23.12.66

Preiss

Illinois Institute of Technology N-M-R Newsletter

No. 98
NOVEMBER, 1966

OVER

Deadline Dates: No. 99 - 15 December 1966
No.100 - 12 January 1967

NOTE

TELEPHONE : MW 0522.

Prof. B.L.Shapiro
Dept. of Chemistry
Illinois Institute of
Technology.

15.10.1966

The University of Sydney
DEPARTMENT OF ORGANIC CHEMISTRY
SYDNEY, N.S.W.

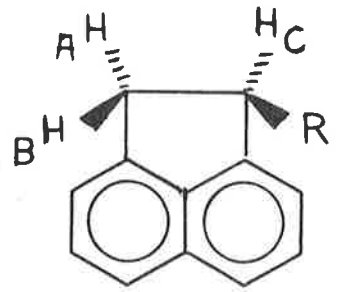
IN REPLY PLEASE QUOTE :

1-SUBSTITUTED ACENAPHTHENES

Dear Barry,

We have carried out some systematic studies on 1-substituted acenaphthenes. The chemical shifts are for approximately 2% solutions in CCl_4 , the coupling constants (ABX approximation) for up to 10% solutions in $CDCl_3$. The data show much poorer correlation with the Dailey electronegativity than those reported by Williamson, although the trends remain the same. There is the usual differentiation between cis and trans vicinal coupling constants and the tendency for eclipsed protons to appear upfield . The latter correlation should be used with caution because of the obvious difficulties associated with the presence of groups which are not cyclindrically symmetrical about the bond joining them to the ring system (cf. e.g. the carboxyl group in this set).

R	δA	δB	δC	J_{AB}	J_{AC}	J_{BC}
-OH	3.70	3.14	5.60	17.8	7.2	2.6
-Cl	3.90	3.63	5.73	18.2	7.4	2.5
-Br	3.93	3.80	5.81	18.8	8.5	1.0
$-NH_2$	3.68	2.92	4.80	17.5	7.6	3.3
-NHAc	3.88	3.05	5.86	18.5	8.0	2.0
-Ph	3.90	3.40	4.85	17.4	8.5	4.1
-CN	3.82	3.72	4.55	17.1	7.5	6.4
-COOH	3.55	3.82	4.55	17.5	9.7	3.3
-OAc	3.72	3.26	6.55	18.0	7.2	2.5



When R = H
$\delta_{A,B,C}$ = 3.36 ppm

We are now looking more closely at these and other data and also at more heavily substituted acenaphthenes.

With best regards, yours sincerely

C.K.Fay
Chay

S.Sternhell
Sev Sternhell

P.W.Westerman
P W Westerman

THE UNIVERSITY OF IOWA
IOWA CITY, IOWA 52240

Department of Chemistry

October 18, 1966

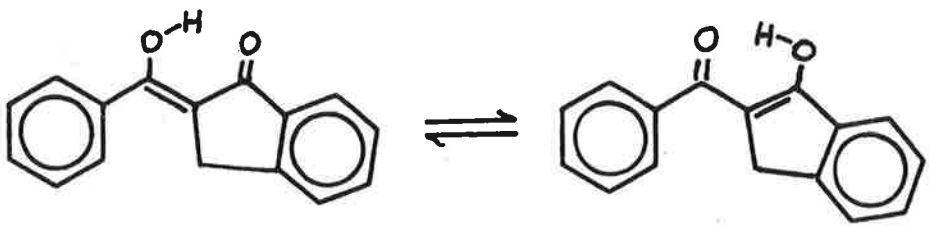
Prof. B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry:

I hope this letter will keep me in good standing as a recipient of your 11TNMR Newsletter. A short title is "Two-site Exchange of a Chelated Proton".

In our studies of unsymmetric enols in which intramolecular hydrogen bonding occurs, it was found that the chelated proton signal, ~ 16 ppm downfield from TMS, was broadened by structural changes that cause an increase in the distance between the two chelating oxygen atoms (11TNMR Newsletter No. 87).

Our recent studies include low-temperature study of 2-benzoylindanone in methylene chloride:

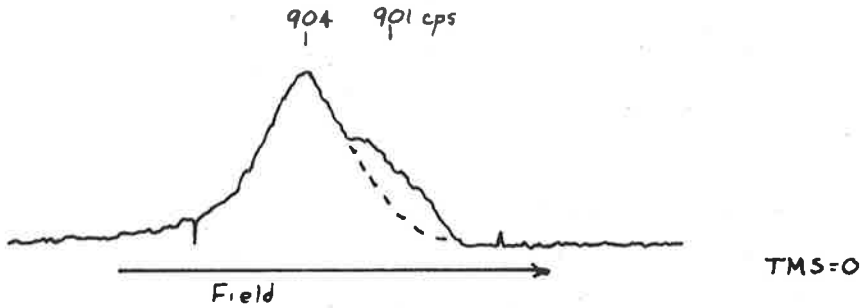


The signal for the chelated enolic proton appears at 904 cps downfield from TMS, in CH_2Cl_2, at room temperature. The signal is broad: width at half-height ($\Delta_{\frac{1}{2}}$) is 17 cps. This value is obtained by doing repeated scans (20-25) on our A-60 with the CAT attached. Earlier data (1) showed a narrower line on a single scan that was of low intensity and difficult to read. The $\Delta_{\frac{1}{2}}$ values for the benzoylindanone enol proton at several temperatures were determined by this method (Table I).

Table I. Enol Proton Signal of Benzoylindanone in CH_2Cl_2

Half Width, $\Delta_{\frac{1}{2}}$	Temperature
17 cps	27°
18 cps	-16
12	-23
6	-37
5.4 cps	-42
4 cps	-56
4 cps	-61

All of these lines have the Gaussian shape, except that at -55° to -60°, the curves show a second signal of weaker intensity. Because we had experienced difficulty with signal drift and trigger jumping during CAT scanning, we repeated the experiment three times over a period of several months, on different samples. In each case, the curve showed two overlapping signals (Fig. 1).



A simple graphical resolution shows that the intensity is 86% in the downfield signal (904 cps) and 14% in the upfield signal (901 cps). The downfield signal shows $\Delta_{\frac{1}{2}}$ = 3.5 cps; the upfield signal (approximate) $\Delta_{\frac{1}{2}}$ = 2 cps. The signal intensity ratio is interpreted to mean that the two species indicated by the structures above are in equilibrium in a ratio of 6:1. The more stable species must be the one with a stronger formal O-H sigma bond. Greater shielding by the sigma orbital should place this proton in resonance upfield from the less stable species, so it is clear that anisotropic deshielding is significantly greater in the more stable species than in the less stable species.

We are now examining this system for a basis for assigning natural line widths so that we can apply the Bloch equation to calculation of proton tunnelling rates. We are also seeking other model compounds for these studies.

Best regards,

Dick

Richard D. Campbell

UNIVERSITY COLLEGE OF SWANSEA.
(UNIVERSITY OF WALES)
SINGLETON PARK, SWANSEA.
Telephone : SWANSEA 25678.

DEPARTMENT OF CHEMISTRY.

PROFESSOR C. H. HASSALL, Ph.D., F.R.I.C.

17th October, 1966

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

Dear Dr. Shapiro,

Asymmetry of R Absorption in AB_2R_p Systems, and the Signs of Some Long-Range Phosphorus Couplings

This contribution is a rider to I.I.T.N.M.R. 88-34 and ref. 1. It was correctly stated[2] some years ago that the R absorption of AB_2R_p systems was in general not symmetric about ν_R. Unfortunately the literature[4] also contains the opposite statement, which was reproduced in a recent text[5]. The purpose of this note is to emphasise, analyse and illustrate this asymmetry.

Let $L \equiv J_{AR} - J_{BR}$, $\delta \equiv \nu_A - \nu_B$. In most of the compounds considered[1], $|L| << |\delta|$ and $|L| << |J_{AB}|$. Accordingly we expand in powers of L, and the energies of the principal transitions are, correct to second order:

No.	Energy
1	$\frac{1}{2}J + J'$
2	$\frac{1}{2}J$
3	$-\frac{1}{2}J$
4	$-\frac{1}{2}J - J'$
5(x)	$+\frac{1}{2}r_+J + \frac{1}{2}(1 - r_+)J' + \frac{1}{2}(x + \frac{1}{2})\, L^2\, (1 - r_+^2)/D_+$
6(x)	$-\frac{1}{2}r_+J + \frac{1}{2}(1 + r_+)J' - \frac{1}{2}(x + \frac{1}{2})\, L^2\, (1 - r_+^2)/D_+$
7(x)	$+\frac{1}{2}r_-J - \frac{1}{2}(1 + r_-)J' + \frac{1}{2}(x + \frac{1}{2})\, L^2\, (1 - r_-^2)/D_-$
8(x)	$-\frac{1}{2}r_-J - \frac{1}{2}(1 - r_-)J' - \frac{1}{2}(x + \frac{1}{2})\, L^2\, (1 - r_-^2)/D_-$

Here $J \equiv J_{AR}$, $J' \equiv J_{BR}$; x can run from $-\frac{1}{2}$ p to $(\frac{1}{2}p - 1)$; $D_{\pm} \equiv (\delta^2 \pm \delta J_{AB} + 9J_{AB}^2/4)^{\frac{1}{2}}$; and $r_{\pm} \equiv (\delta \pm \frac{1}{2}J_{AB})/D_{\pm}$. In these spectra, lines differing only in the value of x are not resolved. Nevertheless, this formulation allows us to determine the magnitude of the coupling constants and their relative signs.

In fig. 1, the spectrum has been annotated in accordance with the above table. When J and J' have the same sign, the sharp transitions 1 and 4 are on the outside of the spectrum: this is clearly the case here. Owing to the presence of the $r_{\pm}$ factors, the R spectrum depends not only on the relative sign of J_{AB} and J, but also on the sign of δ. These signs affect the nature of the asymmetry, and also the breadth of the absorptions 5(x), 6(x) as opposed to 7(x) and 8(x). The ^{31}P spectrum has shown[1] that $\delta < 0$; hence the shape of fig. 1 is only consistent with J_{AB} and J having the same sign. From the positions of peaks a, c and e, we obtain $J_{AR} = +16.2$, $J_{BR} = +0.4$ c/s. (The ^{31}P spectrum gave $\delta = -5.8$ p.p.m., $J_{AB} = +63.3$ c/s).

In fig. 2, it is clear that the asymmetry is exactly reversed. But, from the ^{31}P data[1], we know $\delta > 0$. The reversed asymmetry is compatible only with all the coupling constants still having the <u>same</u> sign.

We can now relate the signs of several coupling constants. Let us assume $^1J_{PF} < 0$. Then from refs. 1 and 3, $^2J_{PNP} > 0$ (and also $^3J_{PNPF} > 0$) in fluorophosphonitrilics. If we assume $^2J_{PNP} > 0$ in all these compounds, then from the preceding discussion $^3J_{POCH} > 0$ and $^3J_{PNCH} > 0$ in agreement with a very recent review[6]. Furthermore, the signs of the small long-range couplings are $^5J_{PNPOCH} > 0$ and $^5J_{PNPNCH} > 0$.

Lastly, we briefly re-consider the asymmetry in the two fluoro-compounds[1,3] where $|L|$ is not small; on the contrary, $|L| > |\delta| >> |J_{AB}|$. Expanding in a power series now in J_{AB}, we find the principal transitions are <u>symmetric</u> about ν_R, correct to second order in J_{AB}: and this is borne out by the published spectra, except at very low field strength. However, the <u>combination</u> bands are asymmetric to first order in J_{AB}. In cases where the principal

transitions give no insight as to the relative signs of J and J_{AB}, such information is readily available from the combination bands, if these can be discerned.

Yours sincerely,

C. W. Haigh

F. Heatley (Manchester University).

Please credit this to the Swansea group.

Errata

I.I.T.N.M.R. 88-34 footnote a: for '$\mp$ 11' read '$\pm$ 11'

Ref. 1, p. 1153, penultimate line: for '$\pm$ 1012' read '$\mp$ 1012'.

References

1. F. Heatley and S. T. Todd, J. C. S. (A) 1152 (1966).

2. P. Diehl and J. A. Pople, Mol. Phys. 3, 557 (1960). See also ref. 3.

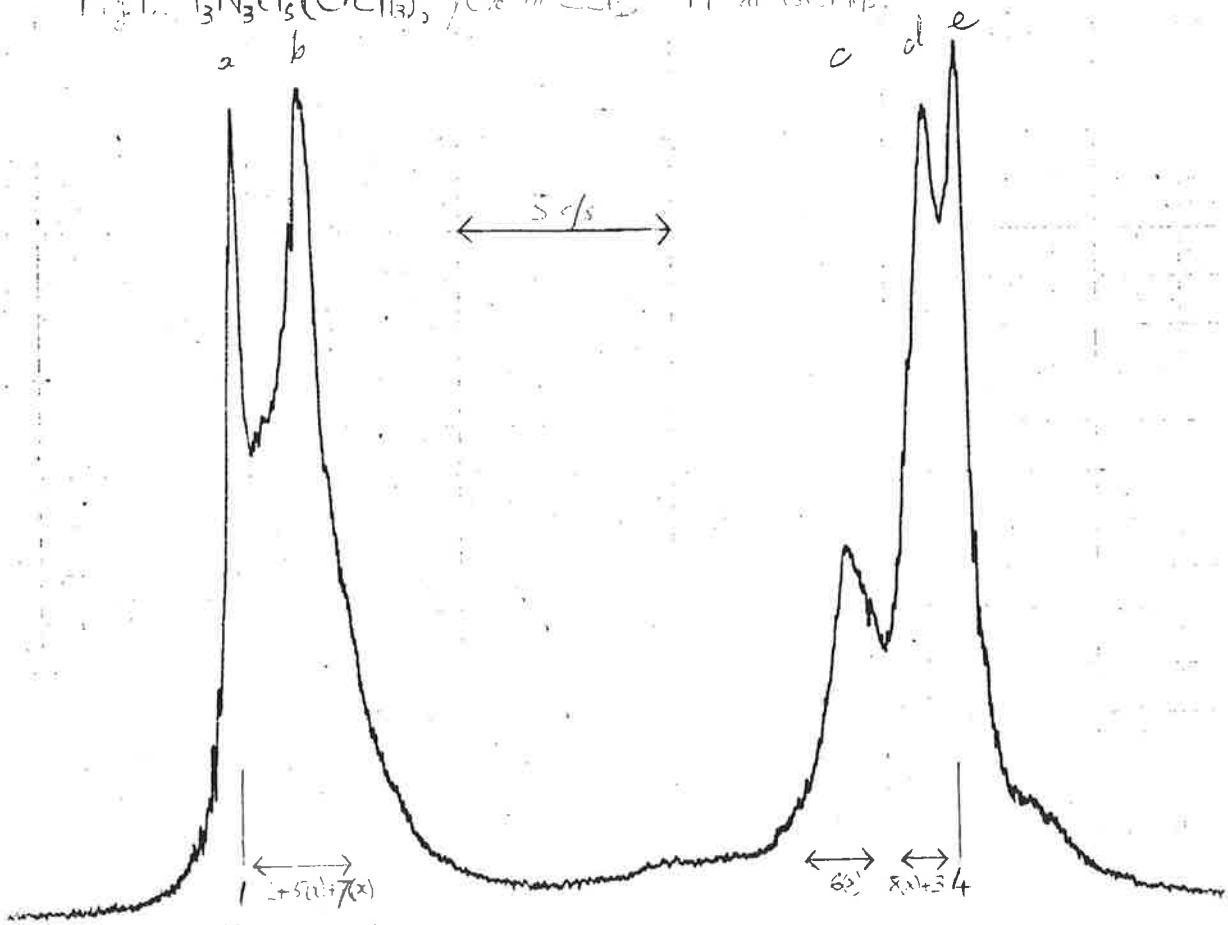
3. M. Heffernan and R. F. M. White, J.C.S. 1382 (1961).

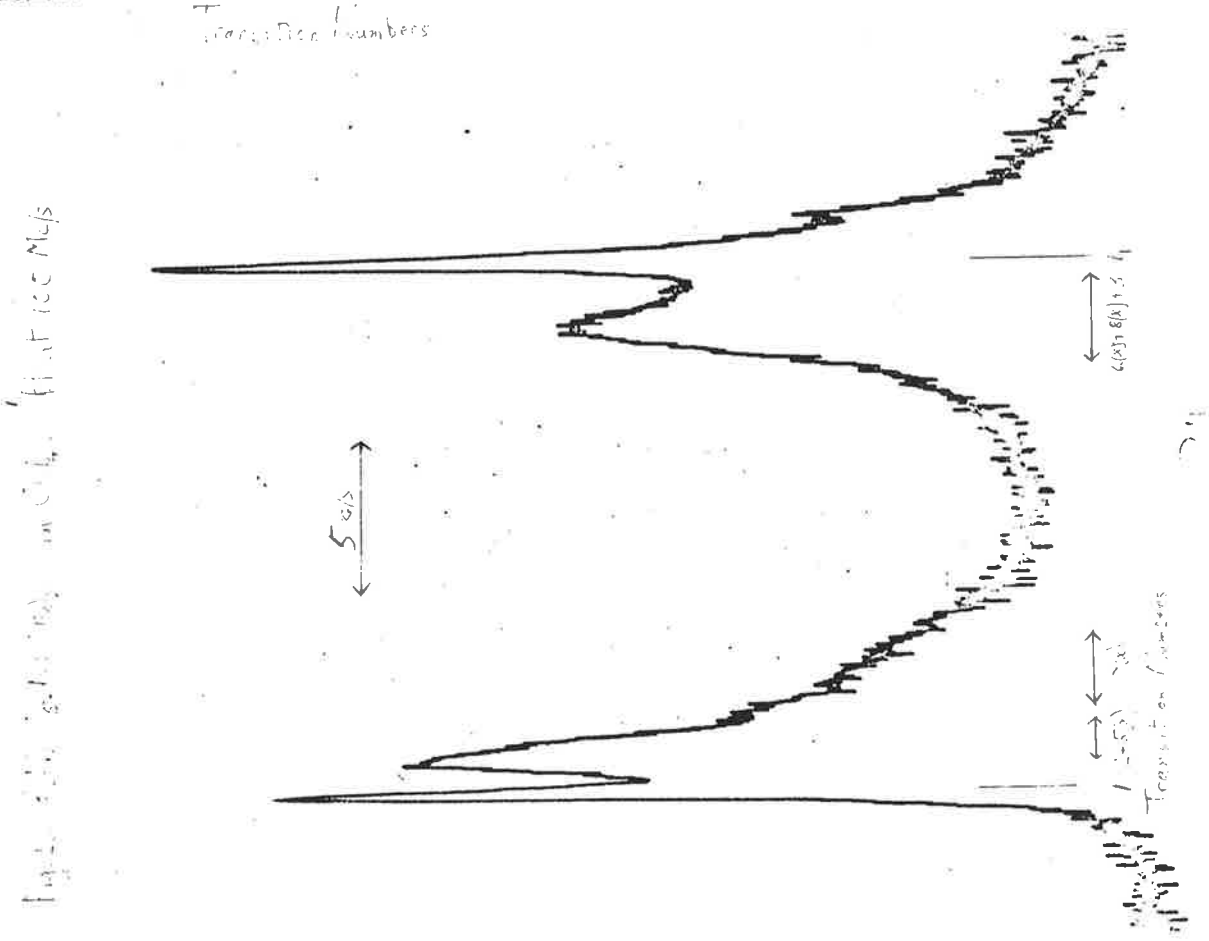
4. P. L. Corio, J. Mol. Spec. 8, 193 (1962) at p.199; see also I.I.T.N.M.R. 86 - 1.

