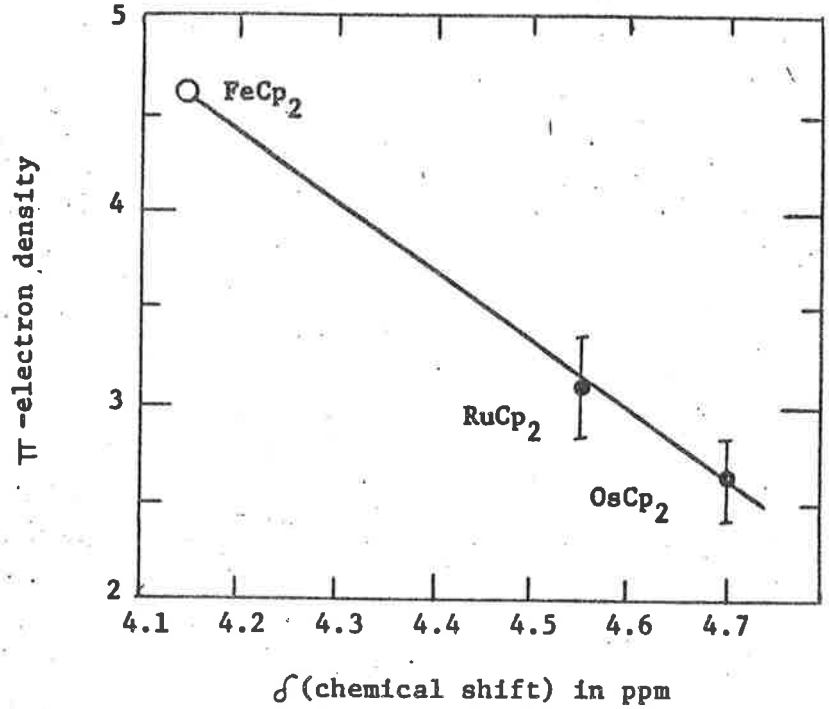
As explained in our note (Mulay et al. J. Chem. Phys. 43, 4522, 1965) the chemical shifts seem to decrease with increasing π-electron density. Thus the protons on the Cp rings may be experiencing "deshielding effects" (from diamagnetic ring currents) which may be masked by some "d-orbital" or "core diamagnetism" effects from the metal atoms (Fe, Ru, and Os, which are respectively 3d, 4d, and 5d atoms) We are now doing some simple "point dipole" calculations to calculate such opposing effects. In spite of these difficulties, it is gratifying to note that our π-electron densities beautifully fit Batsanov's M.O. data. (see figure on attached sheet)

I will keep in touch with you concerning the progress in our wok. We all enjoy reading your excellent newsletter and wish to express our appreciation for your spendid efforts.

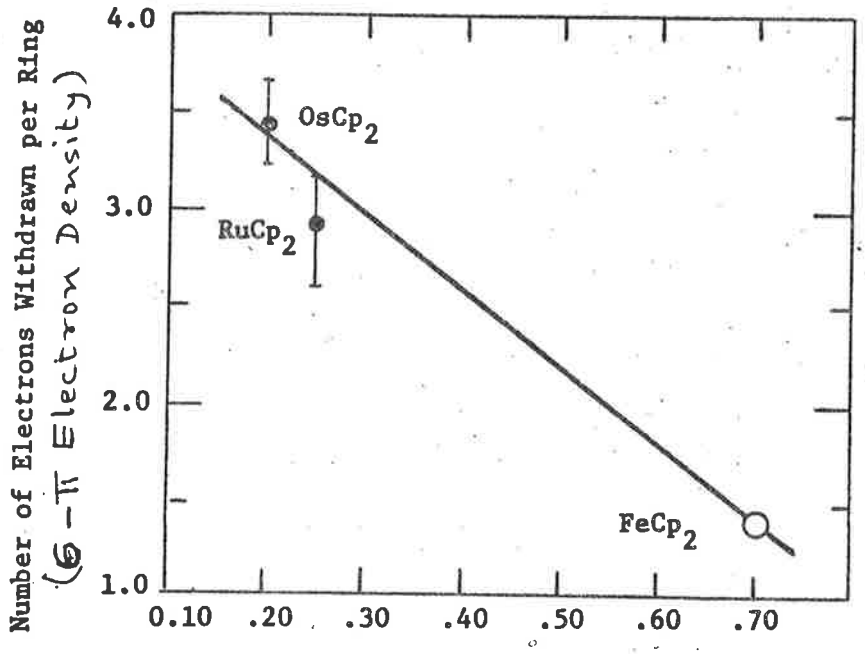
Yours sincerely,

L. N. Mulay
Associate Professor

LNM/blg



π-Electron Density of Cp Ring Vs. Chemical Shift



Number of Electrons Withdrawn per Ring Vs. Charge on Metal Atom

(Mulay et al. J. Chem. Phys. 43, 4522 (1966))

University of East Anglia

From Professor N. Sheppard

School of Chemical Sciences
Wilberforce Road, Norwich NOR 77H
Telephone Norwich 52651

22nd November, 1966.

Professor B.L. Shapiro,
Department of Chemistry,
Illinois Institute of Technology,
Technology Center,
Chicago, Illinois 60616.

Dear Barry,

I am writing to ask if I could renew my subscription to the IITNMR Newsletter. During the last year and a half I have been busy settling down in a new laboratory and have also, until recently, had rather limited NMR facilities. However, about nine months ago we obtained a Varian HA100 spectrometer and we are now hard at work again on NMR problems using this excellent instrument. As my new contribution I report below on some recent work on alkyl fluorides that Dr. Don L. Hooper, Mr. Chris Woodman and I have been recently concerned with.

Following our elucidation of the curious splitting patterns observed in the spectra of the n-alkyl fluorides[1], we have completed an examination of the 100 Mc/s spectra of n-propyl, n-butyl, n-amyl, n-hexyl and n-heptyl fluorides. All the spectra were complicated, and it was only possible to perform a usefully detailed analysis on the spectrum of n-propyl fluoride, but this gave some interesting results. The vicinal H,F coupling constant is 23.55 c/s, which is quite close to that in ethyl fluoride (25.2 c/s), confirming the microwave result[2] that there is only a small energy difference between the trans and gauche forms. The pure gauche isomer would give the larger coupling constant, i.e. $J_{HF} = \frac{1}{2}(J^g + J^t)$, while for the trans isomer, $J_{HF} = J^g$. However, at -41^oC, the mean coupling has increased to 23.94 c/s, indicating that the gauche isomer is more stable (as found by the microwave experiments), and that the fully-averaged coupling constant $(2J_g + J_t)$ must be considerably lower than that in ethyl fluoride, perhaps around 22 c/s. Since CH_3 is generally regarded as electropositive with respect to H this is in the reverse

direction to that suggested by electronegativity considerations.

In analysing the spectrum of n-propyl fluoride, we treated it as an $A_3B_2C_2X$ case. (Considerable inaccuracy resulted if the CH_2F protons were regarded as "M_2", even though they were shifted 300 c/s (at 100 Mc/s) from the remaining protons.) Fitting of the $-CH_2$ region was achieved with LAOCOON II, and to obtain the long-range couplings to the CH_3 group we compared the observed band contour with that calculated assuming a Lorentzian line-shape with constant half-width. Some trial computations were made taking into account the non-equivalence within the CH_2 groups (i.e. as an $A_3BB'CC'X$ system), but this produced only very slight variations in the line positions, and the fit was not usefully improved. This is in accordance with the writings of Abraham and Bernstein[2] on deceptively simple spectra: provided L^2/M is sufficiently small, no dependence of the spectrum on L will be detected. We are not sure however that Cavanaugh and Dailey, who analysed the spectra of other propyl compounds using the same assumption[3], were dealing with such favourable cases.

The parameters found for propyl fluoride were as follows:

$CH_3CH_2CH_2F$

$A_3B_2C_2X$

$\tau_A = 9.03$	$^3J_{AB} = 7.45 \pm 0.02$	$^2J_{CX} = 47.35 \pm 0.2$
$\tau_B = 8.32$	$^3J_{BC} = 6.20 \pm 0.02$	$^3J_{BX} = 23.55 \pm 0.05$
$\tau_C = 5.70$	$^4J_{AC} = -0.12 \pm 0.06$	$^4J_{AX} = +0.38 \pm 0.04$
$f^*_X = 218.7 \pm 0.4$		

(1) D.L. Hooper, N. Sheppard and C.M. Woodman, J.Chem.Phys. 45, 398 (1966).

(2) E. Hirota, J.Chem.Phys. 37, 283 (1962).

(3) J.R. Cavanaugh and B.P. Dailey, J.Chem.Phys. 34, 1094 (1961).

With kind regards,

Yours sincerely,

Norman

N. Sheppard.

99-40

Dr. Naegele/Dr. Wendisch in Fa.

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Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Technology Center

Chicago, Illinois 60616
USA

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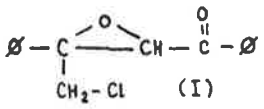
		Dr. N-Dr. We/Wi	Lev. 30	11.11.66

Dear Professor Shapiro!

"Magnetic Nonequivalence in a Methylene Group".

We wish to report an investigation into the nonequivalence of the two protons in the methylene group of the substituted epoxide I. The methylene protons adjacent to the asymmetric carbon atom exhibit a strongly solvent and temperature dependent AB-spectrum:



(I)

	I in DMSO-d_6 solution[x)]	I in trichloracetonitrile solution
C	328.0 c.p.s.	361.5 c.p.s.
J_{AB}	12.3 c.p.s.	12.3 c.p.s.
ν_1	298.7 c.p.s.	348.2 c.p.s.
ν_2	311.0 c.p.s.	360.5 c.p.s.
ν_3	345.0 c.p.s.	362.5 c.p.s.
ν_4	357.3 c.p.s.	374.8 c.p.s.
$\nu_0\delta$	± 44.64 c.p.s.	± 7.30 c.p.s.
ν_A	305.68 c.p.s. (= 5.095 τ)	357.85 c.p.s. (= 5.964 τ)
ν_B	350.32 c.p.s. (= 5.839 τ)	365.15 c.p.s. (= 6.086 τ)

x) T = 29°C; TMS = 600.0 c.p.s.

