Joseph B. Lambert

Illinois Institute of Technology N-M-R Newsletter

No. 105
JUNE, 1967

OVER

PLEASE NOTE DATES ! ! ! !

Deadline Dates: No. 106 - 10 July 1967
No. 107 - 9 August 1967

A monthly collection of informal private letters from laboratories of NMR. Information contained herein is solely for the use of the reader. Quotation is not permitted, except by direct arrangement with the author of the letter, and the material quoted must be referred to as a "Private Communication".

New, Temporary Mailing Address for Contributions

to the IIT NMR Newsletter

I will be on a leave of absence from IIT for approximately one year beginning in August. Through the kindnesses of the PHS and the hospitality of John Baldeschwieler, my address will be:

Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

Please address all Newsletter contributions, enquiries, complaints, checks, etc., to me at the above address, beginning August 10, 1967. The Newsletter will continue to appear monthly without interruption (and hopefully without more than the usual difficulties) during my absence from Chicago.

BLS

Bernard L. Shapiro
26 June 1967

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May 19, 1967

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

Dear Barry:

Re: Tuning and Performance of a Varian HA-100 Spectrometer

I have been checking out our HA-100 spectrometer which we recently converted from a DP-60. Some of the results may be of interest to readers of the NMR newsletter.

Tuning - After setting the seven field homogeneity controls according to the procedure outlined in the instruction manual (chloroform signal on the oscilloscope with the instrument in the HR recurrent-sweep mode) we found the y, curvature, and fourth order always required further adjustment using the recording of the acetaldehyde quartet in the HA mode to get really good performance. As usual, the traces from forward and reverse sweeps were superimposed to show the line shape free of ringing. The pattern indicates what changes must be made to optimize the field shape. The y-setting has very little effect on symmetry, but changes peak height and width (Figure 1). Curvature and fourth order have more effect on symmetry of the pattern. More curvature lowers the forward peak, makes the forward ring larger in amplitude and the aft turn sharper (Figure 2). The fourth order control has the opposite effect and was found to be 2.4 times more sensitive (Figure 3). Thus, an increase of 5 units in fourth order requires an increase of 12 units in curvature setting to maintain symmetry.

The fourth order control affects a larger area of the magnet and has less effect in the center of the spot than the curvature does. Optimizing the curvature first for best symmetry then the y for best signal amplitude gives a skewed pattern (Figure 4) when the fourth order setting is incorrect. The forward-reverse pattern shows ringing of approximately the same peak-to-peak amplitude on each side but the right side is displaced upward. This indicates that the curvature is optimized but the fourth order setting is too low. Most of the active field is flat but the edges drop off to make a flat-topped dome. The loosely-coupled nuclei farther from the center of the magnet resonate at higher applied field and the forward trace has a small amount of tailing near the baseline. This is more readily seen in the right-hand trace

of Figure 4 where a sweep rate of 5000 sec. was used. The two central lines tail off badly. A high forth-order setting has the opposite effect, the active field is dished at the edges, and the trace is a mirror image of Figure 4. At an extremely high forth order setting, the trace actually shows humps to the left of the two central lines (Figure 5). At optimum setting, the lines of the spectrum are symmetrical and the trace returns almost to the baseline between the peaks (Figure 6).

Performance - Our spectrometer gives a signal-to-noise of 30-35, spinning side bands of 0.2 per cent, chloroform line width measured at the top of the C^{13} satellites = 3.9 cps, at one-fifth this height = 6.5 cps, line width at half height of first line of acetaldehyde quartet = 0.13 cps at 5000 second sweep, 0.28 cps at 500 second rate, 0.3 cps measured on line 5 of ODCB at 500 second sweep rate.

Very truly yours,

George Slomp

George Slomp, Ph.D.
Physical and Analytical Chemistry

GS/elm

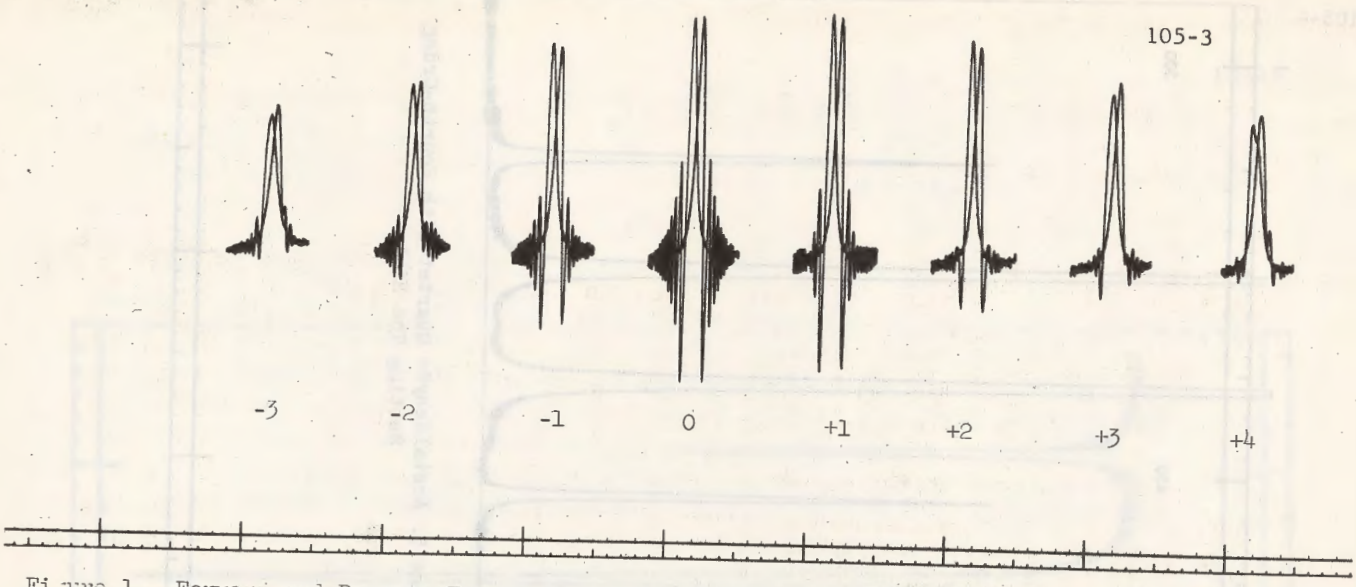

Figure 1. Forward and Reverse Traces of the First Line of the Acetaldehyde Quartet After Turning the Y Control the Indicated Number of Units.

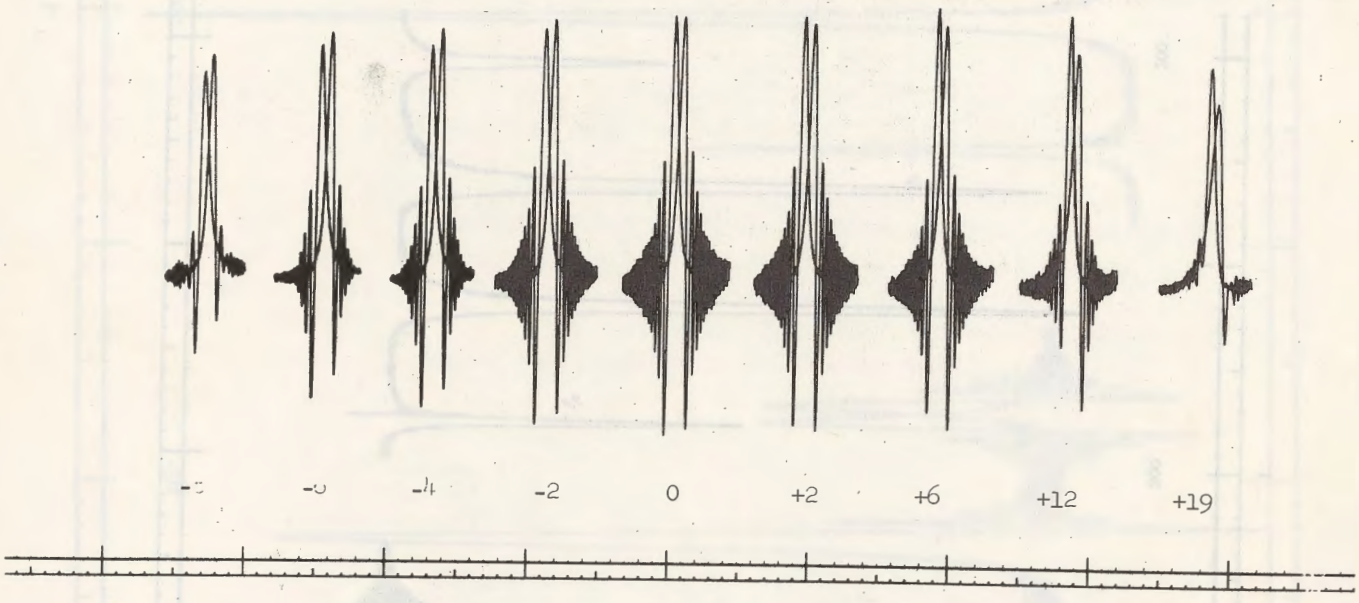

Figure 2. Forward and Reverse Traces of the First Line of the Acetaldehyde Quartet after Turning the Curvature Control the Indicated Number of Units.