5. J. W. Emsley, J. Feeney and L. H. Sutcliffe, High Resolution N.M.R. Spectroscopy, Pergamon 1965, I p. 368.

6. S. L. Manatt, G. L. Juvinall, R. I. Wagner and D. D. Elleman, J.A.C.S. 88, 2689 (1966) at p. 2697.

Fig. 1. $P_3N_3Cl_5(OCH_3)$, 70% in CCl_4, ^1H at 60 Mc/s.

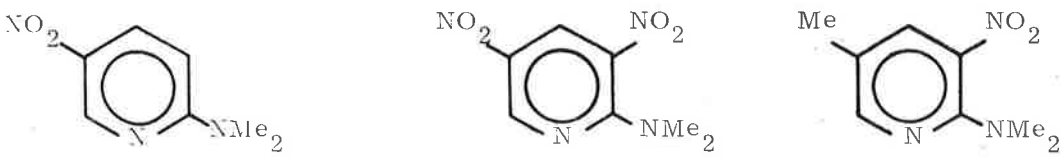




UNIVERSITY OF EAST ANGLIA
School of Chemical Sciences,
University Plain,
Norwich, NOR 85C.

Dear Dr. Shapiro,

In July's Newsletter (I.I.T.N.M.R. 94, p.28), H. Fritz reported hindered internal rotation of an N methyl amino group in substituted (s)-triazines. We have studied the temperature dependence (-60 to +30) of the N.m.r. spectra of the substituted dimethyl amino pyridines A to C and have found similar behaviour of the NMe_2 group in A and B.



A B C

At -60° two peaks are observed for the NMe_2 group in A and B, these collapse to a singlet at higher temperatures. The values of the chemical shift difference between the two methyl peaks at -60^{0} ($\Delta\nu$), together with the coalescence temperatures (Tc), and values of $\Delta G^{\ddagger}$ calculated [1] from these are given in the table.

TABLE

Compound	Solvent	Tc	$\Delta\nu$ [a]	$\Delta G^{\ddagger}$ [b] (Kcal/mole)
A	$CDCl_3$	-34°	12.3	12.3 ± 0.5
A	CH_2Cl_2	-30	15.5	12.4 ± 0.5
B	$CHCl_3$	-50°	47	10.9 ± 0.5
C	$CDCl_3$	-	-	< 10.4 [c]

a) in cps at 100 Mc/s.

b) Calculated using equation from ref. 1

c) Calculated assuming coalescence temperature of -60° with $\Delta\nu$ the same as for compound B.

At first sight the lowering of the barrier to rotation in B is surprising, but this is presumably due to steric interactions between the 3-NO_2 group and the NMe_2 group.

Work is in progress at the moment to obtain more accurate values of $\Delta G^{\ddagger}$ by a complete line shape analysis of the temperature dependence of the NMe_2 peak. We are also looking at 2NHMe, derivatives, and at related substituted benzenes and pyrimidines.

REFERENCES

1 R.K.Harris Ph.D. Thesis, Cambridge 1962.

A.R.Katritzky

G.J.T.Tiddy

IN REPLY PLEASE QUOTE
RÉFÉRENCE À RAPPELER

98-10

DCBRL: 904-1

DEFENCE RESEARCH BOARD CONSEIL DE RECHERCHES POUR LA DÉFENSE

DEPARTMENT OF NATIONAL DEFENCE
MINISTÈRE DE LA DÉFENSE NATIONALE
CANADA

DEFENCE CHEMICAL, BIOLOGICAL AND RADIATION LABORATORIES
OTTAWA 4, ONTARIO

LABORATOIRES CHIMIQUES, BIOLOGIQUES ET RADIOLOGIQUES DE LA DÉFENSE
OTTAWA 4, ONTARIO

12 October, 1966.

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

Dear Dr. Shapiro,

Recently we have adapted the programmes NMREN-1, NMREN-2, and NMRIT of Reilly and Swalen (1) for use with a Control Data 3200 computer. The largest change necessary was the reduction from 8 to 7 spins. NMRIT has been modified to enable trace adjustment of the NMREN 2 output without repunching cards. It should be noted that the subroutine SPINXuses a variable X different from the X which is in COMMON. This can cause difficulties when the EQUIVALENCE statement is modified, and this variable should then be renamed.

The Lusebrink programmes (2) PLOT, BLINE, SCALE, and DECOMP have been adapted to the above computer with no special changes or difficulties. Details of all adapted programmes are available upon request.

Yours sincerely,

Robert A. McIvor

R.A. McIvor

(1) J.D. Swalen and C.A. Reilly, J. Chem. Phys. 37, 21(1962).

(2) T.R. Lusebrink, Ph.D. Thesis, University of California, 1965.
W.D. Keller, T.R. Lusebrink and C.H. Sederholm, J. Chem. Phys. 44, 782(1966).

98-11

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NMR Specialties

RESONANCE SPECIALTIES
INCORPORATED

1410 Greensburg Road
New Kensington, Pa. 15068
U.S.A.
Area Code 412
361-0300 337-7727

October 19, 1966

Professor Bernard L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago 16, Illinois

Dear Professor Shapiro:

Decoupled Spectrum of 4,4'-Difluorobiphenyl

In a previous study,[1] the 60 MHz proton magnetic resonance spectrum of 4,4'-Difluorobiphenyl was analyzed as an ABX spin system. Since then I have had the opportunity to investigate the spectrum more completely, employing irradiation at 56.4 MHz in order to decouple the fluorine nuclei. The resulting AA'BB' spectrum has been analyzed by means of a technique devised by Dischler and Maier.[2] The derived parameters are K = 4.7 Hz, L = 8.5 Hz (estimated), M = 0 Hz, N = 9.1 Hz, and $\nu_0\delta$ = 21.5 Hz.

The undecoupled spectrum consists of the superposition of two AA'BB' spin systems, one arising from coupling to fluorine possessing α spin, and the other from coupling to fluorine possessing β spin. The analysis is identical to that of interpreting the spectra of two AA'BB' spin systems, possessing apparent chemical shifts $\nu_0\delta + \frac{1}{2}(J_{AX} - J_{BX})$, and $\nu_0\delta - \frac{1}{2}(J_{AX} - J_{BX})$. The interval N appears twice in each pattern, and the separation of the pattern centers is equal to $\frac{1}{2}\left|J_{AX} + J_{BX}\right|$. The magnitudes of J_{AX} and J_{BX}, determined from the undecoupled spectrum, are 9.05 Hz and 5.29 Hz, respectively; while the magnitude of N is 9.09 Hz.

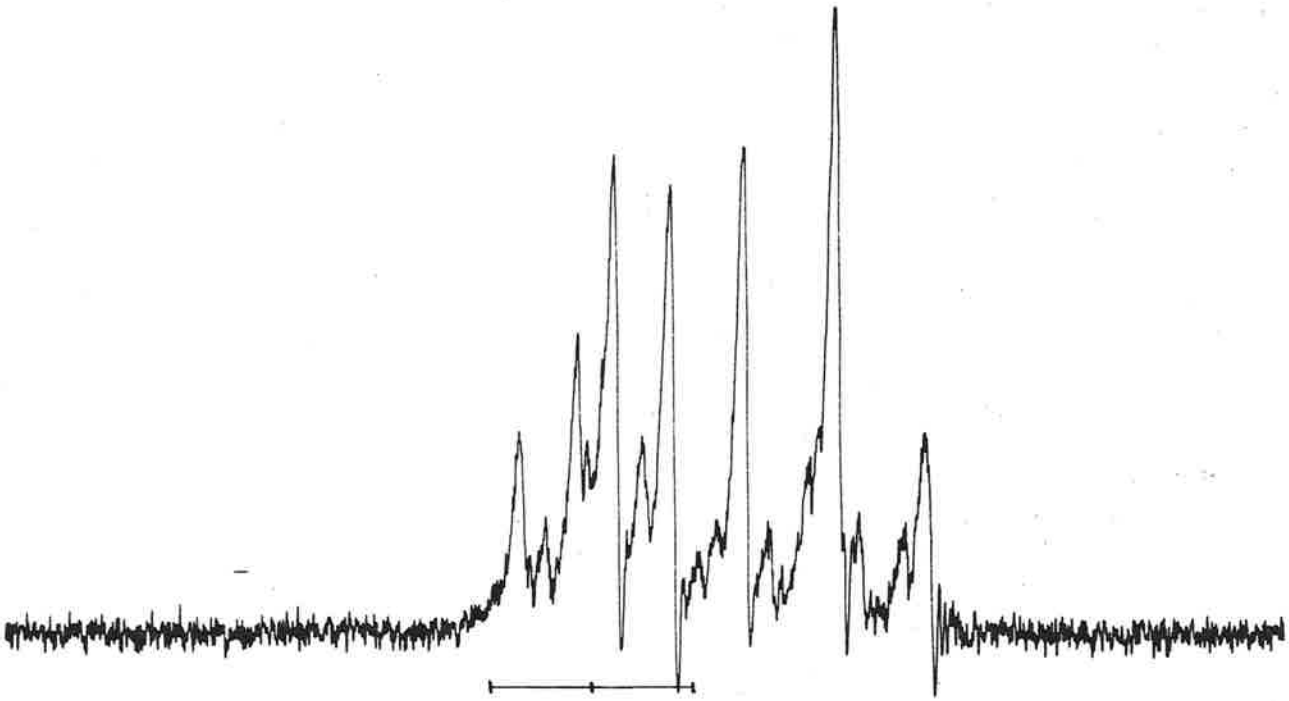
Sincerely,

William B. Wise

William B. Wise

WBW/ck

References:
(1) R. J. Kurland and W. B. Wise
J. Am. Chem. Soc. 86, 1877 (1964).
(2) B. Dischler and W. Maier
Z. Naturforsch. 169, 318 (1961).



60 MC PROTON NMR. SPECTRUM

DATE 6/14/66 SPECTRUM x

TEMP. 30 °C OPERATOR W.B.W.

SOLVENT CHLOROFORM DSTD: Me.Si ✓

SAMPLE 4,4'-DIFLUOROBIPHENYL

SWEEP WIDTH	250	CPS
SWEEP OFFSET	375	CPS
SWEEP TIME	250	SEC
FILT. BAND.	4	CPS
R.F. FIELD	.08	MG
SPEC. AMP.	5.0	
INT. AMP.	off	
REMARKS:		

NUCLEAR MAGNETIC RESONANCE SPECIALTIES
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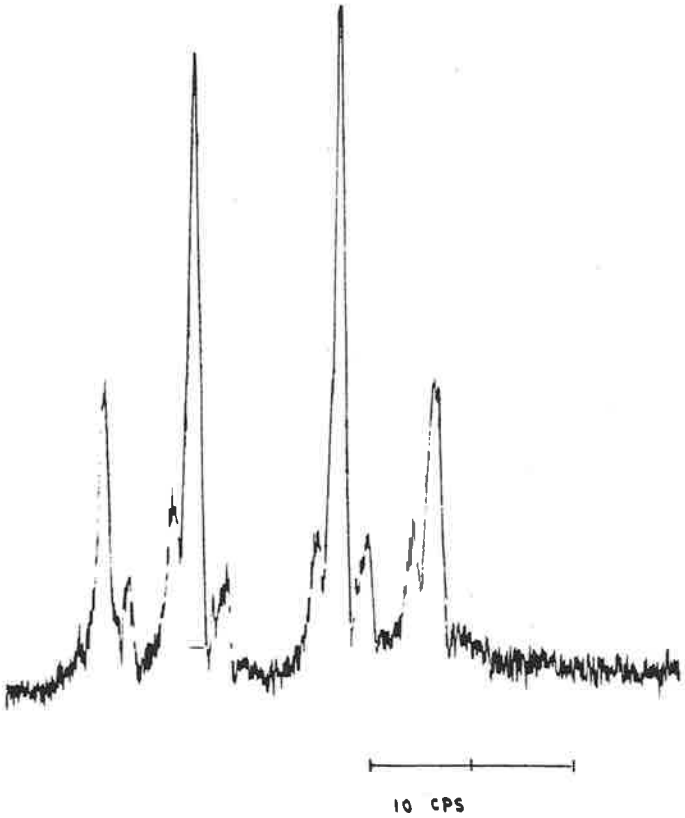


60 MC PROTON NMR. SPECTRUM

DATE 6/13/66 SPECTRUM DECOUPLING

TEMP. 30 °C OPERATOR W.B.W.

SOLVENT CHLOROFORM-D STD: Me_4Si ✓

SAMPLE 4,4'-DIFLUOROBIPHENYL

SWEEP WIDTH	250	CPS
SWEEP OFFSET	365	CPS
SWEEP TIME	250	SEC
FILT. BAND.	4	CPS
R.F. FIELD	.12	MG
SPEC. AMP.	4.0	
INT. AMP.	off	

REMARKS:

NUCLEAR MAGNETIC RESONANCE SPECIALTIES
INCORPORATED
145 GREENSBURG ROAD • NEW KENSINGTON, PA.

Bradford Institute of Technology
BRADFORD 7, ENGLAND.
Principal E G EDWARDS PhD BSc FRIC Bradford 29567
DEPARTMENT OF CHEMICAL TECHNOLOGY
Professor R L Elliott BSc PhD FRIC FTI FSDC

DWJ/JH

18th October, 1966.

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

Dear Dr. Shapiro,

In broad-line N.M.R. spectroscopy, one often wishes to measure absorption line shapes, for example of a solid polymer, over a wide temperature range. We have been working on a system whereby the ordinates of the first-differential output from a modified Robinson radiofrequency (29 Mc/s) spectrometer are punched on paper tape. For each measurement, the tape also carries an indication of temperature, derived from a thermostat control and mechanical digitizer, and a termination character. Thus, the mean second moments for a sequence of N.M.R. absorption curves, appropriately corrected for base-line and time-constant effects, may be computed so as to give directly a graph of second moment versus temperature.

Sample temperature is governed by contact with a heated copper block which forms one terminal of heat leak to a cold reservoir. The method of temperature control may be of interest to readers. It has been found that platinum resistance coils of sufficient resistance are exceedingly brittle when astatically wound in a very confined space. Further, hunting occurs unless the coil is in contact with the heater, in which case another sensor is needed for sample temperature measurement. Both functions can be accomplished with a thermocouple. In a spot galvanometer circuit, the thermocouple e.m.f. is balanced by a 1 kΩ precision potentiometer across a 1.35 volt mercury cell in series with a 69 kΩ tin oxide resistor. The potentiometer is attached mechanically to a shafted digitizer which provides analogue-to-digital conversion of temperature. By means of the circuit shown, sample heater current is reduced when an OCP 71 phototransistor, positioned in the light path of the galvanometer, is illuminated. The lower part of the figure shows the safety circuit to protect against galvanometer lamp failure. An additional circuit (not shown) prevents overshooting of the light beam and consequent loss of control; heater current is switched off as light falls on one "en stop" photocell and is restored only when a second one is illuminated.

Current high-resolution interests include:

(i) chemical shifts in polycyclic aromatic molecules;
(ii) proton resonance in cyclic organophosphorus compounds; and
(iii) amino-acid and protein solutions.

Finally, may we express our appreciation to Dr. A. Bothner-By and his colleagues for the continued appearance of Mellon Institute Bibliography in the I.I.T. N-M-R Newsletter.