The energy barrier between the two exchanging sites of the AB-system was determined by measuring the temperature dependence of I in DMSO-d_6 solution. The calculations are based on the ALEXANDER-density-matrix-treatment of exchanging AB-systems[1] taking into account the coupling constant J_{AB}. Following the suggested procedure of R. KURLAND and W. WISE [2] we have written a somewhat modified computerprogram in FORTRAN IV. After approximately determining the maximum of the programmed signal-shape functions for absorption and dispersion mode we have set up a subroutine SEARCH in order to get clear-cut criteria for the convergence of the procedure. Determination of the energy barrier between the two exchanging sites yields a value of $E_a = 3.87 \pm 0.10$ Kcal/mole.

This value agrees well with the one obtained using the GUTOWSKY/HOLM-method[3] with the following modification : at each temperature (well above the point of coalescence) the chemical shifts ν_A and ν_B were determined separately and used instead of the usual two frequencies of a non-coupled system. The calculations were carried out on an IBM 7070/74 computer.

We hope this note will satisfy the conditions for being put on the mailing list of the IIT-NMR-Newsletters.

Sincerely Yours

W. Naegele D. Wendisch

(W. Naegele) (D. Wendisch)

---

1) S. ALEXANDER, J. Chem. Phys. 37, 967 (1962)

2) W. WISE and R. KURLAND, IIT-NMR-Newsletter 70, 62 (1964); private communication to one of us (D. W.) by Dr. R. KURLAND, Carnegie Institute of Technology, Pittsburgh Pa. (13/10/64).

3) H. S. GUTOWSKY and C. H. HOLM, J. Chem. Phys. 25, 1928 (1956).

99-42

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DEPARTMENT OF CHEMISTRY RIVERSIDE, CALIFORNIA 92502

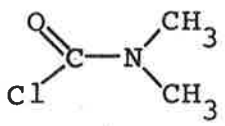
November 28, 1966

Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

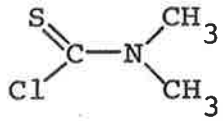
Title: Hindered Rotation in Amides and Thioamides

Dear Barry:

We are in the process of preparing a manuscript for publication dealing with hindered internal rotation in N,N-dimethylcarbamoyl chloride (I) and N,N-dimethylthiocarbamoyl chloride (II).



I



II

Our spectra have been recorded using an A-60 spectrometer equipped with the standard variable temperature equipment. Rate constants as a function of temperature have been determined by comparing the experimental spectra with calculated spectra generated on a Cal-Comp Plotter (Western Data Processing Center, UCLA) using the Gutowsky-Holm line shape equations. Care was taken to insure reproducibility of results and T_2 values for <u>each</u> spectrum were determined from the resonance signal of <u>internal</u> TMS.

Our data for neat dimethylcarbamoyl chloride give $E_a = 16.9 \pm 0.5$ k cal/mole and log A = 12.9 ± 0.4. Our first computer comparisons for I in CCl_4 give about the same values of E_a and log A. A summary of previous results is given in the Table below.

Activation Parameters for Neat DMCC

E_a	Log A	Method	Ref
7.3 ± 0.5	6.1 ± 0.3	Intensity Ratio	1
9.7 ± 0.5	7.7 ± 0.4	" "	2
8.6 ± 1.7	6.9 ± 1.2	Peak Separation	2
14.0 ± 0.9	10.9 ± 0.6	Spin Echo	2
16.9 ± 0.5	12.9 ± 0.4	Total Line Shape	Our Data

These data certainly seem to imply that kinetic studies using methods other than spin echo or total line shape analysis can be very misleading.[3]

The thio compound II is a solid, and was studied in CCl_4. Total line shape analyses on II are presently in progress. The preliminary data indicate a significantly higher rotational barrier[4,5], however the non-exchanging chemical shift is quite temperature dependent making the necessary line shape comparisons more tedious and less certain.

These studies are part of our larger program to compare amides, thioamides, amidines and amidinium ions.[5]

Sincerely yours,

Bob

Robert C. Neuman, Jr.
Assistant Professor of Chemistry

References

1.) M. T. Rogers and J. C. Woodbrey, J. Phys. Chem., 66, 540 (1962).

2.) A. Allerhand and H. S. Gutowsky, J. Chem. Phys., 41, 2115 (1964).

3.) A. Allerhand, H. S. Gutowsky, J. Jonas and R. A. Meinzer, J. Am. Chem. Soc., 88, 3185 (1966).

4.) A. Lowenstein, A. Malera, P. Rigny and W. Walter, J. Phys. Chem., 68, 1597 (1964).

5.) R. C. Neuman, Jr., and L. B. Young, ibid, 69, 2570 (1965).

99-44

CORPORATE HEADQUARTERS
8100 34TH AVENUE SOUTH, MINNEAPOLIS, MINNESOTA 55440 / 612-888-5555

CONTROL DATA

November 28, 1966

Professor Barry Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois

Dear Barry:

MATRIX DIAGONALIZATION ROUTINE

I think many of your readers probably would be interested in a faster method for diagonalizing matrices, whether it be for NMR Spectrum Calculations or for other similar problems.

Most people use Jacobi Method Routines which take a time proportional to N^3 for solution where N is the order of the square symetric real matrix being diagonalized. Faster routines, routines based on the Householder method, have been available for several years. These are particularly attractive for matrices of large order since the method takes a time proportional to N^2. However, the method had the disadvantage that matrices with multiple eigenvalues gave identical eigenvectors associated with multiple eigenvalues.

Recently, the Biostatistics Laboratory at the University of Miami has developed a variant on the Householder method which gives orthogonal eigenvectors for multiple eigenvalues while retaining the speed advantage of the Householder method.

The author, Richard Sherin, has given his permission that it be released. I will be glad to send a listing to anyone interested.

Sincerely,

CONTROL DATA CORPORATION
Data Centers Division

Charles

Charles B. Krabek
Analytical Services Department

lh

DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
FOOD AND DRUG ADMINISTRATION
WASHINGTON, D.C. 20204

December 1, 1966

Prof. B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Computer Retrieval of NMR Abstract Cards. A Progress Report.

Dear Barry,

This is a follow-up on the work of the retrieval of NMR information from Preston cards by computer, which was described in ITTNMRNL 91-24 (April 1966) by Ted Becker.

We have extended the coverage of our system to all cards put out by Preston, that is, also to abstractless cards. Further additions are key words and journal codes; their appearance on the printout makes it easier to decide which abstracts to look up in the actual card file.

For help in working up the abstractless cards and in providing key words and publication codes, we are indebted to W.B.Moniz and C.Poransky (NRL), and to T. Alexander, N. Duy, S. Koch, and M. Lake (FDA).

At the bottom of the page there is a partial printout of a listing of cards having Preston code number 3.

Best regards and wishes.

Sincerely yours,

Ernest Lustig

a	b		c	d
6A-1401 BARLOW	NIXON		AMINODIFLUOROPHOSPHINES	J30
6A-1445 LIPPMAN			O,O-DIMETHYL AND O,O-DIETHYLDITHIOPHOSPHORIC FROM PHO	J59
6A-1446 RAMIREZ	PATWARDHAN	SMITH	UNSYMMETRICALLY -DIKETONES OXYPHOSPHORANE	J58
6A-1451 PINKUS	WILDREP		COUPER,S COMPOUND PHOSPHOROUICHLORIDATE STRUCTURE	J58
66-1487 RAMIREZ	MADAN	SMITH	TRIVALENT PHOSPHORUS COMPOUNDS WITH A,B-UNSATURATED K	T03
66-1504 AKITT	CRAGG	GREENWOOD	PH2PH, PHPH2 AND PHPC12	C15
6A-1519 GRUSHKIN	SANCHEZ	ERNEST	MCCLAN PHENYLPHOSPHONITRILIC CH AND DIMETHYLAMIDE DERIV	I27
66-1524 COSKRAN	VERKADE		2,6,7-TRIOXA-1,4-DIPHOS BICYCLO-2.2.2 OCTANE AND DERI	I27
6A-1597 PETERSON			ADDITIONS OF ORGANOLITHIUM COMPOUNDS TO VINYLPHOSPHIN	J58
66-1604 PRICE	PARASARAN	LAKSHMINARAYAN	1-HYDROXY-1,2,4,6-TETRAPHENYLPHOSPHORABENZENE	J01
66-1706 ANDREW	WYNN		P MAGNETIC RESONANCE SHIFTS AND FINE STRUCTURE	P50
66-1724 MESMER	CARROLL		MECHANISM OF THE HYDROLYSIS OF PYROPHOSPHITE	J01

a: id. number b: authors c: keywords d: publication code

99-46

University of Strathclyde

Professor: Patrick D. Ritchie
Professor: Peter L. Pauson
Professor: Manfred Gordon
Professor: D. W. A. Sharp

Department of Pure and Applied Chemistry

Thomas Graham Building,
Cathedral Street, Glasgow, C.1 Tel: Bell 4400

2nd December, 1966.

Professor B. L. Shapiro,
Department of Chemistry,
Illinois Institute of Technology,
Technology Center,
Chicago, Illinois 60616,
U.S.A.

Dear Barry,

Orthotrifluoroacetates, Cyclic Sulphite Spectra.

We have been using fluorine n.m.r. in looking for further examples of orthotrifluoroacetates similar to (I) (cf[1]), and we find that we can recognize this type of structure and the products of further acylation quite easily. One particular example, the product (II) of the action of trifluoroacetic anhydride on 16-epioestriol (3,16β,17β-trihydroxyoestrane) showed three trifluoromethyl resonances of equal intensity. A sharp singlet at ϕ+75.37 is due to the phenolic trifluoroacetate, while two others at ϕ+75.64 and +75.98 are quartets with splittings of 1.8 c/s. The quartets themselves are unsymmetrical but together they show mirror image symmetry about the mid point. We take this coupling as evidence for the ortho ester structure (II) in which the trifluoromethyl and trifluoroacetyl fluorines are separated by six bonds. The alternative open chain structure (III) would imply a nine bond coupling!

Norman Brown, before he left to take up an I.C.I. Fellowship at Glasgow University, completed analyses of the spectra of ethylene sulphite (IV) and trimethylene sulphite (V). His results (below) add to an already voluminous and controversial

literature on the n.m.r. of cyclic sulphites. In the case of trimethylene sulphite his results are consistent with the axial conformation of the S=O bond (contra Edmundsen[2]), and the assignment of the lowest field resonance to the axial protons adjacent to the oxygens (cf. [3, 4, 5]).