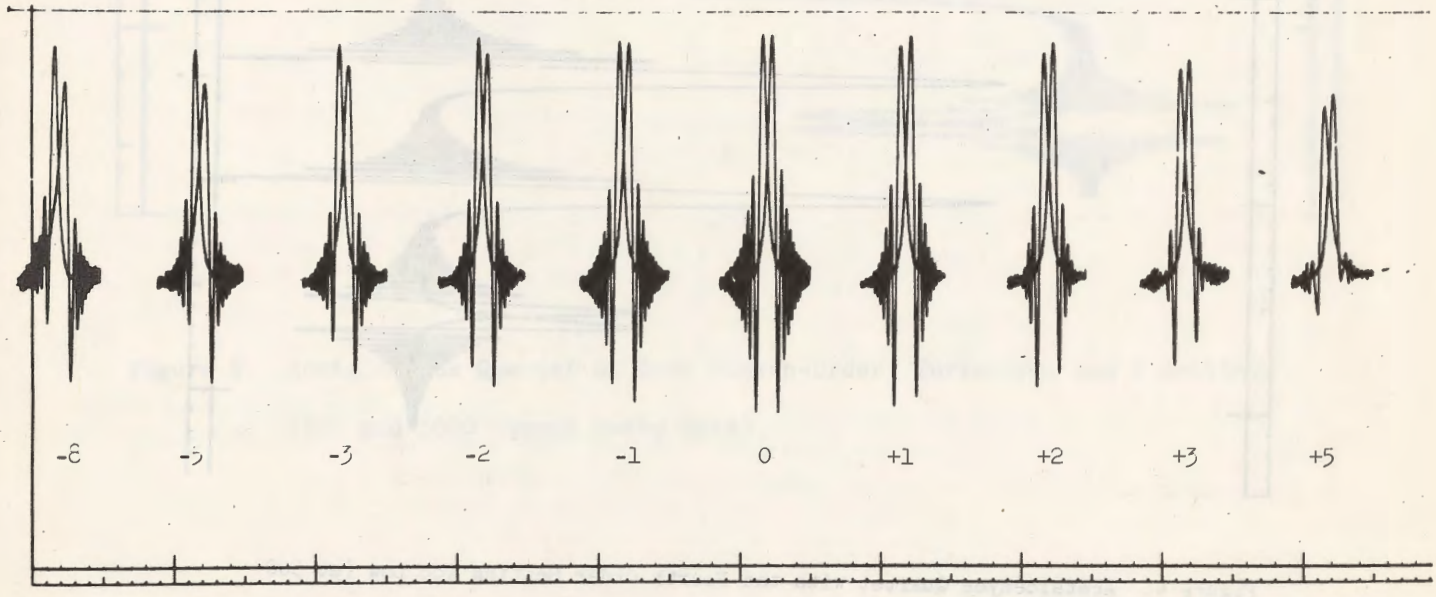

Figure 3. Forward and Reverse Traces of the First Line of the Acetaldehyde Quartet after Turning the 4th Order Control the Indicated Number of Units.

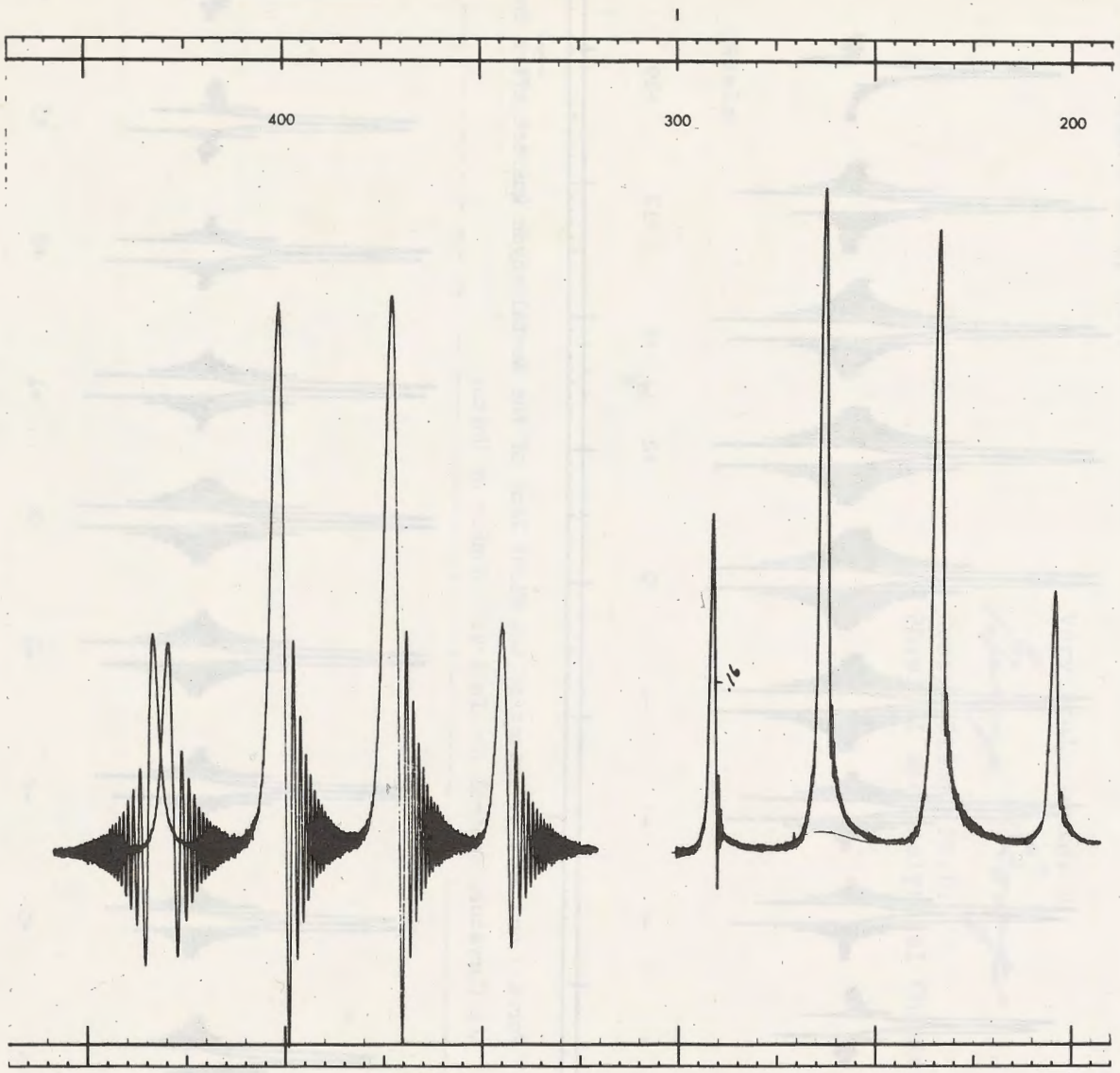

Figure 4. Acetaldehyde Quartet with the Fourth-Order Setting Too Low (at 500 and 5000 Second Sweep Rate).

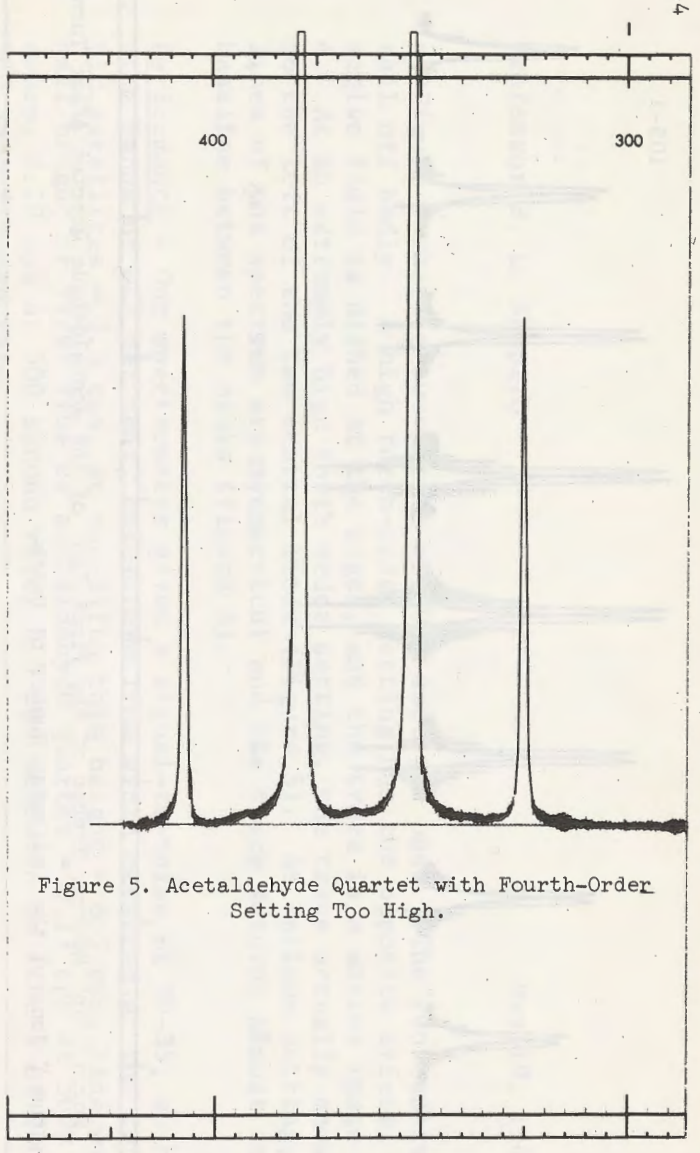

Figure 5. Acetaldehyde Quartet with Fourth-Order Setting Too High.

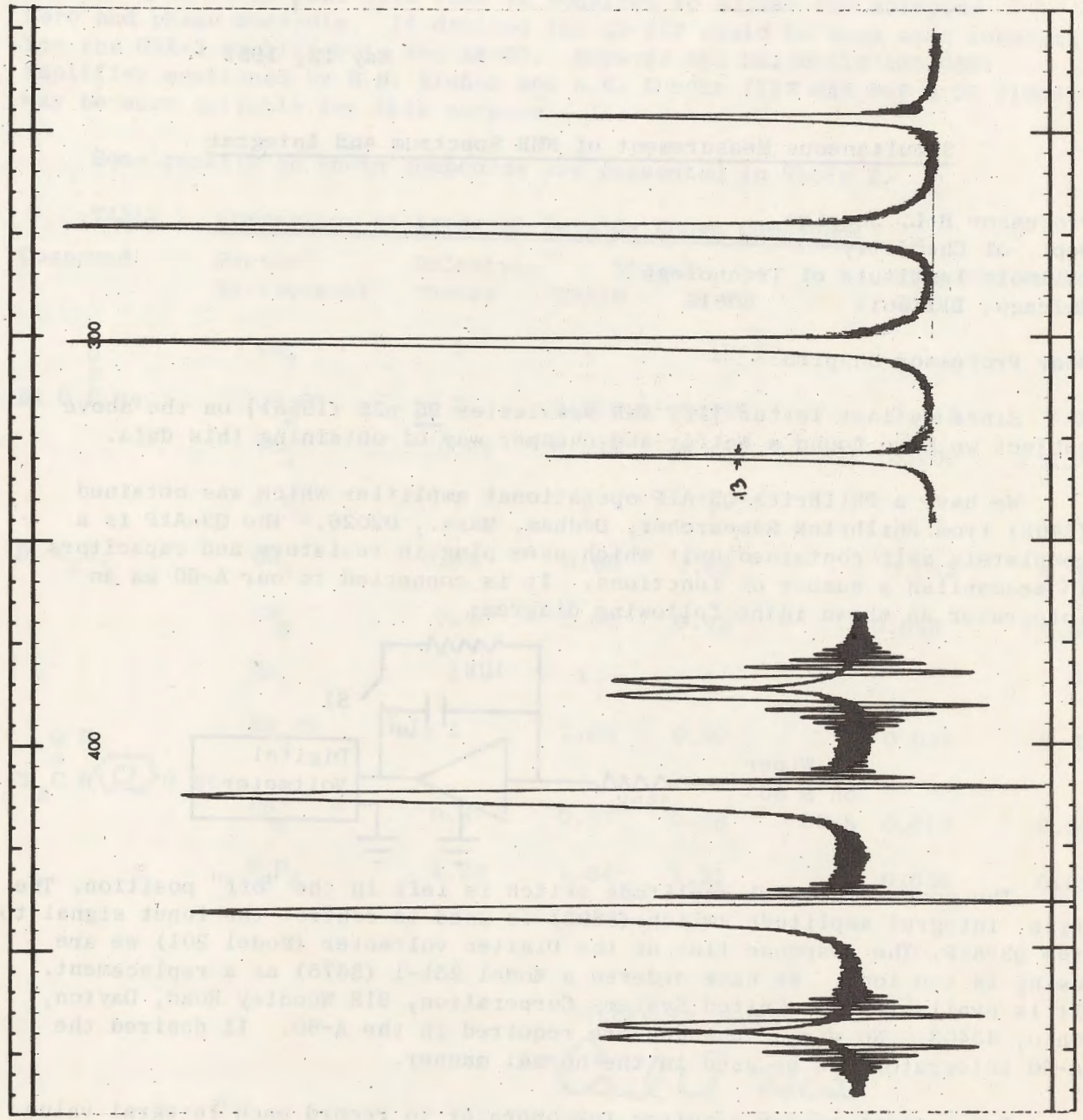

Figure 6. Acetaldehyde Quartet at Best Fourth-Order, Curvature, and Y Settings (500 and 5000 Second Sweep Rate).