Yours sincerely,

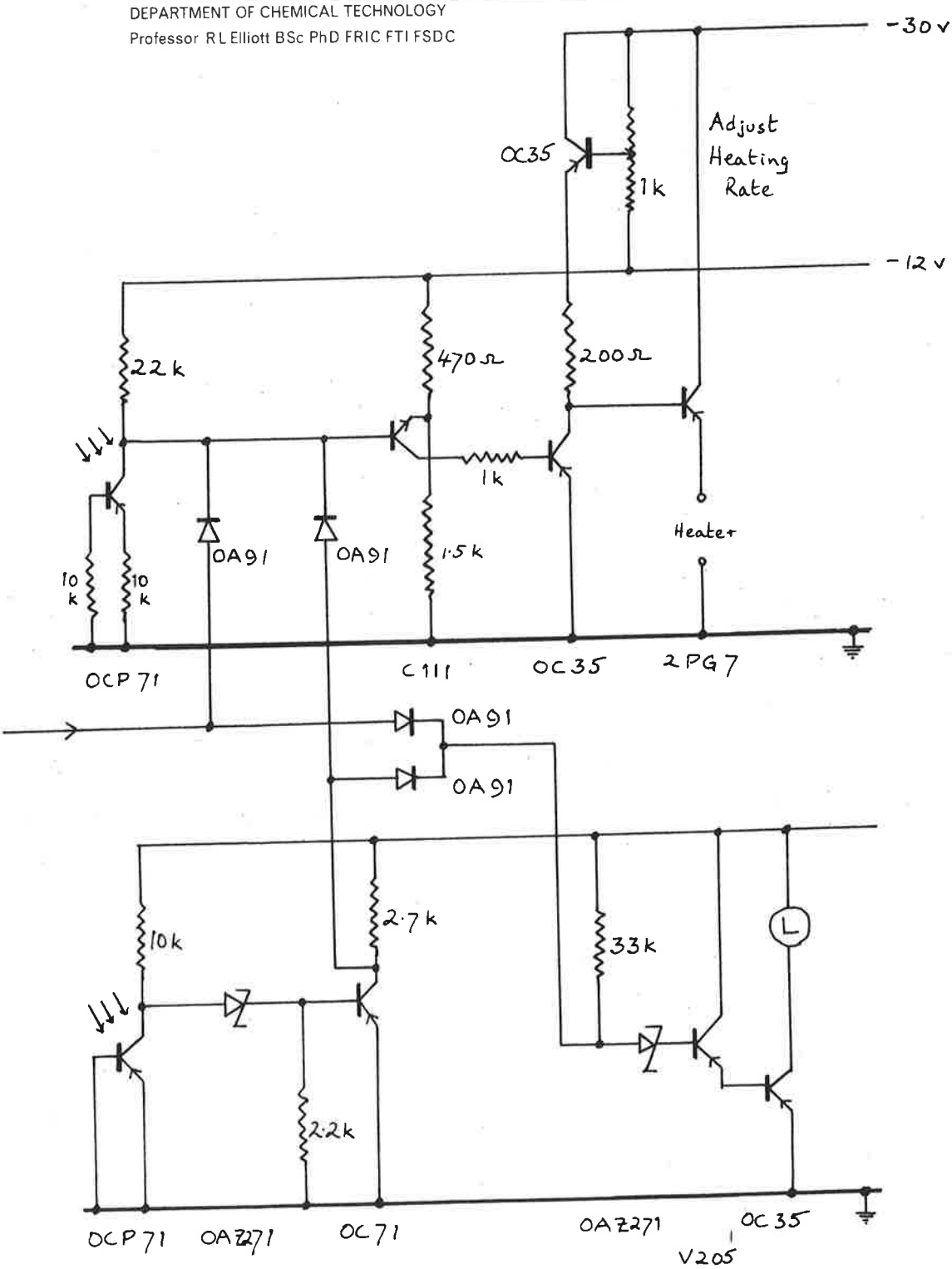
K.D. Bartle
K.D. Bartle

D.W. Jones
D.W. Jones

J.E. Pearson
J.E. Pearson

98-15

Bradford Institute of Technology
BRADFORD 7, England
Principal E G EDWARDS PhD BSc FRIC Bradford 29567
DEPARTMENT OF CHEMICAL TECHNOLOGY
Professor R L Elliott BSc PhD FRIC FTI FSDC



POLITECNICO MILANO
ISTITUTO DI CHIMICA INDUSTRIALE

Milano, October 24, 1966

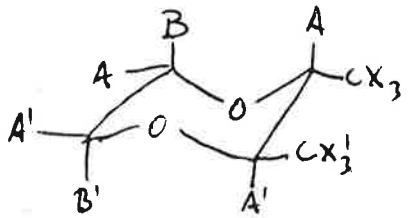
Professor B.L. Shapiro
Department of Chemistry
ILLINOIS, Chicago
Illinois Institute of
Technology

CONFORMATION OF 2,3-DIMETHYL-DIOXANE-1,4

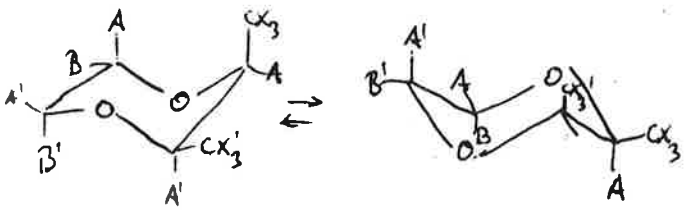
Dear Professor Shapiro:

We have recently studied the n.m.r. spectra of both isomers of 2,3-dimethyldioxane-1,4. The spectral parameters obtained and the assigned structures are as follows :

trans isomer :
diequatorial chair



cis isomer :
ea, ae interconverting chairs



For the $-CCH_2CH_2O-$ fragment :

J_{AB}	- 11.66		- 11.83	
$J_{AB'}$	2.66		6.40	
$J_{AA'}$	0.62		3.18	
$J_{BB'}$	11.51		3.18	
$\Delta\nu$	11.10	in DMS	24.66	in benzene
	[illegible]	in acetone	27.04	in acetone

continued...

Milano, October 24, 1966

For the $-OCHCH_3CHCH_3O-$ fragment :

$J_{AA'}$	8.46	3.20
J_{AX}	6.32	6.75
$J_{AX'}$	-0.33	-0.09

We hope that his contribution will bring our subscription up to date.

Sincerely yours,

Giuseppe Patti

Annalaura Segre

THE UNIVERSITY OF SUSSEX
THE CHEMICAL LABORATORY FALMER BRIGHTON SUSSEX

Professor J. N. MURRELL

Telephone: Brighton 66755

20th October, 1966.

Dr. B. Shapiro,
IIT NMR Newsletter,
Illinois Institute of Technology,
Urbana,
Illinois,
U.S.A.

Dear Dr. Shapiro,

Mr. M. A. Jensen and Dr. E. O. Bishop of this laboratory have begun an experimental and theoretical study of $Si^{29}H$ coupling constants. They have collected data for silanes with substituents such as the halogens, OMe, Ph, NMe_2 and alkyl. Data on thirty of these compounds were available in the literature and they have added data on another thirty five. Details will be published shortly, but Mr. Jensen will be pleased to supply data in advance of publication on request.

For the substituents Ph, Me, Cl and H all possible compounds have been studied and the coupling constants fall as well into a Malinovsky relationship

$$J_{SiH}(SiHABC) = J(AB) + J(BC) + J(CA)$$

as do CH coupling constants in the corresponding methanes.

Yours sincerely,

John Murrell

98-19

U. S. NAVAL RESEARCH LABORATORY
WASHINGTON 25, D. C. 20390

IN REPLY REFER TO

I. Postdoctoral Positions

The Naval Research Laboratory, in cooperation with NAS-NRC, annually offers a number of postdoctoral resident research associateships to U. S. citizens. Opportunities are available in the Chemistry Division for high-resolution, broadline and EPR studies, and in the Solid State Division for broadline, spin-echo and EPR studies.

Additional information may be obtained from the undersigned. The filing deadline is Feb. 1, 1967 for tenures during 1967-68.

II. NMR Review Reprints

A few reprints of the review article on NMR (coauthored by Ernie Lustig) which appeared in the April Review issue of Analytical Chemistry are still available.

W. B. MONIZ, Head
NMR Spectroscopy Section
Chemistry Division

STEVENS INSTITUTE OF TECHNOLOGY

HOBOKEN, NEW JERSEY

Department of
Chemistry and Chemical Engineering

October 20, 1966

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

BULK MAGNETIC SUSCEPTIBILITIES DETERMINED FROM
NMR SPINNING SIDEBANDS USING COAXIAL CELLS

Dear Prof. Shapiro:

I am sure that many of the IITNMR Newsletter readers will be surprised to learn that the spinning sidebands, which occur when one uses coaxial cells, can be used to determine bulk susceptibility. It is generally believed that these sidebands arise because of imperfections in the glass cells. In actuality the sidebands of the material in the annular region are a property of the coaxial system.

The effect of spinning the coaxial system causes the molecules in the annular region to experience an oscillatory field, ΔH_a, due to the bulk susceptibility difference between the sample and external standard; namely,

$$\Delta H_a = \frac{2\pi H_o}{r^2} [a^2(x_c - x_g) + b^2(x_g - x_a)]$$

The frequency, ν_m, of this oscillatory field is exactly twice the frequency of the spinner frequency, ν_s. That is $\nu_m = 2\nu_s$. Sidebands appear at $\nu_o \pm n\nu_m$, where ν_o is the fundamental resonance and n = 1, 2, ---. From the William-Gutowsky theory of field modulation we learn that the intensity, I_n, of the n-th order sideband is directly proportional to $J_n^2(k)$, where J_n is the n-th order Bessel function of the first kind and the argument

$$k = \frac{\gamma \Delta H_a}{2\pi\nu_m} .$$

One can measure the intensity of the center band, I_o, and the intensity of the first sideband. The ratio of intensities is simply

$$\frac{I_o}{I_1} = \frac{J_o^2(k)}{J_1^2(k)}$$

The value of k can be obtained from tables of Bessel functions. The frequency separation between these two bands is ν_m.

When the coaxial cells are stationary the signal of the material in the annular regions spreads into a U-shaped form. The frequency separation, Δ, between the two peaks of the U is related to k and ν_m by the following expression, derived for a 60 Mc/s spectrometer,

$$\Delta = \frac{k\nu_m}{30}$$

This method should prove useful for those investigators currently involved in measuring bulk susceptibilities.

Yours sincerely,

Edmund R. Malinowski

Edmund R. Malinowski
Associate Professor of Chemistry

ERM:jef

Dr. W. Brügel i.Fa.
Badische Anilin- & Soda-Fabrik AG · Ludwigshafen am Rhein
Hauptlaboratorium

BASF 1865 1965

Luftpost

Herrn
Prof. Dr. B.L. Shapiro

Department of Chemistry,
Illinois Institute of
Technology

Chicago, Illinois 60616

U S A

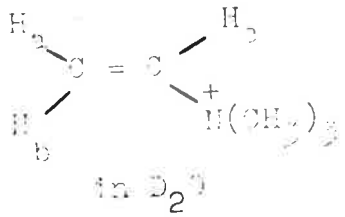
Ihre Zeichen	Ihre Nachricht vom	Unsere Zeichen	Fernsprecher-Durchwahl	Telex	67 Ludwigshafen
		Dr.Brü/Fa	(06 21) 600 . . .	4 64 . . .	26. Okt. 1966

Betreff Analysis of the neurin bromide spectrum

Sehr geehrter Herr Dr. Shapiro!

Vor einiger Zeit berichtete ich in IITNMR Newsletter No. 83 (Aug. 1965) über die Analyse des Protonresonanzspektrums von Neurinbromid (Trimethylvinylammoniumbromid). Dabei waren die Vorzeichen der Kopplungskonstanten der drei Vinylprotonen untereinander alle als positiv angegeben worden. Wie mir Herr Dr. Stanley L. Manatt vom Jet Propulsion Laboratory des CALTEC freundlicherweise mitteilt, hat er mit dieser Substanz u.a. einige thickling-Experimente angestellt und gefunden, daß das Vorzeichen der Kopplungskonstante der beiden geminalen Protonen der Vinylgruppe negativ sein muß.

Tatsächlich gibt es bei der unmittelbaren Analyse des zugrunde liegenden 100 MHz-Spektrums eine Lösung dieser Art, die das gemessene Spektrum ebensogut darstellt, wie die seinerzeit mitgeteilte:



(int. Stand. CH_3-signal)

ν_a = 2.228 ppm; J_{ab} = -4.27 Hz;
ν_b = 2.447 ppm; J_{ac} = 8.32 Hz;
ν_c = 3.139 ppm; J_{bc} = 15.10 Hz;

$|J_{a,N}|$ = 5.61Hz; $|J_{b,N}|$ = 2.52Hz; $|J_{c,N}|$ = 3.52Hz.

Badische Anilin- & Soda-Fabrik AG

Empfänger
Dr. B.L. Shapiro

Unsere Zeichen
Dr.Brü/Fa

67 Ludwigshafen
26.10.66

Betreff

Die Entscheidung über die Richtigkeit der einen oder anderen Lösung anhand der Linienintensitäten ist beim 100 MHz-Spektrum wegen zu geringer Unterschiede nicht möglich, zumal durch die zusätzliche Kopplung mit dem N-Kern Linienüberlagerungen auftreten. Auch das 60 MHz-Spektrum läßt keine zweifelsfreie Entscheidung zu, weil hier die Überlagerungen noch stärker sind.

Das angeführte Beispiel ist ein weiterer deutlicher Hinweis, daß die gewöhnliche, nur auf Linienlagen und Linienintensitäten basierende Analyse eines Spektrums häufig zu Fehlschlüssen führt und unbedingt durch andere Techniken, wie C^{13}-Satelliten, Thickling usw., ergänzt werden muß.

Herr Dr. Manatt hat bei seinen Experimenten, über die er vermutlich bald selbst ausführlich berichten wird, natürlich auch das Vorzeichen der drei Kopplungskonstanten mit dem N-Kern bestimmt und positiv gefunden. Ich bin ihm für seine Mitteilung sehr zu Dank verpflichtet.

Mit den besten Grüßen

Ihr sehr ergebener

W. Brügel

(Dr.W. Brügel)

Computer Triggering

We also have been trying out the idea(1) of reducing the gain of the N.M.R. signal before triggering a computer. In the computer collection of sequential spectra, when memory sweep is actuated by an input signal, the accuracy with which the x axis is located depends on how accurately the y trigger level corresponds to the desired x value. When the spectrometer is operated on high gain, there is a variation due to noise and a variation due to any baseline (i.e. DC level) change. The accuracy of location could be improved by sharpening the trigger signal (i.e. by increasing $\frac{dy}{dx}$): however, beware making the mistake of trying to achieve this by switching from a large spectral sweep width before triggering to the required one after. We have been interested in observing the meta splitting in a 540 c/sec. sweep width (at 60 Mc/sec.) Using our own-blown spherical microcells in a R 10 and a NS-544 computer(2) Using the circuit shown in fig.I, which switches the phase-detector gain from 1 to 8 on triggering, we have obtained the result shown in fig.II. The sample was 403 mg. of acetone 2:4-dinitrophenylhydrazone; a pre-triggering field shift of +10 p.p.m. was introduced, using TMS as the trigger signal. Sweep rate was 1.6 c/sec^2. The system allows a ready check on the sweep width(IIa) and the results accumulating 377 spectra [redacted] is shown in IIb and IIc. IIc was obtained using the same computer output speed as IIb but with the recorder paper at twice the speed.

Gordon Hall — Gordon Hall
Brian Braithwaite — B A Braithwaite
David Frost — D.J. Frost

Colworth House,
Sharnbrook.
Bedford.
England.

(1) Colebrook, Newsletter, 94-44
(2) Frost, Hall, Leane and Green, Chem. & Ind., in press.

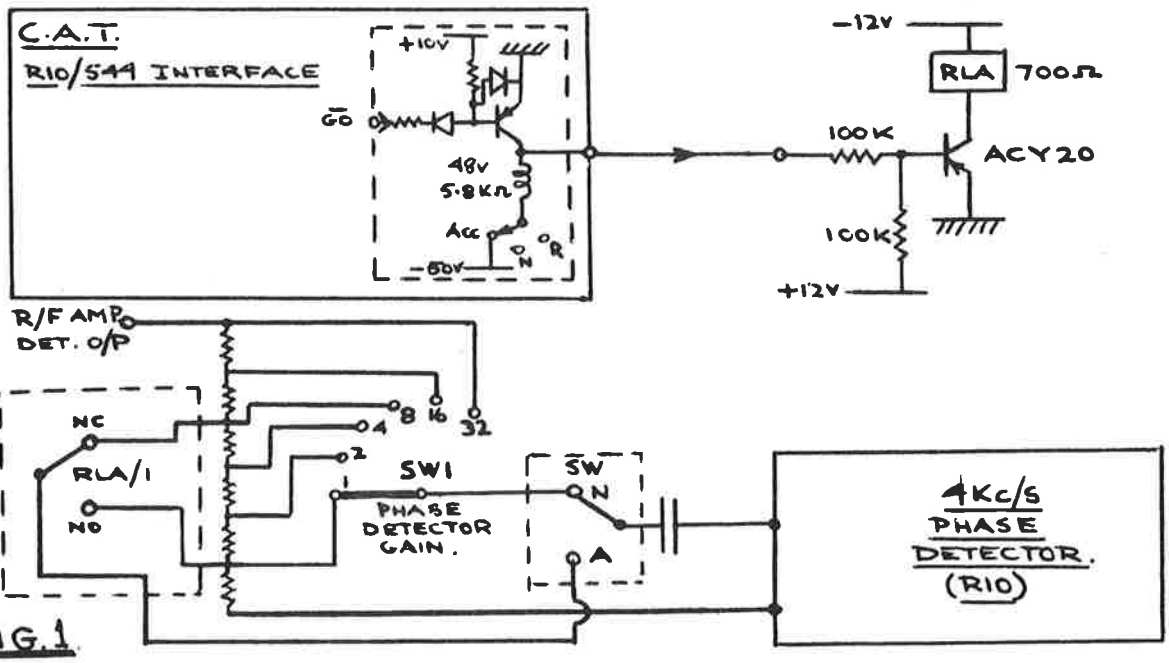
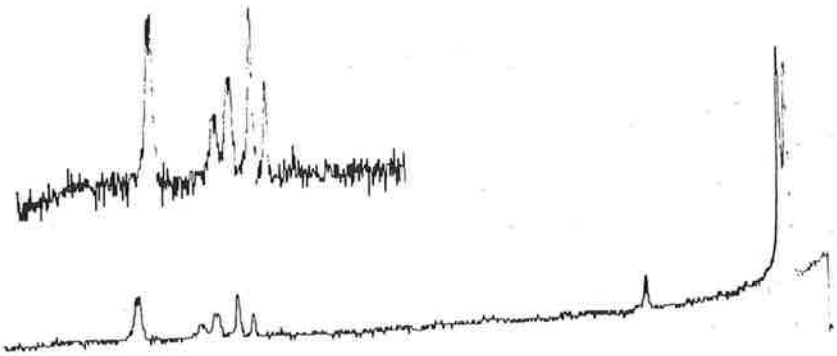


FIG.1

(a)

(b)



(c)

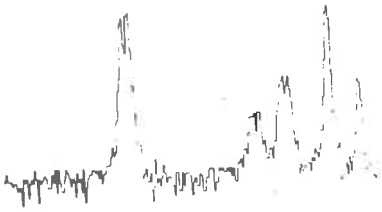


FIG II

PERKIN-ELMER

THE PERKIN-ELMER CORPORATION
NORWALK, CONNECTICUT 06852
TELEPHONE: (203) 847-0411
CABLE: PECO-NORWALK

18 October 1966

Dr. Barry Shapiro
Department of Chemistry
Illinois Inst. of Technology
Chicago, Illinois

Dear Barry:

TITLE: CARBON-13 ENHANCEMENT

We recently ran some Carbon-13 enhancement experiments in England that we would like to report.