Ethylene sulphite (IV)

In $CDCl_3$ at 40 Mc/s and 60 Mc/s

τ_A	τ_B
5.36	5.66

J_{AB}	$J_{AB'}$	$J_{AA'}$	$J_{BB'}$	
-8.48	+6.41	+6.84	+6.84	c/s

Trimethylene sulphite (V)

In $CDCl_3$ at 40 Mc/s and 100 Mc/s

τ_A	τ_B	τ_C	τ_D
5.11	6.20	7.50	8.43

J_{AB}	J_{AC}	J_{AD}	J_{BC}	J_{BD}	J_{CD}	
- 11.5	12.3	2.5	4.8	2.5	-14.5	c/s

Pentaerythritol disulphite (VI)

Saturated solution in CD_3CN at 40 Mc/s

τ_A	τ_B	τ_C	τ_D
5.50	6.25	5.20	5.59

J_{AB}	J_{CD}	J_{BD}	$J_{CC'}$	
(-) 11.8	(-) 12.0	(-)2.4	(-) 0.8	c/s

A further cyclic sulphite that we have looked at is the disulphite of pentaerythritol (VI). The spectrum of this compound yielded the preliminary results of a first order analysis shown. In particular there is present a homoannular-long range coupling between the equatorial protons B and D, and a heteroannular long range coupling between the symmetrically equivalent axial protons C and C', a coupling similar to the first of these is probably present in trimethylene sulphite.

Yours sincerely,

Peter Bladon.

P. Bladon

G.C. Forrest

G. C. Forrest

D. R. Rae

D. R. Rae.

References.

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2. R. S. Edmundsen, Tetrahedron Letters, 1965, 1649.
3. D. G. Hellier, J. G. Tillett, H. F. Van Woerden and R. F. M. White, Chem. and Ind., 1963, 1956.
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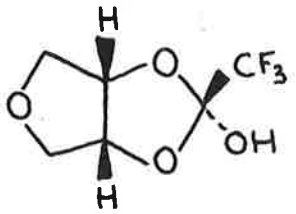

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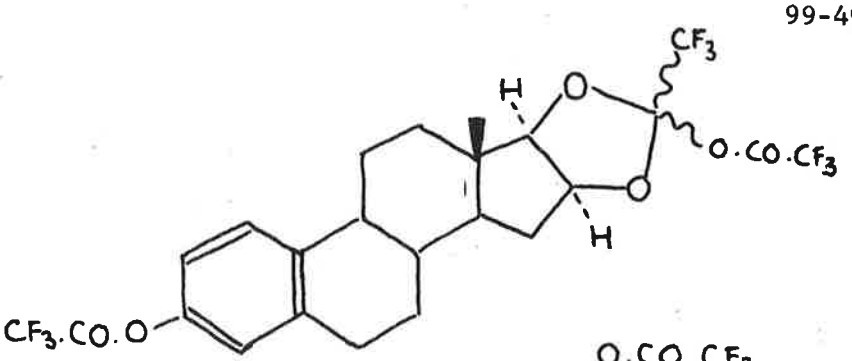

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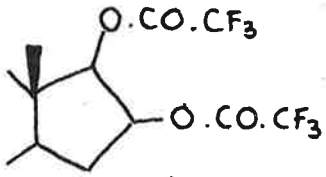

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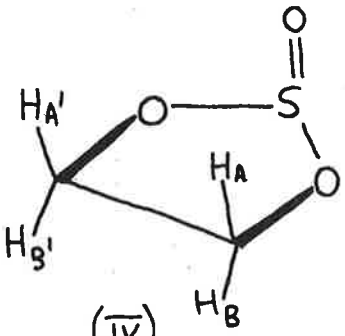

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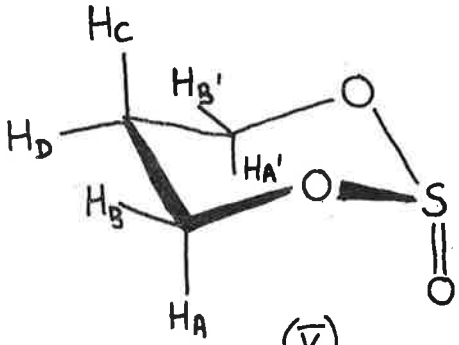

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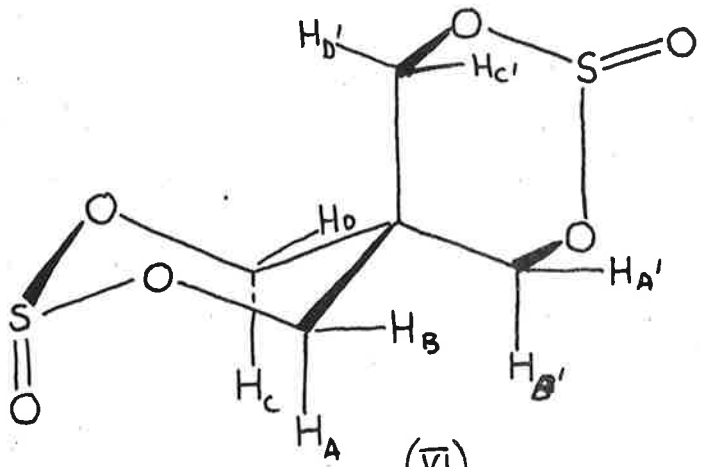


(VI)

99-50

UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
WESTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
800 BUCHANAN STREET
ALBANY, CALIFORNIA 94710

December 7, 1966

AIRMAIL

Professor Barry Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Varian V-3521 NMR Integrator Modification: Replace USA-3 with Equivalent Solid-State Unit

Dear Barry:

We have experienced increasingly frequent difficulties with the Philbrick USA-3 operational amplifiers as used in the Varian Associate V-3521 NMR Integrator. Most common symptoms have been base line instability and sudden onset of USA-3 saturation.

Initially the problems were corrected by chopper and/or tube replacement, especially the 6U8. Sometimes tube pin and socket cleaning corrected the trouble. Finally, as often happens with tube type circuit boards, flexing of tube sockets with tube replacement and heat conduction to the board caused intermittents to develop in the solder and printed circuit connections to components. General deterioration of the circuit board continues until replacement of the USA-3 is the only practical answer. This has been our story with two integrators.

When a second replacement became necessary recently, a new development came to our attention - a solid-state direct replacement for the USA-3 tube type amplifier. Fairchild Instrumentation is marketing a special version of their type AOO-9 operational amplifier called the AOO-9S21. The packaging is comparative with the overall Philbrick USA-3 dimensions, and the characteristics are practically identical to those for the USA-3 except that noise and drift are very appreciably better.

We recently installed one of these units and its operation appears to be completely satisfactorily. The USA-3 board was removed and a cover plate was substituted. A mating connector to the Fairchild was located on this cover plate so that the AOO-9S21 occupies the former USA-3 space. The amplifier was located so that the case does not touch any surrounding components on the V-3521 chassis. The unit is grounded only through a connector pin, thus minimizing any operational stability problems associated with ground loops. To improve mechanical stability, the amplifier case was attached to the Integrator cover plate with a plastic screw and spacer.

All circuit connections removed from the USA-3 were routed with no difficulty to the plug-in connector. One modification was made: the amplifier balance or offset adjust circuit suggested for the AOO-9S21 was used rather than the amplifier balance circuit of the Varian V-3521. A 1 megohm pot physically replaces R411 and all connections to the original R411 were tied off. This modification affords a greater range of amplifier balance and also utilizes the additional power supply regulation packaged with the AOO-9S21.

This same amplifier change could probably be made in the Integrator of the A-60.

Sincerely yours,

Rick

R. H. Elsken
Electrical Engineer

Bob

R. E. Lundin, Chemist
Molecular Structure Investigations
Wool and Mohair Laboratory

99-52

UNIVERSITY OF CALIFORNIA

LAWRENCE RADIATION LABORATORY
BERKELEY 4, CALIFORNIA

December 6, 1966

Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Technology Center
Chicago, Illinois 60616

Dear Barry:

Fourier Transform NMR Spectroscopy of P^{31}
Fourier Transformation by Analogue Methods

We would like to present some preliminary results of the application of Fourier transform nmr spectroscopy to P^{31} and also to indicate how the transformation may be achieved analogically.

The work of Ernst and Anderson[1] points out that a significant saving in time is one of the rewards of the transform method. That this is indeed the case in illustrated in the accompanying figures. Figure one shows a series of P^{31} spectra of trimethylphosphite with sweep times indicated for each spectrum. It is apparent that at least two minutes are required to obtain a fully resolved spectrum.

Figure 2A is the impulse response of 1.25 second duration following a single 90° pulse, and Figure 2B is the Fourier transformed spectrum of this impulse response. If one ignores the time spent in carrying out the transformation, the actual saving in nmr running time is about two orders of magnitude.

The experiments were performed in the following manner. The 24.3 MHz V-4311 rf unit was modified to permit gated or pulsed operation, and the transmitter section pulsed at the 1.25 second period with Tektronix 161 and 162 pulsers. With maximum rf from the V-4311, a 90° pulse requires a pulse width of 5×10^{-4} seconds. The impulse response was recorded in a Northern Scientific NS-544 averager triggered by the 161 pulse.

The Fourier decomposition was accomplished by displaying the stored impulse response at a 1600 Hz rate while feeding the analogue output of the NS-544 to a Nelson-Ross spectrum analyzer. This latter unit is a plug-in accessory for Tektronix oscilloscopes which accept the letter series plug-ins, and was used since we were able to borrow it. The spectrum analyzer was scanned at a sufficiently slow rate that the NS-544 made many passes through the stored data

for each resolution width of the analyzer. The output of the spectrum analyzer fed a Moseley x-y recorder.

Since this spectrum analyzer, as well as most others, employ amplitude detection, the phase information is lost and an "absorption" mode signal is always obtained irrespective of the phase of the rf synchronous detector.

To exploit the full dynamic range of the memories of averaging computers the Fourier transformations should be carried out numerically in a digital computer, for which the programming philosophy of Ernst and Anderson[1] may be followed. The digital-to-analog converters in the averaging computers decode only the three most significant digits (or their binary equivalents) which could limit the usable data range.

It is exceedingly useful, however, to be able to have a spectrum immediately available while awaiting the inevitable turn-around time of the large computer. One may also decide on the bases of the analog transform that the data do not warrant the expense of a computer run (the wrong sample was in the probe). There may even be some nmr installations without easy access to computer centers which may be encouraged to undertake transform spectroscopy when it may be carried out without computers.

The matched filter routine incorporated into Ernst's computer program can be simulated satisfactorily by analogue techniques by employing operational multipliers and logarithmetic amplifiers. The half-width of the Lorentzian line or the e-folding time of the exponential decay can be set to correspond with the impulse response which is actually recorded.

