

Joseph B. Lambert

Illinois  
Institute of  
Technology  
N - M - R  
Newsletter

No. 97  
OCTOBER, 1966

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Deadline Dates: No. 98 - 15 November 1966  
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A monthly collection of informal private letters from laboratories of NMR. Information contained herein is solely for the use of the reader. Quotation is not permitted, except by direct arrangement with the author of the letter, and the material quoted must be referred to as a "Private Communication".

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U.S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS  
WASHINGTON, D.C. 20234

IN REPLY REFER TO: 313.01

16 September 1966

\*

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

Dear Barry:

COMPUTER PROGRAM PERTRB  
SAFE BULB REPLACEMENT IN SUPER-STABILIZER

We would like to offer a recently written computer program, PERTRB, to any of your readers who might be interested. It will calculate spectra for 2, 3, or 4 groups of magnetically equivalent nuclei where the coupling constant is small compared to the chemical shift. The molecules might be represented as  $A_m M_n R_p X_q$  where m, n, p, q may be as high as nine. The formulae used in the program are taken from "High-Resolution Nuclear Magnetic Resonance," by Pople, Schneider, and Bernstein, pp. 151-153.

The program uses third-order perturbation theory for systems with two or three kinds of nuclei; second-order theory when there are four kinds of nuclei. There is no provision for iteration; but in cases where this program is useful, that is, nearly-first-order spectra, an iterative procedure is not really necessary. The lines in the calculated spectra are labelled according to the quantum numbers of the species involved. This feature aids in the interpretation of double resonance experiments. Listings are available with the usual disclaimer,--the program has been tried with several different problems and I know of no remaining bugs; however, I accept no responsibility in case of wrong answers. The program is written in Fortran IV and has been run on the Bureau's IBM 7094 computer. It uses about 20,000 words of storage.

The second point I would like to mention concerns replacement of the galvanometer lamp on the Varian Super Stabilizer. The lamp circuit is about -180 v. with respect to ground, and I have received numerous shocks in the process of adjusting new bulbs. Now I have connected a 6.3 v. filament transformer to a Jones plug which mates with the plug in the Varian unit and drive the lamp bulb from this while adjusting it. This expedient eliminates the shock hazard.

Yours very truly,

*Rolf B. Johannesen*

Rolf B. Johannesen  
Inorganic Chemistry Section



UNIVERSITÉ DE NANTES  
FACULTÉ DES SCIENCES  
B. P. 1044 NANTES

Laboratoire de Chimie Organique-Physique  
38, Boulevard Michelet - Nantes

Nantes, le 14 septembre 1966

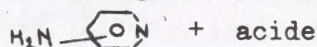
Chemical shifts and  $\pi$  electronic density in protonated aminopyridines.

Cher Professeur Shapiro,

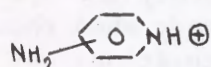
En collaboration avec des théoriciens qui s'intéressent au calcul des densités électroniques dans les molécules conjuguées (1 et 2), nous avons entrepris un travail sur le problème fréquemment soulevé (3) de la relation entre déplacements chimiques et charges  $\pi$ .

Dans un premier stade nous avons sélectionné la série des hétérocycles aminés et de leurs dérivés protonés.

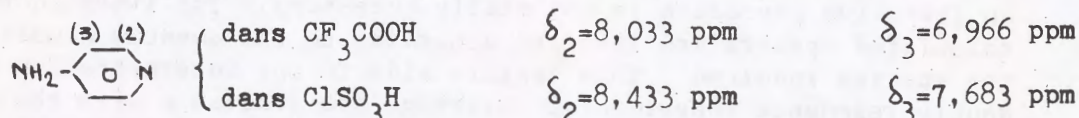
La détermination des déplacements chimiques dans les composés protonés pose des problèmes complexes. En effet, en raison de l'équilibre dynamique entre les différentes espèces présentes dans le système:



il est difficile d'obtenir les valeurs de  $\delta$  correspondant à une forme isolée



. D'autre part les molécules protonées subissent de forts effets de solvants de la part de l'acide et nous montrons que les  $\delta$  sont très sensibles à la nature de l'anion antagoniste:



Par ailleurs les densités électroniques calculées sont fortement tributaires des paramètres de départ et du type d'approximation utilisés (1). Cependant, la figure 1 montre qu'une relation grossièrement linéaire se manifeste entre les déplacements chimiques mesurés dans un même acide ( $CF_3COOH$ ) et les charges calculées par la méthode de Pariser et Parr (1). Ces résultats sont donc encourageants si l'on tient compte des approximations consenties et nous nous proposons de les préciser

- en considérant les facteurs perturbateurs tels que les effets polaires ou d'anisotropie magnétique des substituants et des hétéroatomes
- en améliorant les paramètres utilisés dans le calcul théorique des charges (comparaison avec les résultats de mesures d'absorption dans l'ultraviolet)

.../...



-en perfectionnant la forme de la relation entre les déplacements chimiques et la répartition électronique(2)

# Références bibliographiques:

- (1) F. Peradejordi Cahiers de Physique 17 393 1963
  - (2) S. Odier et F. Tonnard C R Acad Sci 262, 1181, 1966
  - (3) A. H. Gawer et B. P. Dailey J Chem Phys 42, 2658, 1965
- et références incluses

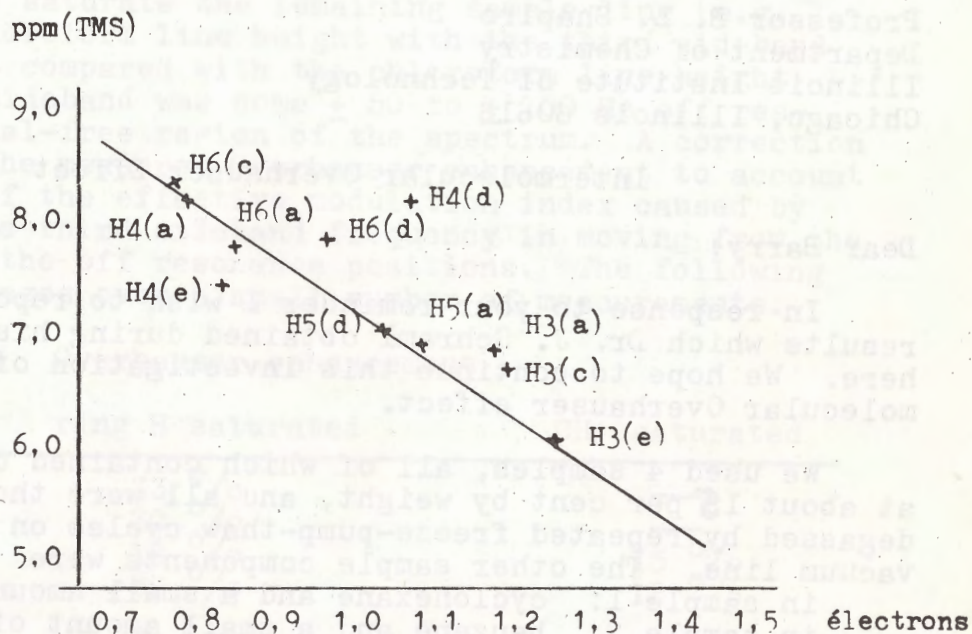


Fig 1: Relation entre déplacements chimiques et charges  $\pi$   
 Aminopyridines:  $\text{NH}_2$  en -2 (a), en-3 (b), en-4 (c)  
 Diaminopyridines:  $\text{NH}_2$  en -2,3 (d), en-2,6 (e)

Bien cordialement,

G Martin

M Martin

J P Dorie



THE UNIVERSITY OF NEW BRUNSWICK  
FREDERICTON, N. B.  
CANADA



DEPARTMENT OF PHYSICS

September 26, 1966

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

Intermolecular Overhauser Effect

Dear Barry:

In response to your reminder I wish to report some results which Dr. J. Schraml obtained during his short stay here. We hope to continue this investigation of the intermolecular Overhauser effect.

We used 4 samples, all of which contained chloroform at about 15 per cent by weight, and all were thoroughly degassed by repeated freeze-pump-thaw cycles on a high vacuum line. The other sample components were

- in sample 1: cyclohexane and a small amount of TMS,
- in sample 2: benzene and a small amount of TMS,
- in sample 3: toluene,
- in sample 4: p-xylene.

Spin-lattice relaxation times  $T_1$  were measured by observation of the recovery of the signals after rapid adiabatic passage inversion. An exponential curve was fitted by the method of least squares, and the resulting accuracy of the  $T_1$  values is about 10 per cent.

Sample	$T_1$ in sec			
	$\text{CHCl}_3$	ring H	$\text{CH}_3$	TMS
cyclohexane	23	11	-	11
benzene	23	14	-	7
toluene	25	13	10	-
p-xylene	23	8	5	-

It is interesting to note that somewhat different  $T_1$  values were obtained when a straight line was fitted by the least squares method to the logarithm of the recovery curve.

We measured the intermolecular Overhauser effect as the



Professor B. L. Shapiro  
September 26, 1966

2

percentage increase of the chloroform resonance line height which results from the saturation of a resonance of another sample component. The measurements were performed by sweeping the observation sideband frequency back and forth through the chloroform resonance line by means of a motor driven small condenser which was connected in parallel with the audio frequency tuning condenser. A second modulation sideband was used to lock the spectrometer field/frequency ratio to a sample resonance (e.g. TMS), and a third (strong) sideband was used to saturate the remaining sample line (e.g.  $C_6H_{12}$ ). The chloroform line height with the third sideband on resonance was compared with the chloroform line height when the third sideband was some  $\pm 50$  to  $\pm 100$  Hz off resonance in a signal-free region of the spectrum. A correction was applied to the measured Overhauser enhancement to account for the change of the effective modulation index caused by the change of the third sideband frequency in moving from the on resonance to the off resonance positions. The following results are averages over a small number of measurements.

Overhauser enhancement

Sample	ring H saturated	CH <sub>3</sub> saturated
cyclohexane	33 %	—
benzene	30 %	—
toluene	18 %	15 %
p-xylene	29 %	13 %

The result for the cyclohexane sample agrees with an earlier measurement (J. Chem. Phys. 42, 1838 (1965)), but the accuracy of these values is not good. We can state with confidence only one conclusion: reliable measurements are difficult to perform with this technique. A pulse method would perhaps be more suitable.

Sincerely yours,

*Reinhold*

R. Kaiser,  
Professor

RK:seb



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BOLLATE 18th September 1966

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Nella risposta citare il N° 3930/Cav/td

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

Subject:  $F^{19}$  NMR spectra of some fluorine-containing aromatic compounds

Dear Professor Shapiro,

the time is due for a new contribution as you have kindly reminded us.

As our contribution to IIT NMR Newsletter we would like to report some of our  $F^{19}$  studies. In connection with structure determination work we have measured some fluoro-containing aromatic compounds, which may be of interest to some of the readers. The data of some of them are summarized herewith below:

	Spin System	$F_1$	$F_2$	$\phi^*$	$F_3$	$F_4$	F, F coupling constant
①	AA'XX'	160.1	166.9		166.9	160.1	$J = \pm 21.2 = J \text{ ortho}$ $J' = \pm 2.9 = J \text{ meta}$ $ J_A  = 21.1 = J \text{ ortho}$ $ J_X  = 6.1 = J \text{ para}$
②	ABX	151.3	159.3		166.9	159.3	$ J_{12}  =  J_{14}  = \sim 0 = J \text{ meta}$ $ J_{13}  = 6.0 = J \text{ para}$ $ J_{23}  =  J_{34}  = 21.5 = J \text{ ortho}$

./.



	Spin System	F <sub>1</sub>	F <sub>2</sub>	F <sub>3</sub>	F <sub>4</sub>	F.F. coupling constant
③						
	A <sub>4</sub>	159.4	159.4	159.4	159.4	
④						
	A <sub>2</sub> MX	125.8	143.2	168.9	143.2	$ J_{12}  =  J_{14}  \approx 0 = J \text{ meta}$ $ J_{13}  = 10.9 = J \text{ para}$ $ J_{23}  =  J_{34}  = 19.4 = J \text{ ortho}$ $ J(\text{F} \cdot \text{CH}_3) _{\text{in ortho}} = 2.1$ $ J(\text{F} \cdot \text{CH}_3) _{\text{in para}} = 1.6$
⑤						
	A <sub>4</sub>	147.0	147.0	147.0	147.0	
⑥						
	AA'XX'M	144.0(AA')	163.0(XX')	156.8(M)		$J = \pm 22.8 = J \text{ ortho}$ $J' = \mp 8.2 = J \text{ para}$ $ J_H  \approx 1.5 = J \text{ meta}$ $ J_X  \approx 1.5 = J \text{ meta}$ $ J_{MX}  = 20.5 = J \text{ ortho}$ $ J_{MA}  = 0 = J \text{ meta}$

The spectra were all recorded at probe temperature ( $\sim 37^\circ\text{C}$ ) with an 56/60 Varian spectrometer. The chemical shifts are expressed in  $\delta^\circ$  values.  $\text{CFCl}_3$  was used as internal reference. The figure herewith attached shows the  $\text{F}^{19}$  spectra of ortho and meta diethoxytetrafluorobenzenes.



The relative signs of some coupling constants were obtained by direct spectral analysis. In the spectrum of ③, being N numerically greater than L, it follows that J and J<sup>3</sup> (J ortho and J meta in this case) have the same relative signs. The spectrum of ④, on the contrary, has N numerically lower than L, implying that J and J<sup>3</sup> (J ortho and J para in this case) have different relative signs. This finding seems to be in agreement with a monotonical variation of J<sub>FF</sub> from ortho, through meta, to para, as it has been recently suggested<sup>1</sup>.

Some of these results will be part of a forthcoming paper.

We are indebted to Dr. Camaggi who kindly supplied us with all the compounds.

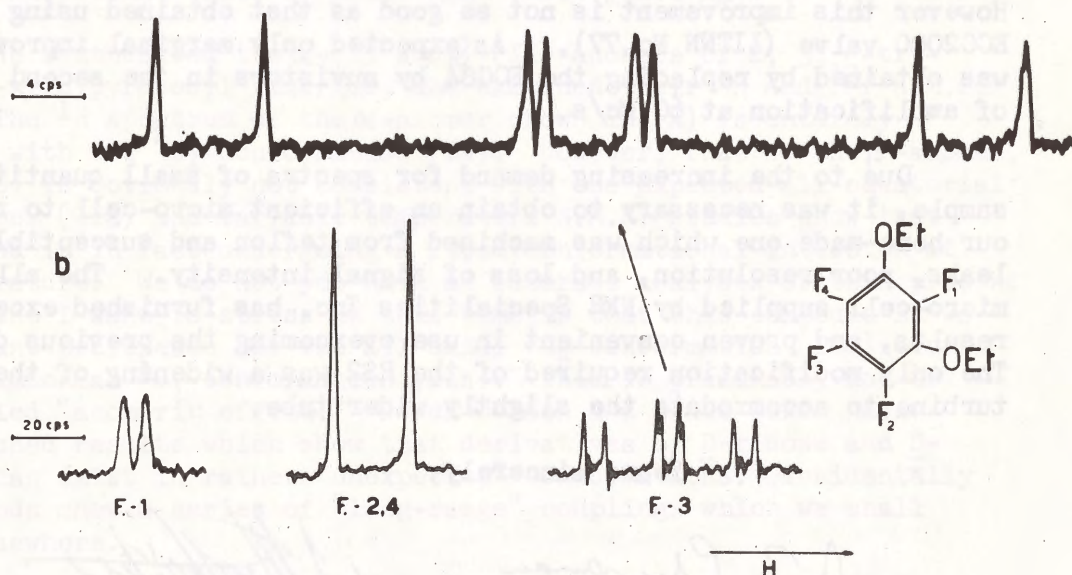
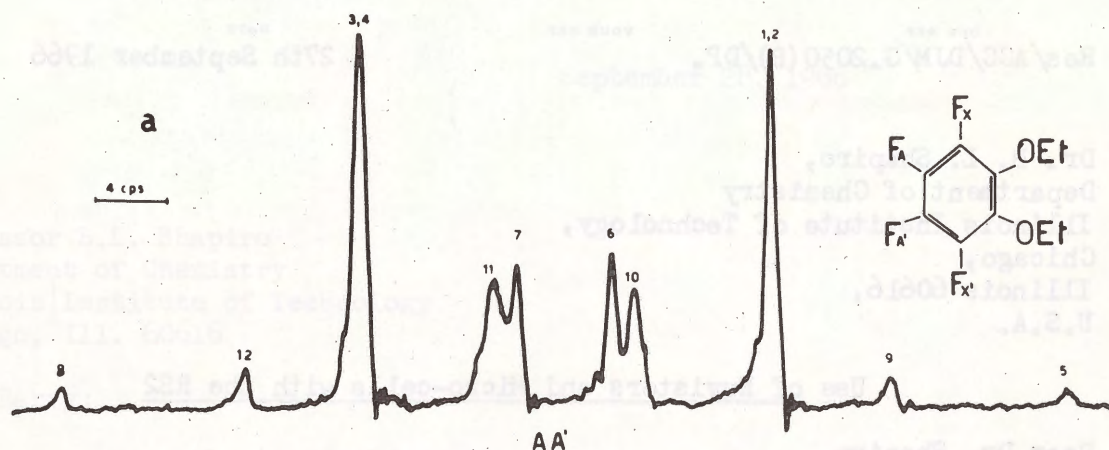
Yours sincerely,

L. Cavalli

*Luigi Cavalli*

*in.*  
Dr. Gb. Cevidalli  
Director of the Laboratory

1 - E. Lustig, P. Diehl, J. Chem. Phys. 44, 2974, 1966





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YOUR REF.

DATE  
27th September 1966

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

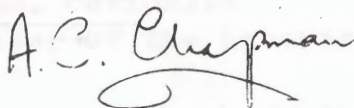
Use of Nuvistors and Micro-cells with the RS2

Dear Dr. Shapiro,

The recent claims made for nuvistor preamplifiers led us to try them at both 25 and 60 Mc/s. At the former frequency no improvement was observed, but at 60 Mc/s the signal/noise ratio was almost doubled compared with that obtained using an ECC84 valve. However this improvement is not so good as that obtained using an ECC2000 valve (IITNN No.77). As expected only marginal improvement was obtained by replacing the ECC84 by nuvistors in the second stage of amplification at 60 Mc/s.

Due to the increasing demand for spectra of small quantities of sample, it was necessary to obtain an efficient micro-cell to replace our home-made one which was machined from teflon and susceptible to leaks, poor resolution, and loss of signal intensity. The all glass micro-cell supplied by NMR Specialities Inc. has furnished excellent results, and proven convenient in use overcoming the previous difficulties. The only modification required of the RS2 was a widening of the spinner turbine to accommodate the slightly wider tube.