The C-13 spectrum absorption mode spectrum of mesitylene is shown. The spectrum was measured at 15.1 mc/s. The range was 200 ppm and the spectrum was run 256 times. These scans were 20 seconds (CRO sweeps) followed by suitable time delay to allow for the relaxation time of the carbon nuclei.

The spectra may be accumulated by means of recorder sweep mode or oscilloscope sweep mode, the latter being more rapid. The computer used was the Northern Scientific NS-544 (1024 channels; 2^{16} bits per channel).

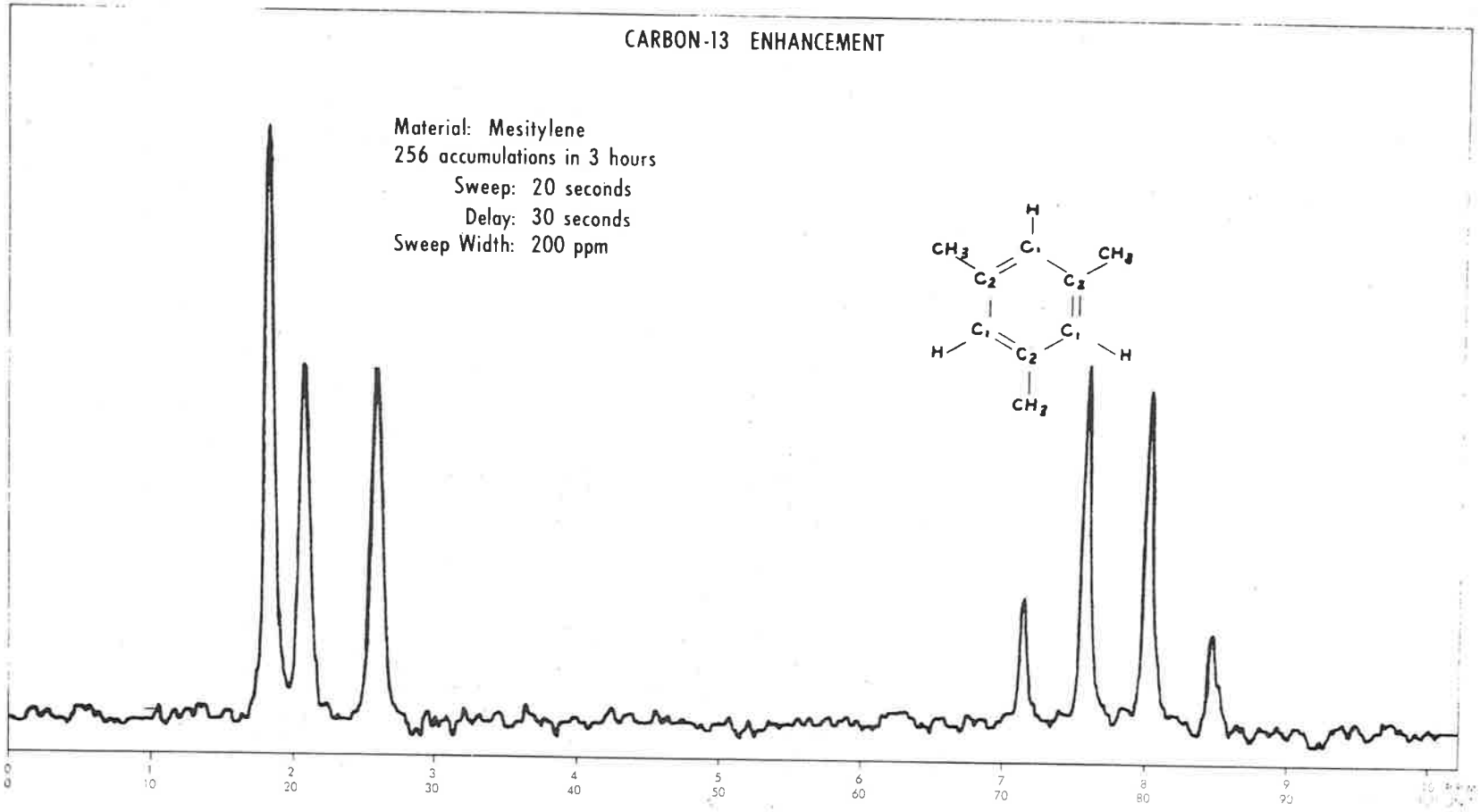
In the C-13 spectrum of mesitylene the high field quartet is the methyl resonance ($^{13}C\text{-}H_3$), the doublet is $-\overset{\overset{\displaystyle O}{\|}}{^{13}C}\text{-}H$ and the single resonance is the substituted aromatic carbons.

Sincerely,

Paul

Paul A. Strauss

PAS:mlt



PERKIN-ELMER

FACULTÉ DES SCIENCES DE MARSEILLE - SAINT-JÉROME
TRAVERSE DE LA BARASSE - MARSEILLE (13e)
TÉLÉPHONE : 64.31.61

DÉPARTEMENT DE CHIMIE ORGANIQUE
LABORATOIRE DE CHIMIE M.P.C.
J. C. MAIRE : MAITRE DE CONFÉRENCES.

le [illegible] 1975

Professor E.L.SHAPIRO
Dept.of Chemistry
Illinois Institute of Technology
CHICAGO 60616 - Illinois (USA)

Dear Professor SHAPIRO,

PMR spectra of stannanes.Sn-C bond magnetic anisotropy

Thank you for the reminder. As our contribution to IITNMR news letter, I would like to give the results we obtained in studying P.MR. spectra of alkyl substituted stannanes : $(alkyl)_n Sn\ H_{4-n}$. It was already known that hydride proton resonance appears lower field with respect to stannane (SnH_4) itself, for example :

	$(n\text{-}Bu)_3\ Sn\ H$	$(n\text{-}Bu)_2 Sn\ H_2$	$n\text{-}Bu\ Sn\ H_3$	SnH_4
τ	5,2	5,47	5,76	6,11

This fact could not be explained by an inductive effet of the alkyl groups which would render the tin bonded protons more negative and lead to an up field shift.

We are thinking now that major effet is the influence of the magnetic anisotropy of Sn-C bonds.

Using Mac-Connell point dipole approximation and giving arbitrary to the Sn-H bond magnetic anisotropy zero value, we calculated for the Sn-C bond $\Delta\chi = 20.10^{-30}$ cm^3 $molecule^{-1}$. Our drastic approximation seems to be justified partly by the fact that τ difference between two stannanes with the same alkyl group and n differing by one (for example : $(n\text{-}Bu)_3Sn\text{-}H$ and $(n\text{-}Bu)_2Sn\text{-}H_2$) is constant (within 0,03 ppm) as illustrated above.

As a further check we are preparing $(CH_3)_3\ Sn\text{-}CH\ (CH_3)_2$ which by comparaison with $(CH_3)_3\ C - CH\ (CH_3)_2$ would give an approximate value for $\Delta\chi$ Sn-C - $\Delta\chi$ C-C.

This work will be shortly reported on in full details in J.organometal. chemistry.

Sincerely yours.

J.C. MAIRE

J.DUFERMONT

98-29

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

October 31, 1966

AIRMAIL

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

Design and Performance of an NMR Microcell Particularly Suited for the HA-100; How to Filter Microsamples?

Dear Barry:

Am sorry to be one of those who are adversely affecting your golf score (to say nothing of your "scientific output and monthly paycheck"!). Will try to be less trouble to you in the future.

We have been quite satisfied by the performance of the spherical glass micro-cells made by NMR Specialties in our A-60. I would estimate that about three-fourths of the two-dozen or so tubes that we have now tested gave satisfactory resolution (linewidth of 5% TMS less than 0.6Hz) either initially or after a careful cleaning, and the manufacturer readily exchanged those which we found to be definitely unsatisfactory. The most serious limitation to the general use of these cells on the A-60 that we have found is their total intolerance of particulate matter of any kind in the sample.

We have experimented with several filter assemblies. The most successful of these to date, though by no means fully satisfactory, consists of a 50 or 100 microliter gas-tight syringe fitted with a luer lock, a 13 mm. Swinny-type polyethylene filter holder made by Millipore and a seven inch Hamilton needle fitted with a teflon hub to reduce the dead volume, which even so is excessively large for the sample volumes involved. So far we have generally used Gelman Alpha-6 regenerated cellulose filters with a 0.45μ pore size. According to the manufacturer these will stand up to all solvents normally used for NMR, and we have certainly not experienced any trouble whatever from them. However, some samples continue to give poor resolution even after several passes through these filters so we are about to try some with 0.2μ pores. I would be most interested to hear how others deal with this problem.

I've emphasized that these cells have performed very well in our A-60. The reason for this emphasis is that at least in our case good A-60 performance in no way guarantees similar performance at 100 MHz. We carefully checked the resolution of six tubes on the A-60 and then, without changing the sample tested them in our variable temperature 100 MHz probe. Only two of them gave really good resolution (better tnan 0.6Hz with ringing) and these were quite difficult to adjust in the spinner so that they did not cause excessive RF modulation when spinning. In fact the only criterion for selection other than actual NMR performance which we have found is the minimum value of spinning modulation which a tube will produce in an HR-100 probe.

The disappointing performance of the all-glass cells at 100 MHz. led us to try a modification of the microcell described by Drs. Frei and Niklaus (IITNMR 67-39) which is shown on the attached figure. Drs. Flath and Teranishi of our Fruit Laboratory made up several of these cells.

They consist of a precision clear NMR sample tube, a machined Teflon chuck and a small glass bulb in which the sample is placed. The spherical sample bulbs are prepared from standard borosilicate melting point tubing. Usually the diameter of the bulb is formed so as to provide a smooth slip fit in the standard NMR tube; this yields a microcell holding approximately 35 microliters of solution. The Teflon holder is turned on a small lathe to provide a snug fit in the standard NMR sample tube (this snug fit, and the considerable difference in coefficients of thermal expansion of Teflon and borosilicate glass will preclude the use of the microcell at elevated temperatures). A 7 mm deep hole is drilled in the lower end of the plug to accept the neck of the micro sample bulb. This hole must be of sufficiently small diameter to grip the neck of the sample bulb firmly and must be concentric with the body of the Teflon holder to minimize sample wobble while spinning. A longitudinal groove is cut in the plug to equalize pressure above and below the plug when it is placed in the NMR tube. The sample bulb and Teflon plug are positioned in the NMR tube for maximum resolution by means of the threaded Kel-F rod which screws into a threaded hole in the top of the Teflon plug. The rod is removed before the cell is placed into the instrument probe. The space around the micro sample bulb, below the Teflon holder, is filled with carbon tetrachloride to further minimize any tendency for the sample bulb to wobble when the assembly is rotated.

We were most pleasantly surprised by the fine performance of these cells in the "100." They appear to hardly degrade resolution at all and require far less readjustment of the homogeneity controls. Since their volume is about 30 percent less than the average volume of our useable all-glass cells, they yield an intensity enhancement over a 0.3 ml sample in a standard tube of about five rather than four. An example of the excellent performance that can be obtained from these cells in an HA-100 is shown in the second figure. The sample consisted of 0.45 mg of trans, trans-humulene in 34 microliters of CCl_4.

I am also enclosing a copy of the quotation from Kontes Glass Company for the fabrication of the entire assembly. As yet, however, we have not had an opportunity to check-out one of these:

	NET PRICE EACH IN LOTS OF			
	12	50	60	200
NMR Tube, interchangeable I.D., per submitted specifications, Ground	2.50		1.50	
Ditto, but polished	3.00		1.90	
Kel-F Rod and Teflon Holder Assembly, complete per submitted sketch and specifications, Rod ONLY	2.50		1.00	
Ditto, but HOLDER ONLY	7.50		2.50	
Sample Tube, approx. 30 ul., per submitted specifications		.90		.60

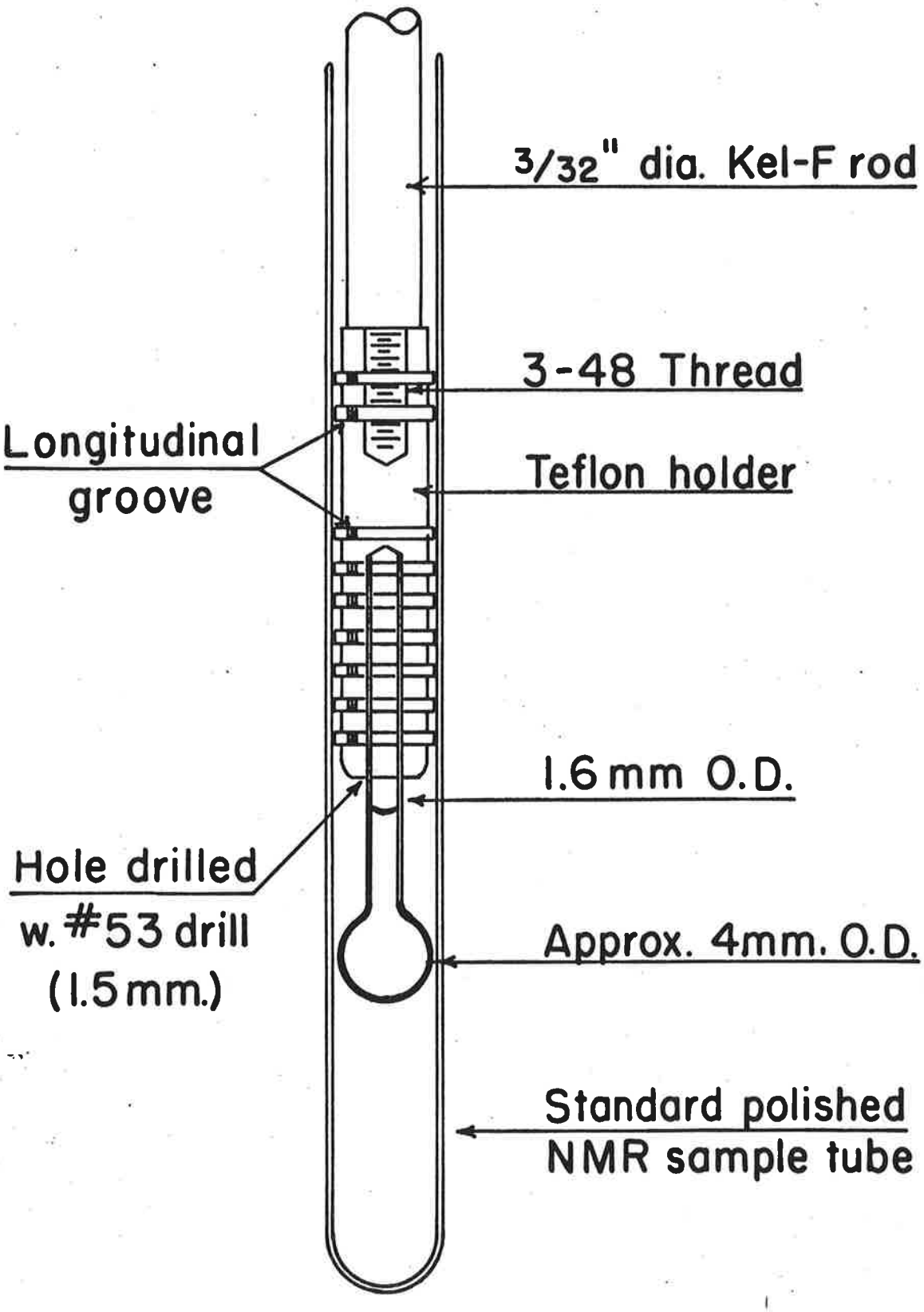
When ordering, kindly place with - Kontes of California
2809 - 10th Street
Berkeley, California 94710
Quote # 26084