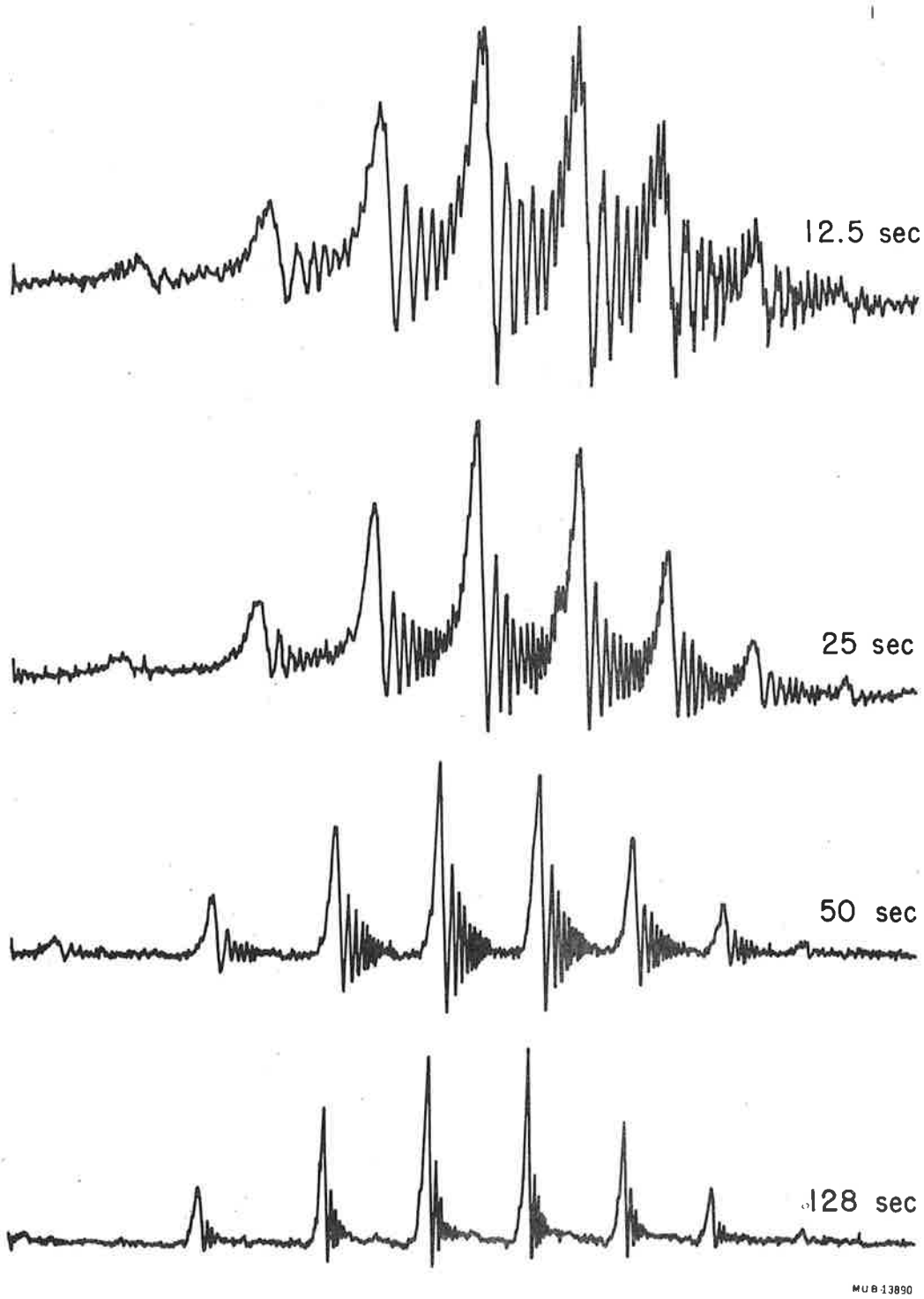
We are intrigued by the potential use of the transform technique for determining reaction kinetics. The impulse responses taken at short time intervals can be digitized and immediately dumped onto either magnetic or paper tape, and then transformed at leisure.

Sincerely yours,

Mel
Don

M. P. Klein
D. A. Phelps

1. R. R. Ernst and W. A. Anderson, Rev. Sci. Inst. 37, 93 (1966).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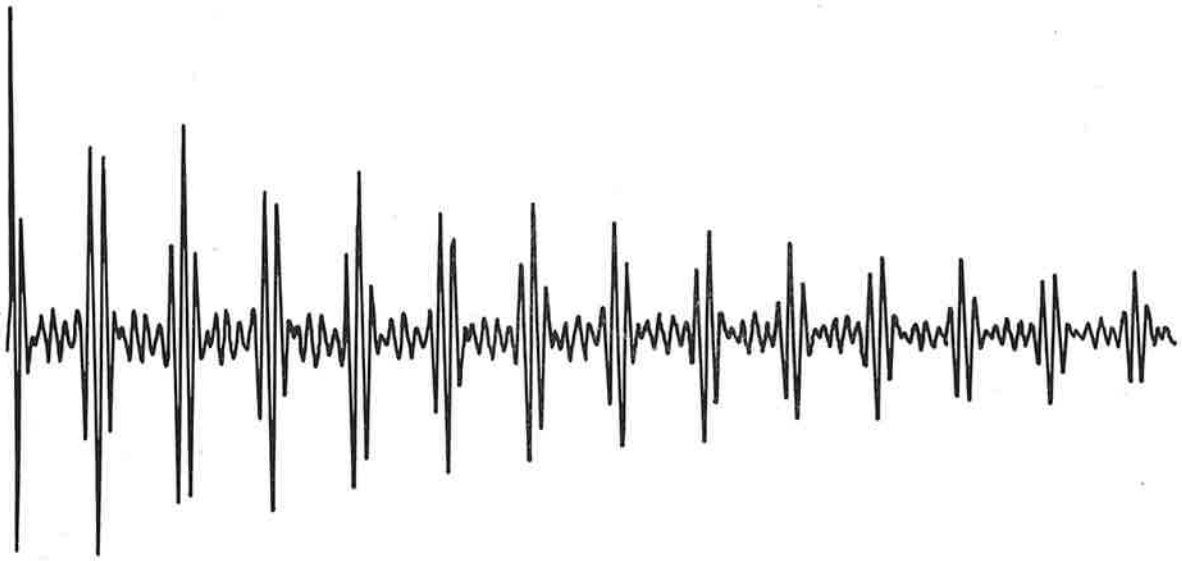

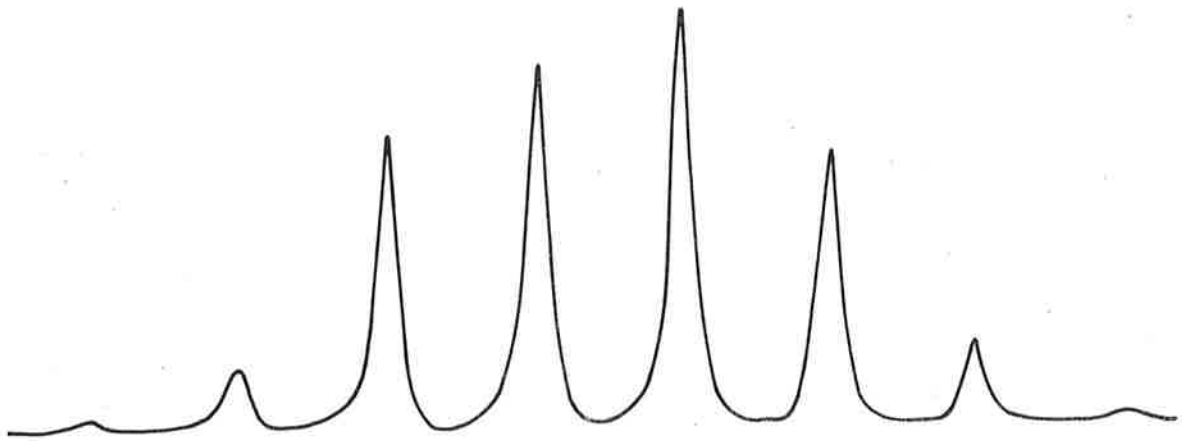

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99-56

TEXAS CHRISTIAN UNIVERSITY
Fort Worth, Texas 76129

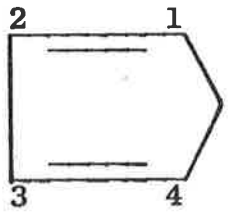
Department of Chemistry

December 8, 1966

Dr. B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Dr. Shapiro:

Recently, we determined the NMR parameters of a series of cyclopentadienylides. The variety of solvents was necessitated by solubility considerations. The triphenylphosphonium compound was done at our request by Dr. C. A. Reilly (I.I.T.N.M.R., 86, 42) to whom we express our appreciation.



= X

X(solvent)	τ^a	Δ_{AB}Hz	J_{12}	J_{23}	J_{13}	J_{14}
Triphenylphosphonium ($DCCl_3$)	3.64	10.2_0	3.8_4	2.8_0	1.8_9	2.1_6
Pyridinium (DMSO)	4.12	23.1_3	4.1_9	2.3_4	1.5_0	2.1_8
Trimethylammonium (Acetonitrile)	5.44	10.6_9	3.8_4	2.9_6	2.2_6	1.7_4
Diazo (CCl_4)	3.67	47.0_6	4.9_3	2.7_0	1.8_7	2.2_0

a. Center of ring proton spectrum.

Our interest in these compounds stemmed from a desire to assess their aromatic character via the NMR route. Regardless of what side one wishes to join the current controversy over the "ring current" effect (see Musher, I.I.T.N.M.R., 83, 18 and J. Chem. Phys., 43, 4081 (1965), practical matters such as solvent effects and the unknown magnetic anisotropy of the substituents rule against any meaningful conclusions from the chemical shifts of these compounds.

The observation of a linear J-bond order relation by Jonathan, Gordon, and Dailey, (J. Chem. Phys., 36, 2443 (1962)) for six-membered rings led us to explore a similar relation in the five membered series shown in the accompanying plot. Bond orders were taken from the literature or calculated by a slightly modified ω'-technique. The least squares fit gave

$$J = 7.12 \times (\text{bond order}) - 1.18$$

Using data available since the original work provided us with a new set of parameters for the six-membered compounds namely

$$J = 8.65 \times (\text{bond order}) + 1.66$$

From the latter relation, one predicts J^{ortho} for benzene to be 7.4 Hz in good agreement with the value recently given by Bernstein of 7.7 (I.I.T.N.M.R., 92, 12). Would you believe we made our prediction before he communicated his number?