Yours sincerely,



A. C. Chapman  
D. J. Mowthorpe  
RESEARCH DEPARTMENT.



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## THE UNIVERSITY OF BRITISH COLUMBIA

VANCOUVER 8, CANADA

DEPARTMENT OF CHEMISTRY

September 28, 1966

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

Dear Barry:

Re: Inverted Conformations of Fluorinated Carbohydrates.

I am sorry that you had to remind me that my subscription was overdue. We have been continuing our studies of specifically fluorinated carbohydrates and in this letter I want to mention a rather interesting conformational result which we found some three years ago.

We synthesised the  $\alpha$ -(I) and  $\beta$ -(II) anomers of 2, 3, 4-tri-O-acetyl-D-xylo pyranosyl fluoride, and examined their  $^1\text{H}$  and  $^{19}\text{F}$  N.M.R. spectra. The  $^1\text{H}$  spectrum of the  $\alpha$ -anomer shown in (A) is entirely consistent with the  $^4\text{C}_1$  conformation (Ia). However, that of the  $\beta$ -anomer, shown in (B) is obviously not consistent with the expected all-equatorial  $^4\text{C}_1$  conformer (IIa) and variable temperature N.M.R. studies show that the compound is in fact undergoing a rapid conformational inversion at room temperature. We do not yet have an accurate analysis of this process but the point I want to stress at this time is that this compound shows a significant preference for the all-axial  $^1\text{C}_4$ -conformation (IIb) over the all-equatorial  $^4\text{C}_1$ -conformation (IIa). This is presumably due to the so-called "anomeric effect." Derek Horton and Bruce Coxon have each published results which show that derivatives of D-ribose and D-arabinose can exist in rather "unexpected" conformations. Incidentally our compounds show a series of "long-range" couplings which we shall discuss elsewhere.

I hope to write to you shortly about the energy parameters of this inversion process.

With best regards,

*Laurie*

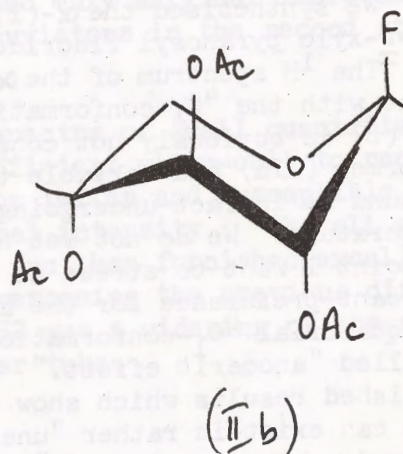
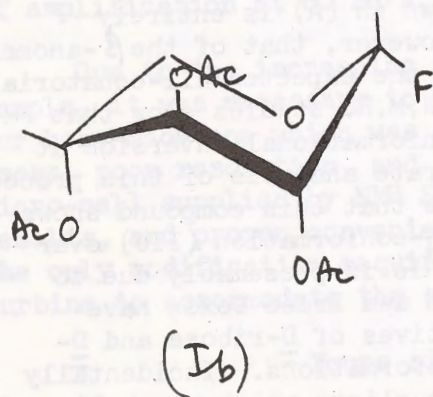
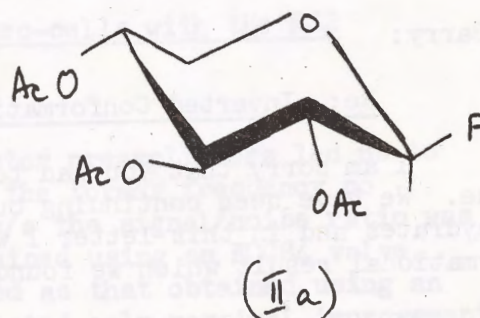
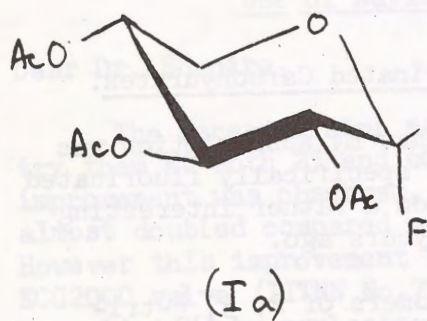
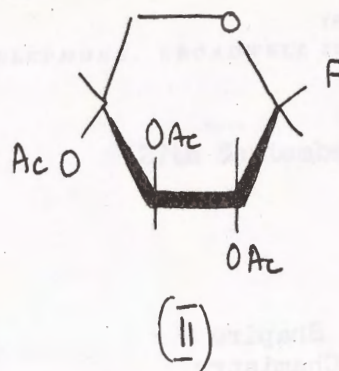
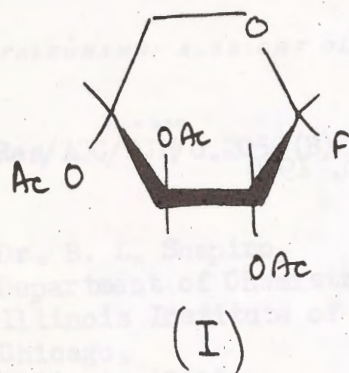
L.D. Hall

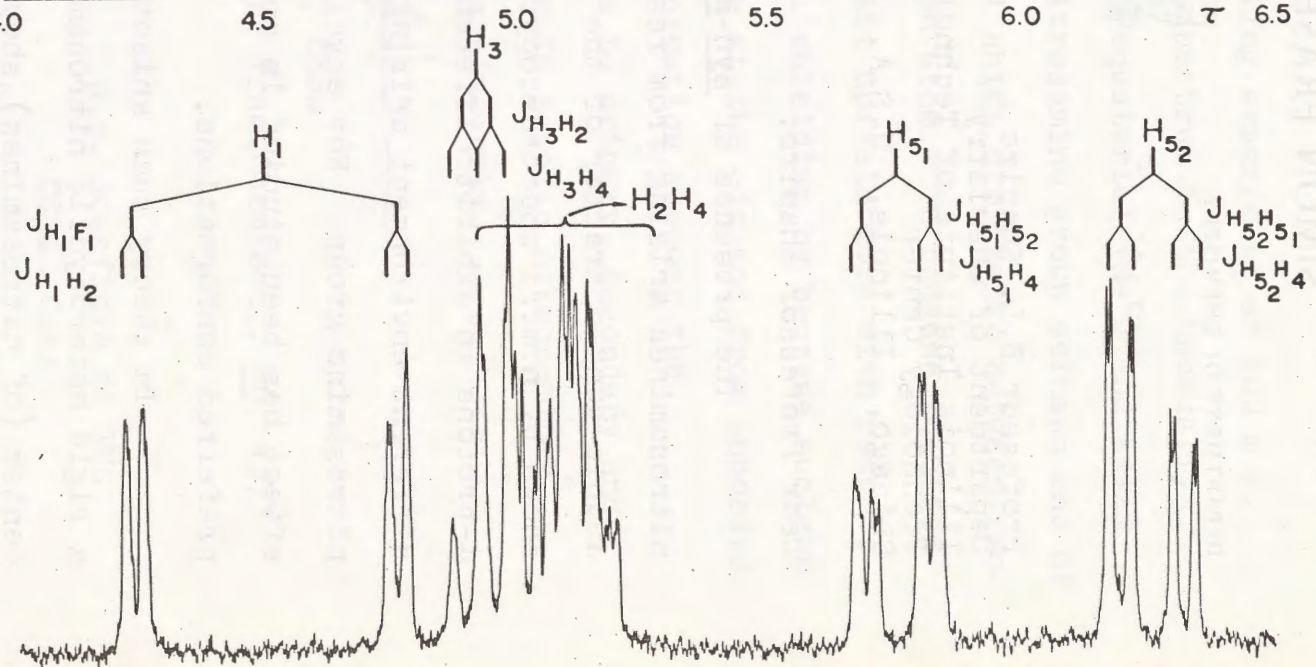
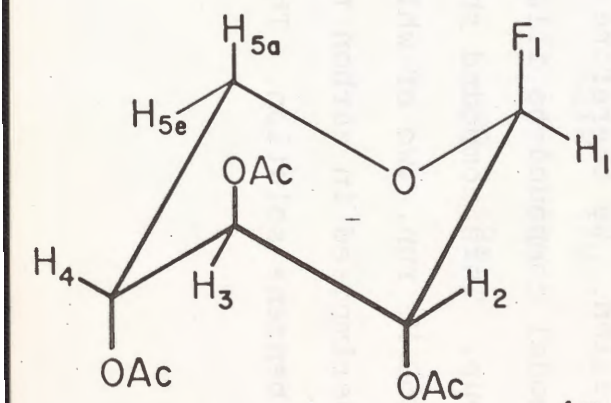
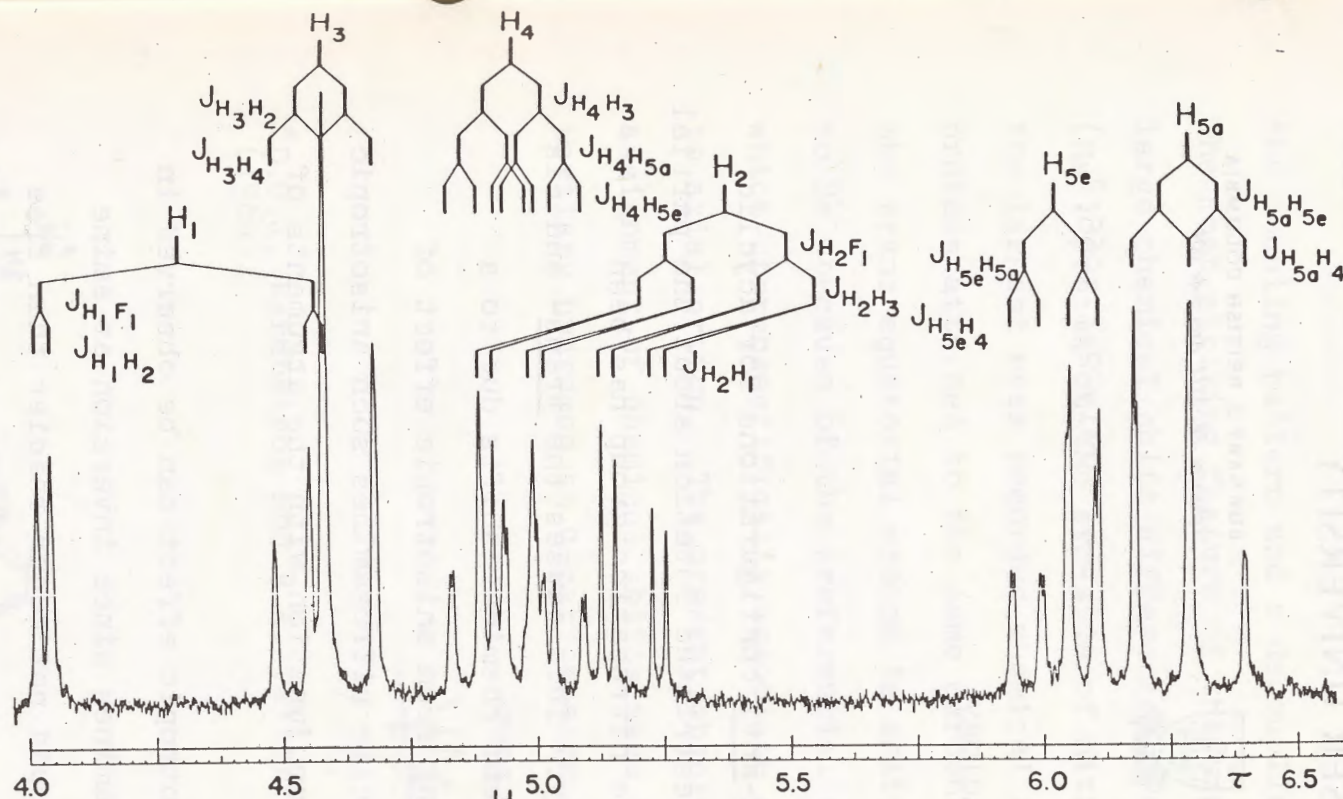
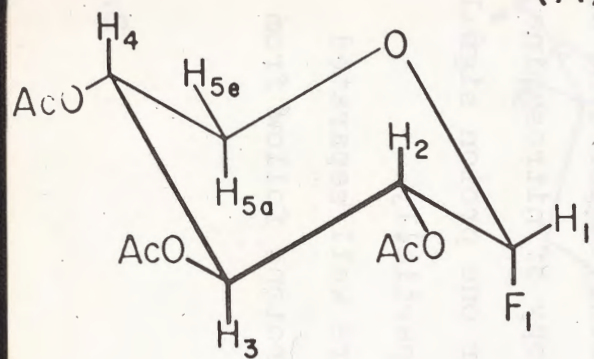
*John Manville*

J.F. Manville

dgh









## SIMON FRASER UNIVERSITY

DEPARTMENT OF CHEMISTRY



BURNABY 2, BRITISH COLUMBIA

Telephone 291-3111 Area code 604

July 21, 1966.

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

Dear Professor Shapiro,

The presence of syn-anti configurations for acyclic nitrosamines arising from restricted rotation about the partial double bond character of the nitrosamino group has been shown by n.m.r. spectroscopy. This causes the trans- and cis  $\alpha$ -protons to exhibit different chemical shifts due to a different environment arising from anisotropic effect of nitrosamino group. For acyclic nitrosamines such anisotropic effect has been studied in conjunction with the arguments of preferred conformations.

In theory such anisotropic effect can be observed in a rigid heterocyclic nitrosamines since inversion at amine center (of nitrosamines) should not occur readier than free rotation. We therefore chose N-nitroso-4-benzylpiperidine as a model compound to illustrate the anisotropy of nitrosamino group. This compound shows (see Fig.) four one proton signals at 60 Mc run, two of which ( $\text{He}^t$ ,  $\text{He}^c$ ) are partially superimposed in carbon tetrachloride but are well separated in benzene solution. The assignments of protons follow from

Professor B.L. Shapiro

July 21, 1966.

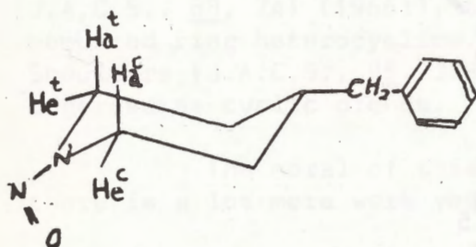
the coupling pattern and a decoupling experiment at 100 m.c. The most striking feature of this spectrum is the unusually large chemical shift difference of equatorial ( $\text{He}^c$ ) and axial ( $\text{Ha}^c$ ) proton on the syn-side of nitrosamino group perhaps one of the largest ever recorded chemical shift difference between two protons attached to the same carbon atom. In benzene solution the trans equatorial proton is shift upfield more with respect to  $\text{He}^c$  because of the preferential shielding by benzene nucleus which stereospecifically associate at the cis and from opposite to axial proton. The present observation support the assignment of chemical shifts made to primary, secondary and tertiary hydrogen at  $\alpha$ -carbon atom in acyclic nitrosamines.\*

Yours sincerely,

*Y. L. Chow*

Y. L. Chow

\*D.G.J. Karabatsos and R.A. Taller, J.Am.Chem.Soc., 86, 4373 (1964).



C.p.s. downfield of TMS

	$\text{CCl}_4$	$\text{C}_6\text{H}_6$
$\text{Ha}^c$	143	117
$\text{Ha}^t$	212	179
$\text{He}^t$	277	263
$\text{He}^c$	290	289



MONASH UNIVERSITY  
DEPARTMENT OF CHEMISTRY

WELLINGTON RD., CLAYTON, VICTORIA. PHONE 5440811

CHAIRMAN OF DEPARTMENT: PROFESSOR R. D. BROWN  
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29 September 1966

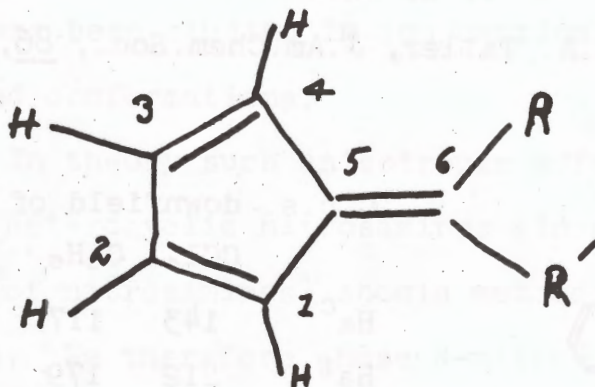
Professor B. J. Shapiro,  
Department of Chemistry,  
Illinois Institute of Technology,  
CHICAGO, 60616, U.S.A.

A Story of Three Fulvenes

Dear Barry,

Thanks for the gentle reminder; we are in the throes of getting our HR-100 converted into an HA-100 and are looking forward to tackling a few of our pet molecules.

One of the things we have been looking at lately is the spectra of some 6,6'-substituted fulvenes. These have rather closely coupled spectra on account of the limited range of chemical shifts which makes it difficult to be sure of the assignments of multiplets to protons. However, by looking at some suitable substituted derivatives and doing some spin decoupling, we have been able to make some progress. The pattern of long-range couplings surprised us, to some extent, and further work is probably necessary of this aspect.



- (a)  $R = H$       $R' = C_6H_5$   
(b)  $R = CH_3$ ,  $R' = C_6H_5$   
(c)  $R = H$ ,  $R' = N(CH_3)_2$

Three compounds studied are (a)-(c), using the above numbering scheme.

(a) 6-Phenyl fulvene

The spectrum of this compound was studied in  $\text{CCl}_4$  and  $\text{Me}_2\text{CO}$  and in the latter case, it was possible to carry out an iterative analysis of the ABCD system in order to obtain the spectral parameters. We found some additional fine structure and line broadening on the multiplets assigned to  $\text{H}_2$  and  $\text{H}_3$ , which was attributed to long-range coupling between  $\text{H}_6$  and these two protons. We have made the assumption that  $\text{H}_1$ , being closest to the phenyl group, will appear at lowest field. The values of the parameters are given in the table.

(b) 6-Methyl-6-phenyl fulvene

The spectrum of this compound was also studied in  $\text{CCl}_4$  and  $\text{Me}_2\text{CO}$  and again the spectrum in the  $\text{Me}_2\text{CO}$  had the greater range of chemical shifts. The experimental and calculated spectra for this case are shown in the figure. The fine structure observable on the multiplets we have assigned to  $\text{H}_2$  and  $\text{H}_3$ , is considered to come from long-range coupling between  $\text{H}_2$  and  $\text{H}_3$  and the methyl protons. This was checked by field-sweep spin decoupling experiments, where the methyl protons were irradiated and the multiplets for  $\text{H}_2$  and  $\text{H}_3$  were observed to sharpen up.