Shipment: in 45 days

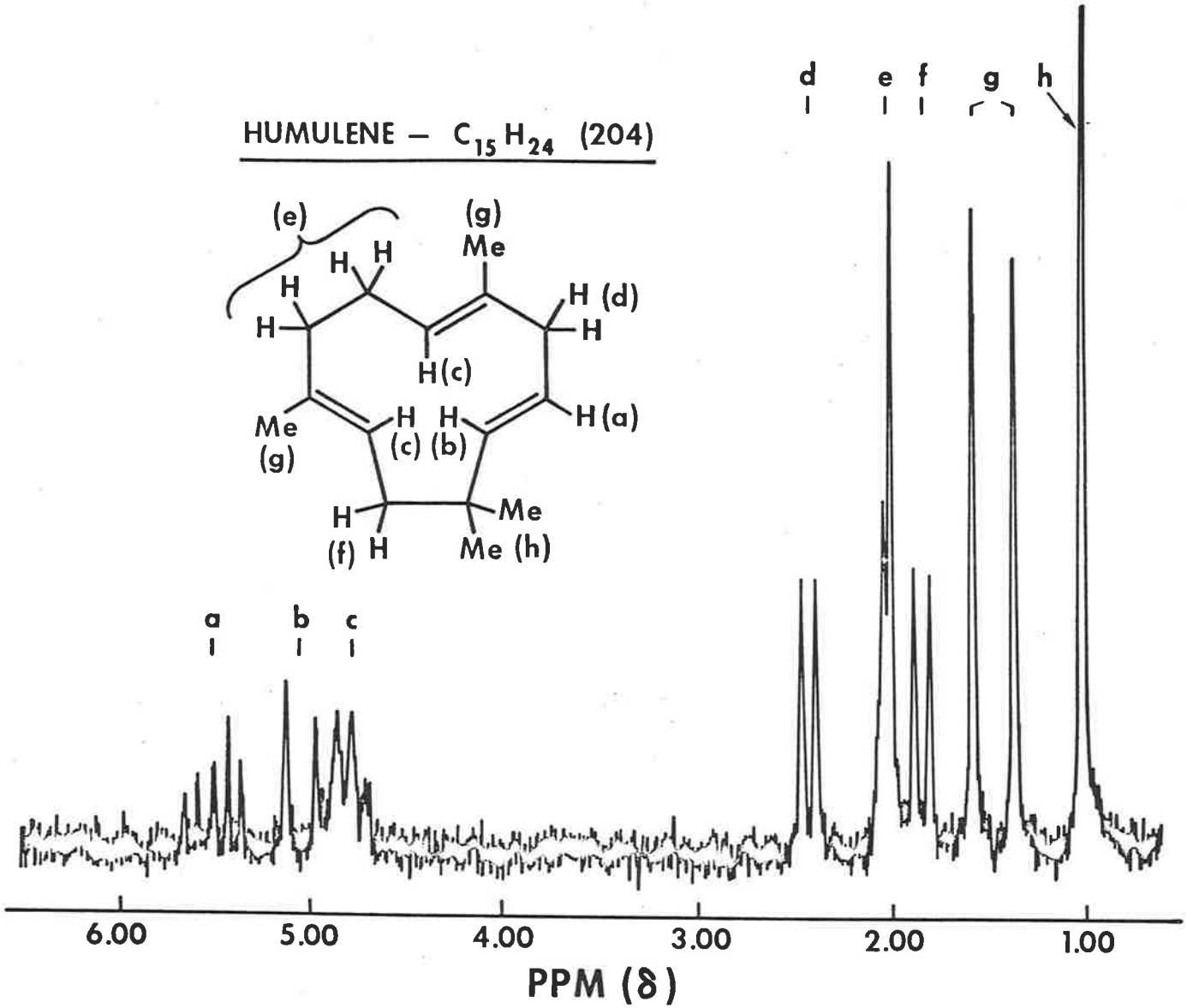
Sincerely yours,

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

Enclosures


3/32" dia. Kel-F rod
3-48 Thread
Longitudinal groove
Teflon holder
1.6 mm O.D.
Hole drilled w. #53 drill (1.5 mm.)
Approx. 4mm. O.D.
Standard polished NMR sample tube

HUMULENE — $C_{15}H_{24}$ (204)



UNIVERSITY OF CALIFORNIA, DAVIS

BERKELEY • DAVIS • IRVINE • LOS ANGELES • RIVERSIDE • SAN DIEGO • SAN FRANCISCO SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY DAVIS, CALIFORNIA 95616

November 4, 1966

^{13}C Spectra at 25.1 MHz. Using Carbon Lock

Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Technology Center
Chicago 16, Illinois 60616

Dear Barry:

We were very interested in LeRoy Johnson's letter in IITNMRN No. 96 (page 43) as we were having approximately the same experience with ^{13}C. Using the standard HA-100 spectrometer system at 25.1 MHz. we have locked on signals from 50% labelled simple organic substances such as $^{13}CH_3OH$, $^{13}CH_3I$ and $^{13}CS_2$ contained in standard melting point capillaries. By employing an external oscillator for the modulation frequency of the control channel when necessary, and by making judicious use of both upper and lower sidebands signals as described by Johnson we are able to cover the usual range of ^{13}C shifts.

Our brief experience seems to indicate that most simple organic molecules are capable of providing reasonable lock signals. We have maintained a stable lock for a twenty-four hour period, enabling time-averaging of several hundred scans. This has allowed us to obtain spectra on natural abundance samples or on dilute solutions of labelled materials, with line-widths sometimes less than one Hz. The technique seems to be relatively straightforward and should open up the field to any laboratory with a standard HA-100 system and access to a 25.1 mc. rf. unit and probe.

We have been applying this technique to the determination of ^{13}C-^{13}C coupling constants, as well as to our continuing study of solvent effects on the ^{13}C shifts of carbonyls. Our group is still searching for bigger and better lock signals and is getting set up to incorporate proton decoupling into the technique.

Sincerely,

Gary E. Maciel
Associate Professor
of Chemistry

GEM:dh

98-35

THE UNIVERSITY OF MANCHESTER

MANCHESTER, 13.

FROM

PROFESSOR GEOFFREY ALLEN
PROFESSOR OF CHEMICAL PHYSICS
TELEPHONE: ARDWICK 3333

1st November 1966

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

Dear Professor Shapiro,

Rotational Averaging of Coupling Constants and Chemical Shifts

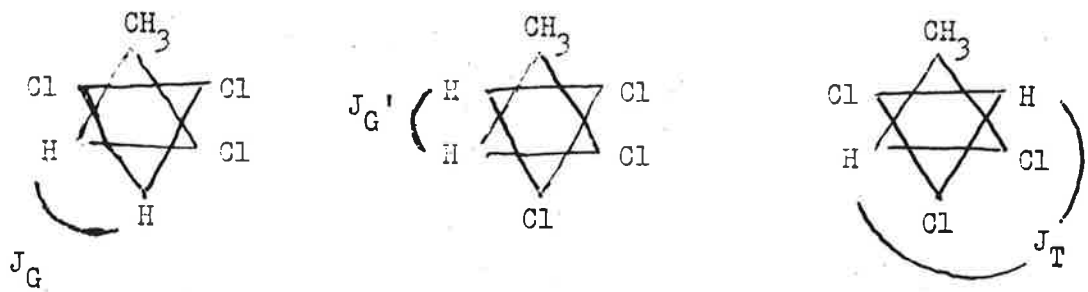
We are currently trying to obtain energy differences between rotational isomers in some substituted ethanes from temperature variation of chemical shifts and vicinal proton coupling constants. The temperature variation of chemical shift is less reliable for this purpose since a significant contribution arises from the effect of varying intermolecular interactions. We tried to allow for this latter effect by choosing reference molecules which do not possess rotational isomers but which contain the chemical group whose proton chemical shift was being investigated.

These references were pentachloroethane for $CHCl_2$ and protons, 1,1,1,2-tetrachloroethane for CH_2Cl and protons and 1,1,1-trichloroethane for CH_3 protons.

These suppositions were not borne out in practice however as shown by the results for 1,1,1,2-trichloropropane in the table below:-

Energy differences between rotational isomers in 1,1,2-trichloropropane

Solvent	$E_T - E_G$ (cals)			J_T cps	(J_G+J_G')cps
	$CHCl_2$ chem shift	CH_3 chem shift	Coup const.		
Pure compound	1400	410	390	14.5	.6
Pentachloroethane	720	710	350	14.9	.7
Acetic anhydride	1630	-	600	14.2	2.5



G G' T

Errors in energy differences are ± 30 cals; errors in coupling constants are ± .3

1,1,2-trichloropropane has 3 non-equivalent rotamers but it was found that a satisfactory fit to the experimental results was obtained by assuming that the G and G' isomers were equal in energy. The energy differences and errors were determined by a method similar to that of Gutowsky, Belford and McMahon[1].

The results from the analysis of coupling constants are straightforward. The agreement between the coupling constants in the three systems is good in view of the approximations involved. This is in marked contrast to a study of the same molecule[3] in CS_2 and acetone where variations of ~2 c/s in the coupling constants were required to fit the results. The large value of $(J_G + J_G')$ in acetic anhydride is probably due to a change in the dihedral angle in the two rotamers caused by solvation effects. The change in J_T would be expected to be much smaller.

The use of a reference for the chemical shift is clearly a failure even though the two solvents are not chemically dissimilar. It shows the importance of :-

(a) intramolecular effects which might cause the chemical shift to be dependent on the angle of rotation so that the chemical shift of an individual isomer, when averaged over the torsional vibration, might vary with temperature, and

(b) specific intermolecular interactions between a particular rotamer and the solvent rather than some "general field effect" which would alter the relative chemical shifts of the three rotational isomers by the same amounts.

With apologies for the delay in sending this contribution,

Yours sincerely,

F. Heatley. G. Allen

F. Heatley Professor G. Allen

References

1. H.S. Gutowsky, G.G. Belford and P.E. McMahon, J.Chem.Phys., 36, 3353, (1962).
2. J.C. Schug, P.E. McMahon and H.S. Gutowsky, J.Chem.Phys., 33, 843, (1960).
3. H.S. Gutowsky, Pure Appl. Chem., 7, 93,

varian associates limited

RUSSELL HOUSE MOLESEY ROAD WALTON-ON-THAMES SURREY

Your Reference:

Our Reference:

Telephone: Walton-on-Thames 28766
Cables: Varian Walton
Telex: 261351

2nd November 1966

Professor Bernard L.Shapiro,
Associate Professor,
Illinois Institute of Technology,
Chicago 60616,
U.S.A.

Dear Barry,

CH DETECTION BY DOUBLE RESONANCE

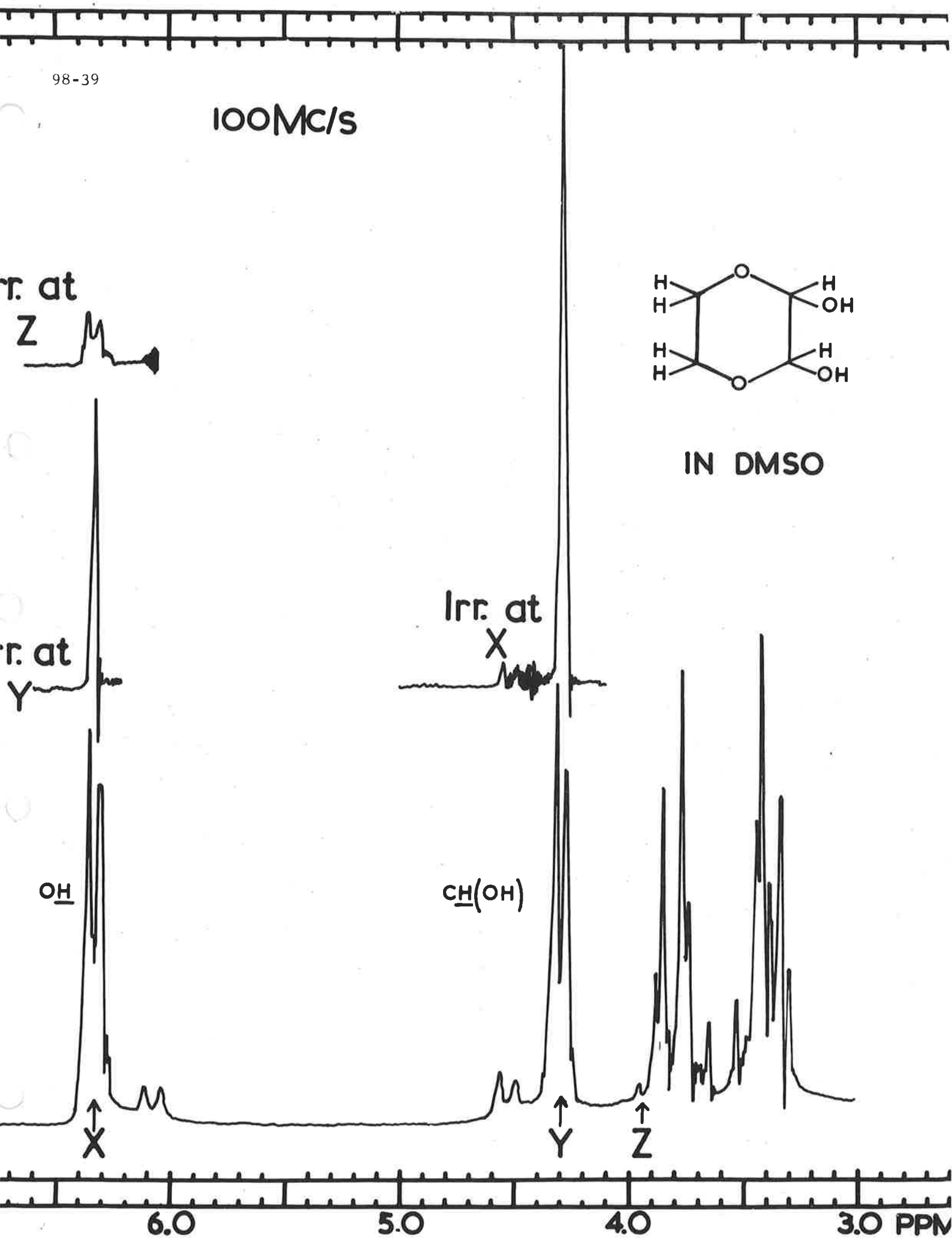
Recently we reported (1) that phenolic protons could readily be detected in solution of phenols in $CDCl_3$ simply by irradiating the small H_2O impurity band in commercial $CDCl_3$ ($\sim$1.5 ppm. downfield from TMS) and observing the collapse of the OH absorption. This effect can also be used when solutions in dimethylsulphoxide are being examined. The spectrum shows the effect of irradiating at $\sim$4 ppm from TMS in a frequency sweep double resonance experiment. Although it is not possible to observe the water absorption at $\sim$ 4 ppm, when this region is irradiated the protons in the water become saturated. The saturated OH groups chemically exchange with the CH protons in the 2,3-dihydroxy 1,4-dioxane and the OH absorption at X is observed to lose intensity. The impurity water absorption is obviously very broad because even when the decoupling irradiation is moved 40c/s from Z (to decouple the CH proton at Y from the OH proton) there is still an appreciable loss in intensity in the decoupled spectrum. It is not known with certainty whether the compound is the cis - or trans - isomer but it is more likely to be in the cis configuration. The small doublets at 4.6 and 6.1 ppm correspond to a small amount of the trans isomer.

(1) Feeney and Heinrich. Chem.Comm.296, (1966).

Yours sincerely,

A. Heinrich Jim Feeney.

A.Heinrich. J.Feeney.


100Mc/s
rr. at
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X
OH
CH(OH)
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Y
Z
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OH
H
H
O
H
OH
IN DMSO
6.0
5.0
4.0
3.0 PPM

הטכניון - מכון טכנולוגי לישראל
TECHNION - ISRAEL INSTITUTE OF TECHNOLOGY

הפקולטה לכימיה
DEPARTMENT OF CHEMISTRY
Physical Chemistry

3.11.1966

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

Dear Professor Shapiro,

Isotope Shifts

We have measured the N^{14} and B^{11} isotope shifts in NH_3D^+ and BH_3D^- ions. The NH_3DNO_3 - NH_4NO_3 system was prepared by dissolving NH_4NO_3 in an acidified 10 percent $D_2O - H_2O$ mixture. The $NaBH_3D$ - $NaBH_4$ system was prepared by equilibrating a $D_2 - H_2$ mixture with $NaBH_4$ at 400°C and about 500 mm/Hg. In both cases enrichment with D was such that mono-substitution to about 30 percent was achieved with minor additions of the higher substituted species. Both N^{14} and B^{11} spectra consisted of a 1 : 4 : 6 : 4 : 1 quintiplet with a superimposed 1 : 3 : 3 : 1 quadruplet (each component of the latter was further split by the D into a 1 : 1 : 1 triplet). The extreme lines were hardly observeable because of their low intensity. The isotopic shift in NH_3D^+ was found to be 0.30 ± 0.04 ppm and 0.17 ± 0.03 ppm in BH_3D^- . Both shifts are to higher fields.

In addition we measured the N^{14} isotope shift between NH_3 and ND_3 (neat liquids at room temperature) and found a value of 2.0 ± 0.3 ppm (to higher field).

We are presently working on other isotopic shifts in similar systems.

Yours, Sincerely

M. Shporer A. Loewenstein

M. Shporer A. Loewenstein

98-41

MOUNT
HOLYOKE
COLLEGE

CARR LABORATORY, DEPARTMENT OF CHEMISTRY

SOUTH HADLEY, MASSACHUSETTS 01075

November 4, 1966

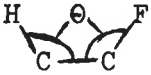
Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois

Dear Barry,

CHANGING CYCLES TILL IT HERTZ

The enclosed clipping comes from the Sunday N. Y. Times of a few weeks ago and may be of some interest to Newsletter readers.

BOND ANGLE AND DIHEDRAL ANGLE DEPENDENCE OF J_{HF}

We've synthesized the six compounds listed on the next page and have found the coupling constants reported in the Table. From this data we have drawn two conclusions: 1) That J_{HF} depends on the dihedral angle in the same way that J_{HH} does except that the values are two to three times larger. This type of dependence has been assumed for some time, but virtually all previous observations of J_{HF}(vicinal) have been on conformationally mobile systems. 2) That J_{HF} is quite dependent on the bond angle Θ: 

Note that the largest value for $J_{HF}(0°)$ is in the unstrained adduct III (31 Hz) and that $J_{HF}(0°)$ decreases to 22 Hz in the anhydride II. It is 20 Hz in the diacid V and decreases to 10 Hz in the anhydride IV. This is in the order that one would expect the angle Θ to increase. On the basis of bond angle Θ we would expect $J_{HF}(0°)$ in the cyclopropane to be even less than 10 Hz. The fact that 18 Hz is observed probably reflects the dependence of J_{HF} on C-C bond length. The C-C bond in cyclopropane is shorter than in the other compounds and would be expected to give a larger vicinal coupling.