The J-bond order relation seems to work for both five and six-membered series though a separate plot is required for each. If one accepts that bond orders and bond lengths are related, then there is a theoretical basis for these observations via the Karplus treatment relating bond length to J (J.A.C.S., 85, 2870 (1963)). Furthermore, bond lengths and aromatic character are related (see Musher above).

Finally, the bond orders for our ylids (both calculated and inferred from the plot) correspond with the known chemistry of these compounds.

Yours sincerely,

W. B. Smith

W. B. Smith
Chairman
Department of Chemistry

WBS/dc

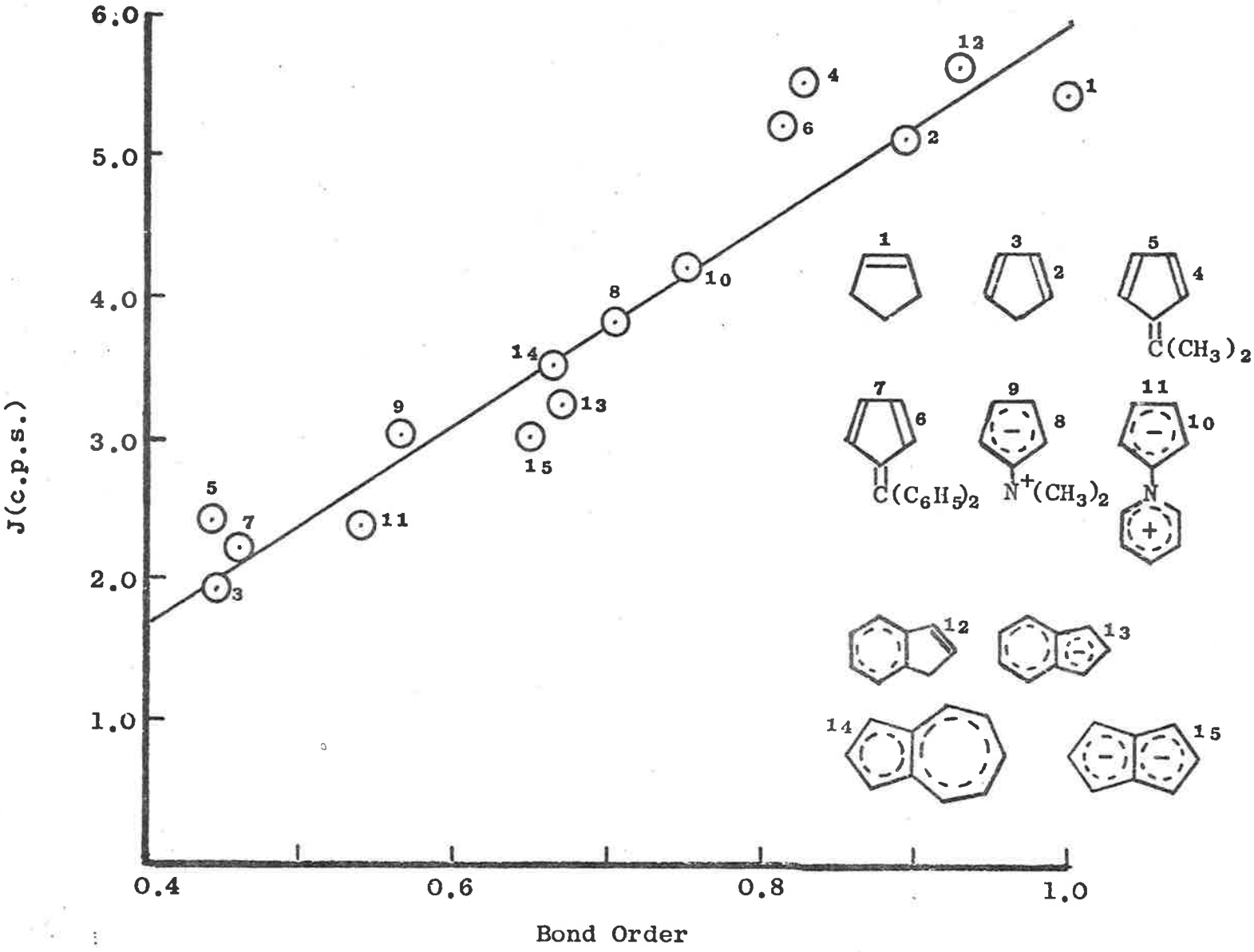


Fig. 2. Plot of proton coupling constants for the five-membered rings vs. the bond orders.

Telephone No. 24161 (Ext.)

Department of Organic Chemistry,

SCHOOL OF CHEMISTRY,
THE UNIVERSITY,
BRISTOL 2.

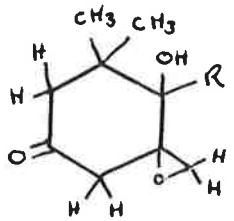
Dr. B.L. Shapiro,
Department of Chemistry,
Illinois Institute of Technology,
Chicago,
Illinois,
U.S.A.

5th December, 1966

Dear Dr. Shapiro,

Would you please consider the following request for information as a contribution from the department.

We have a natural product for which we favour structure (I). In the spectrum two overlapping AB quartets at 7.25-7.71 τ (each with J, 18 c/s) are assigned to the methylene protons adjacent to carbonyl (in a 6-ring). One A of these AB-systems is coupled (J, 3 c/s) to the A proton of a third AB system at 6.11, 6.42 τ (J, 7.5 c/s). We are not confident in ascribing this 3 c/s coupling to a long-range interaction between one of the protons of the terminal epoxide in (1) and one of the methylene protons. We ask if anyone has observed such a large long-range coupling in terminal epoxides. Alternative structures (2) and (3) (less attractive for other reasons) do not help to explain the magnitude of this coupling.



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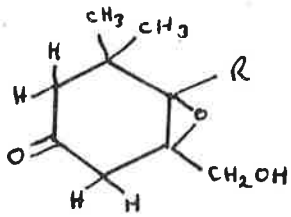

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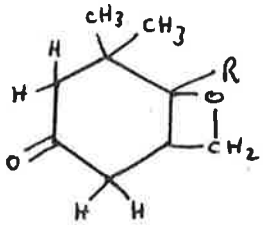

(3)

Yours sincerely,

J. MacMillan.

R. J. Pryce.

J. MacMillan
R.J. Pryce

99-60

varian/611 hansen way/palo alto/california 94303/u.s.a./415/326-4000

December 13, 1966

Professor B.L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

TITLE: POSITIONS AVAILABLE

Dear Barry:

We have openings in our Palo Alto and Pittsburgh NMR Applications laboratories for chemists with experience in operation of NMR spectrometers and interpretation of NMR spectra. Duties in these positions involve a mixture of technical sales support and applications research related to our present and near future instruments.

Interested persons should contact me for further information.

Yours sincerely,

Roy

LeRoy F. Johnson, Manager
Spectroscopy Applications Laboratories
Analytical Instrument Division

LFJ:jc

HARVARD MEDICAL SCHOOL
THE DEPARTMENT OF PHARMACOLOGY
25 SHATTUCK ST., BOSTON, MASS. 02115

December 12, 1966

Dear Barry,

Our group will submit its next newsletter under a different letterhead. We are moving next month to Rahway, New Jersey, to head and partly staff the newly created Department of Biophysics and Pharmacology at Merck & Co. During this transition period we are completing several research projects including one entitled:

HELIX-COIL TRANSITIONS IN POLYAMINO ACIDS

We are among a number of people who are discovering that high molecular weight homopolymers of amino acids give PMR spectra of interest in both the random coil and helical forms. The interconversion between these two structural extremes is so rapid that single peaks representing the average of signals from protons in the two environments are observed. We have studied the chemical shifts and line-widths of spectra of a number of polyamino acids throughout the random-coil to helix transition range. Poly-L-glutamic acid becomes helical at pD 4.8 when its carboxylic acid side chains are titrated with DCl. All other polyamino acids studied are soluble in trifluoroacetic acid-chloroform mixtures and become helical as the percent chloroform is increased.

The polyamino acids give remarkably sharp spectra in the random form; in many cases the splitting fine-structure was observed. As the polymers become helical all peaks broaden out. The resonance peaks of protons on the backbone α-carbon and nitrogen atoms broaden at a faster rate than peaks corresponding to side-chain groups. The backbone N-H resonance is always the first to disappear.

Chemical shift results are summarized in the table below. In all the polymers studied, the protons on the backbone α-carbon atoms showed a significant upfield shift on helix formation. We attribute this upfield shift to the anisotropy of the π-electrons in the adjacent peptide bond. Two factors influence the chemical shift of a proton bound to a peptide nitrogen upon helix formation:

(1) In the helical form the proton takes part in a hydrogen bond which tends to shift the resonance to lower fields.
(2) In the helical form the proton is influenced more strongly by the peptide anisotropy which tends to shift the resonance to higher fields.

All the polyamino acids we studied showed a pronounced downfield shift of the amino proton resonance except poly-β-benzyl-L-aspartate. Side chain interactions in this polymer are thought to force the chain into a left-handed helix, rather than the right-handed α-helix which is usual for poly-L-amino acid polymers. Hydrogen bonding may be less important in the left-handed helix.

At present, we are looking into the effects which temperature, polymer concentration, and degree of polymerization have on the phenomenon of helix formation.

Yours sincerely,

Oleg Jardetzky John L. Markley Donella H. Meadows

POLYAMINO ACID	DEGREE OF POLYMERIZATION	CHANGE IN CHEMICAL SHIFT (at 60 Mc.) ACCOMPANYING TRANSITION TO HELIX	
		proton on backbone α-carbon	proton on backbone nitrogen
poly-L-alanine	280	25 cps upfield transition not sharp	slightly downfield in two stages
poly-β-benzyl-L-aspartate	760	13 cps upfield complete transition not observed	5 cps upfield then very slightly downfield
poly-γ-benzyl-L-glutamate	340	35 cps upfield	downfield as far as could be followed
poly-L-glutamic acid	680	5 cps upfield complete transition not observed	not observed in D_2O solutions used
poly-L-methionine	280	25 cps upfield	downfield as far as could be followed

Paris, le 12 Décembre 1966

Professor B. L. SHAPIRO
Department of Chemistry
Illinois Institute of Technology
CHICAGO - Illinois 60.616
U.S.A.

Subject : Medium effects and dissolved gases.