(c) 6-Dimethylamino fulvene

The spectrum of this compound in  $\text{CCl}_4$  was very closely coupled and resembled that of the parent compound fulvene. In  $\text{Me}_2\text{CO}$ , the transitions are spread over a greater range and an iterative analysis to determine the parameters was possible. The main feature that requires some comment is the greater asymmetry in the coupling constants  $J_{12}$  and  $J_{34}$ , which presumably arises from a greater perturbation of the electron distribution in the fulvene ring on account of the presence of an electron-donating substituent. The pattern of long-range coupling constants is harder to determine but it is possible that in this compound all the ring protons are coupling with  $\text{H}_6$  to about the same extent.

The value of the coupling constant  $J_{23}$  in these fulvenes is much closer to the value of 1.94 c/s obtained for the corresponding bond in cyclopentadiene (Manatt and Elleman quoted in Ganter and Roberts, J.A.C.S., 88, 741 (1966)), than to the higher values obtained in five-membered ring heterocyclics. This supports the conclusions of Smith and Shoulders (J.A.C.S., 86, 3188 (1964)) that these compounds are best regarded as cyclic dienes.

The moral of this little story (and others like it) is that there is a lot more work yet to be done.

Yours sincerely,



Alan J. Jones



Michael L. Heffernan

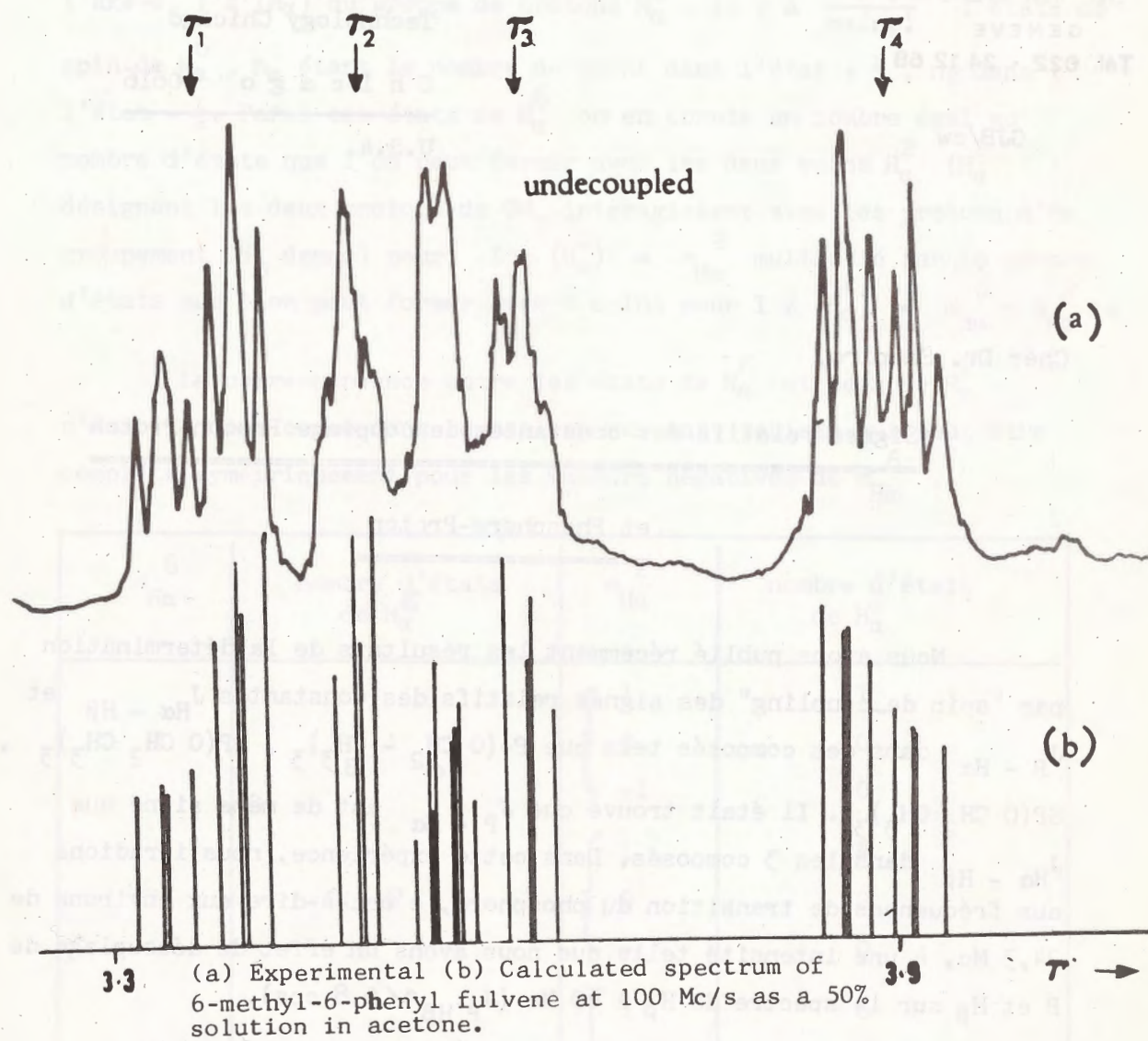


TABLE: CHEMICAL SHIFTS AND COUPLING CONSTANTS FOR FULVENE  
DERIVATIVES (100 Mc/s)

Parameter	6-phenyl fulvene		6-methyl-6-phenyl fulvene		6-dimethylamine fulvene	
	CCl <sub>4</sub>	Me <sub>2</sub> CO	CCl <sub>4</sub>	Me <sub>2</sub> CO	CCl <sub>4</sub>	Me <sub>2</sub> CO
$\tau_1$	3.37	3.291	3.523	3.390	3.72	3.596
$\tau_2$	3.37	3.373	3.588	3.520	3.91	3.711
$\tau_3$	3.56	3.543	3.678	3.598	3.91	3.804
$\tau_4$	3.76	3.689	3.946	3.881	3.72	3.930
$J_{12}$		5.5 <sub>7</sub>	5.5 <sub>6</sub>	5.4 <sub>3</sub>		4.6 <sub>9</sub>
$J_{13}$		1.3 <sub>1</sub>	1.8 <sub>5</sub>	1.8 <sub>6</sub>		1.7 <sub>9</sub>
$J_{14}$		1.4 <sub>4</sub>	1.0 <sub>5</sub>	1.5 <sub>1</sub>		1.7 <sub>3</sub>
$J_{23}$		1.8 <sub>8</sub>	1.8 <sub>5</sub>	1.8 <sub>7</sub>		1.9 <sub>2</sub>
$J_{24}$		2.0 <sub>2</sub>	1.7 <sub>7</sub>	1.6 <sub>8</sub>		1.7 <sub>8</sub>
$J_{34}$		5.8 <sub>6</sub>	5.2 <sub>9</sub>	5.4 <sub>3</sub>		3.5 <sub>5</sub>
$J_{26}$		~ 0.6				
$J_{2-Me}$			~ 0.4	~ 0.4		
$J_{3-Me}$			~ 0.4	~ 0.4		
Concn. (% w/v)	4	5	50	50	6	20

Michael J. Hoffner

Michael J. Hoffner







# ECOLE DE PHYSIQUE

UNIVERSITE DE GENEVE

Geneva, September 27, 1966

INSTITUT DE PHYSIQUE  
EXPERIMENTALE

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GJB/cw

Dr. B.L. Shapiro  
Associate Professor  
Illinois Institute of  
Technology Chicago

Chicago 60616

U.S.A.

Cher Dr. Shapiro,

Signes relatifs des constantes de couplage Proton-Proton

et Phosphore-Proton

Nous avons publié récemment les résultats de la détermination par "spin de couplage" des signes relatifs des constantes  $J_{H\alpha - H\beta}$  et  $J_{P - H\alpha}$  dans des composés tels que  $P-(OCH_2 - CH_3)_3$ ,  $OP(OCH_2 - CH_3)_3$ ,  $SP(OCH_2 - CH_3)_3$ . Il était trouvé que  $J_{P - H\alpha}$  est de même signe que  $J_{H\alpha - H\beta}$  dans les 3 composés. Dans cette expérience, nous irradiions aux fréquences de transition du phosphore, c'est-à-dire aux environs de 24,3 Mc, à une intensité telle que nous ayons un effet de découplage de P et  $H\beta$  sur le spectre de  $H\beta$  à 60 Mc ( $J_{P - H\beta} \sim 0,8$  cps).

Dans cette lettre, nous voulons montrer que, bien que le spectre du phosphore soit du type  $P H_\alpha^6 H_\beta^9$ , le phosphore interagissant avec 3 groupements  $CH_2 - CH_3$  équivalents; et celui des protons du type  $P H_\alpha^2 H_\beta^3$ , les protons n'interagissant pratiquement pas entre groupements dans une molécule, il est en effet possible de déterminer les signes relatifs de  $J_{P - H\alpha}$  et  $J_{H\alpha - H\beta}$ .

La correspondance entre les raies de P et celles de  $H_\beta$  se déduit de ce qui suit. Pour une valeur  $m_{H\alpha}^6$  du moment cinétique de spin suivant l'axe  $z$ ,  $I_z (H_\alpha^6)$  du groupe de protons  $H_\alpha^6$ , il y a  $\frac{6!}{n_\alpha! n_\beta!}$  états de spin de  $H_\alpha^6$ ,  $n_\alpha$  étant le nombre de spins dans l'état  $+\frac{1}{2}$ ,  $n_\beta$  dans l'état  $-\frac{1}{2}$ . Parmi ces états de  $H_\alpha^6$  on en compte un nombre égal au nombre d'états que l'on peut former avec les deux spins  $H_\alpha^2$  ( $H_\alpha^2$  désignant les deux protons de  $CH_2$  interagissant avec les protons d'un groupement  $CH_3$  donné) pour  $I_z (H_\alpha^2) = m_{H\alpha}^2$  multiplié par le nombre d'états que l'on peut former avec 4 spins pour  $I_z (H_\alpha^4) = m_{H\alpha}^6 - m_{H\alpha}^2$ .

La correspondance entre les états de  $H_\alpha^6$  et ceux de  $H_\alpha^2$  s'établit donc comme dans le tableau suivant, tableau qui peut être complété symétriquement pour les valeurs négatives de  $m_{H\alpha}^6$ .

$m_{H\alpha}^6$	nombre d'états de $H_\alpha^6$	$m_{H\alpha}^2$	nombre d'états de $H_\alpha^2$
3	1	$\begin{cases} 1 \\ 0 \\ -1 \end{cases}$	$\begin{matrix} 1 \\ 0 \\ 0 \end{matrix}$
2	6	$\begin{cases} 1 \\ 0 \\ -1 \end{cases}$	$\begin{matrix} 4 \\ 2 \\ 0 \end{matrix}$
1	15	$\begin{cases} 1 \\ 0 \\ -1 \end{cases}$	$\begin{matrix} 6 \\ 8 \\ 1 \end{matrix}$
0	20	$\begin{cases} 1 \\ 0 \\ -1 \end{cases}$	$\begin{matrix} 4 \\ 12 \\ 4 \end{matrix}$



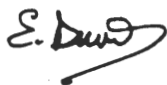
Donc en pratique nous observons une évolution des 3 multiplets de  $H_{\beta}^3$  suivant la fréquence d'irradiation et au lieu d'avoir un effet de découplément complet aux fréquences  $\nu_P + J_P - H_{\alpha}$ ,  $\nu_P$  et

$\nu_P - J_P - H_{\alpha}$  pour les multiplets de  $H_{\beta}^3$  correspondant aux 1,0 et -1 de  $m H_{\alpha}^2$ , nous avons un maximum de cet effet.

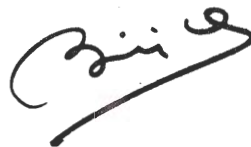
\* E. Duval et E.A.C. Lucken, Mol. Phys. 10, 499 (1966)

E. Duval, Thèse non publiée.

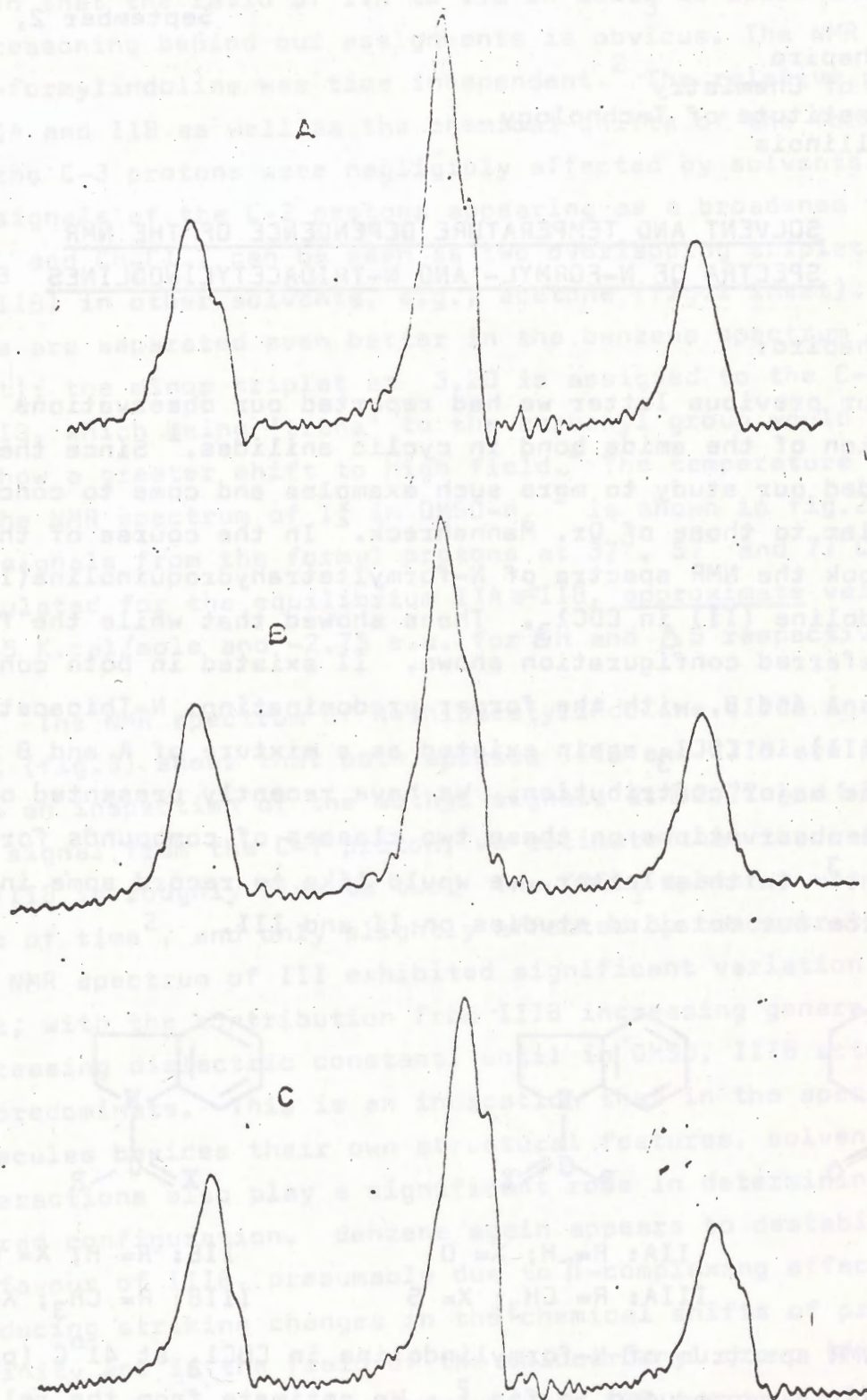
Sincères salutations.



Dr. E. Duval



Prof. G.J. Béné



Evolution du spectre  $H_2$  du triéthyl phosphate à 60 MHz avec la fréquence du champ d'irradiation  $H_2$ .

A :  $\nu_2 = 24,287545$  MHz.

B :  $\nu_2 = 24,287555$  MHz.

C :  $\nu_2 = 24,287565$  MHz.



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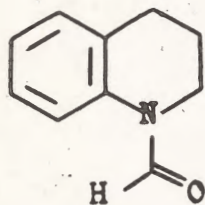
September 2, 1966

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

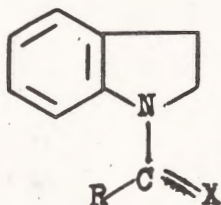
SOLVENT AND TEMPERATURE DEPENDENCE OF THE NMR  
SPECTRA OF N-FORMYL- AND N-THIOACETYLINDOLINES

Dear Dr. Shapiro:

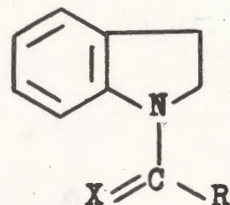
In our previous letter we had reported our observations on the configuration of the amide bond in cyclic anilides.<sup>1</sup> Since then we have extended our study to more such examples and come to conclusions similar to those of Dr. Mannshreck.<sup>2</sup> In the course of this work, we took the NMR spectra of N-formyltetrahydroquinoline(I) and N-formylindoline (II) in  $\text{CDCl}_3$ . These showed that while the former had the preferred configuration shown, II existed in both configurations A and B, with the former predominating. N-Thioacetylindoline (III) in  $\text{CDCl}_3$  again existed as a mixture of A and B with A having the major contribution. We have recently presented our qualitative observations on these two classes of compounds for publication.<sup>3</sup> In this letter, we would like to record some interesting data from our detailed studies on II and III.



I



IIA: R = H; X = O  
IIIA: R =  $\text{CH}_3$ ; X = S



IIB: R = H; X = O  
IIIB: R =  $\text{CH}_3$ ; X = S

The NMR spectrum of N-formylindoline in  $\text{CDCl}_3$  at  $41^\circ\text{C}$  (probe temperature) is reproduced in fig.1. We estimate from the relative intensities of the signals from the formyl proton at  $\delta 8.47$  and  $\delta 8.88$  (all shifts in ppm relative to TMS internal standard), as well as



- 2 -

from the strength of the broad doublet at  $\delta 8.05$  due to the C-7 proton that the ratio of IIA to IIB in  $\text{CDCl}_3$  is approximately 3:1. The reasoning behind our assignments is obvious. The NMR spectrum of N-formylindoline was time independent.<sup>2</sup> The relative proportions of IIA and IIB as well as the chemical shifts of the formyl proton and the C-3 protons were negligibly affected by solvents. However the signals of the C-2 protons appearing as a broadened triplet in  $\text{CDCl}_3$  and  $\text{CH}_2\text{Cl}_2$ , can be seen as two overlapping triplets (from IIA and IIB) in other solvents, e.g., acetone (fig.1 inset). These triplets are separated even better in the benzene spectrum (fig. 1 inset); the minor triplet at  $\delta 3.20$  is assigned to the C-2 protons of IIB, which being 'trans' to the carbonyl group would be expected to show a greater shift to high field.<sup>4</sup> The temperature dependence of the NMR spectrum of II in  $\text{DMSO}-d_6$ <sup>5</sup> is shown in fig.2. Using the signals from the formyl protons at  $37^\circ$ ,  $57^\circ$  and  $77^\circ\text{C}$ , we have calculated for the equilibrium  $\text{IIA} \rightleftharpoons \text{IIB}$ , approximate values of  $-1.35$  K.cal/mole and  $-2.75$  e.u. for  $\Delta H$  and  $\Delta S$  respectively.