GENERAL ELECTRIC COMPANY

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May 12, 1967

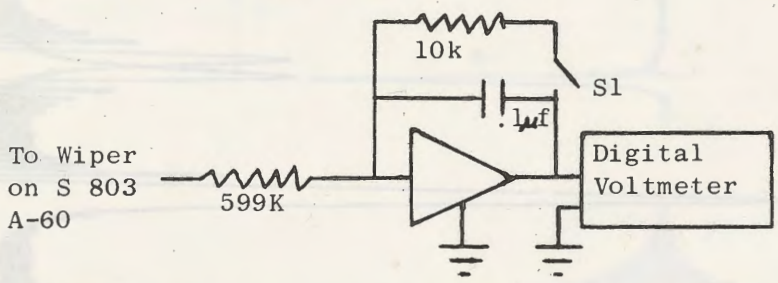
Simultaneous Measurement of NMR Spectrum and Integral

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

Dear Professor Shapiro:

Since my last letter [IIT NMR Newsletter 95 p25 (1966)] on the above subject we have found a better and cheaper way of obtaining this data.

We have a Philbrick Q3-A1P operational amplifier which was obtained ($408) from Philbrick Researcher, Dedham, Mass., 02026. The Q3-A1P is a completely self contained unit which uses plug in resistors and capacitors to accomplish a number of functions. It is connected to our A-60 as an integrator as shown inthe following diagram:



The coarse integral amplitude switch is left in the "off" position. The fine integral amplitude switch (S803) is used to control the input signal to the Q3-A1P. The response time of the Digitec voltmeter (Model 201) we are using is too long. We have ordered a Model 251-1 ($675) as a replacement. It is available from United Systems Corporation, 918 Woodley Road, Dayton, Ohio, 45403. No wiring changes are required in the A-60. If desired the A-60 integrator can be used in the normal manner.

Our present set-up requires the operator to record each integral value as it is obtained. A foot switch(S1) is used to re-set the integrator to zero. On closely spaced peaks the hold feature on the digital voltmeter is used to retain readings until they are recorded. Eventually we hope to record the output of the digital voltmeter on paper tape for computer input.

The visual readout of integral values is often faster than measuring them from a recording. This is particularly true in our quantitative work where we measure a number of integral values for best results.

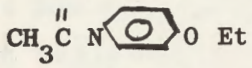
GENERAL ELECTRIC

May 12, 1967

We have also noted that less time is required to adjust the detector zero and phase controls. If desired the Q3-A1P could be used as a substitute for the USA-3 amplifier in the AF-60. However the Fairchild A00-9521 amplifier mentioned by R.H. Elshen and R.E. Lundin (IIT NMR #99 p 50 (1966)) may be more suitable for this purpose.

Some results on known compounds are presented in Table I.

TABLE I Comparison of Integral Data On Known Compounds

Compound	Proton Environment	Relative Signal Theory	Q3A1P	A-60	Standard Deviation Q3A1P	A-60
$Et\,O\,\overset{O}{\overset{\|\|}{C}}\,Me$	CH_3	1	1	1		
	CH_3CO	1	0.992	0.996	0.012	0.012
	CH_2	0.67	0.663	0.652	0.007	0.011
$Et\text{-}C_6H_5$	C_6H_5	1	1	1		
	CH_3	0.60	0.59	0.63	0.042	0.043
	CH_2	0.40	0.36	0.42	0.048	0.058
	CH_3	1	1	1		
	CH_3CO	1	1.00	0.97	0.017	0.025
	CH_2	0.67	0.67	0.68	0.012	0.013
	C_6H_4	1.33	1.34	1.31	0.034	0.023

Sincerely yours,

Carl A. Hirt

Carl A. Hirt
Analytical Chemist

/ml

SCHEIKUNDIG LABORATORIUM
DER VRIJE UNIVERSITEIT
AMSTERDAM-Z.

De Lairessestraat 174 Telefoon 717451

AMSTERDAM, 16 May 1967

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

Dear Dr. Shapiro,

In this note some n.m.r. results are reported of molecules with D_{2h}-symmetry when dissolved in an anisotropic liquid. An example is para-dichlorobenzene in the nematic phase of 4-4-dihexyloxyazoxybenzene. We confine ourselves to a discussion of the spectra in terms of the molecular geometry. The dipolar coupling between the orthoprotons dominates the spectrum and the line positions and intensities can then be given in the following form (see also ref. 1):

Transition	Line positions relative to center	Intensity
$(A_1)_2 - (A_1)_1$	$\frac{3}{4}D^o + \frac{3}{4}D^m + \frac{3}{4}D^p$	2
$(A_1)_1 - (A_1)_0$	$\frac{3}{4}D^o - \frac{1}{4}D^m - \frac{1}{4}D^p + \frac{1}{2}J^m + \frac{1}{2}J^p$	2
$(A_1)_0 - (A_1)_{-1}$	$\frac{3}{4}D^o - \frac{1}{4}D^m - \frac{1}{4}D^p - J^m - J^p$	1
$(B_1)_1 - (B_1)_0$	$\frac{3}{4}D^o + \frac{3}{4}D^m - \frac{3}{4}D^p$	1
$(B_2)_1 - (B_2)_0$	$\frac{3}{4}D^o - \frac{3}{4}D^m - \frac{3}{4}D^p$	1
$(B_3)_1 - (B_3)_0$	$\frac{3}{4}D^o - \frac{3}{4}D^m + \frac{3}{4}D^p$	1

The positions of lines 2 and 3 are correct to first order; second order corrections cause line shifts of a few cycles. These lines could not be resolved experimentally giving a total intensity of 3. The table gives only one half of the spectrum.

D^o = dipolar coupling between orthoprotons

D^m = " " " meta "

D^p = " " " para "

The spectrum consists of 5 doublets [1] with intensity ratio 1:1:3:2:1 symmetrically placed about the center. D^o, D^m an D^p can be extracted from the line positions in the spectrum. These parameters depend on the molecular geometry and the orientation of the molecule in the liquid. In view of the molecular symmetry only two parameter suffice to describe the orientation, which in Snyders [2] notation, are $C_{3z^2-r^2}$ and $C_{x^2-y^2}$. A particular experiment at + 70°C gave the result:

$$D^o = C_{3z^2-r^2} \cdot g^o_{3z^2-r^2} + C_{x^2-y^2}\, g^o_{x^2-y^2} = -4542 \text{ cps} \quad (1)$$

$$D^m = C_{3z^2-r^2} \cdot g^m_{3z^2-r^2} + C_{x^2-y^2}\, g^m_{x^2-y^2} = +275 \text{ cps} \quad (2)$$

$$D^p = C_{3z^2-r^2} \cdot g^p_{3z^2-r^2} + C_{x^2-r^2}\, g^p_{x^2-y^2} = -12{,}6 \text{ cps} \quad (3)$$

The g's are constants for a chosen geometry. In the $(C_{3z^2-r^2}, C_{x^2-y^2})$ plane these equations represent three lines which, of course, should give one unique intersection point within experimental error. The assumption of hexagonal symmetry is not consistent herewith. By introducing a deviation from hexagonal symmetry this condition can be fulfilled: for instance by bending out the C-H bonds in the direction of the chlorine nuclei over 23'. Another way to give a proper description of the n.m.r. spectra is to keep the C-C distances constant while squashing the carbon skeleton in the direction of the chlorine nuclei; the necessary deviation from 120° of the hexagonal angle near the chlorines is 4°18'.
Other paradisubstituted benzenes give similar results.

Sincerely yours,

J. Bulthuis C.W.Hilbers C.MacLean

c/o

References

1. G.Englert und A.Saupe, Z.Naturforschung 19a, 172-177, (1964)

2. L.C.Snyder J.Chem.Phys. 43 4041 (1965).

University of East Anglia

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

School of Chemical Sciences
University Plain
Norwich, Norfolk NOR 85C
Telephone Norwich 52651

16th May, 1967.

Dear Barry,

In response to the usual stimulus for I.I.T.N.M.R. here are some notes on a couple of the activities concerning us at present.

Band Shapes

PART 1. The critical condition for two lines to be resolved.

The problem of obtaining data from absorption lines or bands which overlap one another is one that is common to most forms of spectroscopy; it is of particularly frequent occurrence for NMR. Moreover the simple case of overlap of two bands of equal intensity often occurs, and it is desirable to obtain a good estimate of the true splitting (usually a small coupling constant). We have considered this problem under the simplifying conditions (a) Lorentzian line-shapes, and (b) the band-shape determined solely by overlap (no cross-relaxation effects) - i.e. field inhomogeneity the dominant factor. There are two cases of interest, namely A. when only a single band is seen (absorption a maximum at its centre), and B. some splitting observed (a central minimum in absorption). We have shown that for a true separation of absorption lines equal to J, with the width of a single line denoted Δ_o, the critical condition between the two cases is given by $J/\Delta_o = 1/\sqrt{3}$, i.e. $J = 0.577\Delta_o$. Moreover, if a single band of width Δ is observed, then $J \leqslant 0.393\Delta$. Thus the limit of "observable" coupling is appreciably lower than is usually assumed ($J \leqslant \Delta_o$), [N.B. $0.393\Delta \leqslant 0.577\Delta_o$]. By use of

suitable calculated plots, such as J/Δ_o vs. Δ/Δ_o, reasonable estimates of J can be obtained for both case A and case B. Clearly the approach can be modified to deal with lines of differing intensity, or for more than two lines, or for non-Lorentzian lines.