Dear Professor Shapiro,

Thanks for your reminder and in response to it I wish to report here some results obtained by Dr. Dayan and Dr. Widenlocher in the same field as previous papers (1.2.3.4.) concerning gases but, here, dissolved in organic solvants. They extend so the experiments of Buckingham and Coworkers ([5]) on CH_4 to simple non polar molecules as H_2, CH_4, C_2H_4 and C_2H_2.

The experimental values given in the following figure show that :

- the effect of organic solvant may be classified by means of H_2-CH_4 curve where these molecules may be considered free.

- The experimental points out of the dotted line (ideal line at 45°) are due probably to electric properties (quadrupole moment, particularly in C_2H_2) and to the site effect proposed by Rummens and Bernstein (6).

Sincerely yours,

R. FREYMANN

Physique Expérimentale
Moléculaire
Faculté des Sciences
9, Quai St Bernard - Paris.

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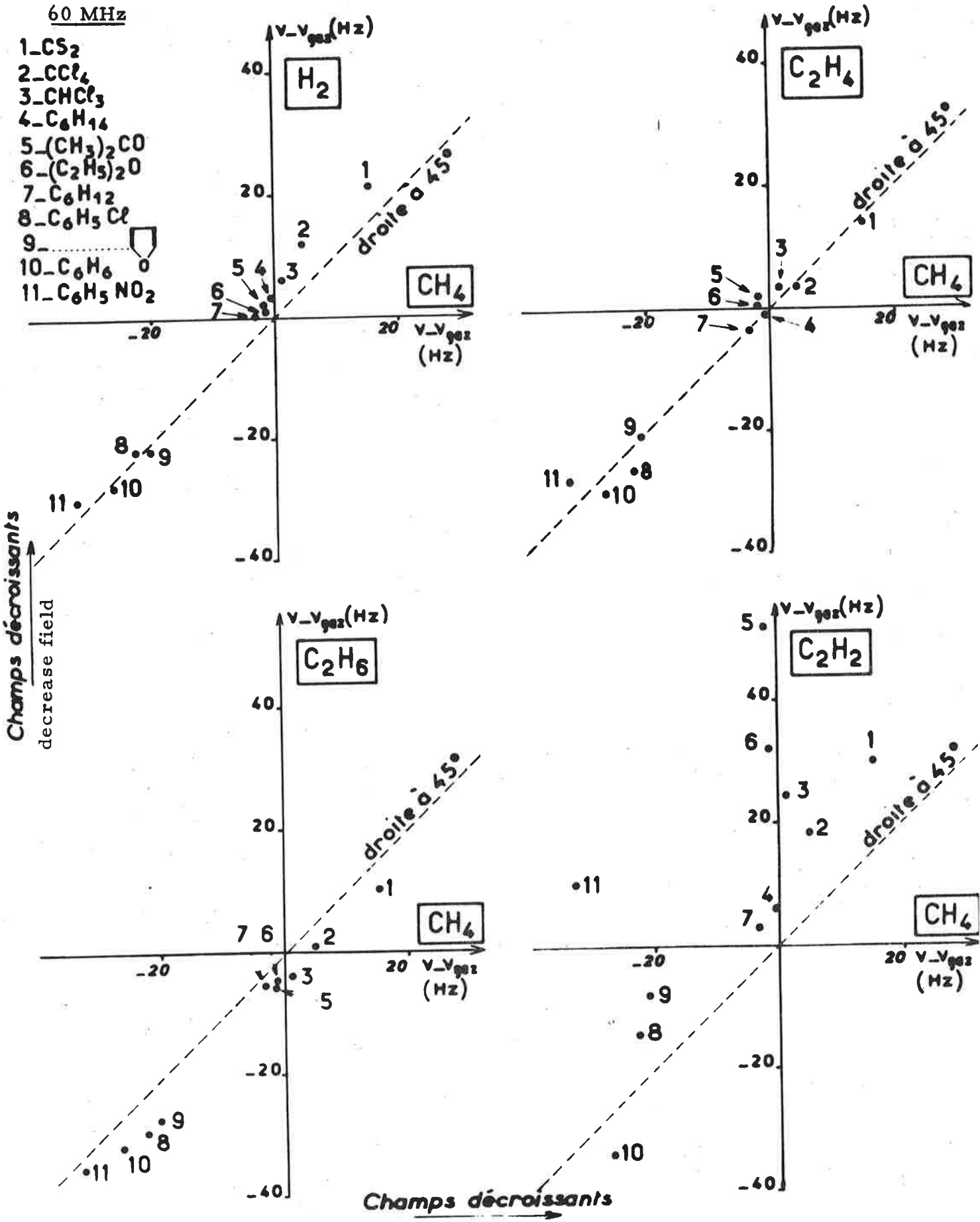
60 MHz
1_CS2
2_CCl4
3_CHCl3
4_C6H14
5_(CH3)2CO
6_(C2H5)2O
7_C6H12
8_C6H5Cl
9_
10_C6H6
11_C6H5NO2
H2
C2H4
C2H6
C2H2
CH4
droite à 45°
ν-νgaz (Hz)
Champs décroissants
decrease field

15_6_66

99-66

THE UNIVERSITY OF UTAH

SALT LAKE CITY 84112

DEPARTMENT OF CHEMISTRY
CHEMISTRY BUILDING

December 13, 1966

Professor Bernard L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Title: The relative sign of the C^{13}-C^{13} coupling constant

Dear Barry,

I would like to present some typical carbon-13 spectra obtained in our Varian AFS-60 spectrometer and make some comments on the relative sign of the C^{13}-C^{13} coupling constant. The sample is acetic acid enriched to approximately 20 atom percent in the carboxyl position. Thus, with normally occurring carbon-13 nuclei there will be four distinctly different molecules in the sample with the following abundances: $C^{12}H_3C^{12}O_2H$, ~ 79 %; $C^{12}H_3C^{13}O_2H$, ~ 20 %; $C^{13}H_3C^{12}O_2H$, ~ 0.8 %; and $C^{13}H_3C^{13}O_2H$, ~ 0.2 %. The first molecule contains only C^{12} atoms and, therefore, does not have a carbon-13 spectra. The third and fourth molecules exhibit carbon-13 methyl signals with a first-order splitting pattern and these are shown in Figure 1. The less abundant species has two carbon-13 atoms, and the coupling between these two nuclei splits the methyl resonance into a doublet of proton induced quartets. With 196 sweeps on the TAD (time averaging device - Varian C-1024) one can just barely recognize the carboxyl carbon-13 satellites on the methyl carbon-13 signal for the two center lines of the quartet expected for the $C^{13}H_3C^{12}O_2H$ molecules. The carboxyl region exhibited in Figure 2 shows all lines expected for the second and fourth molecules listed above. Here the spectrum of the $C^{12}H_3C^{13}O_2H$ molecule is observed on a single sweep because of the relatively high isotopic abundance of this species. It was necessary, however, to accumulate 400 sweeps on the TAD to observe the methyl carbon-13 satellites for the carboxyl signal.

Figure 3 contains three spectral traces of the carboxyl spectral region while utilizing our heteronuclear proton-decoupler. The lower spectrum shows the collapse of the strong center multiplet with the decoupler adjusted to the most favorable proton-decoupling frequency, fo. With the proton decoupler set at fo - 66 cps, the lower frequency C^{13} satellite enhances more than the higher frequency multiplet. (Note: Frequency increases to the left in all of the figures so as to be consistent with former field-sweep spectra.) With the proton-decoupler set at fo + 66, it is the high frequency multiplet that completely collapses. These data indicate that the $J_{C^{13}-C^{13}}$ is of the same relative sign as the $J_{C^{13}-H}$ coupling, and one might, therefore, suspect that the coupling mechanism is similar in both cases.

The observance of carbon-13 spectra of molecules in only 0.2% abundance gives a fair indication of the present state of the art. With a concentration limit at this level, carbon-13 spectroscopy can now be expected to compete more favorably with PMR studies.