The NMR spectrum of N-thioacetylindoline (III) in  $\text{CDCl}_3$  at  $41^\circ\text{C}$  (fig.3) shows that both species IIIA and IIIB are present. From an inspection of the methyl signals at  $\delta 2.77$  and  $\delta 2.95$ , and the signal from the C-7 proton, we estimate that the ratio of IIIA to IIIB is roughly five to two. The  $\text{CDCl}_3$  spectrum was independent of time<sup>2</sup>, and only slightly affected by concentration. But the NMR spectrum of III exhibited significant variation with solvent, with the contribution from IIIB increasing generally with increasing dielectric constant, until in  $\text{DMSO}$ , IIIB actually begins to predominate. This is an indication that in the spectra of such molecules besides their own structural features, solvent - solute interactions also play a significant role in determining the preferred configuration. Benzene again appears to destabilize IIIA in favour of IIIB, presumably due to  $\pi$ -complexing effects, besides producing striking changes in the chemical shifts of protons in the vicinity and in the field of the thiocarbonyl group (fig. 3 inset). A temperature dependence study<sup>5</sup> of the NMR spectrum of III in  $\text{DMSO}-d_6$  indicated coalescence of the methyl signals to a broad singlet ( $\delta 2.83$ ) at  $100^\circ$ , which sharpened at  $120^\circ\text{C}$ . Calculation of



$\Delta H$  and  $\Delta S$  was complicated because the relative intensities of the methyl signals (by themselves more reliable than the signal due to the fraction of the C-7 proton appearing for IIIB) could not be satisfactorily determined, due to partial overlapping from other signals. We feel that  $\Delta H$  and  $\Delta S$  would be roughly 1.8 K. cal/mole and 5.1 e.u. respectively for the equilibrium  $\text{IIIA} \rightleftharpoons \text{IIIB}$ . We hope that these results would be of some interest for your readers, although they are less exciting compared to Dr. Mannshreck's report<sup>6</sup> of his successful separation of rotational isomers of thioamides and nitrosoamines!

With regards,

Yours sincerely,

K. Nagarajan

(K. Nagarajan)

M. D. Nair

(M.D. Nair)

References:

1. K. Nagarajan and M.D. Nair, IITNN, 87, 34 (1966)
2. A. Mannshreck, IITNN, 68, 44 (1964)
3. K. Nagarajan, M.D. Nair and P.M. Pillai, paper submitted for publication
4. L.A. Laplanche and M.T. Rogers, J. Am. Chem. Soc., 85, 3728 (1963)
5. This was kindly carried out by Dr. Stuber of CIBA, Basle
6. A. Mannshreck, IITNN, 85, 41 (1965)

FIG. 1

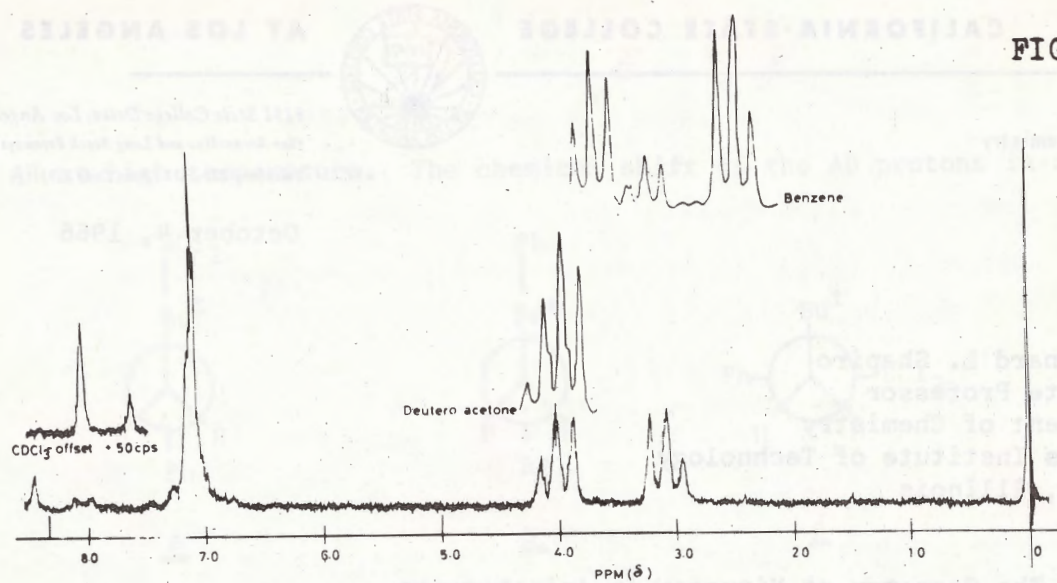


FIG. 2

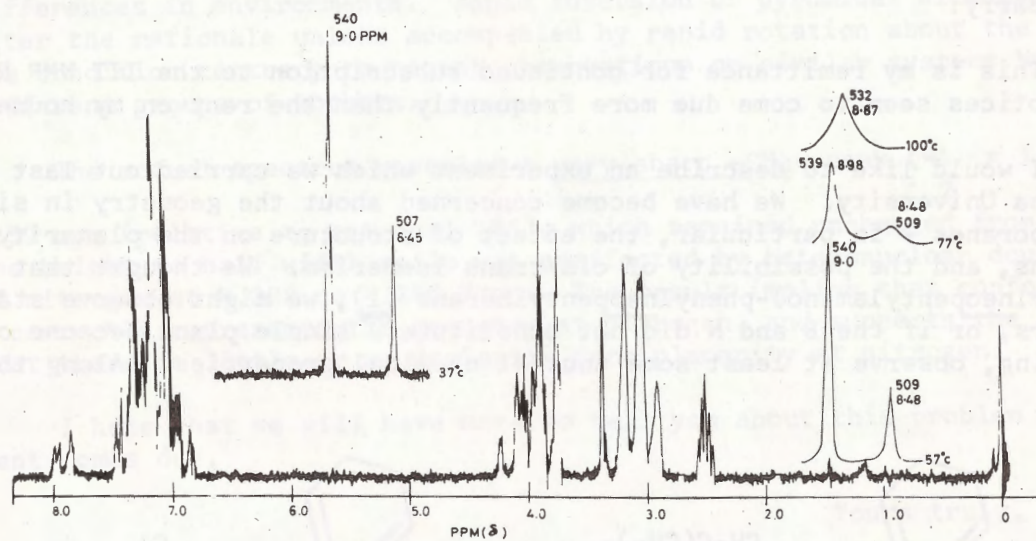
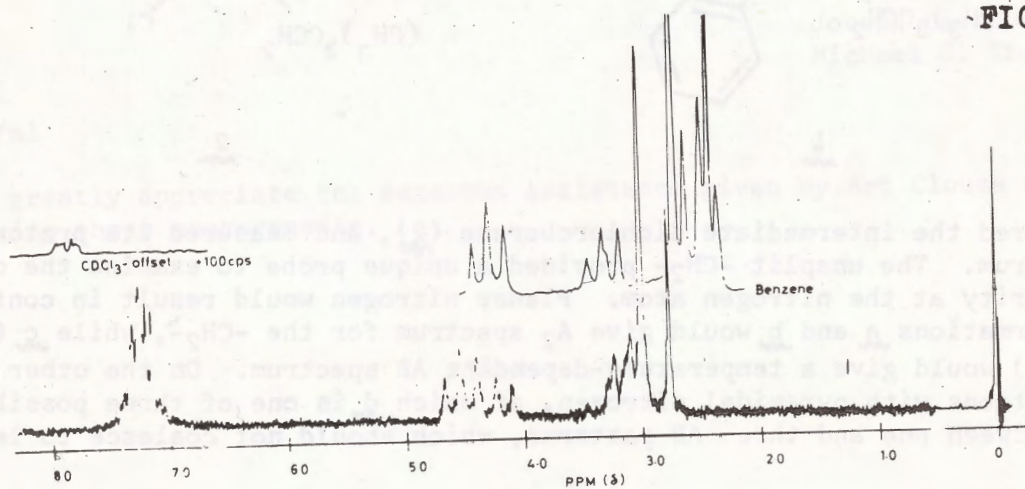


FIG. 3







Department of Chemistry

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October 4, 1966

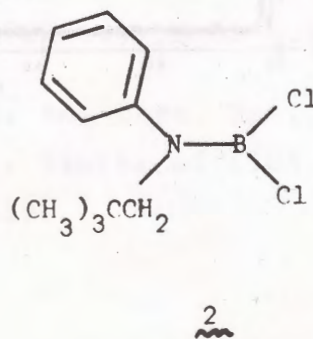
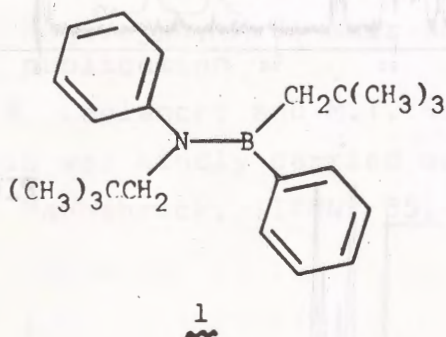
Dr. Bernard L. Shapiro  
 Associate Professor  
 Department of Chemistry  
 Illinois Institute of Technology  
 Chicago, Illinois  
 60616

Title: The Geometry at Nitrogen in Aminoboranes

Dear Barry:

This is my remittance for continued subscription to the IIT NMR Newsletter. The notices seem to come due more frequently than the rent on my house.

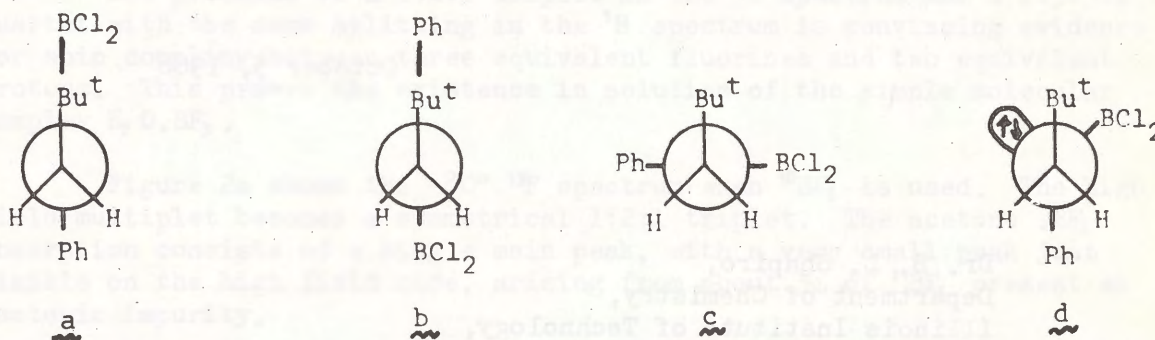
I would like to describe an experiment which we carried out last summer at Indiana University. We have become concerned about the geometry in simple aminoboranes - in particular, the effect of structure on the planarity, and bond lengths, and the possibility of cis/trans isomerism. We thought that by examining (phenylneopentylamino)-phenylneopentylborane (1), we might observe stable cis/trans isomers, or if the B and N did not constitute a single plane because of over-crowding, observe at least some unusual chemical properties. Along the route we



prepared the intermediate dichloroborane (2), and measured its proton resonance spectrum. The unsplit  $-\text{CH}_2-$  provided a unique probe to examine the question of planarity at the nitrogen atom. Planar nitrogen would result in conformers a - d. Conformations a and b would give  $A_2$  spectrum for the  $-\text{CH}_2-$ , while c (and its mirror image) would give a temperature-dependant AB spectrum. On the other hand, conformations with pyramidal nitrogen, of which d is one of three possible, would lead to between one and three AB patterns, which should not coalesce to less than one

- 2 -

AB at high temperature. The chemical shift of the AB protons in conformations



such as d are intuitively expected to be rather different because of gross differences in environments. Rapid inversion of pyramidal nitrogen would not alter the rationale unless accompanied by rapid rotation about the B—N bond - a condition contrary to recent observations on similar systems by several different groups of workers.

The actual spectrum revealed a very sharp  $-\text{CH}_2-$  peak ( $\frac{\text{Wl}}{2} = 1.29$  cps) at 3.59 ppm (relative to external TMS), which remained unchanged from  $-50^\circ$  to  $+60^\circ$ . The height to half width ratio was unaffected by heteronuclear double irradiation of nitrogen at 4.328 to 4.340 Mcps. The result implies that conformation a or b correctly represents the geometry at nitrogen, and supports the current view that there is little or no deviation from planarity at nitrogen.

I hope that we will have more to tell you about this problem when the next rent comes due.

Yours truly,

*Joe Casanova, Jr.*

Joseph Casanova, Jr.  
Michael J. Thomas

JC/ml

We greatly appreciate the generous assistance given by Art Clouse at I.U. in making these measurements.



## MCMMASTER UNIVERSITY

HAMILTON COLLEGE

HAMILTON, ONTARIO, CANADA

October 5, 1966

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

The  $^1\text{H}$  and  $^{19}\text{F}$  N.M.R. Spectra of the Adduct  $\text{H}_2\text{O} \cdot \text{BF}_3$

Dear Professor Shapiro,

We have recently obtained the  $^1\text{H}$  and  $^{19}\text{F}$  n.m.r. spectra of the molecular complex  $\text{BF}_3 \cdot \text{H}_2\text{O}$ . They provide strong evidence to support the usual assumption that the adduct is the simple donor-acceptor complex  $\text{H}_2\text{O} \rightarrow \text{BF}_3$ .

Low temperature ( $-80^\circ$ ) proton and fluorine NMR spectra of a solution of water and  $\text{BF}_3$ , in a molar ratio of 1:2, (0.44M and 0.86M) in acetone solvent are shown in Figure 1. The proton spectrum (Figure 1a) has two small peaks, to low field of the solvent acetone peak, which can be attributed to species complexed with  $\text{BF}_3$ . A small peak at  $2.81\delta$  arises from acetone complexed to  $\text{BF}_3$ ; the 0.64 p.p.m. shift of the acetone peak to low field on complex formation is in reasonable agreement with the value of 0.59 p.p.m. that we have found for solutions of acetone and  $\text{BF}_3$  in methylene chloride. A small low field peak at  $12.42\delta$  has a 1:3:3:1 quartet fine structure with a spacing of  $2.93 \pm 0.1$  c.p.s. The fluorine spectrum (Figure 1b) consists of two major peaks of chemical shifts 146.05 and 146.59 p.p.m. to high field of  $\text{CFC}_2$ , each further split by fine structure. The low field peak is assigned to the 1:1 complex of  $\text{BF}_3$  with acetone, since this is the major peak in samples of  $\text{BF}_3$  in acetone containing only a trace of water. This peak is split by a  $^{10}\text{B}$ - $^{11}\text{B}$  isotope shift. The high field  $^{19}\text{F}$  peak has a skewed quartet structure with unequal splittings. The splittings are similar in size to those of the  $^1\text{H}$  low field quartet, and to the isotope shift on the low field  $^{19}\text{F}$  peak ( $3.72 \pm 0.1$  c.p.s. at 56.4 Mc./sec.).

The skewed quartet structure of the high field peak can be explained by an isotope shift of 0.066 p.p.m., as for the low field peak, and also a splitting of each of the F-on- $^{10}\text{B}$  and F-on- $^{11}\text{B}$  peaks

Dr. B. L. Shapiro

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October 5, 1966

into a 1:2:1 triplet with splittings of 2.93 c.p.s. Figure 1c shows the theoretical spectrum based on these assumptions, together with the observed spectrum. Agreement is good.

The presence of a 1:2:1 triplet in the  $^{19}\text{F}$  spectrum and a 1:3:3:1 quartet with the same splitting in the  $^1\text{H}$  spectrum is convincing evidence for spin coupling between three equivalent fluorines and two equivalent protons. This proves the existence in solution of the simple molecular complex  $\text{H}_2\text{O} \cdot \text{BF}_3$ .

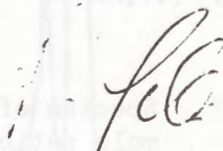
Figure 2a shows the  $-80^\circ$   $^{19}\text{F}$  spectrum when  $^{10}\text{BF}_3$  is used. The high field multiplet becomes a symmetrical 1:2:1 triplet. The acetone  $\cdot\text{BF}_3$  absorption consists of a single main peak, with a very small peak just visible on the high field side, arising from about 3% of  $^{11}\text{BF}_3$  present as isotopic impurity.

The use of  $\text{BF}_3$  containing approximately 50% of  $^{10}\text{B}$  and 50% of  $^{11}\text{B}$  yielded the low temperature spectrum shown in Figure 2b. The high field multiplet is a 1:3:3:1 quartet resulting from the overlap of two 1:2:1 triplets of almost equal intensity, due to  $\text{H}_2\text{O} \cdot ^{10}\text{BF}_3$  and  $\text{H}_2\text{O} \cdot ^{11}\text{BF}_3$ . The acetone  $\cdot\text{BF}_3$  peak is a doublet, the peaks having almost equal intensities. Figures 2a and 2b confirm the postulated contribution of the isotope shift to the skewed quartet structure of the high field peak of the  $^{19}\text{F}$  spectrum of Figure 1.

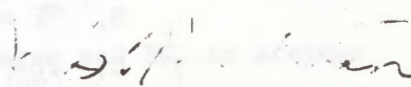
Proof that the  $^{19}\text{F}$  1:2:1 triplet (or overlapping triplets) arises from coupling with protons was obtained from a solution of natural abundance  $\text{BF}_3$  in acetone- $d_6$  containing  $\text{D}_2\text{O}$ . In the  $-80^\circ$   $^{19}\text{F}$  NMR spectrum (Figure 2c), both the acetone- $d_6 \cdot \text{BF}_3$  and  $\text{D}_2\text{O} \cdot \text{BF}_3$  peaks were split into two peaks of relative intensities 1:4 by an isotope shift of 0.066 p.p.m.; no additional splitting was present on either peak. Thus deuterium substitution destroys the 1:2:1 triplet structure, proving that the triplet arises in the  $^{19}\text{F}$  spectrum of  $\text{BF}_3 \cdot \text{H}_2\text{O}$  from coupling with protons.