PART 2. Coupling constants to quadrupolar nuclei.

We have been attempting to see if a simple plot of temperature vs. some function of line-width for the resonance of a spin-$\frac{1}{2}$ nucleus coupled to a spin-1 nucleus would furnish a value for the coupling constant between the nuclei when direct measurement is impossible due to quadrupolar broadening. It appears that unless some departure from overall Lorentzian shape can be detected, it is only possible to obtain $10\pi T_1 J^2$, where T_1 is the spin-lattice relaxation time for the quadrupolar nucleus. Unfortunately, in most cases a quantitative measure of non-Lorentzian character cannot be obtained. In particular, the compound we were studying, 2,6-difluoro-3,4,5-trichloropyridine, gave a Lorentzian band in ^{19}F resonance even at 160°C for the neat liquid, when the band has a half-width of 18 c/s at 100 Mc/s. However we were able to reverse the process originally envisaged, measuring $^2J_{^{15}NF}$ = 52.2 c/s from the ^{15}N satellites, and obtain values for the correlation time τ_c in the solution, thus giving in principle information about molecular motion. We believe measurement of line-widths may be of considerable use in investigating relative magnitudes of coupling constants, as for $^2J_{NF} : {}^3J_{NF}$ in 2,3-difluoro-4,5,6-trichloropyridine. We are currently investigating such cases.

I hope the above will keep us "solvent" as far as I.I.T.N.M.R. is concerned for the next few months.

Best wishes

Robin Tony

R. K. Harris, A.V. Cunliffe.

THE UNIVERSITY OF ROCHESTER
COLLEGE OF ARTS AND SCIENCE
RIVER CAMPUS STATION
ROCHESTER, NEW YORK 14627

DEPARTMENT OF CHEMISTRY

May 16, 1967

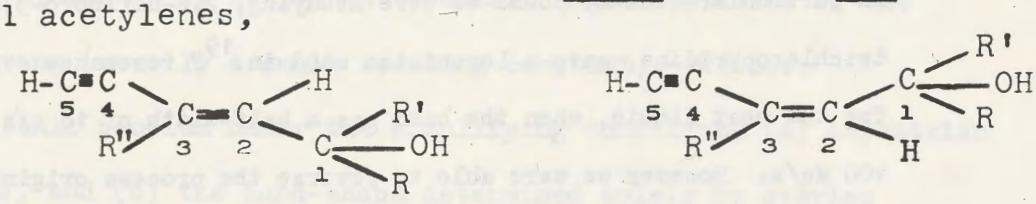
Dr. B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry:

Spectra of Vinyl Acetylenic Alcohols

Mrs. A. Turner has been analyzing the spectra of a number of acetylenes. This project is nearing completion and we now have chemical shifts and coupling constants for many of our compounds. Data for six vinyl acetylenic alcohols are presented below. The spectra of $CDCl_3$ solutions of these compounds were determined on an A-60 spectrometer. Satisfactory fitting of experimental and calculated spectra has been achieved in these cases. Part I of LAOCOON II was used to calculate the spectra for up to seven spins, spectral fitting being done by the laborious eye-ball method since the complete LAOCOON II will not fit on our 7074 computer.

The compounds are derivatives of trans-(I) and cis-substituted (II) vinyl acetylenes,



I II

the pairs (3)(5) and (4)(6) being geometric isomers.

(1) I, R = R" = H, R' = CH_3
(2) I, R = R' = CH_3, R" = H
(3) I, R = R' = H, R" = CH_3
(4) I, R = R' = R" = CH_3
(5) II, R = R' = H, R" = CH_3
(6) II, R = R' = R" = CH_3

Chemical shifts are given in Table I and coupling constants in Table II. It should be noted that the chemical shifts of the acetylenic protons (5-H) are concentration dependent. Table II also lists the number of bonds through which coupling is observed,

Dr. B. L. Shapiro May 16, 1967

the greatest long range coupling which can be measured in these compounds being through six bonds. Differences between corresponding coupling constants in the cis- and trans- compounds are small. Structural differences appear to be more important in determining chemical shifts in these compounds than configuration about the double bond.

Yours sincerely,

Laurie

L. D. Colebrook

LDC/pn

Table I: Chemical Shifts (Hz) from TMS (60 MHz Spectra)

	1-H	1-Me	2-H	3-H	3-Me	5-H*
(1)	259.6	76.0	375.5	340.3	---	174.6
(2)	---	79.2	379.2	340.9	---	171.3
(3)	251.6	--	363.2	---	108.9	171.4
(4)	---	84.6	356.6	---	112.9	201.2
(5)	259.5	--	356.4	---	113.9	194.2
(6)	---	84.0	355.5	---	111.8	202.0

*Concentration Dependent

Table II: Coupling Constants

	J_{1H-1Me}	J_{1H-2H}	J_{1H-3H}	J_{1H-3Me}	J_{1H-5H}	J_{2H-3H}	J_{2H-3Me}	J_{2H-5H}	J_{3H-5H}
(1)	6.5	5.5	-1.4	--	-0.5	16.0	--	0.6	-2.3
(2)	-	-	--	--	--	16.1	--	0.6	-2.3
(3)	-	6.7	--	0.8	-0.3	--	-1.5	0.6	--
(4)	-	-	--	--	--	--	-1.6	0.6	--
(5)	-	6.6	--	1.2	a	--	-1.5	0.8	--
(6)	-	-	--	--	--	--	-1.5	0.8	--
No. of bonds	3	3	4	5	6	3	4	5	4

a $|J_{1H-5H}| < 0.2$ Hz

105-14

Dr. B. Hampel, Dr. L. Pohl
in Fa.
E. MERCK · DARMSTADT
AKTIENGESELLSCHAFT

Anschrift: E. Merck AG, 61 Darmstadt, Frankfurter Straße 250
Hauptleitung Chemische Forschung

LUFTPOST

Herrn
Professor Dr. Bernhard L. SHAPIRO
Department of Chemistry
Illinois Institute of Technology

C h i c a g o 60616

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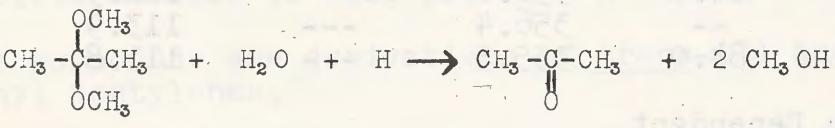
Ihre Zeichen	Ihr Schreiben vom	Hausruf	Unsere Zeichen	DARMSTADT
				16. Mai 1967

2,2-Dimethoxypropan als Reagens zur NMR-spektroskopischen Wasserbestimmung

Sehr geehrter Herr Professor Shapiro!

2,2-Dimethoxypropan geht folgende Säure-katalysierte Reaktion mit H_2O ein:

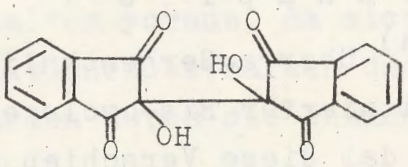


$$CH_3-C(OCH_3)_2-CH_3 + H_2O + H \longrightarrow CH_3-\underset{\underset{O}{\|}}{C}-CH_3 + 2\ CH_3OH$$

Diese Reaktion wurde daher zur Trocknung von Extrakten und biologischen Proben[1] oder Substanzen für IR-Untersuchungen[2] benutzt, ebenso zur Wasserbestimmung mit Hilfe der Gas-Chromatographie[3,4] oder IR-Spektroskopie[5], besonders dann, wenn die betreffenden Verbindungen leicht OH abspalten und daher unzuverlässige Werte nach anderen Methoden liefern.

Einfacher und schneller geht jedoch die Wasserbestimmung mit Hilfe der Kernresonanz, wobei zur Gehaltsbestimmung jedes der 4 Methyl-Signale im Spektrum herangezogen werden kann.

Als Beispiel sei die Bestimmung des Hydratwassers von Hydrindantin angeführt, die so erfolgte, daß in einer mit HCl-Gas schwach angesäuerten (pH 5 - 6), trockenen Lösung von CCl_4 und DMF Hydrindantin aufgeschlämmt und das integrierte Lösungsspektrum mit dem Blindwert der Hydrindantin-freien Lösung korrigiert wurde. Die Messung ergab, daß Hydrindantin als



. 2 H_2O

vorliegt.

Zur Zeit prüfen wir die Genauigkeit und die Anwendungsgrenzen dieser Methode.

Mit freundlichen Grüssen,

B. Gampel
L. Pohl

1) W.F. Bonsquet, Anal.Biochem. 3, 519, 1962
2) I.S. Erley, Anal.Chem. 29, 1564, 1957
3) M. Hager, G. Baker, Proc.Montana Acad.Sci. 22, 3, 1963
4) J.M. Martin, A.M. Knevel, J.pharm.Science 54, 1464, 1965
5) F.E. Critchfeld, E.T. Bishop, Anal.Chem. 33, 1034, 1961

ANORGANISCH-CHEMISCHES LABORATORIUM
DER
TECHNISCHEN HOCHSCHULE MÜNCHEN

8 MÜNCHEN 2, den 17.5.1967
Arcisstraße 21
Ruf-Nr. 5592/330
331

Herrn
Professor Bernard L. S h a p i r o
Illinois Institute of Technology
C h i c a g o , 60616
USA

^{1}H-KMR-Spektren substituierter Bis-cyclopentadienyl-metall-Komplexe.

Sehr geehrter Herr Professor S h a p i r o !