I'm eagerly awaiting the arrival of my HA-100 spectrometer, due in January. I'm especially pleased to be getting the new low impedance magnet and solid state power supply.

Sincerely yours,

Kenneth L. Williamson
Asst. Professor of Chemistry

Changing Cycles Till It Hertz

By JACK GOULD

WITH complete returns in the broadcasting season still some days away, there is just time to report on the worldwide controversy involving the late Heinrich Rudolf Hertz. As a result of misguided zeal to honor the German physicist who died in 1894, the cause of precision in speech and writing faces its severest setback in years.

The word "hertz" is rapidly supplanting the word "cycle" as an electronic definition of a recurring period of time in which phenomena repeat themselves in the same order and at the same intervals. The label for the standard unit of measuring frequency, in short, is being switched so subtly and quietly by scientists that enlightened etymologists probably do not know that it may already be too late to fight.

If the layman regards such a prospect as one of life's lesser worries, let him be forewarned. Unless the connoisseurs of clarity in word usage rise to instant opposition, electricity in the home will not long be rated at 110 volts and 60 cycles per second. The current of tomorrow will be alternating at 60 hertz.

The radio dials of the future are similarly threatened by modification. The numbers will remain the same but they will stand for kilohertz (thousands of cycles) on the AM dial and megahertz (millions of cycles) on the FM dial. Moreover, such is the contagion of meddling with good words that the hertzian menace is apt to spread. In another generation, the child's tricycle may be known as the trihertz.

Surrender

Many of the most influential scientific societies in several countries have already substituted "hertz" for "cycles" in their publications. Hugo Gernsback, the grand old man of popular technical publishing, has just announced his abject surrender in an obscure corner of the September issue of Radio Electronics. His magazine is the latest of a large number of American publications to make the change.

Even worse, the new unabridged edition of the Random House Dictionary of the English Language, just out, includes the word "hertz" and defines it as "a unit of frequency, equal to one cycle per second."

A last-ditch stand against the determination of scientists to make themselves a little harder to understand may, however, still be fruitful. The Federal Communications Commission is still holding out against the hertz as of this writing, and QST, the journal of the American Radio Relay League, organization of amateur radio operators, remains sensibly partial to cycles.

The movement to honor Hertz in the most awkward kind of way was initiated a few years ago by learned scientific groups. They believe the physicist is the only major creative pioneer in electronics not to have his name inscribed in the patois of their art. In 1888, Hertz was the first to demonstrate the transmission and reception of a radio wave across a room and he also measured the length and frequency of electromagnetic waves.

Shabby Treatment

There is no doubt Hertz has been shabbily treated. Count Alessandro Volta has been remembered by the volt, the unit of electromotive force; James Watt, the British scientist, by the watt, the unit of power; Georg Simon Ohm, the German physicist, by the ohm, the unit of resistance; André Marie Ampère, the French physicist, by the ampere, the unit of current; Michael Faraday, the British physicist, by the farad, the unit of capacitance; Joseph Henry, the American physicist, by the henry, the unit of inductance; James Clerk Maxwell, the British scientist who developed the theory of electromagnetic waves in 1864, by the maxwell, the unit of magnetic flux.

The argument against adding a hertz to the list rests on whether the act of paying homage to a genius should take precedence over the preservation of a word that means what it says and is not only comprehensible within the scientific community but also outside.

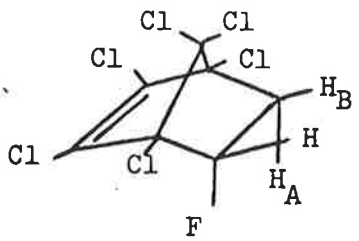
All the other scientists cited above won a permanent niche in the language because they discovered units of measurements that could not be defined accurately by any other existing words. The use of their names was as good a solution as any.

But, in measuring radio waves, Hertz encountered tough luck semantically. Every wave — ocean, sound, radio or light — undergoes a series of repetitive changes most handily summarized as a cycle. A count of the number of such cycles in a second is the basis for fixing the frequency of the wave. The prevailing language, in other words, could accommodate the gist of the findings of Hertz and, for almost 80 years, has been so used in all electronics literature from primer to esoteric thesis. Now the word "cycle" is being arbitrarily dumped to make language a vehicle of history rather than a tool of communication.

Writing in the April bulletin of the Radio Society of Great Britain, Paul Harris, a divertingly articulate member of the society from Sussex, argues that kilohertz might be construed as a fatal number of hertz. He also suggests that if hertz takes the place of cycles, then telephones soon will be known as bells, and screws as archimedeans. Here in New York it would seem only reasonable to drop the word channels from television's lexicon and substitute commercial and noncommercial sarnoffs.

If posterity has been unfair to Hertz, surely the remedy does not lie in making it harder to understand and appreciate what he did. The advantage of using "cycles" is that the word is self-explanatory and can be applied uniformly and consistently to the study of all kinds of waves. Now a word that has no meaning for the layman will have to be accompanied by a definition every time it is used. Dictionaries and encyclopedias are being revised and the supremely simple abbreviation of "kc" for kilocycle is being changed to the puzzling "kHz" for kilohertz.

Since references to frequency are exceptionally numerous in electronics, it would seem that the late-starting Heinrich Hertz may wind up with the best credit line since Count Volta. But it is a pity the scientific community could not ease its guilty conscience without trying to destroy a perfectly good word. It is not too late to start a whole new hertz of thinking on the subject.



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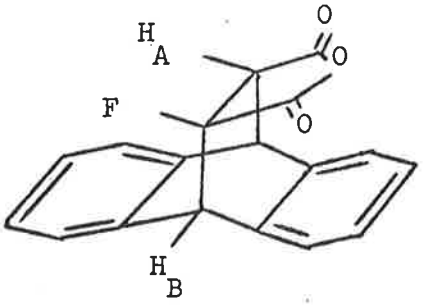

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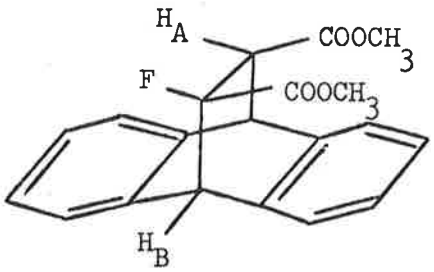

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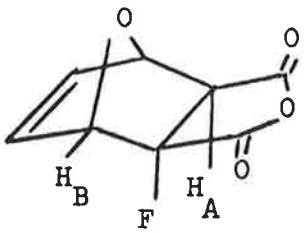

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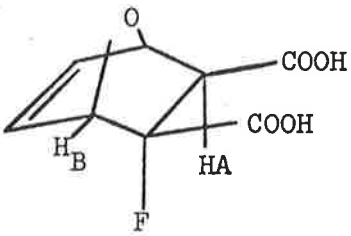

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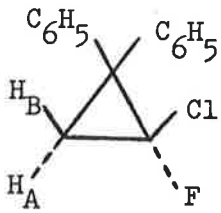


VI

Table I

Vicinal Proton-Fluorine Coupling Constants (Hz)

Approximate Dihedral Angles	$0^o(J_{AF})$	$60^o(J_{BF})$	$90^o(J_{BF})$	$120^o(J_{BF})$	$132^o(J_{BF})$
I	24.7			12.2	
II	22.5	6.0			
III	30.8	3.8			
IV	10.55		2.3		
V	19.8		ca. 2.0		
VI	+17.7				+6.3

University of Toronto
TORONTO 5, CANADA

DEPARTMENT OF CHEMISTRY

1 November, 1966.

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

Dear Dr. Shapiro,

Solvent Effects: Ar-X-Me types in benzene solution

Bowie, Ronayne and Williams (J. Chem. Soc. 1966, B, 785) have recently reported an unusual electronic dependence of the solvent effect of benzene on the methoxyl protons of anisoles. We have observed the same kind of behaviour for the methyl protons of N,N-dimethylanilines (Aust. J. Chem. 19, 835 (1966)) and I have been busy extending my work to anisoles, thioanisoles and N-methylanilines.

All show the same behaviour.....the extent of the upfield shift on moving from deuterochloroform to benzene follows the order of electron withdrawing ability of a group para to the one under study.

	Upfield shift (c/s) of methyl protons			
4-substituent	Me_2N	MeNH	MeS	MeO
NMe_2	-	-	10.4	19.1
OMe	18.7	-	-	23.0
Me	20.3	25.9	24.8	24.8
H	24.2	30.2	28.8	29.1
NO_2	57.7	58.0	50.3	52.8

The trends are explicable in terms of the now-classical model of Klinck and Stothers, in which the solvation complex comprises a molecule of benzene overlaying a solute molecule and located near a centre of positive charge. In a compound Ar-X-Me, the heteroatom X is electron deficient due to donation of electrons to the aromatic ring, and becomes increasingly so

as electronegative substituents increase the capacity of the ring to absorb electronic charge. The increased fractional charge on the heteroatom results in a greater stability constant for the complex and hence greater shielding of the methoxyl etc. protons, as is observed.

There is some supporting evidence for a model of this type. A para methyl substituent might be expected to be much less affected than the group under study, due to its distance from the anisotropic benzene molecule. The upfield shift of the p-methyl protons in each series was: NMe_2, 2.3; NHMe, 2.5; OMe, 9.1; SMe, 15.6 c/s.

The same differential should be observed in the shifts of the aromatic protons. I have made no measurements myself, but the data of Hutton and Schaefer (Can. J. Chem. 43, 3116 (1965)) can be used to show that the carbon tetrachloride - benzene shifts for p-nitroanisole are: protons ortho to methoxyl, 34.1; meta 13.8 c/s.

It is interesting to examine some cases where steric factors might disrupt the complex and upset the shielding patter, but so far the ones I have tried have been unexceptional, the shifts being simply those that one might have predicted on electronic grounds.

2,6,N-trimethyl aniline	20.1	c/s
2,4,6-trichloro anisole	26.2	
2,4,6-tribromo anisole	25.4	
2,4,6-triiodo anisole	24.9	

Yours sincerely,

Ian D Rae

(Ian D. Rae)

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

3 November 1966

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

Dear Barry,

Line Width Transfer by Double Resonance

One is forced to agree with Ed Randall[1] that anthropomorphic expressions such as "spin tickling" are out of place in serious scientific publication. Such needless frivolity may be avoided by replacing the expression throughout the text by some suitable brief descriptive phrase, e.g.

> "A double resonance experiment in which a single NMR line (ω_{pq}) is irradiated with a radiofrequency field ω_2 near exact resonance $\left(|\omega_2 - \omega_{pq}| < \gamma H_2\right)$ with the level of the rf field chosen to be weak compared with the separation from the next-nearest line in the spectrum $\left(\gamma H_2 < |\omega_{pq} - \omega_{st}|\right)$, but strong enough to cause an observable splitting of any connected transition ω_{qr}."

Better still, a less offensive terminology may be substituted. For example, Jim Ferretti[2] has described a double resonance experiment where transitions are identified through the observations of transient "Torrey oscillations". Since the essential characteristics are tickling + oscillation, this is clearly "Spin titillation".

We observed "line-width transfer" quite by accident one day while t*ckl*ng one of the ^{13}C lines of chloroform and recording the splitting of one of the ^{13}C satellites of the proton spectrum during a routine calibration of the rf field strength H_2. It was noticed that the individual components of the proton doublet were appreciably broader than the width of the singlet observed in the absence of H_2. The effect was not merely of instrumental origin since under similar conditions the ^{13}C satellite of bromoform split into a sharply resolved doublet.

The explanation lies in the fact that the ^{13}C lines of chloroform themselves have an appreciable natural line width attributable to spin coupling to the chlorine nuclei that are rapidly relaxed through electric quadrupole interaction with the environment. Although transverse relaxation (T_2) of a ^{13}C nucleus does not affect a coupled proton in the absence of H_2, in a double resonance experiment the axes of quantization of ^{13}C and ^{1}H are no longer the same, and transverse relaxation of ^{13}C generates a randomly varying Z-component of the local field at the proton, causing a

line broadening. It is as if the diffuseness of the offset parameter for ^{13}C $(\omega_2-\omega_{pq})$ has been "transferred" to the proton resonance. Similar effects can be obtained by irradiating a sharp resonance with an incoherent rf field.

Richard Ernst[3] has treated this effect theoretically by the density operator method[4] and has proved that for the spin tickling approximation a much simpler treatment due to Wes Anderson[3] is valid and gives identical expressions for the observed line profiles. In the simple treatment the irradiated line is divided into a large number of narrow vertical slices, each with a characteristic value of the offset parameter $(\omega_2-\omega_{pq})$, and each generating a corresponding doublet in the observed spectrum calculable from the expressions[5]

$$\text{Frequency} = \omega_{qr} \pm \tfrac{1}{2}\{(\omega_2-\omega_{pq}) \pm [(\omega_2-\omega_{pq})^2 + (\gamma H_2)^2]^{\frac{1}{2}}\}$$

$$\text{Relative intensity} = \tfrac{1}{2} \mp \tfrac{1}{2}(\omega_2-\omega_{pq})/[(\omega_2-\omega_{pq})^2 + (\gamma H_2)^2]^{\frac{1}{2}}$$

The required line profile is obtained by integration over all possible values of the offset parameter, allowing for the natural line-width of the observed nuclei—a convolution integral. For the simple case of irradiation in the center of a broad resonance with an rf field γH_2 strong compared with the width of this resonance, the components of the doublet acquire half the width of the irradiated line. The experiment can clearly be used to measure line widths indirectly.

For chloroform the results indicate that the two ^{13}C resonance lines have a natural width of 1.8 cps; we were able to confirm this later by direct observation of the ^{13}C slow passage signal after time averaging. Measurement of the ^{35}Cl line width of chloroform then permits an order-of-magnitude estimate of $|J(^{13}C^{35}Cl)| \approx 50$ cps.

Sincerely yours,

Ray

Ray Freeman
Analytical Instrument Research

1. E. W. Randall and D. Shaw, I.I.T.N.M.R. 91, 38 (1966).
2. J. A. Ferretti and R. Freeman, I.I.T.N.M.R. 88, 40 (1966); J.C.P. 44, 2054 (1966).
3. To be published in J. Chem. Phys.
4. We found a related density operator treatment while browsing through EESTI NSV TEADUSTE AKADEEMIA TOIMETISED [V. Sinivee and E. Lippmaa, Communications of the Estonian Academy of Science, 15, 64 (1966)].
5. R. Freeman and W. A. Anderson, J.C.P. 37, 2053 (1962).

STERLING-WINTHROP RESEARCH INSTITUTE
A DIVISION OF STERLING DRUG INC.
RENSSELAER, NEW YORK

November 7, 1966

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

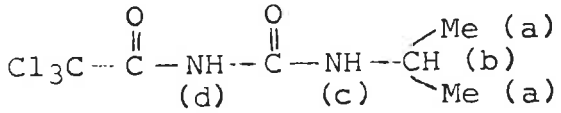
Dear Professor Shapiro:

Characterization of Amines

We are presently examining the application to amines of Goodlett's[1] method for characterization of alcohols. (This has also been applied recently to thiols.)[2]

Goodlett added trichloracetylisocyanate[3] to alcohols to produce the trichloracetyl carbamates and found (10% acetone solution) an amide NH signal at about 10 ppm and α hydrogen shifts for primary alcohols of about 0.7 ppm and for secondary alcohols of about 1.1 ppm.

As an example, we added to a $CDCl_3$ solution of isopropyl amine (Me doublet 1.05 ppm, NH_2 1.17 ppm, CH heptet 3.1 ppm), successive amounts of trichloracetylisocyanate until no forther change of the spectrum was noted. The NMR spectrum of the final product,



showed: (a) Me doublet 1.25 ppm (b) CH octet 4.03 ppm, (c) NH doublet (broad peaks) 7.83 ppm, and (d) a broad NH at 10.2 ppm (all J's about 7 cps).

This experiment is being carried out for a variety of amines and the results will be reported at a later date.

Sincerely,

R. K. Kullnig

R. K. Kullnig

C. M. Martini

C. M. Martini

mwg

[1] V. W. Goodlett, Anal. Chem. 37 431 (1965)

[2] P. E. Butler, W. H. Mueller, Anal. Chem. 38 1407 (1966)

[3] Commercially available from Distillation Products Industries, Division of Eastman Kodak.

PRINCETON UNIVERSITY
DEPARTMENT OF CHEMISTRY
PRINCETON, NEW JERSEY 08540

Frick Chemical Laboratory

November 7, 1966

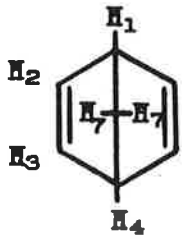
Dr. B.L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Technology Center
Chicago, Illinois

Dear Barry:

Bicycloheptadiene Spectral Analysis: Comparison of '66 Vintage with Earlier

Reilly (IITNMR 97-48) has reported preliminary results of the analysis of the proton coupling in bicycloheptadiene (I), using a 100 MHz spectrometer, decoupling techniques, and ^{13}C satellites. His investigations were prompted by the observation of discrepancies from the published analysis of Mortimer at 40 MHz (J. Mole. Spectry., 3, 528 (1959)).

We have already observed this error in Mortimer's work (Laszlo and Schleyer, J. Am. Chem. Soc., 85, 2017 (1963); 86, 1171 (1964)). Tori, Muneyuki and Tanida (Can. J. Chem., 41, 3142 (1963)) have also reported on the analysis of the spectrum of norbornadiene. It is gratifying that both of these studies, at 60 MHz, are in good agreement with Reilly's report, as the summary below indicates.