The evidence obtained from the isotopic substitution experiments is sufficient to remove any doubts that the species observed is the simple 1:1 complex,  $\text{H}_2\text{O} \rightarrow \text{BF}_3$ , with  $J_{\text{H-F}} = 2.93 \pm 0.1$  c.p.s. The absence of any visible effect arising from coupling with boron is presumably due to rapid quadrupole relaxation of the boron nuclei.

Yours sincerely,



R. J. Gillespie



J. S. Hartman

RJG/pm



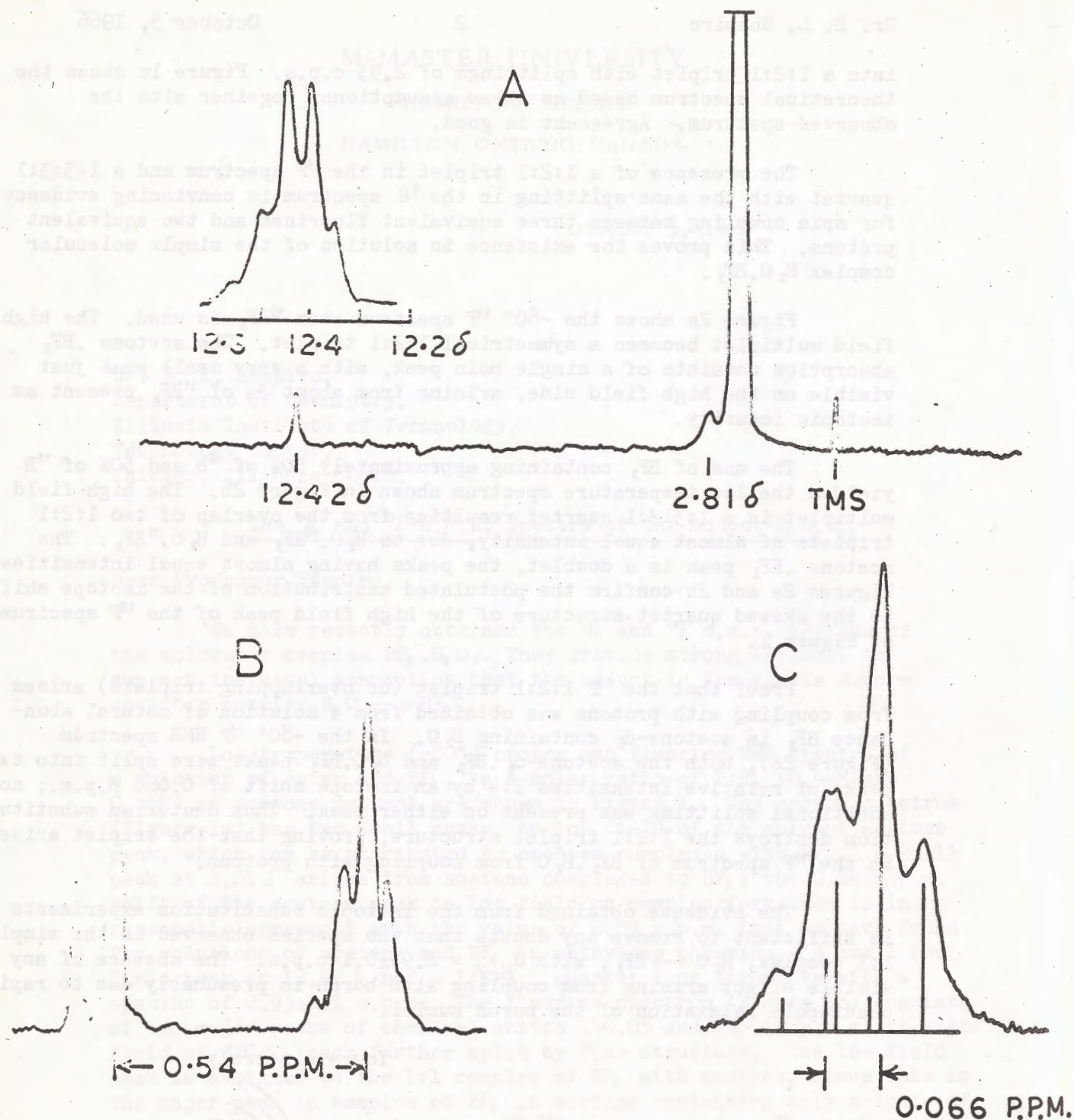


Figure 1. Spectra of an acetone solution of water and  $\text{BF}_3$ , in the mole ratio 1:2 at  $-80^\circ$

- A.  $^1\text{H}$  spectrum (60 Mc.p.s.)
- B.  $^{19}\text{F}$  spectrum (56.4 Mc.p.s.)
- C. The high field multiplet of the  $^{19}\text{F}$  spectrum, with the theoretical spectrum superimposed.

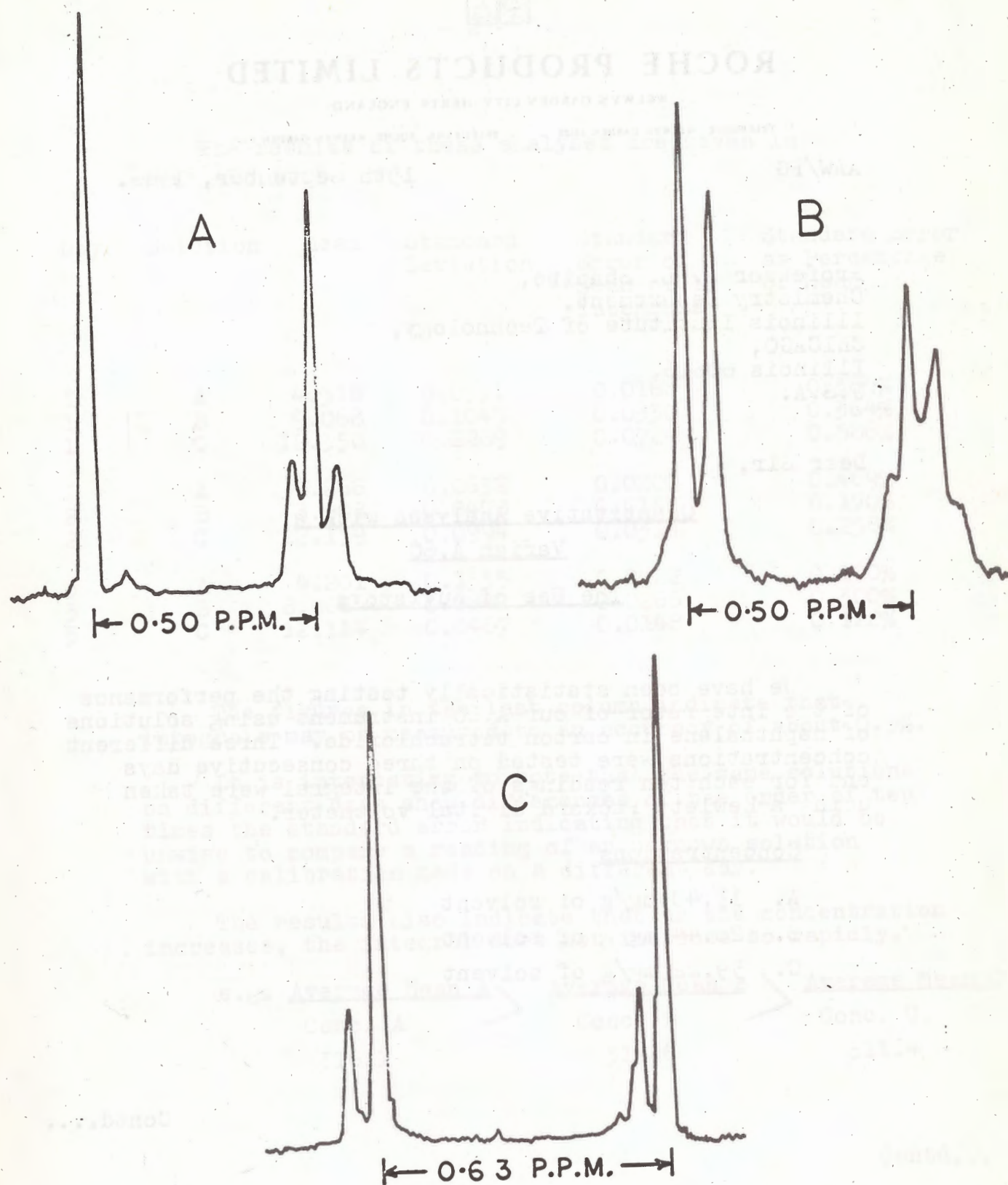


Figure 2.  $^{19}\text{F}$  spectra of solutions of water and  $\text{BF}_3$  in acetone at  $-80^\circ$ , such that  $[\text{H}_2\text{O}] < [\text{BF}_3]$ .

A.  $^{10}\text{BF}_3$ ; B.  $^{10}\text{BF}_3$ ;  $^{11}\text{BF}_3$  = 53:47;

C. natural abundance  $\text{BF}_3$  and  $\text{D}_2\text{O}$  in acetone- $\text{d}_6$





# ROCHE PRODUCTS LIMITED

WELWYN GARDEN CITY, HERTS, ENGLAND

TELEPHONE: WELWYN GARDEN 28128

TELEGRAMS: ROCHE, WELWYN GARDEN CITY

AAW/PG

15th September, 1966.

Professor S. L. Shapiro,  
Chemistry Department,  
Illinois Institute of Technology,  
CHICAGO,  
Illinois 60616,  
U.S.A.

Dear Sir,

## Quantitative Analyses with a

Varian A.60

## The Use of Nuvistors

We have been statistically testing the performance of the integrator of our A.60 instrument using solutions of naphthalene in carbon tetrachloride. Three different concentrations were tested on three consecutive days and for each ten readings of the integral were taken using a Hewlett Packard digital voltmeter.

### Concentrations

- A. 13.43 mg/g of solvent
- B. 23.44 mg/g of solvent
- C. 39.22 mg/g of solvent

Contd....

- 2 -

The results of these analyses are given in  
Table 1:

Day	Solution	Mean	Standard Deviation	Standard Error of Mean of 10 Integrals	Standard Error as Percentage of Mean
1	A	4.316	0.0531	0.0163	0.389%
1	B	9.068	0.1045	0.0330	0.364%
1	C	12.358	0.2239	0.0724	0.586%
2	A	4.266	0.0632	0.0200	0.469%
2	B	3.903	0.0478	0.0151	0.170%
2	C	12.159	0.0994	0.0314	0.258%
3	A	4.201	0.1335	0.0422	0.100%
3	B	3.866	0.0842	0.0266	0.300%
3	C	12.114	0.0467	0.0148	0.122%

The figures in the last column indicate that integrals may be measured to an accuracy of about 0.5%.

It is interesting to note that the same solutions on different days show differences of the order of ten times the standard error indicating that it would be unwise to compare a reading of an unknown solution with a calibration made on a different day.

The results also indicate that as the concentration increases, the integral does not increase so rapidly.

e.g. Average Mean A > Average Mean B > Average Mean C  
           Conc. A                      Conc. B                      Conc. C.  
           316.1                          314.6                      311.4

Contd...



- 3 -

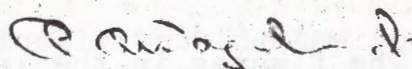
These differences are significant and suggest that comparisons of controls and unknowns should be carried out at similar concentrations. It is, however, possible that concentration should be measured in mass of solute per unit volume of solution and we are currently following this up.

Incidentally, we found that it is unimportant whether or not the sample tube is spinning, a result which was somewhat unexpected.

#### The Use of Nuvistors

We have recently installed two Nuvistors in the first stage of our A.60 sample receiver. We have not succeeded in increasing the S/N ratio by any significant amount and would be grateful for any tips from anybody who has successfully carried out this modification.

Yours faithfully,  
ROCHE PRODUCTS LIMITED



A. A. Wagland.



## UNIVERSITY OF PITTSBURGH

PITTSBURGH-PENNSYLVANIA-15213



DEPARTMENT OF CHEMISTRY

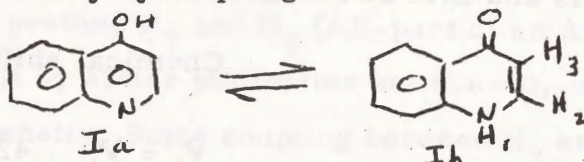
5 October, 1966

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

Dear Barry:

Vicinal Couplings in 4-Quinolones

The spectra of a number of examples of potentially tautomeric hydroxypyridines have been studied by Katritzky; on the basis of chemical shift arguments, it was concluded that the pyridone tautomers predominate for the 2- and 4-isomers. We have been examining the spectra of a series of substituted hydroxyquinolines and find that for the 4-isomers (I) predominance of the quinolone tautomers (Ib) can be demonstrated readily by coupling constant considerations. The C(2)-C(3)



$\pi$ -bond order of Ib should be significantly higher than that of Ia and, consequently,  $J_{2,3}$  for Ib should be of greater magnitude than for Ia. This expectation was realized in three examples of I [(i) 6-methyl; (ii) 8-methyl; (iii) 6,8-dimethyl] for which  $J_{2,3} = 7.2-7.9$  c.p.s. For model quinolines incapable of this tautomerism,  $J_{2,3}$  the corresponding coupling constants were 4.0-4.1 c.p.s. The quinolone assignment for Ii-iii, as well as for 3-carbethoxy-8-methyl-4-hydroxyquinoline, was further supported by the observation of couplings with  $H_1$ :  $J_{1,2} = 7.4-8.3$  c.p.s.;  $J_{1,3} = 0.8-1.1$  c.p.s. (for Ii-iii).

Corresponding studies of the potentially tautomeric 5- and 7-hydroxyquinolines are in progress.

Sincerely,

C. E. Griffin

W. E. Byrne



INSTITUT FÜR ORGANISCHE CHEMIE  
DER UNIVERSITÄT KÖLN

5 KÖLN, September 29, 1966

ZÜLPICHER STRASSE 47

TELEFON: 20 24 239

Dr. H. Günther

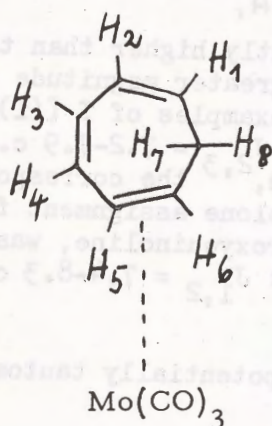
Professor Dr. B. L. Shapiro  
Illinois Institute of Technology  
Department of Chemistry  
Chicago, Illinois 60616  
U.S.A.

Dear Barry,

I have always been interested to see, how accurately your reminder-system works. Now I know! Thanks and here it goes:

Analysis of CHT-molybdenumtricarboxyl spectrum

In connection with other studies of cyclic 1.3.5-trienes we have analyzed the nmr spectrum of cycloheptatriene-molybdenumtricarboxyl using sub-spectral analysis and LAOCOON II. Results are shown below:



Chemical shifts: (\*)

	A	B
$\nu_1 = \nu_6$	$424.2 \pm 1.0$	$424.2 \pm 1.0$
$\nu_2 = \nu_5$	$345.1 \pm 1.0$	$345.1 \pm 1.0$
$\nu_3 = \nu_4$	282.15	282.14
$\nu_7$	492.06	492.06
$\nu_8$	464.30	464.30
$J_{12}$	$8.4 \pm 0.2$	$8.4 \pm 0.2$
$J_{13} + J_{14}$	1.38	1.38
$J_{16}$	$< 0.3$	$< 0.3$
$J_{23}$	6.81	6.82
$J_{24}$	0.77	0.77
$J_{25}$	0.34	- 0.59
$J_{34}$	8.43	8.44

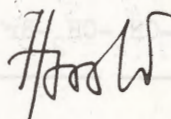
(\*) in  $C_6D_6$ ; TMS (= 600.00 cps)  
as internal reference.

	C	D
$J_{17}$	2.75	2.74
$J_{18}$	8.70	8.70
$J_{27}$	- 1.32	1.17
$J_{28}$	0.94	0.80
$J_{78}$	-14.14	- 14.14

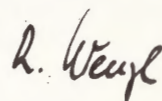
The spectrum has been treated as two independent 6-spin-cases (protons  $H_1 - H_6$  and  $H_1, H_2, H_5 - H_8$  respectively). Since coupling between protons  $H_3, H_4$  and  $H_7, H_8$  is negligible, most of the information for each system has been extracted from the lines of these protons. The line-positions for protons  $H_3$  and  $H_4$  (AA' part of an AA'BB'XX'-system) are equally well fit by either parameter set A or B (rms error 0.07 cps), but A gives the better agreement with intensities. We therefore believe  $J_{25}$  to be positive. The lines for protons  $H_7$  and  $H_8$  (AB-part of an ABMM'XX'-system) are equally well fit by either parameter set C or D, with respect to position as well as intensity. Since coupling between  $H_2$  and  $H_7$ , according to their steric relationship, is mainly based on the allylic  $\pi$ -mechanism,  $J_{27}$  should be negative. In contrast,  $J_{28}$  should have only a small  $\pi$ -contribution and a large (positive)  $\sigma$ -part. Parameter set C therefore seems the correct one.

A detailed discussion of these results, together with a refined analysis for cycloheptatriene will be published shortly in Z. Naturforsch. (b).

Sincerely yours,



(H. Günther)



(R. Wenzl)



## National Chemical Research Laboratory

of the South African Council for Scientific &amp; Industrial Research

NCRL

OF THE CSIR

Telegrams NAVORSCHM

Telephone 74-6011

P.O. Box 395, Pretoria

AIR MAIL

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Our letter

Your file

Your letter

6-16-66

Prof. B.L. Shapiro,  
 Illinois Institute of Technology,  
CHICAGO,  
 Illinois 60616,  
 U.S.A.

Solvent Dependence of Coupling Constants  
in 1,2-Dibromo-ethane and 1,2-Dichloro-ethane

Dear Prof. Shapiro,

We have analysed the  $C^{13}$  - satellites of the title compounds in various solvents to obtain the proton-proton coupling parameters N and L. Using a modified reaction field theory<sup>1,2</sup> to calculate the energy difference between the rotamers, we derived the N and L values for the two forms (trans and gauche) and the corresponding vicinal coupling constants for the individual isomers. The experimental N and L parameters and the theoretical curves fitted by least-squares are shown in the two figures. The coupling constants for the two rotational isomers are given in the following table:

Compound	trans isomer		gauche isomer	
	$J^t$	$J^g$	$J^t + J^{g'}$	$J^g$
$Cl-CH_2-CH_2-Cl$	13.6	4.8	15.1	2.3
$Br-CH_2-CH_2-Br$	13.7	5.1	14.7	2.7

These / .....