Wir berichteten kürzlich [1-4] über außergewöhnliche ^{1}H-KMR-Kontaktverschiebungen substituierter Bis-cyclopentadienyl-metall-Komplexe. Es wurde gezeigt, daß diese Verschiebungen weder durch einen wesentlichen pseudo-Kontaktbeitrag noch durch einen schon früher vorgeschlagenen Polarisierungsmechanismus [5] zu erklären sind. Um die ungewöhnlichen und unerwartet grossen Linienbreiten in den experimentellen KMR-Spektren einiger dieser Komplexe aufzuklären, haben wir insbesonders die Verbindungen Bis-(methyl-cyclopentadienyl)-kobalt(II) und -nickel(II) eingehender in der Schmelze bei verschiedenen Temperaturen untersucht.
Hierbei zeigt der Nickel-Komplex im festen Zustand Linienbreiten im Bereich von einem Gauß, die sodann beispielsweise für die Cyclopentadienylprotonen in der Schmelze sowie in benzolischer Lösung auf etwa 650 Hz zurückgehen. Wird langsam auf 40°C aufgeheizt, so verbreitern sich zunächst die Signale wieder enorm um dann über 40°C aufzuspalten. Eine eindeutige Erklärung für diese Aufspaltung haben wir bislang noch nicht, sie könnte eventuell durch eine Dimerisierung oder eine intramolekulare Umlagerung hervorgerufen werden.
Wie bereits früher erwähnt [3,6], kommt in den ^{1}H-KMR-Spektren des Bis-(methyl-cyclopentadienyl)-kobalt(II) der starke elektronische und sterische Einfluss der CH_3-Gruppen auf die Anordnung der Liganden zum Ausdruck. Eine Verzerrung des ursprünglich axialsymmetrischen Systems (etwa eine Abwinkelung Ring-Metall-Ring sowie zusätzlich eine Abwinkelung in jedem Liganden um die C_2-C_5-Linie) wird angenommen, auch das UV-Spektrum der Verbindung liefert

einen Hinweis hierfür.
Wir sind der Auffassung, daß die außergewöhnlichen Verschiebungen in der Reihe der Bis-(cyclopentadienyl)-metall-Komplexe auf einer starken Wechselwirkung zwischen den ungepaarten Elektronen in praktisch nichtbindenden Metallbahnfunktionen mit den sehr nahe stehenden Ringprotonen resultieren. Dieses Modell erklärt für $V(C_5H_5)_2$ und $Cr(C_5H_5)_2$ die Verschiebung nach tieferen Feldern durch direkte Übertragung ungepaarter Spindichte von Metall auf die Ligandenprotonen. Für $Co(C_5H_5)_2$ und $Ni(C_5H_5)_2$ sagt es ein quasi-"normales" Verhalten voraus, da sich hier die ungepaarten Elektronen in Ligandenfunktionen aufhalten und sich ihre Delokalisierung über den Liganden durch die bekannten Gleichungen wiedergeben lässt.

Abschließend möchten wir für die Übersendung der I.I.T. N.M.R.-Newsletters herzlich danken und unsere Freude darüber aussprechen, daß das Weiterbestehen dieser von Ihnen unter großen persönlichen Opfern geschaffenen und für uns unbezahlbaren Einrichtung nun doch gesichert erscheint.

Mit freundlichen Grüßen

Heinz P. Fritz — Heinz J. Keller — Karl Schwarzhans

(H.P.Fritz) (H.J.Keller) (K.E.Schwarzhans)

1) H.P.Fritz, H.J.Keller, K.E.Schwarzhans; J.organomet.Chem. 6, 652 (1966)
2) H.P.Fritz, H.J.Keller, K.E.Schwarzhans; J.organomet.Chem. 7, 105 (1967)
3) H.P.Fritz, H.J.Keller, K.E.Schwarzhans Zeitschr.f.Naturforschg. 21b, 809 (1966)
4) H.P.Fritz, H.J.Keller, K.E.Schwarzhans; Zeitschr.f.Naturforschg., im Erscheinen
5) D.A.Levy, L.E.Orgel; J.Mol.Phys. 3, 583 (1961)
6) H.J.Keller, H.Nawersik; J.orgenomet.Chem. 7, 185 (1967)

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

BASF

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 am Rhein
		Dr.Brü/Fa	(0621) 60...	464...	22. Mai 1967

Betreff Spectra of 2.6-Dimethylmorpholines

Sehr geehrter Herr Professor Shapiro!

BOOTH u. GIDLEY (Tetrah. 21 (1965), 3429) haben vor einiger Zeit die Spektren von cis- und trans-2.6-Dimethylmorpholin untersucht und analysiert. Im Rahmen einer ausgedehnten Untersuchung N-substituierter 2.6-Dimethylmorpholine habe ich diese Messungen wiederholt. Meine Ergebnisse stimmen mit denen von BOOTH u. GIDLEY ausgezeichnet überein, jedoch fand ich eine von diesen Autoren nicht erwähnte Aufspaltung des Signals des 3eq-Protons der cis-Verbindung in der Größe von ungefähr 0.7 Hz, die zusätzlich zu den bei dem vorliegenden $ABXY_3$-System zu erwartenden Aufspaltungen auftritt. Sie erscheint ebenso in allen von mir untersuchten N-substituierten cis-2.6-Dimethylmorpholinen in einer Größe von 1.3 bis 1.7 Hz. BOOTH (priv. Mitt.), der dies an derselben Verbindungsklasse ebenfalls fand, glaubt, daß es sich dabei um eine weitreichende Kopplung über 5 Bindungen zwischen dem 6ax- und dem 3eq-Proton handele, und stützt diese Vermutung auf ein Doppelresonanzexperiment, weil diese Aufspaltung bei Einstrahlung mit der chemischen Verschiebung der 2ax- und 6ax-Protonen verschwindet (zusammen natürlich mit der Kopplung 2ax/3eq). BOOTH wird darüber selbst in einiger Zeit berichten. Am Signal des 6ax-Protons ist eine entsprechende Aufspaltung jedoch nicht zu bemerken, obwohl Linienbreite und Linienform des außerdem noch durch die Methylgruppe aufgespaltenen Signals dies ermöglichen sollten. Für einige trans-Verbindungen zeigen die Signale beider 3-Protonen ebenfalls eine zusätzliche Aufspaltung; sie beträgt für das 3ax-Proton ungefähr 0.5 bis 0.6 Hz, während die sich beim 3eq-Proton nur als Linienverbreiterung manifestiert.

Badische Anilin- & Soda-Fabrik AG

Empfänger	Unsere Zeichen	67 Ludwigshafen am Rhein	Blatt
Prof.Dr. B.L. Shapiro	Dr.Brü/Fa	22. Mai 1967	2

Betreff

Die Spektren der N-substituierten 2.6-Dimethylmorpholine zeigen eine Reihe weiterer Besonderheiten, auf die kurz hingewiesen sei. Sieht man von der vorerwähnten Kopplung und der von den Methylgruppen herrührenden ab, so bleibt ein ABC-System für die Ringprotonen übrig, das bekanntlich bestimmte Regelmäßigkeiten für wiederkehrende Linienabstände aufweisen sollte. Diese Regeln sind für die cis-Verbindungen in erstaunlichem Ausmaß durchbrochen: die von der geminalen Kopplung der 3-Protonen herrührende Aufspaltung ist für das 3ax-Signal um 0.6 bis 1.4 Hz größer als für das 3eq-Signal! Auch für R=H ist schon eine kleine Diskrepanz von 0.4 Hz feststellbar. Die naheliegende Annahme, daß diese Diskrepanz durch eine technische Unzulänglichkeit der Apparatur (HA-60 und A-60), nämlich durch eine Nichtlinearität des Sweep hervorgerufen sei, wurde schon durch die Tatsache widerlegt, daß für die gleichzeitig untersuchten trans-Verbindungen kein solcher Effekt auffindbar war. Außerdem bewies eine exakte Ausmessung der Linien mit einem elektronischen Zähler, daß es sich um eine reelle Beobachtung handelt. Mir ist völlig unklar, wodurch diese Diskrepanz hervorgerufen wird. Sie macht die exakte Analyse der Spektren als ABC-System unmöglich. Bei den Ergebnissen, die auf der beigefügten Tabelle zusammengestellt sind, handelt es sich um Werte, die durch Mittelwertbildung der beiden gemessenen Aufspaltungen gewonnen wurden. Sie sind daher für die cis-Verbindungen weniger genau angegeben als für die trans-Verbindungen, was sich besonders bei den Kopplungskonstanten auswirkt.

Ein Blick auf diese Tabelle zeigt, daß für die trans-Verbindungen die chemical-shift-Differenz der geminalen Protonen 3ax und 3eq praktisch unabhängig vom N-Substituenten den Wert 0.30 bis 0.33 ppm hat mit Ausnahme des Substituenten R=H. Hingegen ist bei den cis-Verbindungen diese Differenz sehr stark vom N-Substituenten abhängig (0.41 ppm für R=H bis 1.32 ppm für R=3.5-Dimethylcyclohexyl). Dieser Effekt wurde auch von BOOTH beobachtet, und er wird darüber ebenfalls in Kürze berichten. Offensichtlich kommen zur Erklärung dieses Effektes ähnliche Einflüsse in Frage, wie sie BOOTH u. LITTLE (Tetrah. 23 (1967), 291) gerade erst für N-substituierte 2.6-Dimethylpiperidine diskutiert haben.

Mit freundlichen Grüßen

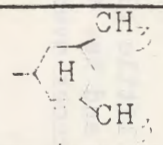
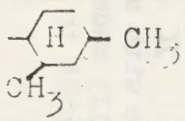
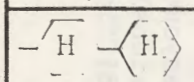
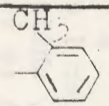
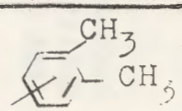
Ihr sehr ergebener

W. Brügel

(Dr. W. Brügel)

NMR data of N-substituted 2,6-dimethylmorpholines in $CDCl_3$

R	conf.	pur. %	conc. %	ν_2 ppm	ν_{3ax} ppm	ν_{3eq} ppm	$J_{2,3ax}$ cps	$J_{2,3eq}$ cps	$J_{3ax,3eq}$ cps	$\nu_{3eq} - \nu_{3ax}$ ppm
-H	cis	>99	20	3.55	2.38	2.79	10.2	2.3	-12.2	0.41
	trans	>95	20	3.899	2.514	2.399	5.94	3.30	-12.13	0.355
$-CH_3$	cis	>99	15	3.55	1.67	2.65	10.1	2.1	-11.1	0.98
	trans	>98	20	4.957	2.035	2.399	5.29	3.31	-10.85	0.316
$-CH_2CH_3$	cis	>99	20	3.71	1.67	2.75	10.4	2.1	-11.1	1.08
	trans	>90	15	4.106	2.177	2.485	5.35	3.59	-11.15	0.308
$-CH(CH_3)_2$	cis	>99	20	3.60	1.67	2.71	10.2	2.1	-10.9	0.84
	trans	>90	20	4.070	2.240	2.562	5.95	3.40	-11.06	0.322
$-CH_2CH_2CH(CH_3)_2$	cis	>99	20	3.68	1.67	2.74	10.1	2.3	-11.0	1.07
	trans	>95	15	4.005	2.145	2.446	5.89	3.07	-11.10	0.301
$-CH_2CH(CH_3)CH_2OH$	cis	>99	20	3.67	2.02	2.76	10.1	2.1	-11.0	0.74
	trans	>95	10	4.025	2.353	2.666	5.94	3.17	-11.11	0.313
$-CH(C_3H_5)_2$	cis	mix-ture 72:28	15	3.60	1.67	2.70	10.4	2.5	-11.6	1.03
	trans			3.95	2.06	2.41	5.8	3.2	-11.6	0.33