Sincerely yours,

David M. Grant
Chairman

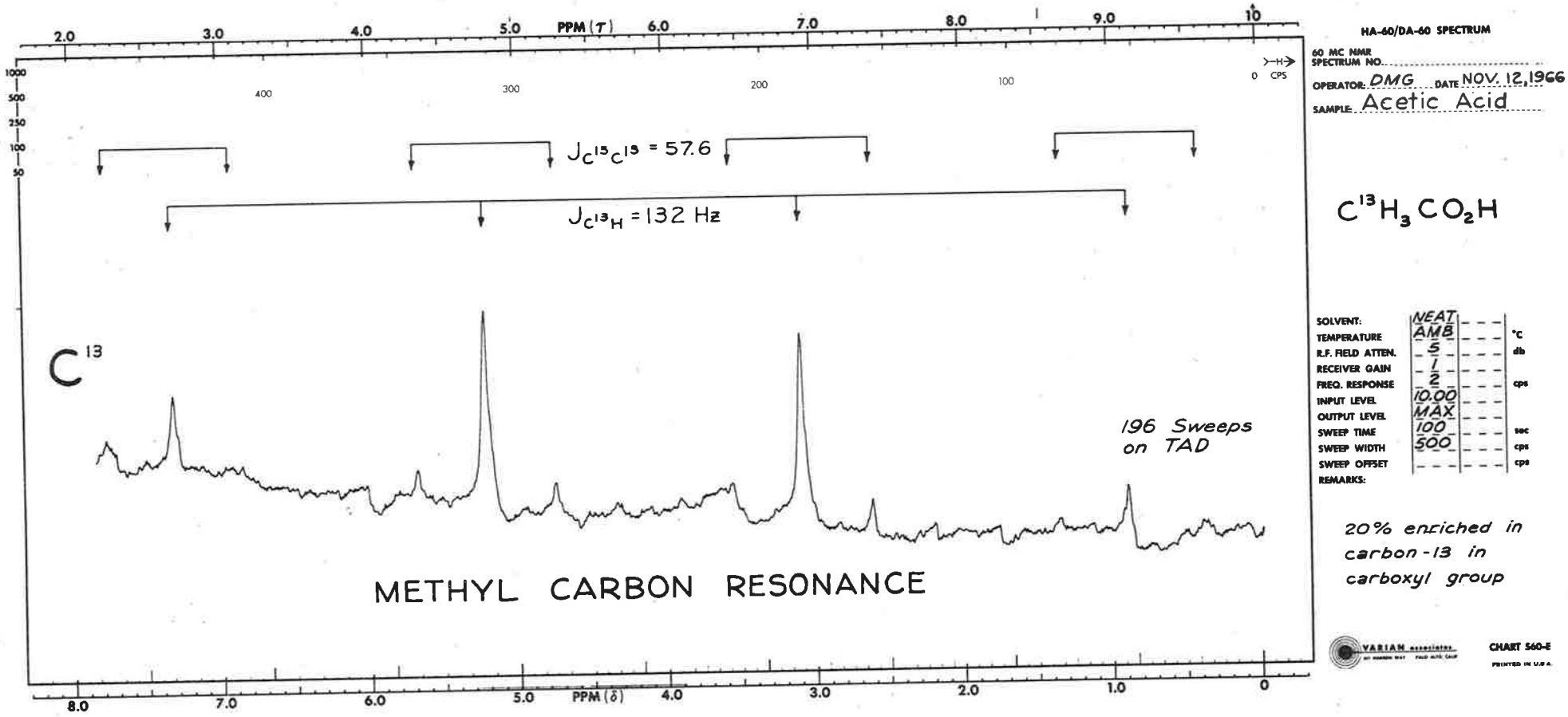
PPM (τ)
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10
400
300
200
100
0 CPS
$J_{C^{13}C^{13}} = 57.6$
$J_{C^{13}H} = 132$ Hz
C^{13}
196 Sweeps on TAD
METHYL CARBON RESONANCE
8.0
7.0
6.0
5.0
PPM (δ)
4.0
3.0
2.0
1.0
0
HA-60/DA-60 SPECTRUM
60 MC NMR SPECTRUM NO.
OPERATOR: DMG DATE NOV. 12, 1966
SAMPLE: Acetic Acid
$C^{13}H_3CO_2H$
SOLVENT: NEAT
TEMPERATURE AMB °C
R.F. FIELD ATTEN. 5 db
RECEIVER GAIN 1
FREQ. RESPONSE 2 cps
INPUT LEVEL 10.00
OUTPUT LEVEL MAX
SWEEP TIME 100 sec
SWEEP WIDTH 500 cps
SWEEP OFFSET cps
REMARKS:
20% enriched in carbon-13 in carboxyl group
VARIAN ASSOCIATES
CHART 560-E
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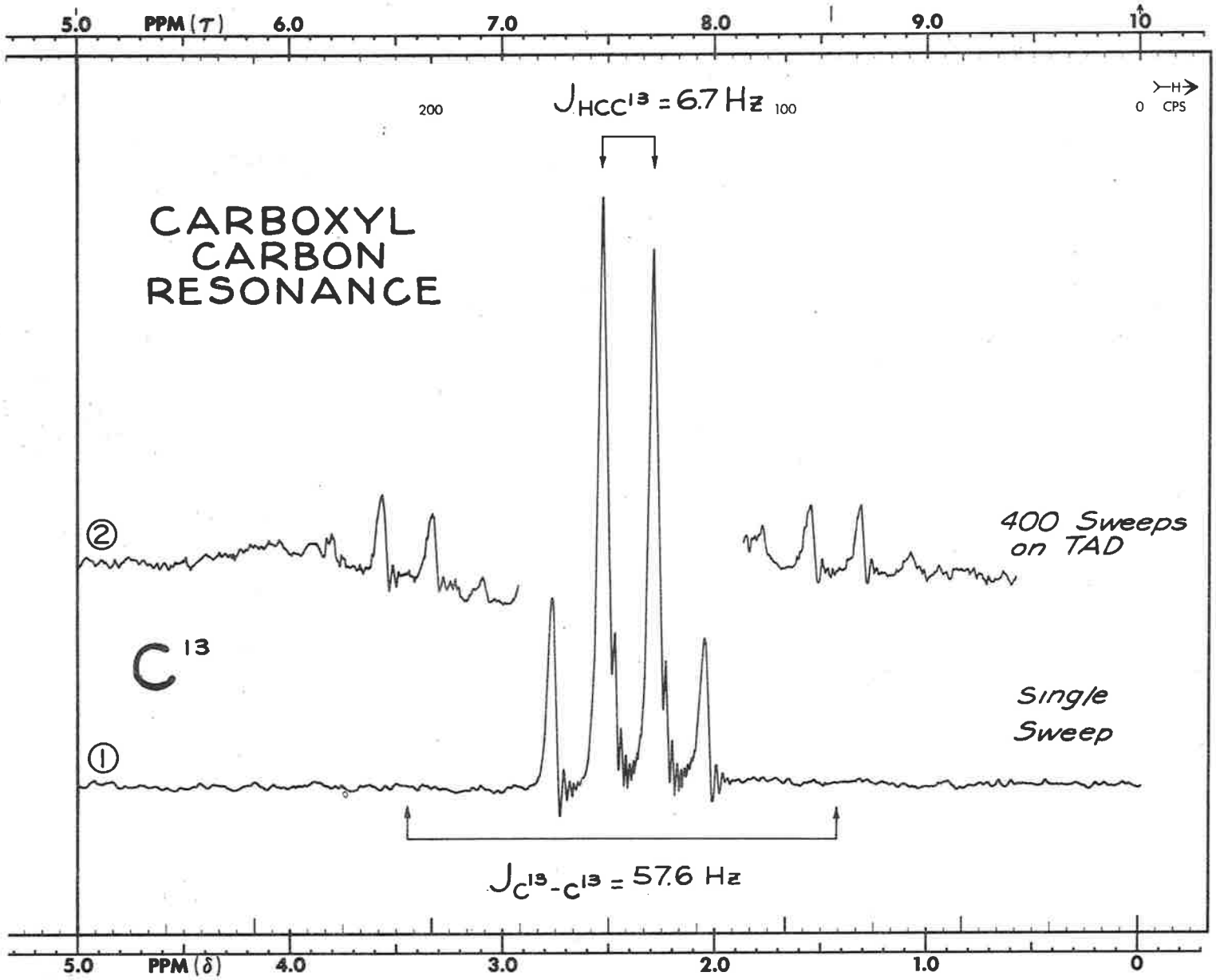
Fig. 1

99-67



HA-60/DA-60 SPECTRUM

60 MC NMR SPECTRUM NO.

OPERATOR: DMG DATE Oct. 14, 1966

SAMPLE: Acetic Acid

$CH_3C^{13}O_2H$

	①	②	
SOLVENT:	NEAT	NEAT	
TEMPERATURE	AMB	AMB	°C
R.F. FIELD ATTEN.	10	10	db
RECEIVER GAIN	2	2	
FREQ. RESPONSE	2	2	cps
INPUT LEVEL	10.00	10.00	
OUTPUT LEVEL	X0.7	MAX	
SWEEP TIME	100	100	sec
SWEEP WIDTH	250	250	cps
SWEEP OFFSET	– – –	– – –	cps
REMARKS:			

20% enriched in carbon-13 in carboxyl group

VARIAN associates 611 HANSEN WAY PALO ALTO, CALIF.

CHART S60-E

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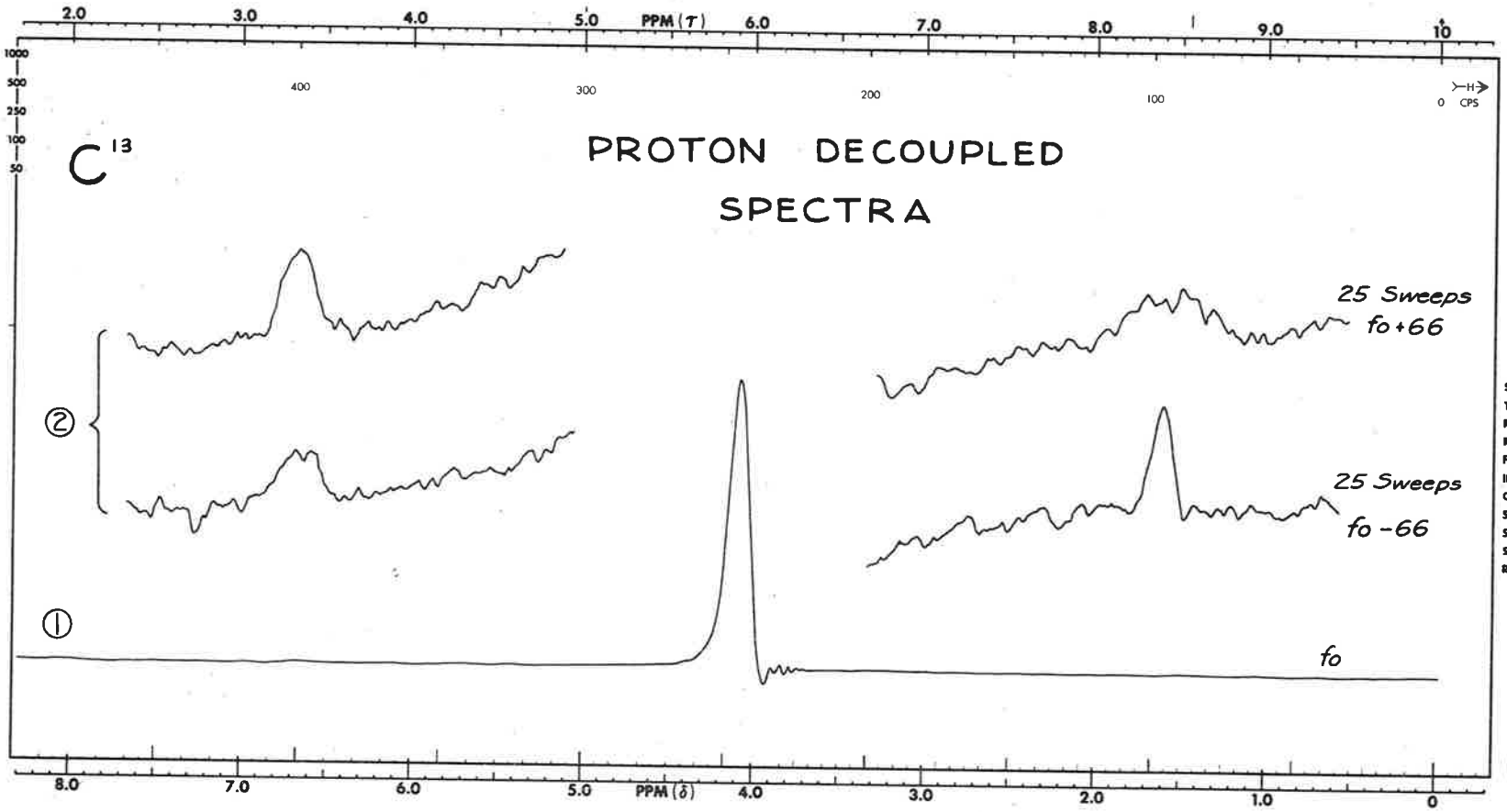
Fig. 2