- 2 -

These couplings agree well with values found for other 1,2-dihalo-ethanes<sup>2</sup> and support the observation that an electro-negative substituent has the maximum effect on a vicinal proton-proton coupling when it is trans and co-planar to one of the coupling protons.<sup>3</sup>

A detailed account of this study will be submitted for publication in due course.

Yours sincerely,

*P.L. Wessels*

P.L. Wessels

*K.G.R. Pachler*

K.G.R. Pachler

ASSISTANT TECHNICAL OFFICER    SENIOR RESEARCH OFFICER  
CHEMICAL PHYSICS GROUP  
NATIONAL CHEMICAL RESEARCH LABORATORY

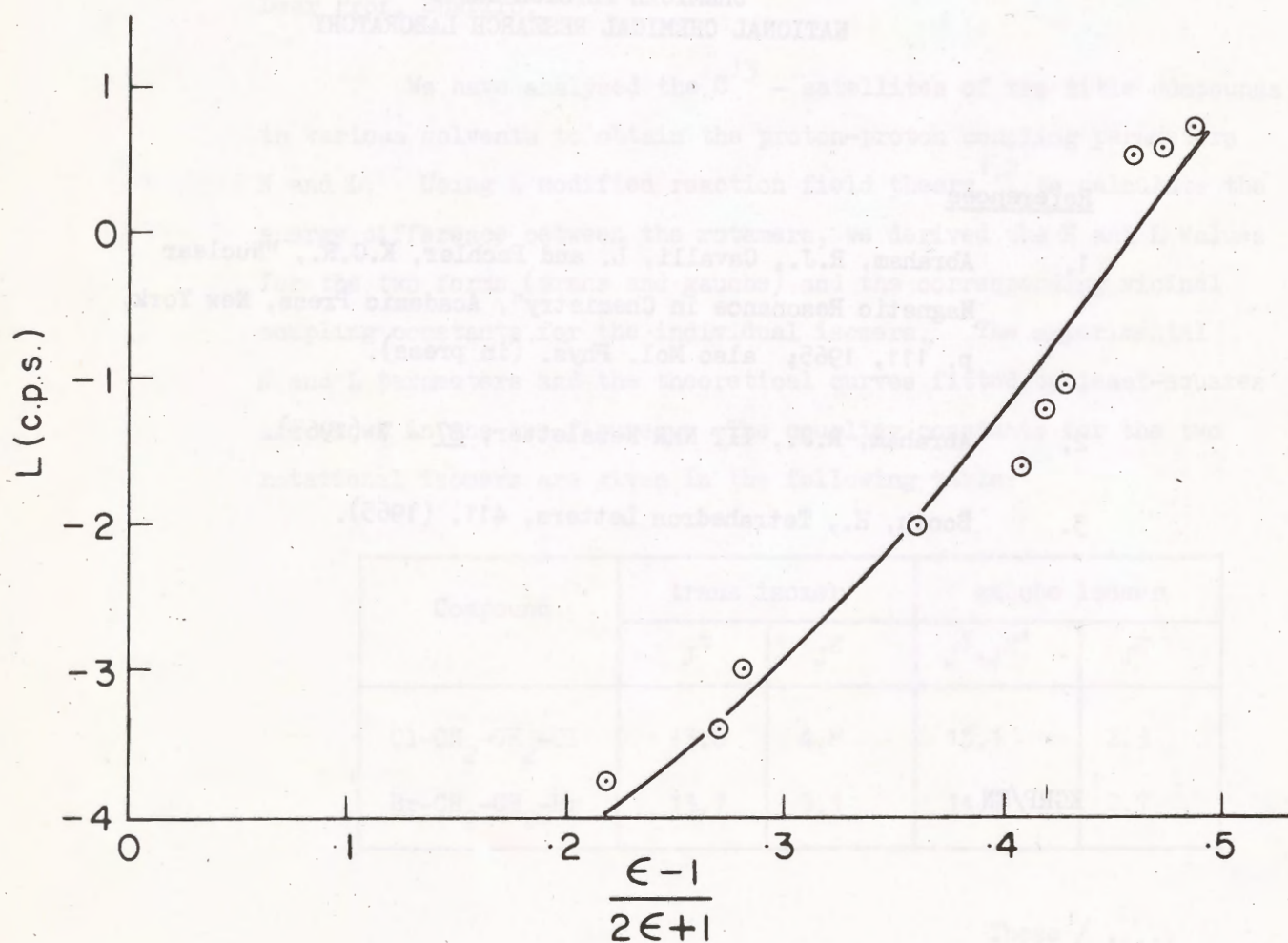
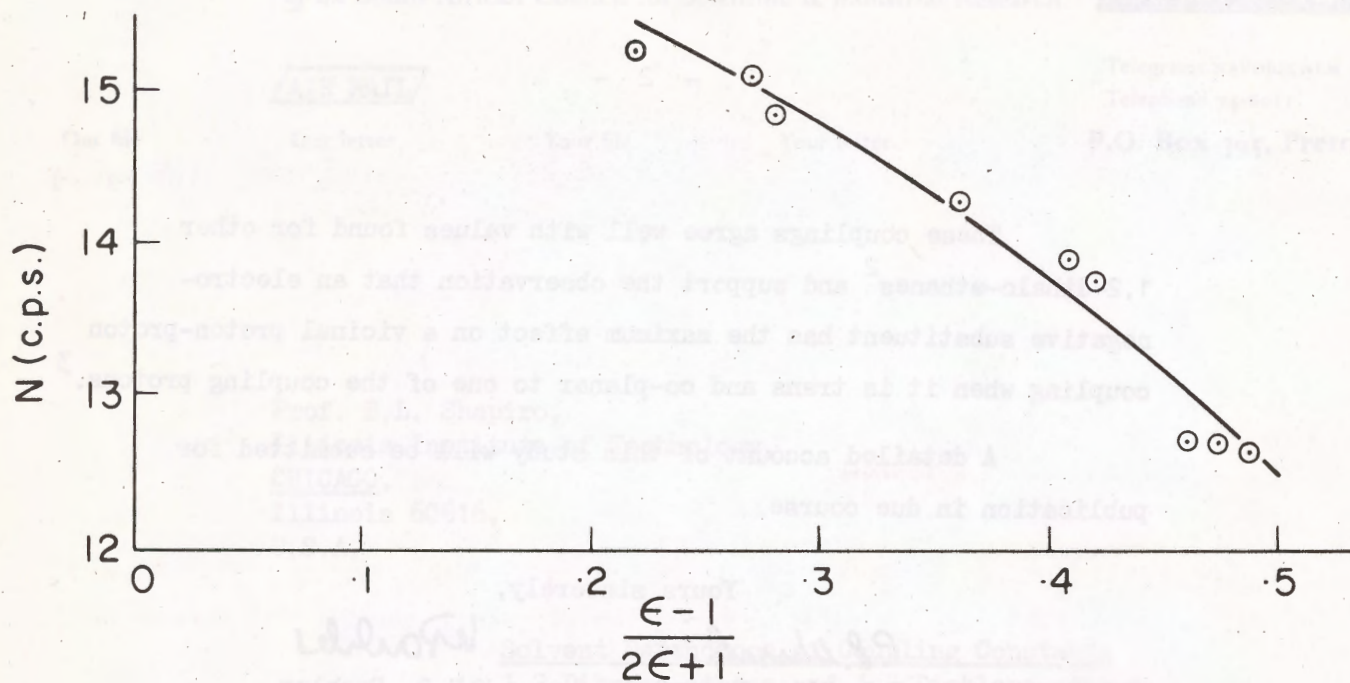
#### References

1. Abraham, R.J., Cavalli, L. and Pachler, K.G.R., "Nuclear Magnetic Resonance in Chemistry", Academic Press, New York, p. 111, 1965; also Mol. Phys. (in press).
2. Abraham, R.J., IIT NMR Newsletter, 87 - 7 (1965).
3. Booth, H., Tetrahedron Letters, 411, (1965).

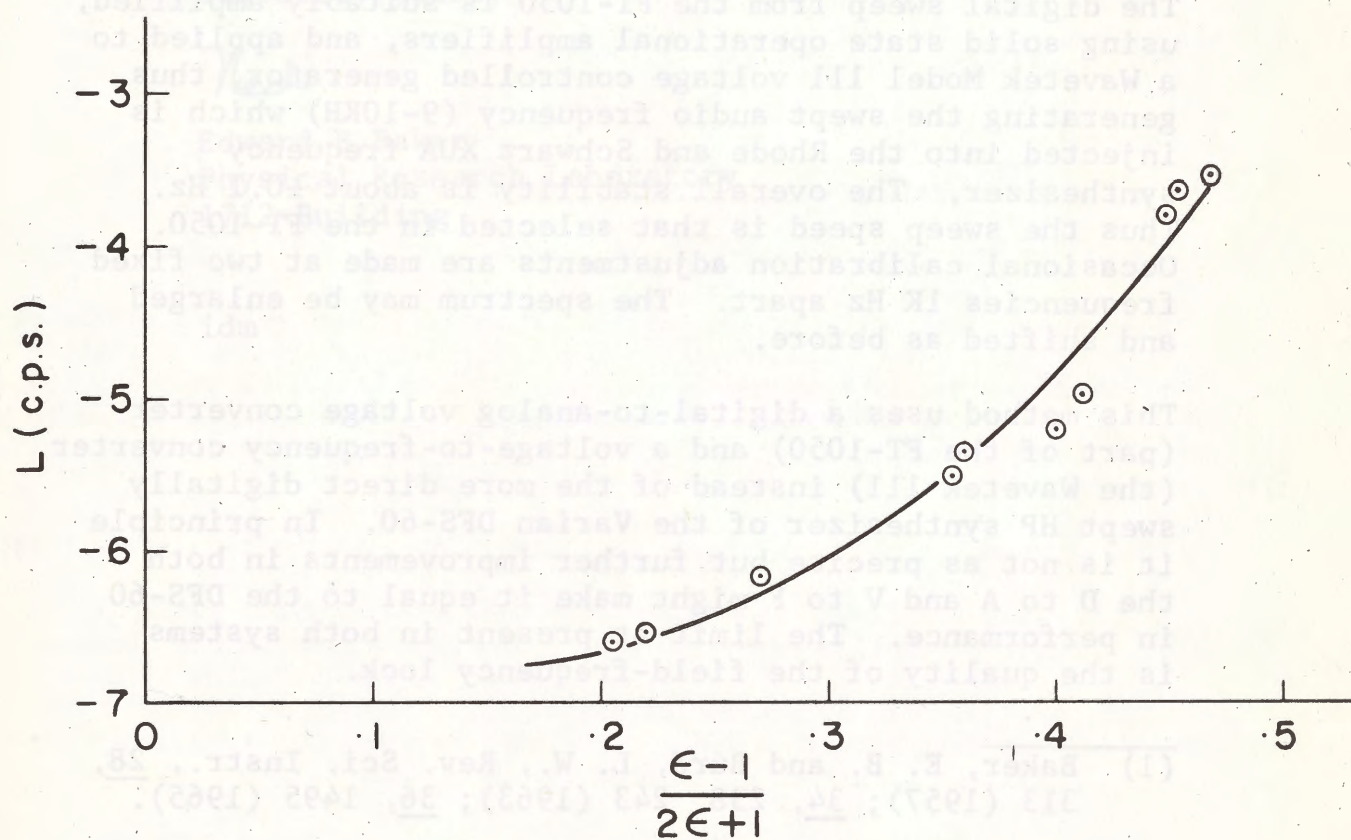
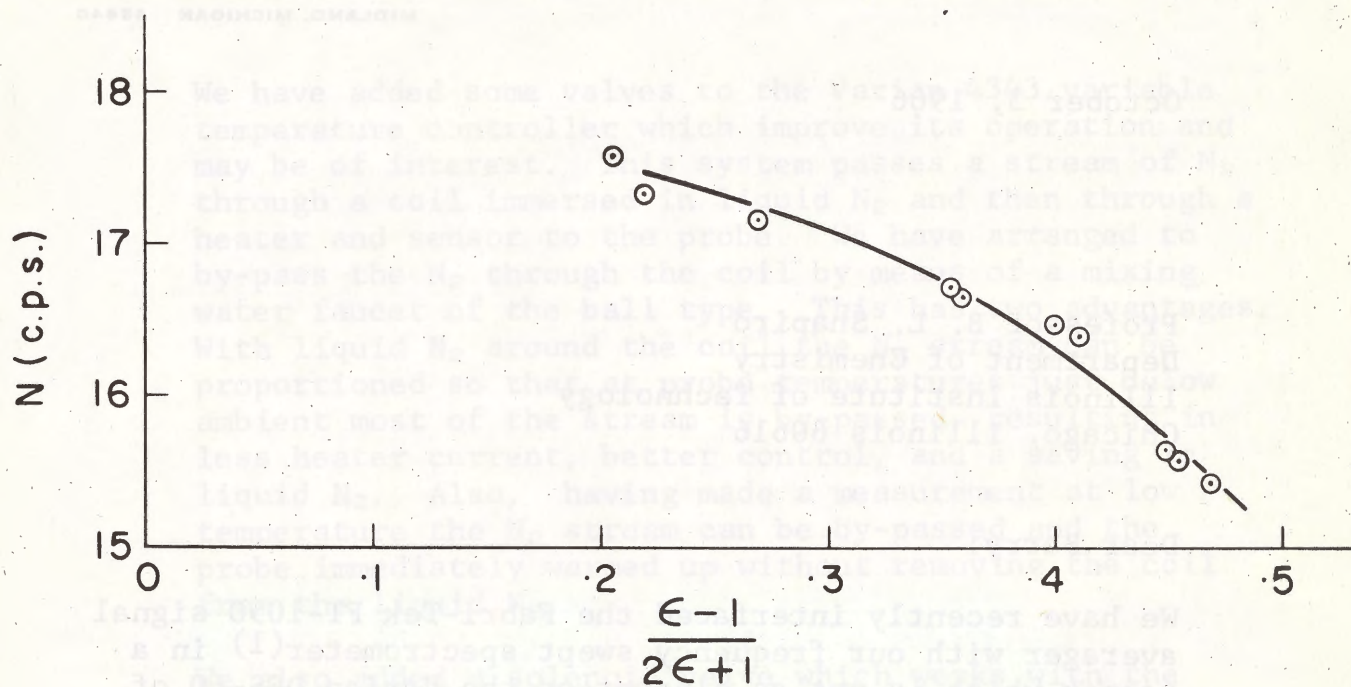
KGRP/EN



## National Chemical Research Laboratory



N- and L- values for 1,2-dichloro-ethane in various solvents



$N$ - and  $L$ - values for 1,2-dibromo-ethane in various solvents





THE DOW CHEMICAL COMPANY

MIDLAND, MICHIGAN 48640

October 3, 1966

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

Dear Barry:

We have recently interfaced the Fabri-Tek FT-1050 signal averager with our frequency swept spectrometer(1) in a manner which is not as elegant as the Varian DFS-60 of Professor Roberts, but is an acceptable and less expensive approach.

The digital sweep from the FT-1050 is suitably amplified, using solid state operational amplifiers, and applied to a Wavetek Model 111 voltage controlled generator, thus generating the swept audio frequency (9-10KH) which is injected into the Rhode and Schwarz XUA frequency synthesizer. The overall stability is about  $\pm 0.1$  Hz. Thus the sweep speed is that selected in the FT-1050. Occasional calibration adjustments are made at two fixed frequencies 1K Hz apart. The spectrum may be enlarged and shifted as before.

This method uses a digital-to-analog voltage converter (part of the FT-1050) and a voltage-to-frequency converter (the Wavetek 111) instead of the more direct digitally swept HP synthesizer of the Varian DFS-60. In principle it is not as precise but further improvements in both the D to A and V to F might make it equal to the DFS-60 in performance. The limit at present in both systems is the quality of the field-frequency lock.

(1) Baker, E. B. and Burd, L. W., Rev. Sci. Instr., 28, 313 (1957); 34, 238, 243 (1963); 36, 1495 (1965).

Professor B. L. Shapiro -2- October 3, 1966

We have added some valves to the Varian 4343 variable temperature controller which improve its operation and may be of interest. This system passes a stream of  $N_2$  through a coil immersed in liquid  $N_2$  and then through a heater and sensor to the probe. We have arranged to by-pass the  $N_2$  through the coil by means of a mixing water faucet of the ball type. This has two advantages. With liquid  $N_2$  around the coil the  $N_2$  stream can be proportioned so that at probe temperatures just below ambient most of the stream is by-passed, resulting in less heater current, better control, and a saving in liquid  $N_2$ . Also, having made a measurement at low temperature the  $N_2$  stream can be by-passed and the probe immediately warmed up without removing the coil from the liquid  $N_2$ .

We also added a solenoid valve which works with the V4343 power switch and shuts off both  $N_2$  streams. (A second stream is used in the probe body.)

Sincerely yours,

*Ned*

Edward B. Baker  
Physical Research Laboratory  
1712 Building

idm





# ESSO RESEARCH AND ENGINEERING COMPANY

BAYTOWN RESEARCH AND DEVELOPMENT DIVISION P. O. BOX 4255, BAYTOWN, TEXAS 77520

CHEMICALS DEPARTMENT  
A. T. WATSON, DEPT. HEAD

October 4, 1966

## Corrosion Inhibition in A-60 Magnet Cooling Systems

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

Dear Barry:

Some difficulty has been experienced by others in formulating the corrosion inhibitor we recommended for A-60 magnet cooling systems in IITNMRN 88-38. This inhibitor is working so well in our instrument it seems worthwhile to pass along the following additional information.

Mr. Roy V. Comeaux, our corrosion expert, says that the compounding of these inhibitor concentrates is tricky. Components must be dissolved in the right order and under controlled conditions as well as in the right concentrations. He suggests that the prepared liquid inhibitor concentrate be purchased from

Wright Chemical Corporation  
1319 Wabansia Avenue  
Chicago, Illinois 60622

Specify the blend containing 33% as  $\text{CrO}_4^{=}$ , 5% as  $\text{Zn}^{++}$ , and 5% as  $\text{PO}_4^{=}$ .

We have found that our A-60 magnet is now so susceptible to corrosion fouling that it will become inoperable in about four months, even on distilled water, unless this corrosion inhibitor is used. If the inhibitor is used without periodic replacement of the water, the magnet will foul with chromic oxide (from inhibitor decomposition) in about 6 months. It is necessary, therefore, to observe a reasonable schedule of water and inhibitor replacement.

The procedure that has proved to be best for our magnet is as follows:

1. Clean and flush the magnet coils with water and air until all major corrosion particles have been removed. It may be necessary to circulate a weak solution of ammonium phosphate for an hour or so, followed by a solution of trisodium phosphate, followed by distilled water. All  $\text{NH}_4^+$  ions must be removed because they will continue to attack the copper.