R	conf.	pur. %	conc. %	ν_2 ppm	ν_{3ax} ppm	ν_{3eq} ppm	$J_{2,3ax}$ cps	$J_{2,3eq}$ cps	$J_{3ax,3eq}$ cps	$\nu_{3eq} - \nu_{3ax}$ ppm
	cis	>99	20	3.68	1.94	2.74	10.1	2.0	-10.7	0.80
	trans	>95	10	4.001	2.277	2.585	6.09	3.04	-10.98	0.308
	cis	>98	20	3.61	1.51	2.83	10.4	2.1	-10.8	1.32
	trans	>95	10	4.010	2.145	2.462	5.63	3.20	-11.43	0.316
	cis	>90	10	3.80	1.72	2.86	10.3	2.4	-11.3	1.14
	cis	>95	20	3.64	1.67	2.82	10.1	2.0	-11.0	1.15
cyclo-C_8H_{15}	cis	>95	20	3.63	1.96	2.59	10.0	2.0	-10.9	0.63
	trans	>90	15	3.972	2.215	2.513	6.02	3.14	-11.16	0.298
cyclo-$C_{12}H_{24}$	cis	>99	20	3.66	2.00	2.64	10.0	2.1	-11.3	0.64
	trans	>98	20	3.971	2.255	2.574	5.64	3.15	-11.03	0.319
	cis	>98	20	3.81	2.39	2.87	10.0	2.0	-10.9	0.48
	trans	>95	20	4.165	2.639	2.935	5.97	3.06	-11.33	0.296
	cis	>98	20	3.86	2.41	2.87	10.0	2.0	-10.7	0.46
	trans	>90	10	4.182	2.602	2.921	5.81	2.92	-11.63	0.319

105-22
ARCO Chemical Company
Division of Atlantic Richfield Company
1900 Crescent Avenue
Anaheim, California 92801
Telephone 714 533 3131

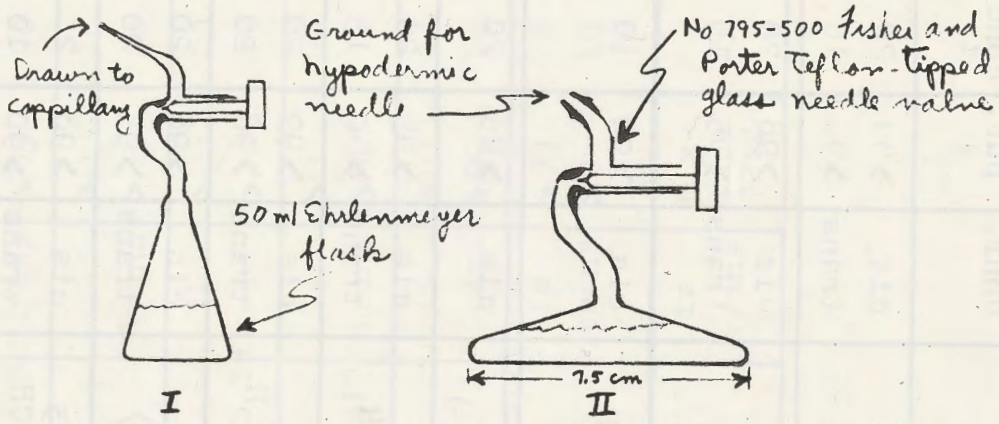
May 24, 1967

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

Dear Dr. Shapiro

Handy Greaseless Container-Dispenser for Volatile Liquids

Handling tetramethylsilane is sometimes inconvenient using conventional bottles. When stored in a refrigerator losses and contamination can occur. We have been using a specially made container-dispenser flask for TMS which, although still not the ultimate, has proved a vast improvement over conventional containers. The container in two of its development stages is shown in the sketches below.



Container I can be made by anyone with a little glassblowing ability while II was professionally made and has the advantage that it is very stable and difficult to knock over.

The containers are filled by evacuating them through a small polyethylene tube slipped over the capillary outlet for I and through a commercially available integral all polyethylene tube-syringe hub placed on the ground outlet for II followed by sucking the desired liquid into the container. The liquid is dispersed using its own vapor pressure by inverting the container and opening the valve. Some liquid is always wasted because of the dead space in the valve but wastage has been far less than with ordinary bottles.

We have found this type container very useful for other volatile liquids such as acetaldehyde but care should be taken that vapor pressures not to exceed about 10 psig in a flat-bottomed thin-walled container. For more volatile liquids we use the same needle valve attached to a heavy-wall glass tube and have had no trouple up to 200 psig.

Sincerely yours,

F. F. Caserio, Jr.
P. G. Sibbald

F. F. Caserio, Jr.
P. G. Sibbald

FFC
PGS:ka

UNIVERSITY OF CALIFORNIA, IRVINE

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

DEPARTMENT OF CHEMISTRY IRVINE, CALIFORNIA 92650

May 29, 1967

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

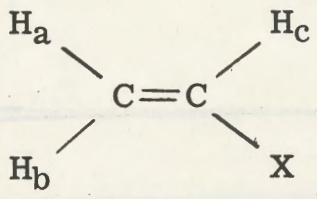
Dear Barry:

We apologize for the delay in submitting our contribution to IIT NMR letters. Herewith in haste is a modest message, the main point of which is to report our analysis of the n. m. r. spectra of methyl vinyl sulfide, methyl vinyl sulfoxide, and methyl vinyl sulfone, and to draw attention to the probable misidentification of the spectrum of methyl vinyl sulfone No. 35 in the Varian NMR Spectra Catalog. If the latter spectrum is compared with the spectra we obtained for the vinyl sulfur compounds (see Figures 1, 2 and 3 of this letter), the resemblance to that of methyl vinyl sulfoxide, Figure 2, is striking. We suggest that spectrum No. 35 in the spectra catalog is not that of methyl vinyl sulfone, but is that of methyl vinyl sulfoxide.

Our sample of methyl vinyl sulfone was obtained from K and K Laboratories. It showed intense bands in the infrared at 1315 cm^{-1} and 1135 cm^{-1}, characteristic of the sulfone group. Methyl vinyl sulfoxide was prepared from β-chloroethyl methyl sulfide by oxidation with hydrogen peroxide followed by dehydrohalogenation with potassium hydroxide. The product had b. p. 80-82^0 at 21 mm and showed intense absorption in the infrared at 1050 cm^{-1}, which band is characteristic of the S=O group of sulfoxides.

The spectra of Figures 1, 2 and 3 were of 10% solutions in deuterochloroform. Reiterative computer analysis using a modified Swalen-Reilly program reproduced the observed spectra with an average deviation between observed and calculated frequencies

of less than 0.1 cps. The "best fit" spectral parameters are listed below:



X	δH_a ppm	δH_b ppm	δH_c ppm	δCH_3 ppm	J_{ab} cps	J_{bc} cps	J_{ac} cps	$J_{CH_3H_a}$ cps	$J_{CH_3H_b}$ cps
SCH_3	5.19	4.97	6.43	2.24	-0.18	16.91	10.42	0.4	0.2
$SOCH_3$	5.92	6.08	6.77	2.61	-0.56	16.71	9.79	0.3	0.0
SO_2CH_3	6.14	6.43	6.76	2.96	-0.45	16.52	9.98	--	--