HA-60/DA-60 SPECTRUM

60 MC NMR SPECTRUM NO. .....

OPERATOR: DMG DATE Oct. 15, 1966

SAMPLE: Acetic Acid

$CH_3C^{13}O_2H$

	①	②	
SOLVENT:	NEAT	NEAT	
TEMPERATURE	AMB	AMB	°C
R.F. FIELD ATTEN.	10	10	db
RECEIVER GAIN	2	2	
FREQ. RESPONSE	2	2	cps
INPUT LEVEL	10.00	10.00	
OUTPUT LEVEL	X.1	MAX	
SWEEP TIME	50	50	sec
SWEEP WIDTH	100	100	cps
SWEEP OFFSET	- - -	- - -	cps
REMARKS:			

20% enriched
in carbon-13
in carboxyl group

VARIAN associates

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Fig. 3

69-69

99-70

College of Arts and Sciences

STATE UNIVERSITY OF NEW YORK AT BUFFALO

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December 14, 1966

Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Subject: Noise Discrimination by Signal Inversion

Dear Professor Shapiro:

To distinguish a weak spectrum from noise, it is advantageous to display the inverse of the spectrum. An example is the NMR spectrum of ethylbenzene. Its spectrum, taken with a Varian A60 NMR spectrometer, is shown in the figure, together with the inverted spectrum, 1/v. Inversion has the advantage that the weak signals are amplified much more than the strong signals. If the signal is very weak, 1/v becomes quite large, and the recorder runs off scale, as seen in the figure. Thus, very weak signals are discriminated against. In order to avoid cutting off true signals by this, the discrimination is adjusted by use of a potentiometer such that some noise comes through, perhaps one noise pulse every few seconds.

From the Bloch equations, it follows that the inverted signal has a parabolic shape,

$$1/v = (1/v)_{max} + T_2(\omega_0-\omega)^2/\gamma H_1 M_0$$

This shape helps to distinguish visually a signal peak from a noise spike.

The inversion was accomplished using a Heath servo-recorder EUW-20A, as shown in the diagram. The servo motor is at balance when the points A_2 and B_3 have the same potential. Thus, $p f v = E$, where p is the position of the pen, and f is the attenuation factor of the input potentiometer. p and f vary from zero to one. The recorder will run off scale when the signal v is smaller than E/f. The system was satisfactory except for the rather unreliable paper drive of the recorder.

Please credit this letter to P. T. Lansbury of our department.

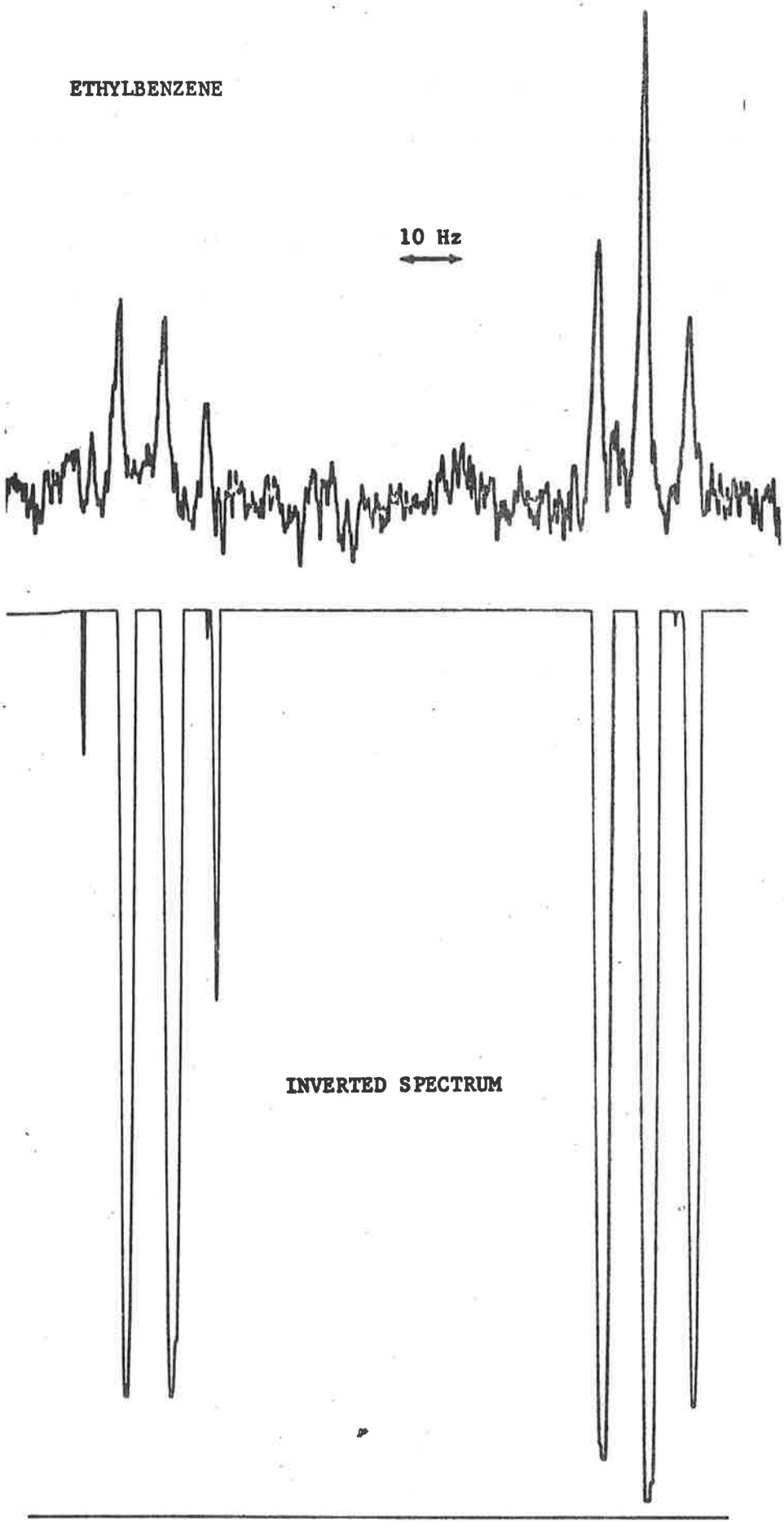
Sincerely yours,

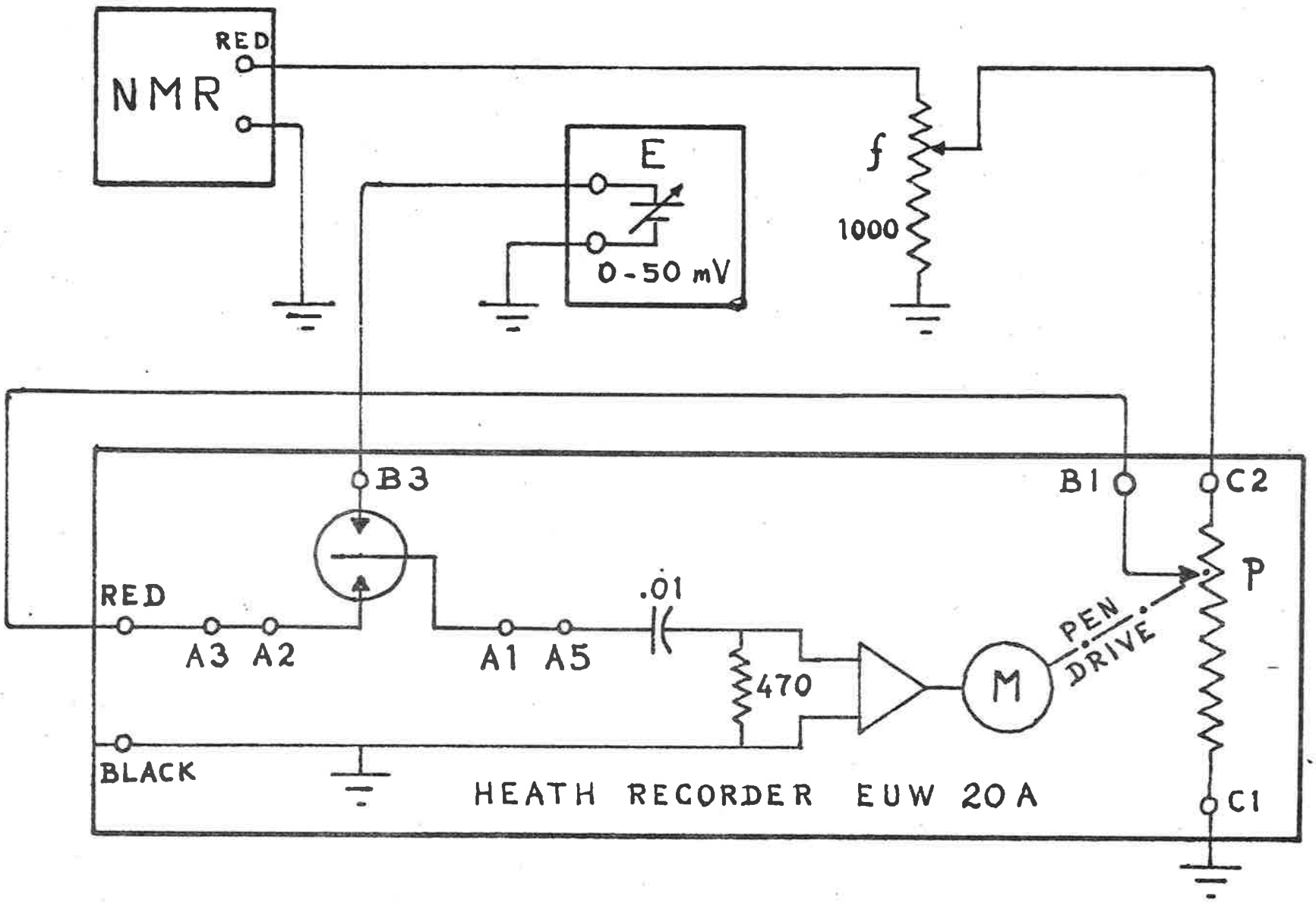
A. L. Van Geet L. Wechsler

A. L. Van Geet
Asst. Professor of Chemistry

L. Wechsler
Electronics Engineer

ALVG/dt


ETHYLBENZENE
10 Hz
INVERTED SPECTRUM



INVERSION OF THE NMR SIGNAL

MELLON INSTITUTE

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