Flush each cooling coil separately to be sure it is open before beginning the circulation of cleaning solutions. Do not use the carbon vane pump supplied with the A-60 cooling system to circulate cleaning solution unless you have a replacement pump. The gritty corrosion products will probably ruin the carbon vanes.



Dr. Bernard L. Shapiro - 2

10-4-66

2. Drain the last flush water from the system and replace with distilled water containing 1 cc of the inhibitor concentrate to each 5 gallons. A sediment of corrosion products will probably continue to collect in the heat exchanger surge tank for several months as the inhibitor solution works its way underneath the remaining scale in the tubes. The tank should be cleaned periodically to prevent this sediment from being drawn into the pump.
3. Maintain a definite yellow color in the circulating water by adding fresh inhibited water as needed. This is best accomplished by withdrawing a gallon of used water and replacing it with fresh solution containing 0.2 to 1.0 cc of inhibitor.

If the circulated water becomes cloudy or greenish in color, replace all of it. This is an indication of excessive buildup of chromic oxide due to inadequate rate of replacement of water.

We have found the best schedule of water replacement for our magnet to be one gallon every two weeks, with the replacement solution containing 0.3 cc of inhibitor. Using this schedule we have had trouble-free operation for seven months, with no sign of fouling, very little sediment in the surge tank, and no pump trouble.

Hope this will work as well for others.

Sincerely,

*Nugen*

N. F. Chamberlain

NFC:osa





## SHELL DEVELOPMENT COMPANY

A DIVISION OF SHELL OIL COMPANY

1400 - 53rd STREET

EMERYVILLE, CALIFORNIA 94608

October 13, 1966

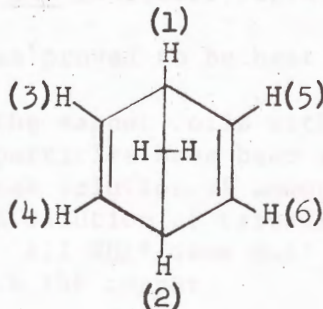
Dr. B. L. Shapiro  
 Department of Chemistry  
 Illinois Institute of Technology  
 Technology Center  
 Chicago, Illinois 60616

Dear Barry,

I delayed writing to you until the last minute in the hope that we would have a complete set of coupling constant values for bicycloheptadiene. But at the moment, the best I can do is to quote some tentative ones.

An NMR spectral analysis of the 40 MHz spectrum was made several years ago by Jack Mortimer [J. Molec. Spectro. 3, 528 (1959)]. As you know the methods for analyzing spectra were not as well developed then as they are now. Better spectra can also be obtained now with the more stable spectrometers available. A comparison of the spectrum observed at 100 MHz with that calculated with Mortimer's coupling constants showed significant discrepancies; a reinvestigation was indicated. Such a study has now been almost completed by R. Reijnhart, a student from the University of Delft in the Netherlands, who is spending the summer here with me.

First of all, in order to reduce the labor involved in the analysis, the protons in the methylene bridge were decoupled from the remaining ones. No change in the multiplet from the olefinic protons could be detected - showing that there is essentially zero coupling to these protons. An analysis of the spectrum from the 6 remaining protons, which form an AA'BB'B''B''' system of spins, is being made.



Dr. B. L. Shapiro

-2-

October 13, 1966

The magnitudes of several couplings were determined from the  $^{13}\text{C}$  satellites of the olefinic protons to be about

$$\begin{array}{ll} J_{15} = 2.7 \text{ Hz} & J_{25} = 0.9 \text{ Hz} \\ J_{56} = 5.2 \text{ Hz} & J_{35}, J_{45} \leq 0.5 \text{ Hz} \end{array}$$

It should be possible to determine values for the remaining coupling constants and all the signs by an analysis of the normal multiplets in the decoupled spectrum. The tentative values found by the not-yet-completed analysis are:

	<u>Mortimer</u>	<u>Present</u>
$J_{12}$	0 Hz	- 2.0 Hz
$J_{13} = J_{15} = J_{24} = J_{26}$	2.9	+ 3.0
$J_{14} = J_{16} = J_{23} = J_{25}$	0.95	+ 0.8
$J_{34} = J_{56}$	3.45	+ 5.2
$J_{35} = J_{46}$	0	$\leq 0.5$
$J_{36} = J_{45}$	0	$\leq 0.1$

I shall write you another letter containing the final results as soon as I can.

Sincerely yours,

*Charlie*

C. A. Reilly

CAR: jel

Suggested short title:

Bicycloheptadiene - NMR Spectral Analysis



## MELLON INSTITUTE

4400 FIFTH AVENUE

PITTSBURGH, PA. 15213

11 October 1966

Long-range  $J_{C-C-H}$  in Aldehydes - H. Dreeskamp

Dear Barry:

In confirming our earlier work (1) on long-range  $C^{13}-H^1$  coupling constants, I have investigated in more detail the 2-bond  $J_{C^{13}-C-H}$  of aldehyde groups. Since these are generally large they are readily measurable and signs might be determined by H- $\{H\}$  experiments relative to the sign of  $J_{C-H}$  in many cases. In (1) it was shown - at least for ethane-like structures - that  $J_{C-C-H}$  may be interpreted as a product of the s-character of the coupling  $C^{13}$  in the C-C-bond directed toward the coupling H and a term being proportional to the hybridization of the "transmitting" carbon. Replacing in acetaldehyde successively hydrogens by chlorine-atoms produces indeed an increase in  $J_{C-C-H}$  (1)(2) (see Table I) in quantitative agreement with the expectations from an increased s-character of the coupling C-atom. This may be estimated à la Frei and Bernstein (3) from the  $J_{C-H}$  in chloromethanes:

Table I:

$J_{C-C-H}$ :			
$J_{C-H}$ :	$(CH_4) = 125$	$(CH_2Cl_2) = 178$	$(CHCl_3) = 209$

Replacing in acetaldehyde the methyl-group by:  $CH_2CH_2-$ ,  $C_6H_5-$ ,  $CH_2CH=CH-$  and  $CH_3C\equiv C-$  groups produces an increase in  $J_{C-C-H}$  which is, however, much smaller than the one expected:

Table II:

$J_{C-C-H}$ :				
$J_{C-H}$ :	$(C_2H_6) = 125$ ;	$(C_6H_6) = 159$ ;	$(C_2H_4) = 157$ ;	$(CH_3CCH) = 248$

From these experimental data it appears that the partial double bond character of the "transmitting" C-C bond gives a negative contribution to  $J_{C-C-H}$ . The MO-theory (4) interprets the large coupling constant  $J_{C-C-H}$  in acetaldehyde by the super-position of two effects: ( $\alpha$ ) an inductive withdrawal of  $\sigma$ -electrons and ( $\beta$ ) a hyperconjugative backdonation of  $\pi$ -electrons in the H-C-C plane due to the unshared electrons centered at the oxygen.

Starting from there several reasons might be given for the "unexpectedly low" values in Table II:

(i) If the inductive effect overrides the hyperconjugative effect polar structures  $\text{>C}^+ - \text{C} = \text{C} - \text{O}^-$  would tend to decrease  $J_{C-C-H}$ .

MELLON INSTITUTE

page 2 - Long-range  $J_{C-H}$  in Aldehydes - H. Dreeskamp

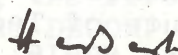
(ii) A shortening of the C-C bond due to its double bond character would increase the interaction of "other bonds" (5) and lead to a decrease of  $J_{C-H}$ .

(iii) The presence of  $\pi$ -electrons in the C-C bond indirectly changes the effective potential for the  $\sigma$ -electrons and hence the MO coefficients in a way to decrease  $J_{C-H}$ .

It will be interesting to see as more data become available whether these observations can be generalized.

May I add that I enjoy the hospitality here at Mellon Institute and gratefully acknowledge useful discussions with Dr. Bothner-By on this letter. I expect to be back in Stuttgart in December.

Sincerely yours,




---

H. Dreeskamp

- (1) E. Sackmann and H. Dreeskamp, Spectrochim. Acta., 21, 2005 (1965).
- (2) N. Muller J. Chem. Phys., 36, 359 (1962).
- (3) K. Frei and H. J. Bernstein, J. Chem. Phys., 38, 1216 (1963).
- (4) J. A. Pople and A. A. Bothner-By, J. Chem. Phys., 42, 1339 (1965).
- (5) J. A. Pople and D. P. Santry, Mol. Physics, 9, 311 (1965).





Corporation

## INDUSTRIAL CHEMICALS DIVISION

405

MORRIS TOWNSHIP CENTER • P.O. BOX ~~77~~ MORRISTOWN, NEW JERSEY 07960  
TEL. (201) 538-8000

October 11, 1966

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

Dear Professor Shapiro:

In my last letter (IITNMR 88 50) I mentioned that I hoped to have an improved version of the computer program ASSIGN completed by this time. The new program is nearly complete but has not been exhaustively checked out, therefore I would like to renew my subscription with a description of another program on which I am working concurrently.

The series of computer programs, DECOMP, ASSIGN, NMREN, and NMRI<sup>1,2</sup> make it possible to proceed directly from a complex spectrum of overlapping peaks to chemical shifts and coupling constants without the necessity of having prior knowledge of the system or making any assumptions about the NMR parameters. To recapitulate; overlapping peaks in a digitized spectrum are resolved with the program DECOMP. Using these resolved lines, ASSIGN, finds all sets of energy levels and transition assignments consistent with frequency and intensity sum rules. These assignments can then be fit to the data by least squares using Swalen and Reillys NMREN to provide more representative sets of energy levels. These sets of energy levels, along with arbitrary starting parameters, are then used directly in the iterative stage of NMRI (N) to yield the final sets of parameters corresponding to the sets of energy levels.



Prof. B. L. Shapiro  
Department of Chemistry

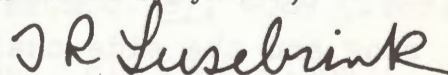
- 2 -

October 11, 1966

Thus, by combining all the above programs together into a single program, it should be possible to obtain chemical shifts and coupling constants directly from a spectrometer coupled to a computer. Alternately, digital spectra can be obtained from the spectrometer and this data used as input to the computer. I have started writing such a program and currently have progressed to the stage where a digital spectrum can be automatically decomposed into its constituent peaks. The only input required is the digitized spectrum plus a few control cards. The program scales the data, identifies all separate groups of peaks which return to within a certain distance of the base line, and finds all shoulders and maxima within each separate group of peaks. The positions and intensities of these shoulders and maxima are then used as the basis for making the first estimates of peak parameters for the subroutine DECOMP. Each group of peaks is decomposed separately; if the fit is not satisfactory, DECOMP adds an additional peak where the fit is poorest and continues in this manner until the fit to the data is satisfactory. The values for the line positions and intensities are stored and DECOMP proceeds to the next group, continuing in this manner until the entire spectrum is decomposed.

The next step will be to normalize the intensities so the line intensities and positions can be used as input for ASSIGN. The sets of energy levels from ASSIGN will then be used in NMRIT(N) with arbitrary starting parameters, as discussed above, to find the chemical shifts and coupling constants. Thus, a complete analysis can be carried out in a single step, yielding chemical shifts and coupling constants as "direct" output from the spectrometer. Of course the limitations of such a program will not be known until it has been tested with actual spectra, however, it is anticipated that it will not require excessive amounts of computer time for 3,4, and perhaps some 5 spin systems. More complex systems will probably require additional information, such as that obtained from double resonance experiments, to reduce the computer time required. I hope to be able to report the availability of these programs before my subscription runs out next time.

Sincerely yours,



T. R. Lusebrink  
Senior Research Chemist

TRL/pl

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ORGANISCH-CHEMISCHES INSTITUT  
DER UNIVERSITÄT

Albrecht Mannschreck

69 HEIDELBERG, October 10, 1966

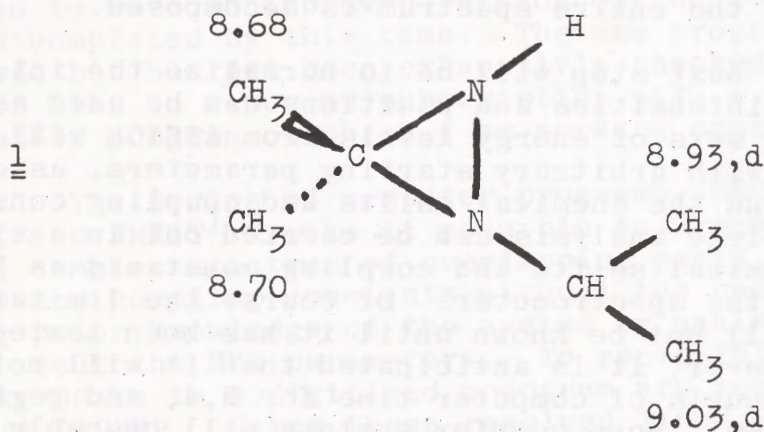
Tiergartenstraße  
Tel. 27121 (über Chirurg. Klinik)

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

A Case of Very Slow Nitrogen Inversion

Dear Dr.Shapiro:

I am stimulated by the current boom in all kinds of "inversions" to report on diaziridines. Pure liquid 1 shows at 135°C the  $\tau$ -values



given with the formula. They do not change much when spectra are taken at lower temperature or in a number of solvents. At higher temperature 1 begins to decompose. The non-equivalence of both the ring and the side chain methyl groups requires the inversion to be slow at one nitrogen atom at least. From the shift difference of 1.2 cps between the ring methyl signals at 135°C one calculates a lower limit of 23 kcal/mole for the inversion barrier  $\Delta F^\ddagger$ . The investigation of six other diaziridines supports this view. Results

obtained in collaboration with scientists of the Deutsche Akademie der Wissenschaften zu Berlin have been sent to the Chemische Berichte<sup>1)</sup>.

Inversion rates are decreased by at least two factors: 1) inclusion of nitrogen into a four- or, still better, into a three-membered ring; 2) attachment of O (or N) to the inverting nitrogen<sup>2)</sup>. Both factors are cooperating in 1,2-oxazetidines<sup>3)</sup>, 1,2-diazetidines<sup>4)</sup>, oxaziridines<sup>5)</sup>, and diaziridines which seem to show the slowest nitrogen inversion hitherto reported. The magnitude of the barriers suggests that suitably substituted diaziridines might be separated into inversion isomers.

Yours sincerely,

*Albrecht Mannschreck*  
Albrecht Mannschreck

- 
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## MELLON INSTITUTE

4400 FIFTH AVENUE

PITTSBURGH, PA. 15213

14 October 1966

Rotational Isomerism in  $\text{CH}_2\text{BrCH}_2\text{CN}$ ;  $J_{\text{meta}}^{\text{FF}}$  in FluorobenzenesRaymond J. Abraham

Dear Barry:

My apologies for once again needing a reminder.

Research at Liverpool has been on two main lines. Dr. Deb has been extending our studies of medium effects on rotational isomerism noted in our last Mellon letter, and has measured the coupling constants in  $\text{CH}_2\text{BrCH}_2\text{CN}$  in various solvents. Our results are summarized in Table I. The remarkably large concentration dependence of the J values in non-polar media is noteworthy. We ascribe it to a monomer<sup>5</sup>dimer equilibrium in dilute solutions, though no doubt larger aggregates could be present in concentrated solutions. In the dimer, the "effective" dielectric constant of the medium is now not the solvent dielectric constant but a much higher value and this alters the proportions of the rotational isomers. To interpret the results fully it is necessary to calculate the quadrupole moments of the isomers, which on our model depends on where the CN dipoles are placed. This we are still considering.

Also, we have been studying the large variations of the meta F-F coupling in fluorobenzenes (-10 to +10 c/sec.).

I won't go into detail as we have already published a short note on this, but the substituent contributions shown in Table II together with the calculated value of  $J_{\text{meta}}^{\text{FF}}$  in meta-fluorobenzene of +6.3 c/sec. gives calculated values in very good agreement with most of the observed couplings. Some interesting values are given below.

Compound	Coupling	Calculated value	Observed value
1,3,5-trifluorobenzene	$J_{13}$	+5.8	+5.6
1,2,3,5-tetrafluorobenzene	$J_{13}$	+5.7	+5.3
hexafluorobenzene	$J_{15}$	+1.7	+1.8
meta-fluorobenzene	$J_{13}$	-2.3	-4 (+ 2?)
pentafluoroaniline	$J_{26}$	+4.3	4.6
	$J_{35}$	-2.5	2.6
	$J_{24}$	-6.3	6.9
pentafluorochlorobenzene	$J_{26}$	-5.3	5.4
	$J_{35}$	-2.4	2.6
	$J_{24}$	+1.2	1.0

In the pentafluoro compounds the AA'XX'R analysis does not give any of the signs nor does it assign  $J_{26}$  and  $J_{35}$  uniquely so we would be very pleased if someone could check our predictions experimentally (provided they agreed with us!)

TABLE I

Spectral parameters of  $\beta$ -bromo propionitrile in different solvents at different dilutions

Solvents	Solvent dielectric constant at 30°C	Concentration in % by volume	N	L	-K	M	$\delta$ (60Mc/s)
Acetonitrile	35.90	10	12.66	2.30	27.92	7.1	33.48
Dimethyl formamide	35.87	10	12.67	2.35	28.0	"	30.28
Acetone	20.21	10	12.69	2.14	27.84	"	32.18
Methyl chloroacetate	12.66	10	12.04	2.00	28.20	"	32.28
1,2-Dichloroethane	10.03	10	13.06	1.60	27.67	"	33.24
		5	13.10	1.58			
		2	13.14	1.57			
		0.5	13.16	1.55			
CH <sub>2</sub> Cl <sub>2</sub>	8.62	10	13.14	1.60	27.90	"	33.26
		5	13.21	1.48			
		2	13.26	1.41			
		0.5	13.32	1.32			
CHCl <sub>3</sub>	4.63	15	13.34	1.28	28.01	"	32.82
		10	13.43	1.20			
		5	13.54	1.00			
		2.5	13.64	0.86			
		1	13.73	0.72			
		0.5	13.80	0.47			
C <sub>2</sub> HCl <sub>3</sub>	3.38	15	13.25	1.44	27.54		33.50
		10	13.50	1.05			33.84
		5	13.67	0.81			34.30
		2	13.83	0.58			34.72
		1	13.93	0.43			
		0.5	14.01	0.31			
CCl <sub>4</sub>	2.22	20	13.21	1.47	27.86	"	33.50
		15	13.22	1.40			33.10
		10	13.23	1.30			33.72
		5	13.66	0.82			34.48
		2.5	14.07	0.22			35.44
		1	14.33	-0.16			35.68
		0.5	14.44	-0.32			



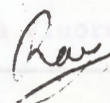
## MELLON INSTITUTE

R. J. Abraham

page 2.

As you can see from the above address, I am now starting my year in Mellon Institute, so could you credit this to Tony Thomas at Liverpool?