Investigators	$J_{1,2}$	$J_{1,3}$	$J_{2,3}$	$J_{1,7}$
Mortimer	2.9 Hz[a]	0.95 Hz[a]	3.45 Hz[a]	1.65 Hz[a]
Laszlo-Schleyer	2.7[b]	0.95[b]	5.05[b]	1.5[b]
Tori, *et al.*	2.8	1.1	5.2	1.6
Reilly	2.7[b]	0.9[b]	5.2[a,b]	-

[a]Complete analysis. [b]Analysis by means of ^{13}C satellites.

Sincerely yours,

Pierre Laszlo
Asst. Professor

Paul von R. Schleyer
Professor

PL-PSmb
cc: C.A. Reilly

98-51

DIVISION OF APPLIED CHEMISTRY
DIVISION DE CHIMIE APPLIQUÉE

CABLE ADDRESS
ADRESSE TÉLÉGRAPHIQUE "RESEARCH

PLEASE QUOTE FILE No.
No DE DOSSIER À RAPPELER

NATIONAL RESEARCH COUNCIL
CONSEIL NATIONAL DE RECHERCHES
CANADA

OTTAWA 2,

8 November 1966.

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

Dear Barry:

We have been re-investigating the fluorine resonance spectra of phosphorus pentafluoride and some of its complexes and have obtained some results which may be of interest to your readers.

From vapor pressure measurements it has been shown that PF_5 behaves ideally in CH_2Cl_2 solution. The fluorine resonance spectrum shows two lines interpreted as five equivalent fluorine atoms spin coupled to phosphorus. The variation of the width of these lines with temperature and concentration is shown in the accompanying Figure. The widening of the lines with decreasing concentration or increasing temperature must be due to either an increasing rate of intermolecular exchange (effectively averaging out the spin coupling) or to a dereasing rate of intramolecular exchange (such that the chemical shift differences between axial and equatorial fluorines are not completely averaged). Since it is difficult to conceive of an intermolecular reaction whose rate is accelerated with decreasing concentration of reactants it is preferable to suppose that a decreased rate of intramolecular exchange is being observed.

The temperature dependance of the line width may then be explained in the following manner. There is approximately an equal volume of liquid and vapor in the sealed N.M.R. tube. With rising temperature more of the PF_5 will be in the vapor phase decreasing the concentration in solution. If the effect of concentration in slowing the rate is greater than that of temperature in increasing it the observed effects can be explained.

.... 2

The decrease of concentration in solution with increasing temperature has been demonstrated for boron trifluoride.

In order for intramolecular exchange to happen a PF_5 molecule must collide with another to form some transient intermediate (probably with fluorine bridging) allowing intramolecular exchange of axial and equatorial fluorines to occur but not intermolecular exchange. It was found that BF_3, which is known to form fluorine bridges, is also effective in causing intramolecular exchange in PF_5.

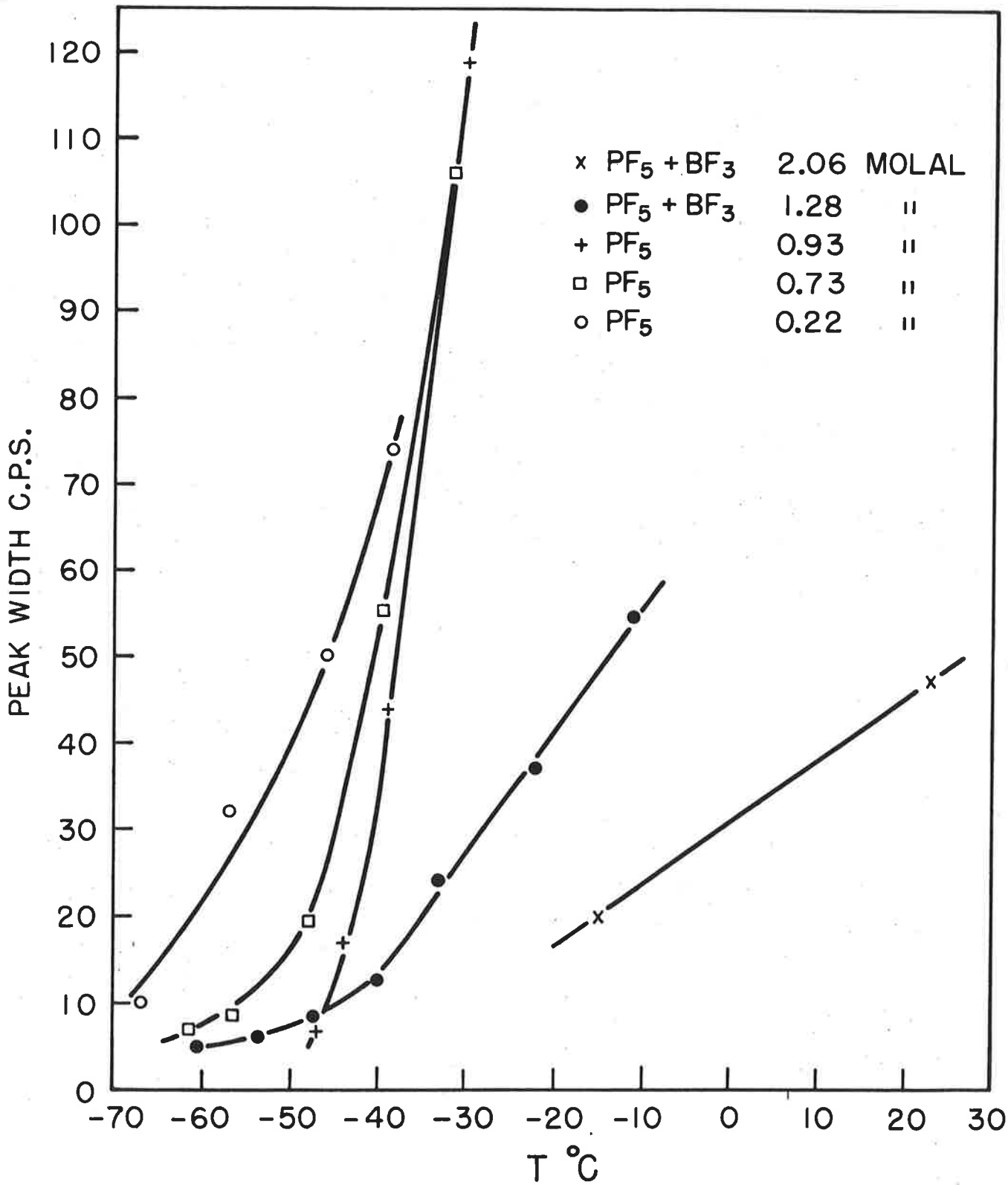
The continued receipt of this collection of letters and bibliography is greatly appreciated.

Yours truly,

Syd.

S. Brownstein.

SB/adg


PEAK WIDTH C.P.S.
T °C
× PF5 + BF3 2.06 MOLAL
● PF5 + BF3 1.28 "
+ PF5 0.93 "
□ PF5 0.73 "
○ PF5 0.22 "
0
10
20
30
40
50
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90
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120
-70
-60
-50
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-20
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NORTH CAROLINA STATE UNIVERSITY

SCHOOL OF PHYSICAL SCIENCES AND APPLIED MATHEMATICS

DEPARTMENT OF CHEMISTRY
BOX 5247 ZIP 27607

November 9, 1966

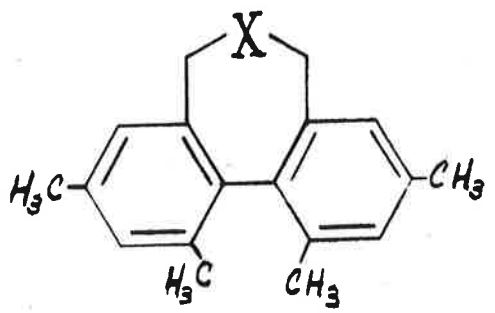
Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

NMR of BIPHENYL METHYL GROUPS - NMR ABSTRACT CARDS

Dear Dr. Shapiro:

We regret the delay in submitting our renewal subscription. Please credit this to the account of Dr. C. G. Moreland.

Continuation of earlier work carried out in collaboration with Professor Mislow [see, J.Am.Chem.Soc.,86,1710(1964).] has led us to the system I. As previously described, the substituent X uniquely determines the dihedral angle and thus the "electronic view" of the distal phenyl ring that each methyl group ortho to the phenyl-phenyl bond experiences. Earlier models (loc.cit.) lacked the methyl groups para to the phenyl-phenyl bond and thus had no internal reference. Thanks to the generosity of Professor M. S. Newman, the compounds I have been synthesized and the pertinent nmr data is collocated in Table I.



I

TABLE I

X	Dihedral Angle	τ ortho Methyl	τ para Methyl	Δτ
single bond	15°	7.79	7.69	0.10
O	44°	7.84	7.64	0.20
NCH_3	46°	7.84	7.63	0.21
S	57°	7.96	7.66	0.30
bimesitylene	(90°)	8.15	7.70	0.45

The trend of increasing chemical shift of the signal due to the ortho methyl group is to be expected. More significantly the correlation of the difference in chemical shift ($\Delta\tau$) of ortho and para methyl groups with calculated dihedral angle is encouraging and rules out any possibility that the substituent X plays a direct role in determining the shielding of the ortho methyl groups.

We hope to compare these findings with predications based on Johnson-Bovey calculations and will submit the completed study for publication in the near future.

We have a subscription to "N-M-R Abstract Cards" and would like to search them directly for nmr studies of Hydrogen Bonding but have been unable to "crack the code". Any suggestions would be gratefully received.

We have recently enhanced our nmr facility (HA-100) by the addition of a Varian C-1024 Computer of Average Transients and a P^{31} unit.

Sincerely yours,

George H. Wahl, Jr.
Assistant Professor

GHW/mt

UNIVERSITY OF ILLINOIS
Department of
CHEMISTRY AND CHEMICAL ENGINEERING
URBANA
61801

The William Albert Noyes Laboratory

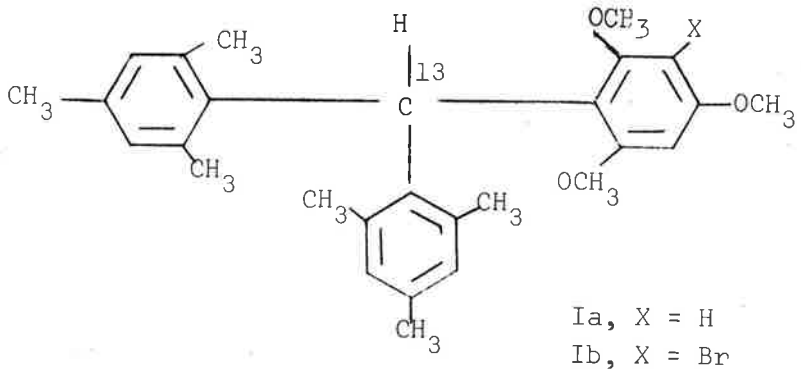
November 9, 1966

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

Dear Barry:

"A Decrease in $J_{^{13}C-H}$ with Increased p-Character in a C-H Bond"

Further work on the sterically hindered triarylmethane, Ia, which was the subject of our last letter to you, has included a study of $J_{^{13}C-H}$ for the methane C-H bond.



Ia, X = H
Ib, X = Br

The value of $J_{^{13}C-H}$ is 126.0 ± 0.2 Hz for triphenylmethane and several substituted analogs, including 2,2',2",6,6',6"-hexamethoxytriphenylmethane. For the more hindered molecule Ia, in a sample containing ca. 65% ^{13}C at the central position $J_{^{13}C-H}$ is 119.6 Hz.

At present Dr. Iain Paul is collaborating with us in a single-crystal X-ray structure determination on Ib, containing the heavy atom, bromine. It is clear from preliminary results that the angles between the central C-C bonds of Ib are considerably greater than tetrahedral. When the refinement of structure is complete we should be able to say more about the possible relation of $J_{^{13}C-H}$ and orbital hycridization in C-H bonds having less than 25% s-character.

Yours,

J. C. Martin (with M. J. Sabacky)

f

98-57

UNIVERSITY OF CALIFORNIA, LOS ANGELES

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DEPARTMENT OF CHEMISTRY
LOS ANGELES, CALIFORNIA 90024

November 9, 1966

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

Subject: Ring Inversion in Cyclohexane-$\underline{d}_{11}$. Double Resonance Measurements of Exchange Rates With A Field-Frequency Lock Spectrometer

Dear Barry:

Your dire warnings lead me to send in this subscription to IIT NMR Newsletter. Despite my apparent reluctance to send you something sooner, let me say that I greatly appreciate receiving the Newsletter and that I would hate to be cut off from the wonderful service that you are performing.

Dr. A. J. R. Bourn and I have measured rate constants for ring inversion in cyclohexane-$\underline{d}_{11}$ using the double resonance method of Forsén and Hoffman.[1] We have modified the fast repetitive field sweep procedure,[1] since with an internal field-frequency lock spectrometer, it is very easy to remain exactly at the center of a line, and thus the intensity of a line can easily be monitored over a period of time. We have used the highest possible resolution (i.e. spinning, cf. ref. 1), and we were careful to avoid saturation of the line whose intensity was being monitored by using very low radiofrequency power.

At -105° the proton spectrum of C_6HD_{11} in CS_2 solution consisted of two very sharp lines when the deuterons were decoupled. The Figure shows the intensity of the high-field (axial proton) line as measured on a strip-chart recorder. At the extreme left in the

Figure, it can be seen that the height of the line remains constant with time (the pen position when the spectrometer is off-resonance is at the "baseline"). At the position marked (1) the low-field line (equatorial proton) was suddenly strongly irradiated, so that the equatorial protons were saturated, i.e. their spin populations became equalized. The intensity of the axial proton band gradually diminishes as a result of the exchange of equatorial for axial protons and vice versa. The intensity does not go to zero, however, because of spin-lattice relaxation, which tends to maintain the thermal equilibrium population of the spin states for the axial protons. As Forsén and Hoffman have shown, it is easy to obtain a relationship between the ratio of the intensity before irradiation to that after irradiation in terms of $\underline{T}_1$ and τ (the average lifetime of protons in one site). Also, the decay of the intensity is a simple exponential which depends on $\underline{T}_1$ and τ, so that both $\underline{T}_1$ and τ can be obtained.

At (2) the irradiation of the equatorial proton was stopped. The intensity of the axial proton line was then observed to increase and ultimately to reach a value that it had at the beginning of the experiment. The recovery is the sum of two exponentials, but in our case of equal $\underline{T}_1$'s at the two sites, it is still only a function of $\underline{T}_1$ and τ.

We have made a number of measurements at temperatures between -97° and -117°. Over this range, τ varied from 3.8 sec. to 240 sec. and $\underline{T}_1$ was about 27 sec.

These results give the same $\Delta\underline{H}^*$ (10.8 kcal/mole) and $\Delta\underline{S}^*$ (2.8 e.u.) as those we obtained at higher temperatures by the use of the conventional line shape procedure. (IIT N-M-R Newsletter No. 87, p.42)

These activation parameters, however, do not agree with those obtained by Allerhand, Chen and Gutowsky with the spin-echo method[2] ($\Delta\underline{H}^*$ = 9.1 kcal/mole, $\Delta\underline{S}^*$ = -5.8 e.u.).

---

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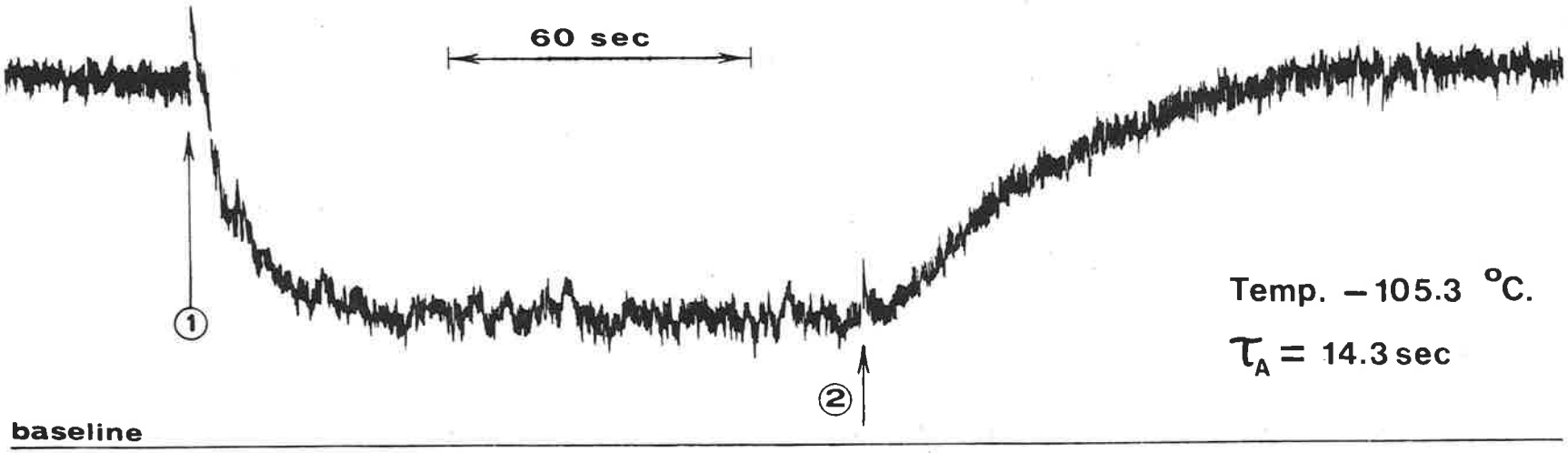
Yours sincerely,

Frank

F. A. L. Anet
Professor of Chemistry

FALA/oh



Decay and recovery of magnetization for the high-field line of cyclohexane-$\underline{d}_{11}$. Arrow 1 shows the onset of saturation of the low-field line, and arrow 2 the removal of the saturating field.