Sincerely yours,

Marjorie C. Caserio

Marjorie C. Caserio

Stephen H. Smallcombe

Stephen H. Smallcombe

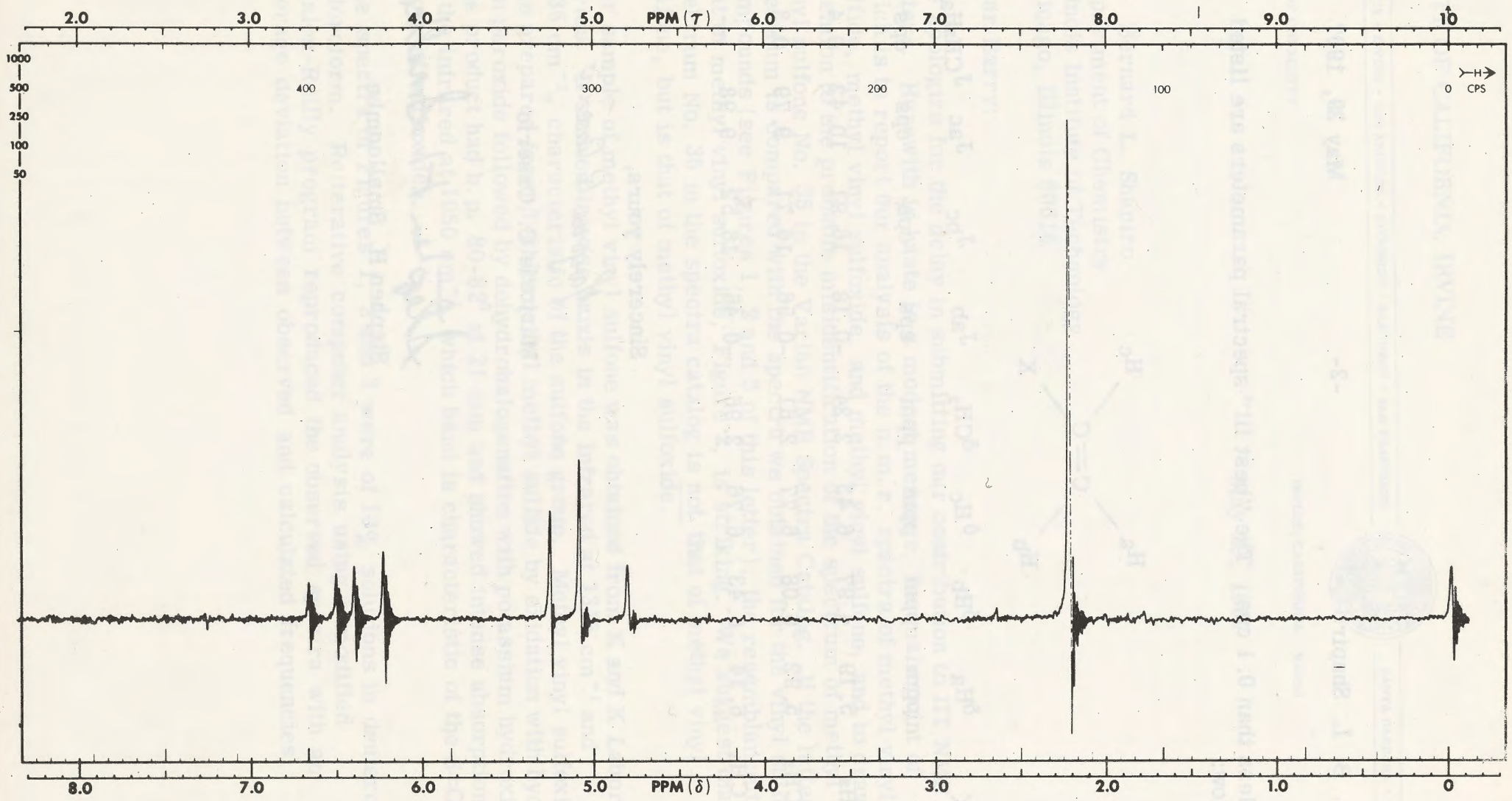


Figure 1. Methyl Vinyl Sulfide

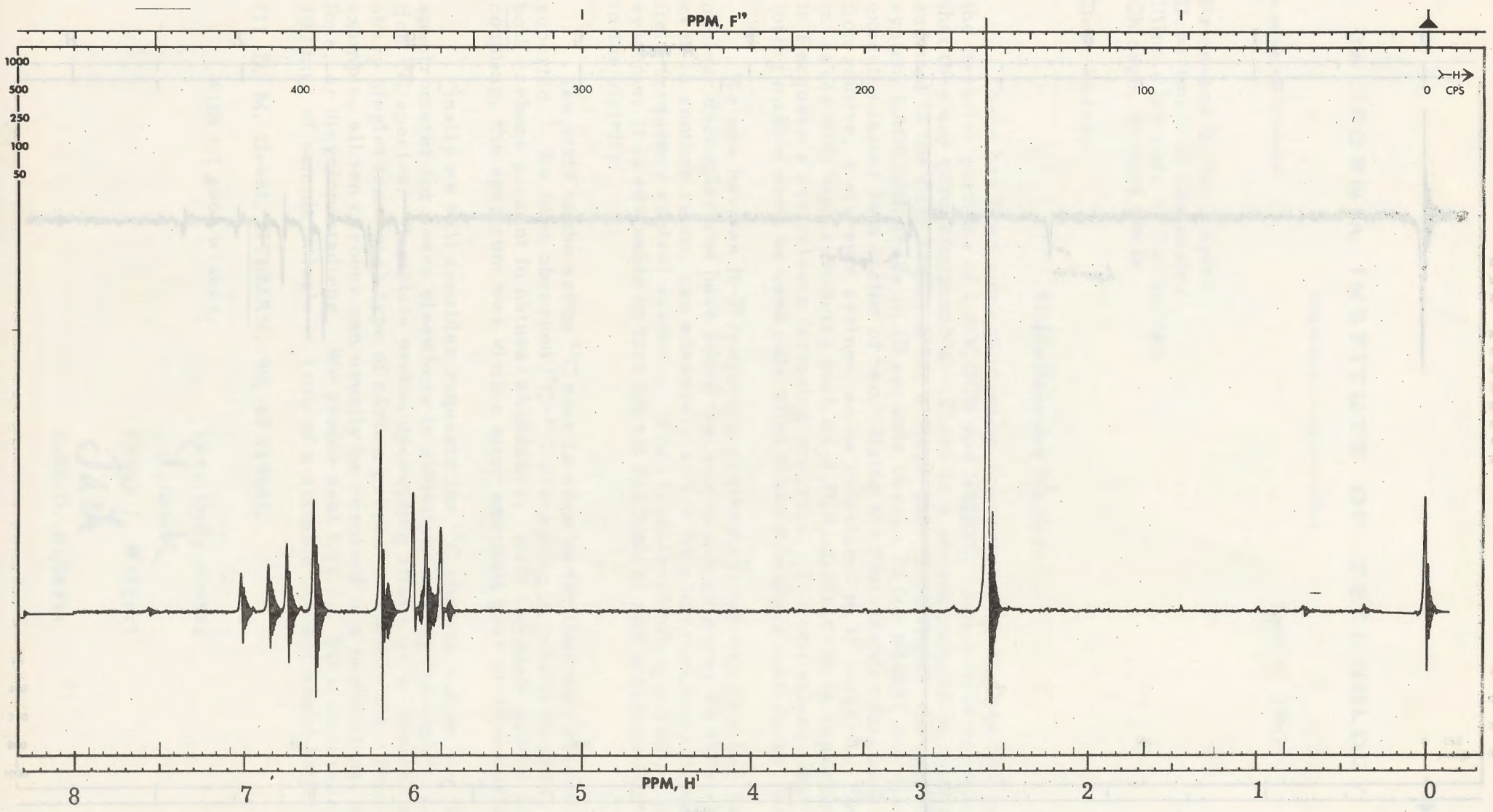


Figure 2. Methyl Vinyl Sulfoxide

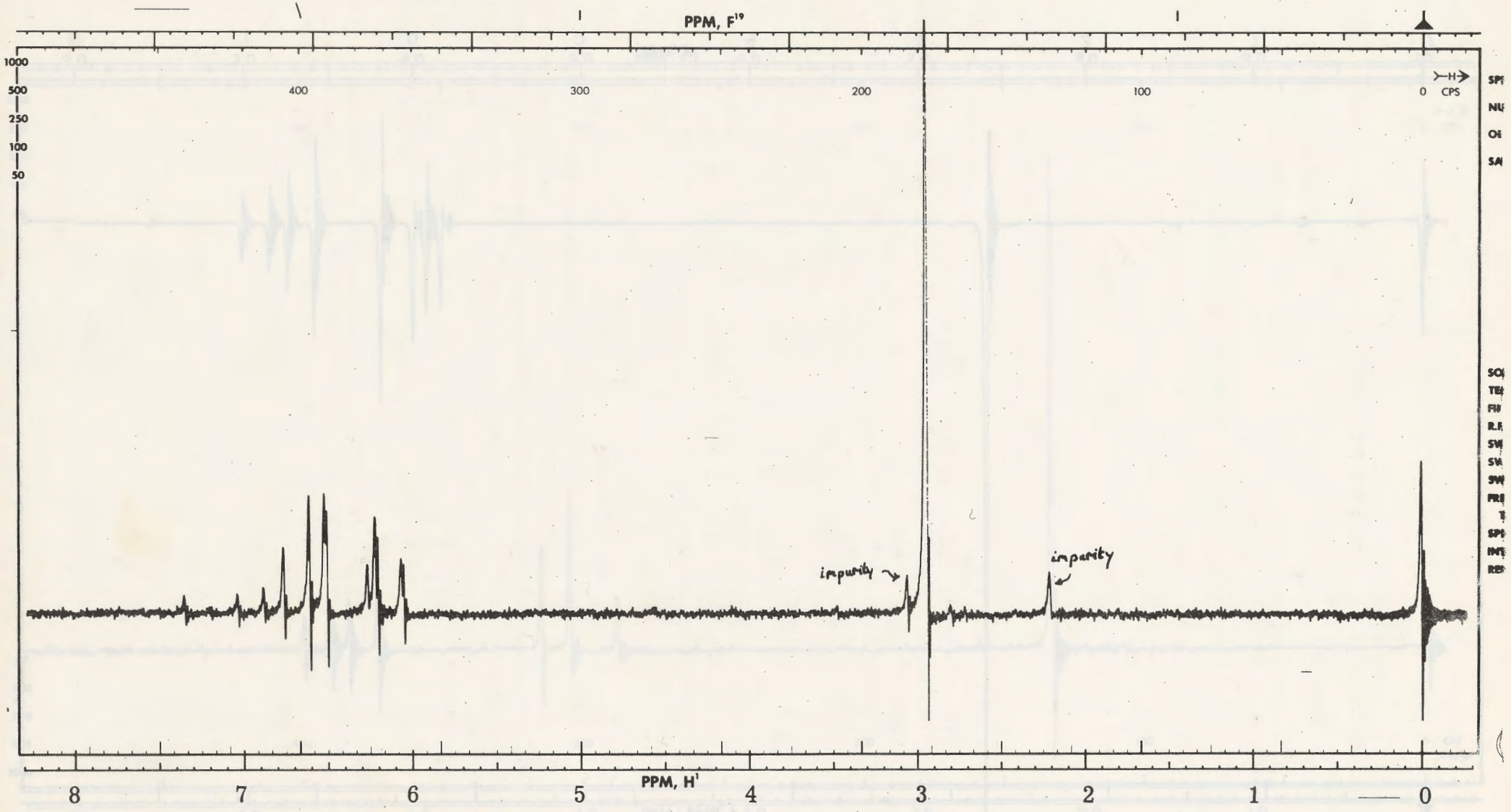


Figure 3. Methyl Vinyl Sulfone

CALIFORNIA INSTITUTE OF TECHNOLOGY

PASADENA, CALIFORNIA 91109

GATES AND CRELLIN LABORATORIES OF CHEMISTRY

June 1, 1967

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

Dear Barry:

Miscellaneous Matters

There has been some discussion concerning the relative merits of the iterative portions of LAOCOON and NMRIT. The conclusion was that they are quite comparable. There is a second point to be considered, and that is the time required for a Stage-One calculation. On our 7094 system LAOCOON loads in 10 seconds versus 30 for NMRIT and also executes faster by a factor of two. Using the Householder diagonalization routine, a six-spin system can be calculated in 15 seconds. Even in cases with high symmetry such as A_3B_2X, LAOCOON is superior to Fergusen's equivalence factoring program. The equivalence factoring method need be used only when there are more than seven spins.

We now have an H-P frequency synthesizer for use as a heteronuclear decoupler and have found that use of a high power 60 MHz source, even in another room, can adversely affect both internal and external field-frequency control systems. When trouble-shooting erratic control systems, it is advisable to turn off all decouplers and spectrometers in the vicinity.

The state of the art in ^{13}C nmr is even better than was recently reported.[1] We have observed ^{13}C-^{13}C decoupling in t-butyl bromide with both carbons present in natural abundance. With complete proton decoupling, the spectrum was visible after one-half hour of time-averaging

Finally we will consider requests for ^{13}C spectra run on our DFS-60 spectrometer for users elsewhere in situations calling for high-resolution ^{13}C spectra. Complete proton decoupling allows us to observe a sharp singlet from each type of carbon present in a molecule. For example, all ten carbons can usually be resolved in a naphthalene, decalone, or dicyclopentadiene. We prefer neat liquids, but if necessary, 100 mg. of sample soluble in 1 ml. of a suitable solvent would suffice.