With best wishes,



Ray Abraham

Table II

Substituent	Contribution to $J_{\text{meta}}^{\text{FF}}$ (c.p.s.)		
	ortho-ortho	ortho-para	meta-meta
H	0.0	0.0	0.0
F	-0.3	-3.8	-0.7
NH <sub>2</sub>	+6.3	-8.1	-0.8
Cl <sup>2</sup>	-3.3	-0.3	+0.1
NO <sub>2</sub>	-7.7	+6.1	+1.9
CN <sup>2</sup>	-6.1	+4.4	-
I	-3.6	+0.6	+0.6

## MELLON INSTITUTE

4400 FIFTH AVENUE

PITTSBURGH, PA. 15213

7 October 1966

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

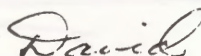
Dear Barry:

I am sorry that I must be twice reminded to pay my subscription dues to your highly useful newsletter. If I had known these extra reminders were causing irreparable damage to your golf score the first reminder would have received greater attention.

In the process of studying rotational isomerism in 1,1-dichloro-3-fluoro-1,3-butadiene ( $\text{CH}_2=\text{CF}-\text{CH}-\text{CCl}_2$ ) we also looked at the relative signs of the HH coupling  $J$  constants. Our "spin tickling" equipment was not working as expected so 2nd order effects were introduced into the spectrum by recording it at 15 MHz on our HR-60 with an f.f. unit loaned to us by Dr. Kurland at Carnegie Tech. At 15 MHz the proton spectrum is still essentially first order but four weak lines sensitive to the relative signs of the HH couplings are predicted in the fluorine spectrum. The four weak lines were easily found by increasingly saturating the strong 1st order lines (Figure 1). Two possible sign combinations are found to fit the four lines,  $J_{\text{gem}}(+)$  or  $(-)$ ,  ${}^4J_{\text{c}}(-)$  and  ${}^4J_{\text{t}}(+)$ . There is little doubt that  $J_{\text{gem}}$  is  $(-)$  since electron withdrawing substituents tend to make this  $J_{\text{gem}}$  coupling constant more negative and its magnitude (3.18 Hz) is greater than that in butadiene (1.74 Hz)<sup>1</sup>. A value of -3.18 Hz for  $J_{\text{gem}}$  also compares favorably with the -3.2 Hz value for this coupling in  $J_{\text{gem}}$  monofluoroethylene<sup>2</sup>. The great similarity of the coupling constants in the analogous 1,1,-dibromo-3-fluoro-1,3-butadiene lead us to believe that it has the same relative signs.

Table I lists a number of 2 (or 3) substituted butadienes for which the relative signs of  ${}^4J_{\text{t}}$  and  ${}^4J_{\text{c}}$  are known. When there is an interior substituent in the butadiene system it appears that a general rule regarding  ${}^4J_{\text{t}}$  and  ${}^4J_{\text{c}}$  can be formulated, that is,  ${}^4J_{\text{t}}$  will be more positive than  ${}^4J_{\text{c}}$ .

Sincerely,



David Koster

DK:hb

Title: Relative Signs of Coupling Constants in Butadienes.



TABLE I

Compound	$^4J_t$	$^4J_c$	Reference
	+0.095	-0.19	3
	-0.18	-0.60	3
	-0.32	-0.76	4
	-0.575	-1.12	4
	-0.034	-1.83	4
	-0.60	-2.20	4
	$\approx 0.6^{(a)}$	$\approx 0.0$	5
	+0.64	-0.31	6
	+0.54 <sup>(b)</sup>	-0.31 <sup>(b)</sup>	6

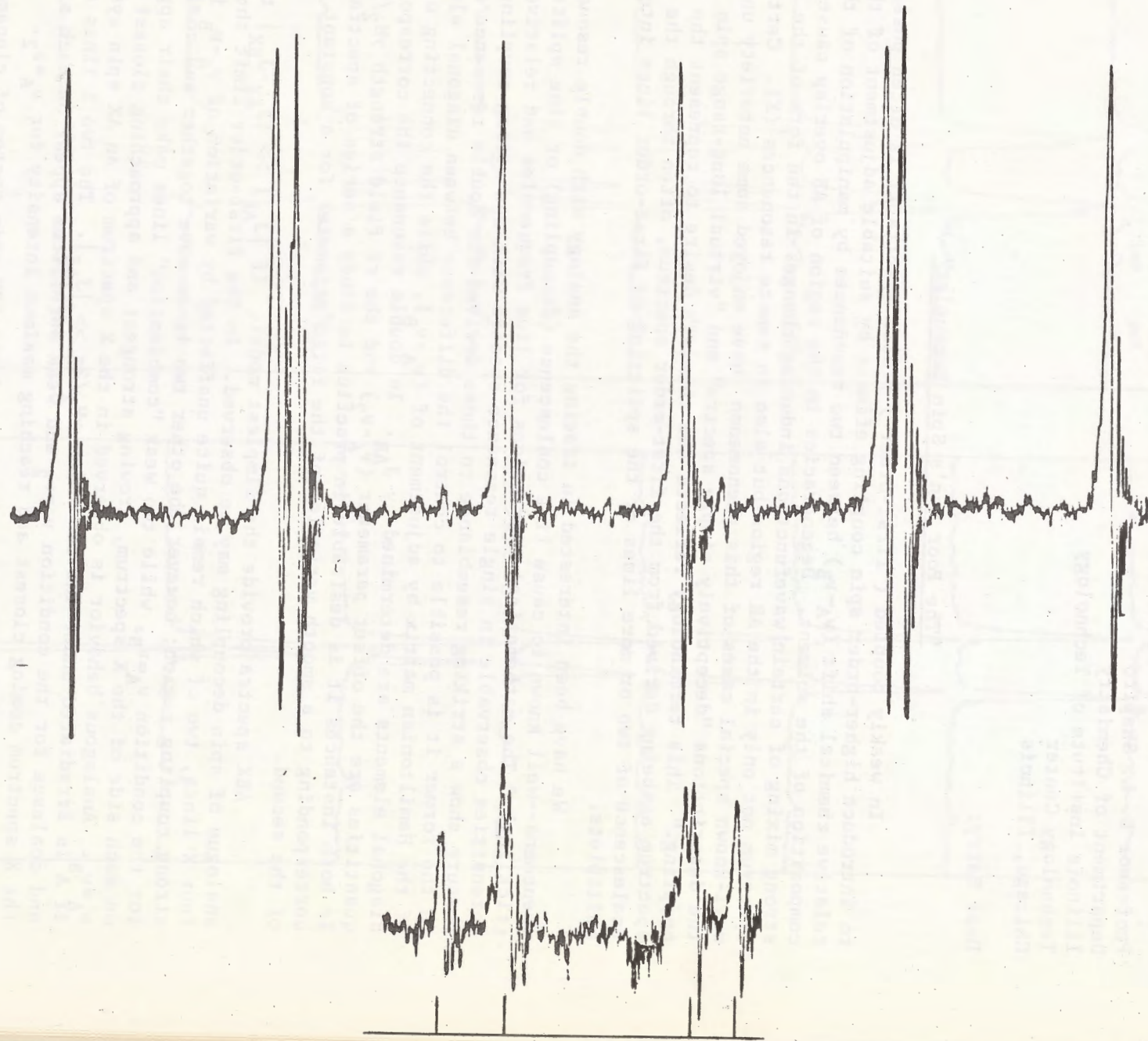
(a) Sign not yet known

(b) Assumed by analogy with dichloro compound.

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# 15 MHz FLUORINE SPECTRUM OF 1,1-DICHLORO-3-FLUOROBUTADIENE

Figure 1.







10 October 1966

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

Dear Barry:

"The Poor Man's Spin Decoupler"

In weakly coupled ("first-order") proton spectra it is often possible to introduce higher-order spin coupling effects by suitable adjustment of the relative chemical shift ( $\nu_A - \nu_B$ ) between two resonances by manipulation of the composition of the solvent. Degeneracies in the region of AB overlap cause strong mixing of certain wavefunctions inducing changes in the form of the spectrum not only in the AB region but also in remote resonances (X). Certain well-known special cases of this phenomenon have enjoyed some notoriety under the descriptions "deceptively simple spectra" and "virtual long-range spin coupling." This terminology reflects the chemist's desire to represent the actual spectrum as being derived from the first-order spectrum, often through the coalescence of two or more lines or the splitting of first-order lines into multiplets.

We have been interested in tracing the analogy with double resonance phenomena--well known to cause line coalescence (decoupling) or line splitting (tickling). The mathematical expressions for line frequencies and relative intensities observable in single resonance spectra with one strong coupling feature show a striking resemblance to those derived for double resonance spectra. In the former it is possible to control the difference between diagonal elements of the Hamiltonian matrix by adjustment of ( $\nu_A - \nu_B$ ), while the connecting off-diagonal elements are determined by  $J_{AB}$ . In double resonance the corresponding quantities are the offset parameter ( $\nu_A - \nu_2$ ) and the rf field strength  $\gamma H_2 / 2\pi$ . In both instances it is desirable in practice to study a series of spectra corresponding to a smooth variation of the first parameter for a constant value of the second.

ABX spectra provide the simplest model. If  $|J_{AB}| \gg |J_{AX} - J_{BX}|$  the analogue of spin decoupling may be observed. In the first-order limit there are four X lines, two of which remain quite unaffected by variation of  $\nu_A - \nu_B$  into the strong coupling region; however the other two lines move together and coalesce for the condition  $\nu_A = \nu_B$ , while two weak "combination" lines make their appearance on each side of the X spectrum, growing strongest and approaching closest for  $\nu_A = \nu_B$ . Analogous behavior is observed in the X spectrum of an AX spin system if A is irradiated under the condition  $\gamma H_2 / 2\pi \gg |J_{AX}|$ . The two X lines approach and coalesce for the condition  $\nu_A = \nu_2$  and weak satellites appear on each side of the X spectrum coming closest and reaching maximum intensity for  $\nu_A = \nu_2$ .

Proton spectra of the X region of the ABX spin system of cinnamalazine in deuteriochloroform/deutero benzene solutions provide a good example of coalescence induced by preferential solvent effects (Fig. 1). This may be compared with the X spectra of an AX spin system (maltol) recorded while the A resonance is irradiated at several different settings of the offset parameter ( $\nu_A - \nu_2$ ) (Fig. 2).



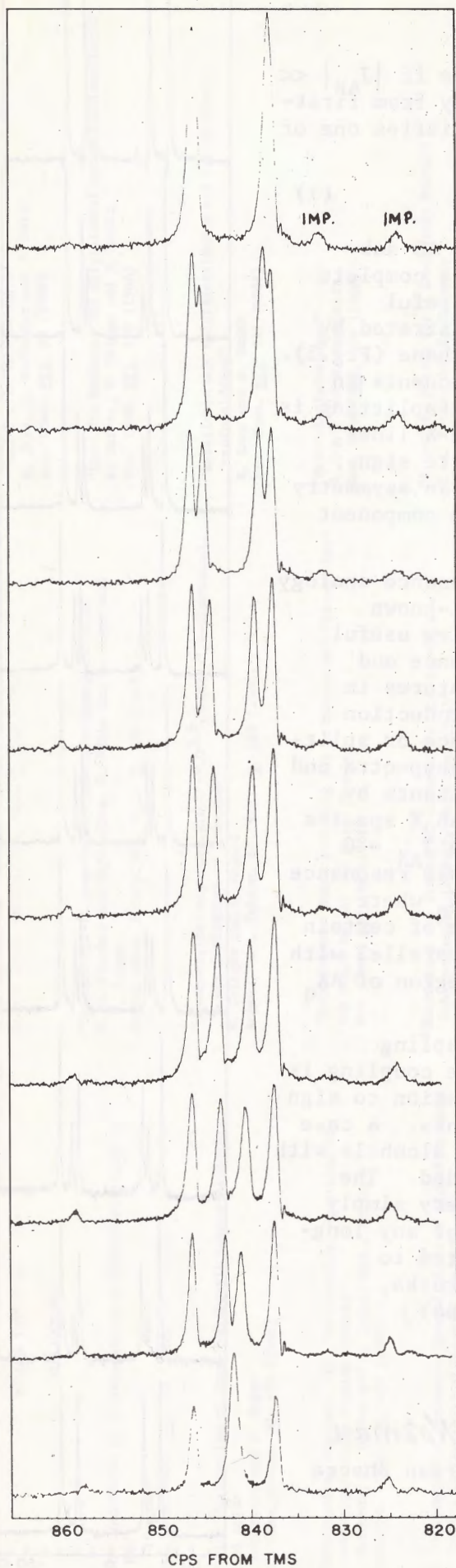


Figure 1

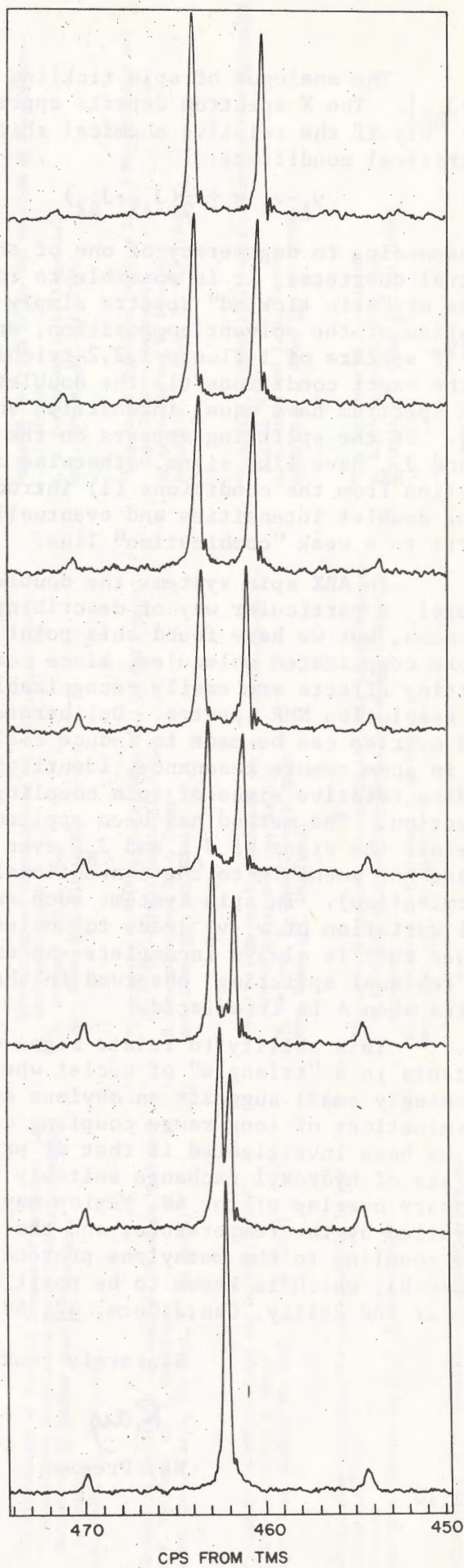


Figure 2



The analogue of spin tickling occurs if  $|J_{AB}| \ll |J_{AX} - J_{BX}|$ . The X spectrum departs appreciably from first-order only if the relative chemical shift satisfies one of two critical conditions:

$$\nu_A - \nu_B = \pm \frac{1}{2}(J_{AX} - J_{BX}) \quad (1)$$

corresponding to degeneracy of one of the two AB sub-spectral quartets. It is possible to follow a complete series of "spin tickled" spectra simply by careful variation of the solvent composition, as illustrated by the  $^{19}\text{F}$  spectra of 1-fluoro-1,2,2-trichloroethane (Fig.3). For the exact conditions (1) the doublet components in the X spectrum have equal intensities and the splitting is  $|J_{AB}|$ . If the splitting appears on the inner X lines,  $J_{AX}$  and  $J_{BX}$  have like signs, otherwise opposite signs. Deviation from the conditions (1) introduces an asymmetry in the doublet intensities and eventually one component reverts to a weak "combination" line.

In ABX spin systems the double resonance analogy is merely a particular way of describing well-known phenomena, but we have found this point of view useful in more complicated molecules, since coalescence and splitting effects are easily recognizable features in high resolution NMR spectra. Deliberate introduction of AB overlap can be made to induce coalescence or splitting in some remote resonance, identifying subspectra and yielding relative signs of spin coupling constants by inspection. The method has been applied to  $\text{AB}_2\text{X}$  spectra to relate the signs of  $J_{AB}$  and  $J_{BX}$  even where  $J_{AX} = 0$  (a case not amenable to the conventional double resonance determination). In spin systems such as  $\text{AB}_2\text{X}_q$  where  $q > 1$  variation of  $\nu_A - \nu_B$  leads to coalescence of certain X lines that is always incomplete--an exact parallel with the "residual splitting" observed in the X region of  $\text{AX}_q$  spectra when A is irradiated.

This ability to relate signs of coupling constants in a "triangle" of nuclei where one coupling is vanishingly small suggests an obvious application to sign determinations of long-range coupling constants. A case that we have investigated is that of primary alcohols with the rate of hydroxyl exchange suitably retarded. The necessary overlap of the  $\text{AB}_2$  region may be very simply controlled by the temperature, and the sign of any long-range coupling to the methylene protons related to  $J(\text{H-C-O-H})$ , which is known to be positive [Hruska, Schaefer and Reilly, Can.J.Chem. 42, 697 (1964)].

Sincerely yours,

Ray

Ray Freeman

Norman

Norman Bhacca

RF/NB:iw

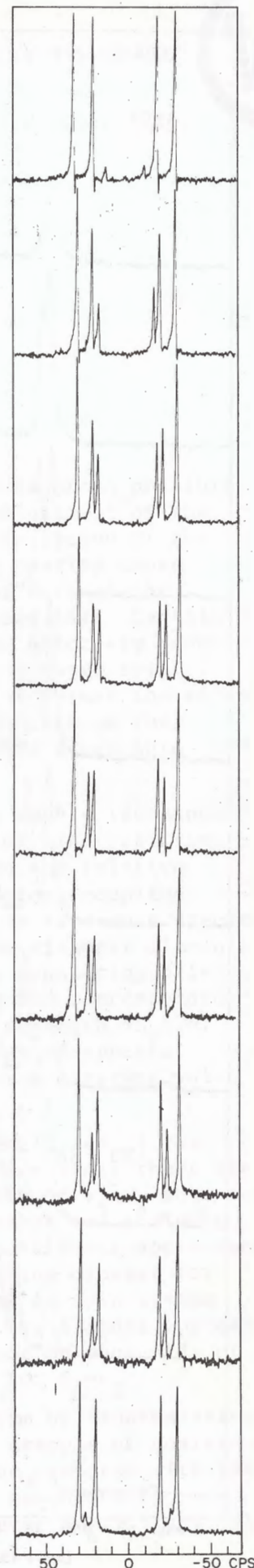


Figure 3



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