8th ENC (E X P E R I M E N T A L N M R C O N F E R E N C E)

8th ENC Committee

J. B. Stothers, Chairman
P. R. Shafer, Chairman-elect
B. L. Shapiro, Secretary Treasurer
A. A. Bothner-By, Local arrangements
A. W. Douglas
E. W. Garbisch

Address Correspondence to:

Prof. B. L. Shapiro,
Dept. of Chemistry,
Illinois Institute of Technology,
CHICAGO, Ill. 60616.

The Eighth Experimental NMR Conference will be held Thursday through Saturday, March 2 - 4th, 1967 at the Mellon Institute, Pittsburgh, Pennsylvania. This conference is devoted to advances in techniques, experimental design and instrumentation; it does not compete with Workshops or courses offering an introduction to the field.

The sessions consist mainly of invited papers, although a limited number of contributed papers may be accepted. Manuscripts or applications to read a paper should be sent to the appropriate session chairman or to the conference chairman* for his decision regarding their disposition. Topics to be included in the 8th ENC are listed below together with the sessional chairmen.

Topic	Chairman	Address
Biological Applications of NMR	R. G. Shulman	Bell Telephone Labs, Murray Hill, N.J., 07971
Computer-Spectrometer Interfacing	C. A. Reilly	Shell Development Co., Emeryville, Calif, 94608
Instructional Methods	P. Bender	Dept. of Chemistry, University of Wisconsin, Madison, Wis. 53706
Less-Receptive Nuclei	R. J. Gillespie	Dept. of Chemistry, McMaster University, Hamilton, Ontario, Canada.
Multiple Resonance	J. H. Noggle	Dept. of Chemistry, University of Wisconsin, Madison, Wis. 53706.
New Developments (i.e. Misc.)	B. L. Shapiro	Dept. of Chemistry, Illinois Inst. of Technology, Chicago, Ill. 60616.
Oriented Molecules	L. C. Snyder	Bell Telephone Labs. Murray Hill, N.J. 07971
Pulse Techniques	L. W. Reeves	Dept. of Chemistry, University of British Columbia, Vancouver 8, B.C., Canada.

* J. B. Stothers, Dept. of Chemistry, University of Western Ontario, London, Canada.

ADDITIONAL INFORMATION AND THE APPLICATION TO ATTEND 8th ENC MAY BE OBTAINED ONLY BY RETURNING THE ENCLOSED POST CARD (DON'T FORGET TO PUT A STAMP ON THE POST CARD!) OR BY OTHERWISE INFORMING EITHER THE CHAIRMAN OR THE SEC'Y TREAS. OF YOUR INTEREST. PLEASE BE SURE TO DO THIS PROMPTLY, IF YOU CONTEMPLATE ATTENDING; IT MAY BE NECESSARY TO LIMIT ATTENDANCE BECAUSE OF RESTRICTED PHYSICAL FACILITIES.

The 1967 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy will be held March 5 - 10, 1967. The program will include a symposium on ^{13}C NMR Spectroscopy."

Instructions for Speakers and Contributors

I. Lengths of invited and contributed papers will be assigned by Session Chairmen so as to leave ample time for discussion in each session.

II. Abstracts and supplementary material (figures, tables, etc.) in no case exceeding 6 pages should be sent, by February 1, to:

Dr. A. A. Bothner-By,
8th ENC Arrangements
Mellon Institute
4400 Fifth Avenue
Pittsburgh, Pennsylvania 15213.

They will be printed and distributed to the conferees. Additional material, either too long or too late to have been printed, may be reproduced by the author and arrangements will be made to distribute it at the Conference. A distribution center will also be provided for commerical material of possible interest to the conferees. Authors are urged to submit detailed abstracts and drawings, schematics, spectra, etc., if at all possible. Participants in past conferences have found such a printed record to be quite valuable. In addition, the presentation of undigestible masses of data, complicated schematics, etc. on slides or blackboard is obviated, saving time and easing communication.

Abstracts of papers and contributions not accepted for formal presentation will be printed in a Supplementary Program and may be discussed at any appropriate session if time permits. The deadline and length limitation are the same as for other abstracts.

Projectors for 3 $\frac{1}{2}$ x 4 inch and 2 x 2 inch slides will be available at all sessions. Unusual requirements of any sort should be transmitted in advance to the Session Chairman.

BP RESEARCH CENTRE

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ENGLAND

CARBON-13 ABSORPTION MODE SPECTRA OF PETROLEUM FRACTIONS

Complex mixtures of compounds such as petroleum stocks can be characterised by hydrogen NMR spectroscopy. However, such characterisations (1) depend on semi-empirical correlations between hydrogen and carbon types present in these stocks and also require additional data in the form of per cent weight carbon, hydrogen, sulphur and a molecular weight.

Carbon-13 NMR spectroscopy seemed to offer (2) a good method for the direct determination of carbon types in these complex mixtures, but until recently suitable instrumentation has not been available. Friedel and Retcofsky (3) have explored the use of rapid passage dispersion mode and have obtained carbon-13 spectra of coal and petroleum fractions. However, this mode of operation gives distorted band shapes which makes accurate integrated spectra difficult to obtain.

Recently computer systems have appeared which enable the weak absorption mode spectra of carbon-13 nuclei to be accumulated. These spectra have the enormous advantage in that they can be accurately integrated. Such a spectrum (obtained on our behalf by Perkin-Elmer Limited of Beaconsfield, Bucks, England) and its integral obtained from a lubricating oil is shown in Figure 1. It can be seen that the aromatic and aliphatic carbon types are clearly resolved and the bands show much fine structure which is ordinarily lost in dispersion mode spectra. The integrated spectrum gives C_A (carbon atoms in aromatic rings) as 21.5 per cent, whilst other spectroscopic methods (infra-red and hydrogen NMR) gave a value of

24 per cent. Absorption mode carbon-13 spectra can also show fine detail resulting from $C^{13}CH$ couplings and the ability to obtain and interpret this hyperfine structure enormously increases the utility of the spectra in molecular structure studies (4). The aliphatic carbon band, Figure 1, shows much fine detail but at this stage of development unambiguous interpretation is difficult.

Holm (2) indicated in his early work the prospect of estimating carbon in naphthene rings (C_N) and in paraffinic chains (C_P). This suggestion needs further exploration however, as a method will have to be devised for the analysis of the aliphatic carbon resonance band. However, there is little doubt that accurate measurement of carbon types and their relative amounts on a routine basis by carbon-13 adsorption mode spectroscopy is a most important advance.

S. Knight

SK/CM

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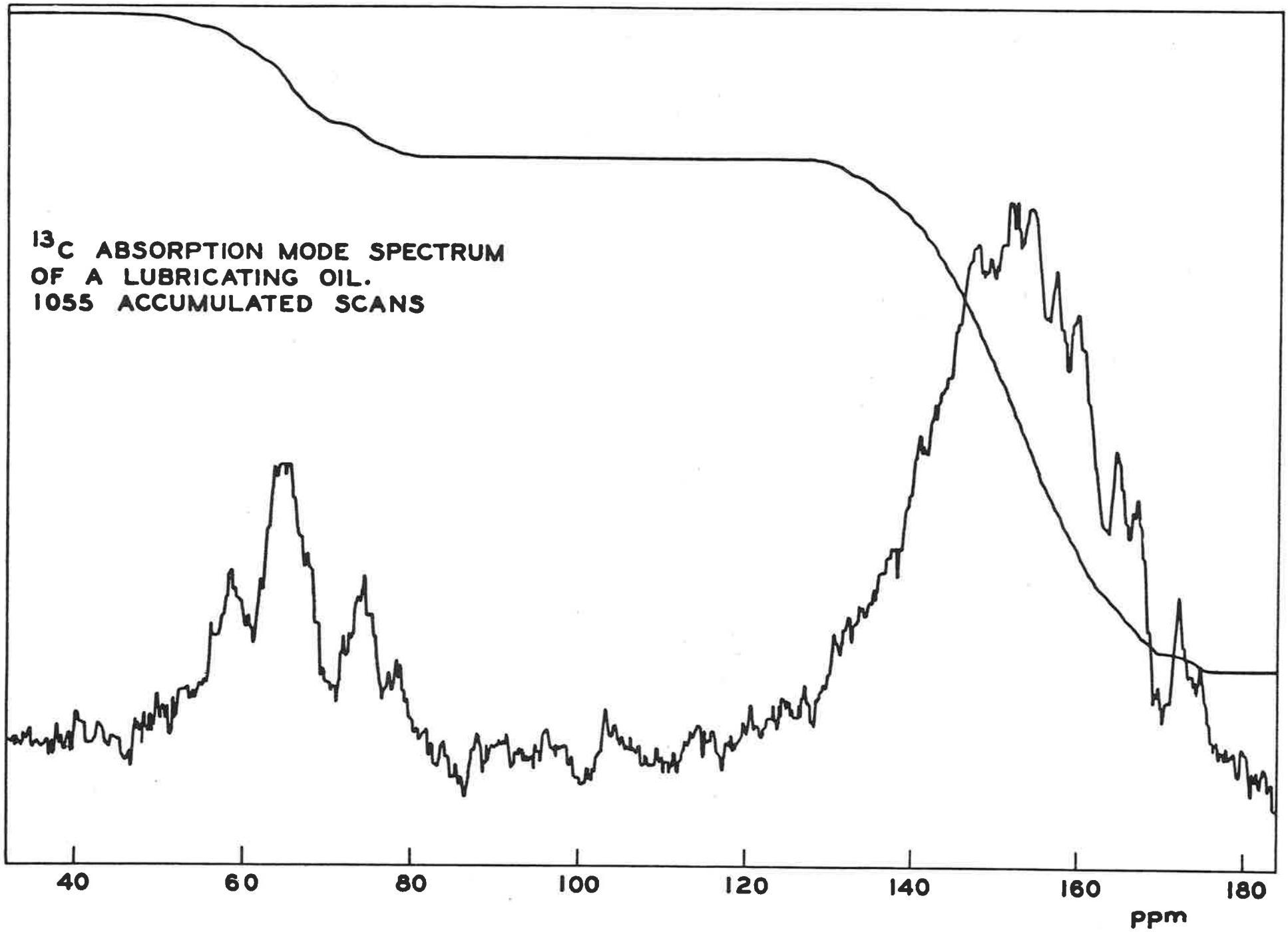

^{13}C ABSORPTION MODE SPECTRUM
OF A LUBRICATING OIL.
1055 ACCUMULATED SCANS
40
60
80
100
120
140
160
180
ppm

98-64

98-65

UNIVERSITY OF VIRGINIA
DEPARTMENT OF CHEMISTRY
COBB CHEMICAL LABORATORY CHARLOTTESVILLE, VA.

November 14, 1966

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

Dear Professor Shapiro:

Two postdoctoral positions available.

We much appreciate your restoring our department to the IIT NMR Newsletter mailing list. Unaccountably our subscription was allowed to lapse last year while I was on leave as program director in the Molecular Biology Section of the National Science Foundation. The importance of receiving the Newsletter is well demonstrated in our case, as we unwittingly and unwillingly duplicated some of the research on chemical shift non-equivalence in aminoacylglycylpeptides reported by Oleg Jardetzky and Asao Nakamura in issue number 88. We shall make a more substantive contribution to the Newsletter in the near future.

In the meantime I should like to bring to the attention of readers two postdoctoral positions that are available in our department. We now have a Varian A-60 instrument,with an HA-100 machine with a time averaging computer due to arrive within two months. The starting dates for both positions are flexible, but dates as early as January 1967 are possible.

Dr. John E. Bloor in our department would prefer candidates with some nmr experience to study the nature of charge transfer complexes in biological systems and conformations of drug molecules. Interested persons may write to him directly at the letterhead address.

I am particularly interested in collaborating with someone in undertaking studies of coenzyme and inhibitor binding to enzyme molecules. There are, of course, many other problems that would also be worth undertaking on our new machine. We also employ circular dichroism as a companion technique.

We were pleased to learn that you and your family on your brief trip to Charlottesville "were most pleasantly struck by your delightful city, area and school." At the same time I am chagrined that I was not here to meet you. On your next trip I hope you will plan to stay longer, and by all means stop in and let us talk.

Sincerely yours,

R. Bruce Martin
Professor of Chemistry

RBM:era

FACULTE DES SCIENCES
DE MONTPELLIER

SERVICE CHIMIE P C 1

R. JACQUIER
Professeur

E N S C M 8, Rue de l Ecole Normale

Tél (67) 72 14 50

MONTPELLIER le 10 Novembre 1966

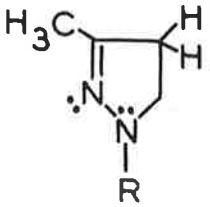
Professeur B.L. Shapiro
Department of Chemistry
Illinois Institute of Technology
CHICAGO, Illinois 60616

INFLUENCE DU DOUBLET D'AZOTE sp^3 SUR UNE CONSTANTE DE COUPLAGE ELOIGNEE DANS LE CYCLE PYRAZOLINE-2.

Cher Professeur Shapiro,

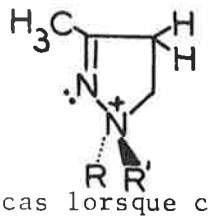
Deux articles récents (1,2) signalent que dans les systèmes hétérocycliques, les constantes de couplage ^2J et ^3J dépendent du recouvrement des liaisons C-H avec les doublets libres des hétéroatomes adjacents.

Nous voulons signaler l'influence du doublet de l'azote en 1 des pyrazolines-2 sur une constante de couplage $^4J_{CH_3-H}$, aucune des liaisons impliquées n'étant adjacente à l'azote :

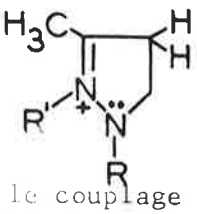


Dans les pyrazolines-2, $^4J_{CH_3-H}$ a une valeur proche de 1 Hz, quel que soit R méthyl, phényl, p-nitrophényl, dinitro-2,4 phényl, trinitro-2,4,6 phényl (3) .

Par contre, dès que l'on supprime le doublet par quaternarisation ($R'=CH_3$) ou par protonation ($R'=H$)



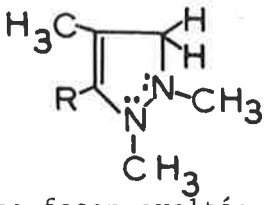
le couplage disparaît, ce qui n'est pas le cas lorsque c'est le doublet sp^2 de l'azote N_2 qui est bloqué :



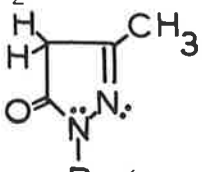
(3,4)

Dans ce dernier cas, cependant, le couplage est difficile à observer car les signaux sont élargis (relaxation quadrupolaire de l'azote $\overset{+}{N}_2$)

Les pyrazolines-3 :

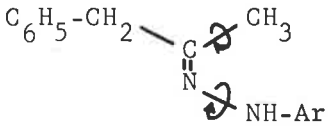


présentent le couplage d'une façon exaltée (5) ($^4J_{CH_3-H}$ = 1,4 Hz)(deux azotes sp^3), tandis que dans les Δ_2-pyrazolones-5 (azote $^3N_1$ de type amide) :



il n'apparaît pas (6,7). R (CH_3,C_6H_5)

Nous avons vérifié que, comme dans le cas de ^2J et ^3J, une certaine géométrie (parallélisme p-σ) est nécessaire à l'intervention du couplage ; ainsi on ne l'observe pas pour des produits tels l'hydrazone suivante :



étant donné la libre rotation autour des liaisons C—C et N—N.

Des recherches en vue de préciser et de généraliser les observations précédentes sont en cours dans notre Laboratoire.

Veuillez croire, Cher Professeur Shapiro, à l'assurance de nos sentiments les meilleurs.

R. JACQUIER.

J.-L. AUBAGNAC.

P. BOUCHET.

J. ELGUERO.

C. MARZIN.

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* * * * *

VARIAN AG • SWITZERLAND

Research Laboratory · Service Center · Klausstrasse 43 · Tel. 051 / 47 24 20 · Zürich 8 · Telex 58 444

Zürich, November 14, 1966

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

U.S.A.

Dear Professor Shapiro,

Attilio has given me your timely reminder and suggested that I might write something for the IIT NMR Newsletter.

Although I am essentially an electron resonance spectroscopist I have recently become interested in NMR because of its ability to measure very small proton hyperfine splittings in stable free radicals. You will recall that the paramagnetic shift of a proton line in a free radical relative to that in a similar diamagnetic species is directly proportional to the isotropic coupling between the unpaired electron and the proton[1,2] and so gives the absolute magnitude of this coupling. Of course the measurement of the spin distribution in a radical is sometimes useful however I have a more specific problem in mind.

The electron resonance spectra of a biradical depends critically on the magnitude of J the scalar exchange interaction between the two unpaired electrons. In the case of the nitroxide biradicals[3] if $J > a^N$ where a^N is the nitrogen hyperfine splitting the EPR spectrum contains five lines whereas if $J < a^N$ the spectrum contains just three lines[4]. Since a^N is about 40 Mc/s if the spectrum does possess just three lines we cannot put a very low limit on J. However the coupling to the protons is only about 0.3 Mc/s and so observation of the NMR spectra of biradicals should allow us to put a much lower value to this limit.

./.

At the moment I am studying experimentally and theoretically both mono-, bi- and tri-radicals kindly given to me by Prof. A. Rassat of Grenoble and Prof. E.G. Rosantsev of Moscow. The preliminary results are not readily interpreted but do indicate that this is a particularly interesting problem.

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Short title: NMR of stable mono-, bi- and tri-radicals.

Yours sincerely,

G.R. Luckhurst

Dr. G.R. Luckhurst
Research Chemist
VARIAN AG

GL/ik

MELLON INSTITUTE
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