(1) D. M. Grant, IITNMRN, 99, 67 (1966).

With all good wishes,

Very truly yours,

Frank

Frank J. Weigert

Jack

John D. Roberts

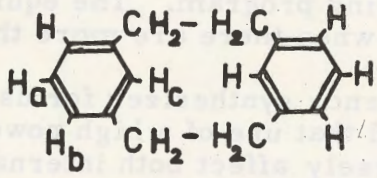
Dr.F.Stuber c/o
CIBA LIMITED

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

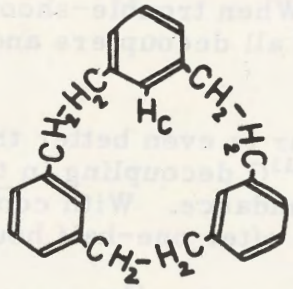
Basle, May 31, 1967

Dear Dr. Shapiro:

I would like to report the chemical shifts of the aromatic protons in metacyclophanes.



[2.2] metacyclophane



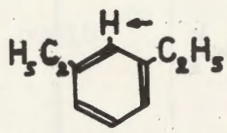
[2.2.2] metacyclophane

The chemical shift of H_c in [2.2] metacyclophane has already been reported[1]. The strong shielding of this proton can be explained by means of the "interatomic current" in the opposite benzene ring.

However, in [2.2.2], [2.2.2.2] and [2.2.2.2.2] metacyclophanes the H_c-signals still appear at high fields which cannot be interpreted as in the previous example, due to the changed geometry. In the spectra of the last three substances (see Figure) the CH_2 groups appear as singlets, all at δ = 2.77 ppm; this result suggests a certain symmetry in these molecules and a degree of rotation of the CH_2 groups, leading to an averaging out of the chemical shifts.

Dr.F.Stuber c/o
CIBA LIMITED

Proton chemical shifts of H_c

metacyclophane	$\delta^{(2)}$ ppm
2.2	4.25
2.2.2	6.14
2.2.2.2	6.51
2.2.2.2.2	6.72
	6.98

For comparison, the chemical shift of the corresponding proton in 1,3-diethylbenzene has been quoted, too, in the table. It is interesting to note that the shielding of the H_c's decreases with increasing ring size. The factors contributing to that situation are, however, not quite understood.

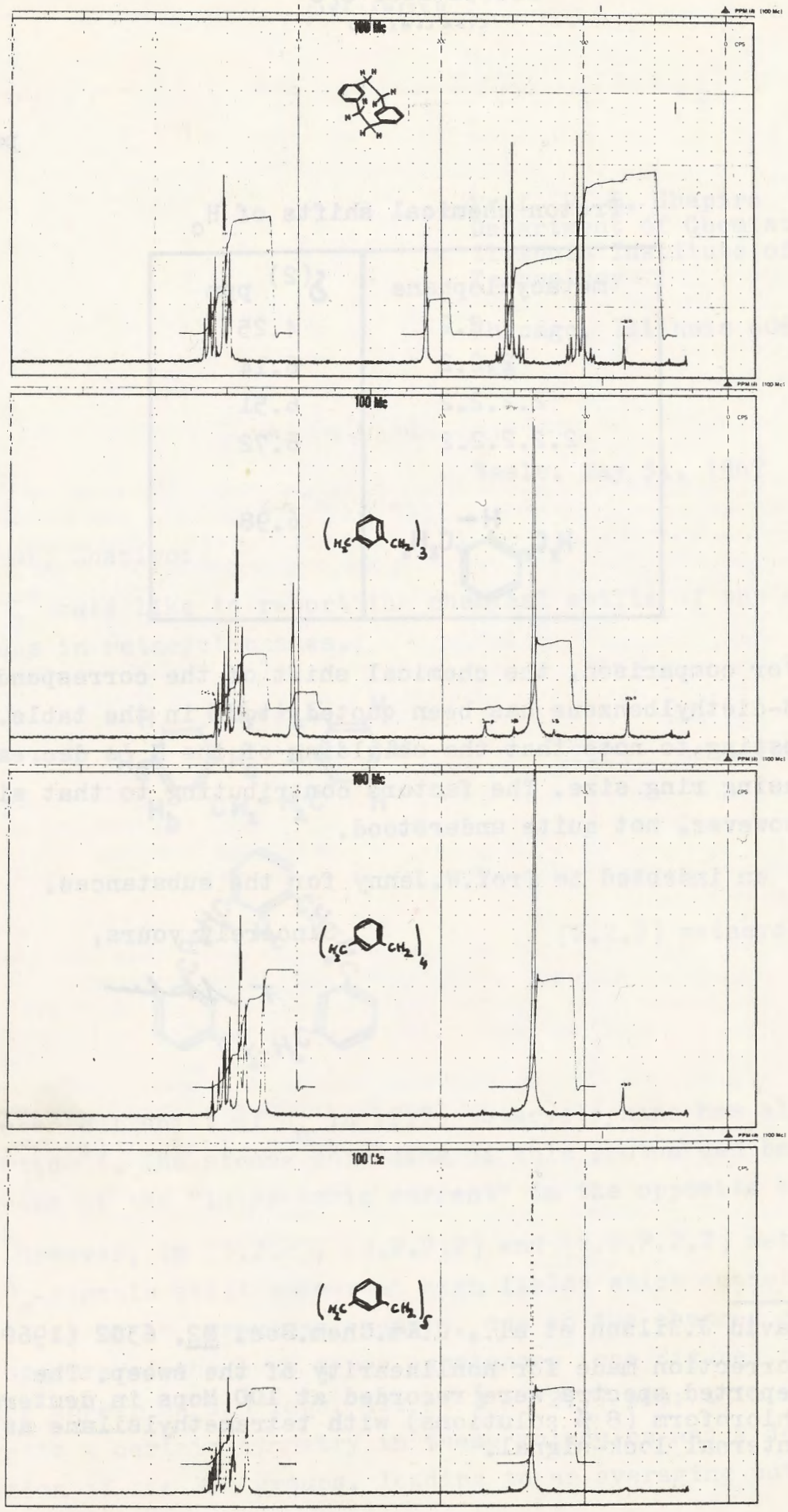
I am indebted to Prof.W.Jenny for the substances.

Sincerely yours,

F. Stuber

(1) David J.Wilson et al., J.Am.Chem.Soc. 82, 6302 (1960)

(2) Correction made for nonlinearity of the sweep. The reported spectra were recorded at 100 Mcps in deuterochloroform (8 % solutions) with tetramethylsilane as internal lock-signal.


100 Mc
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DEPARTMENT OF CHEMISTRY DAVIS, CALIFORNIA 95616

May 31, 1967

^{15}N Spectra at 10.135 MHz. Using Nitrogen Lock

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

Dear Barry:

We have recently tried, with some success, to duplicate with ^{15}N what we previously reported (IITNMR 98-34) for ^{13}C. Accordingly, using a nearly standard HA-100 spectrometer system at 10.135 MHz. we have locked on a sample of $KC^{15}N$ aq. (99% ^{15}N label) in a capillary tube with outside diameter 2.2 mm. The lock seems to be quite stable, permitting the time-averaging of the ^{15}N spectra of samples contained in the outer annulus of the 5 mm., spinning sample tube system.

George Gray is employing this method at the present to determine the ^{15}N chemical shifts of labelled nitriles; and we are setting up to use it in studies of solvent effects, hydrogen bonding and non-hydrogen bonding.

Sincerely,

Gary E. Maciel

Gary E. Maciel
Associate Professor of
Chemistry

GEM:amm

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TÉL. 87-59-11

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

RÉFÉRENCE A RAPPELER : G COP-1/67-490/mjc

VOTRE RÉF. :

VOTRE LETTRE DU

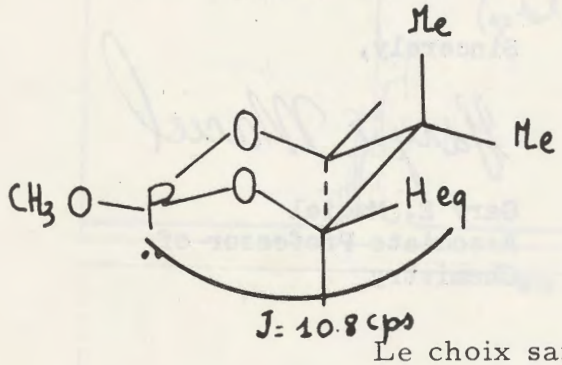
GRENOBLE LE 6 juin 1967

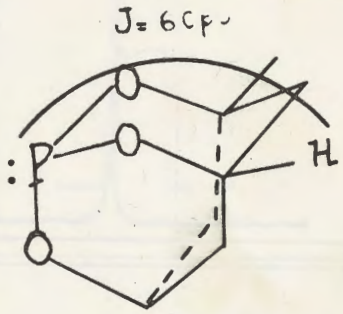
^{4}J(POCH) coupling constants : Influence of lone pair orientation and nature of substitution.

Cher Professeur Shapiro,

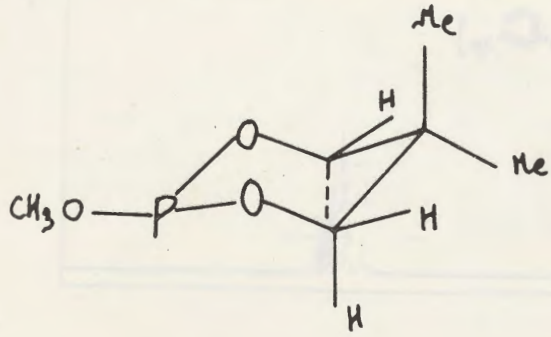
Deux résultats peuvent être signalés pour le couplage ^{4}J phosphore-proton dans des dérivés hétérocycliques :

1) La disposition spatiale du doublet libre est illustrée par exemple dans la comparaison suivante :





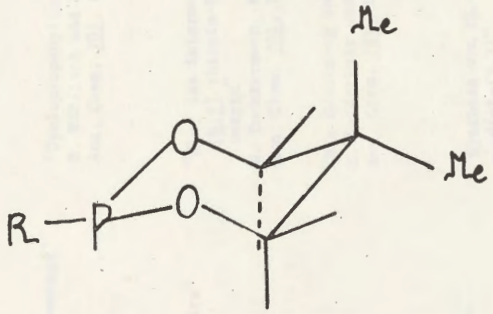
Le choix sans ambiguité des protons axiaux et équatoriaux dans le premier exemple résulte de deux couplages à longue distance :



$J_{H_{eq}-H_{eq}}$ = 2,7 cps
(vu sur satellites C_{13})

$J_{CH_3-H_{ax}}$ = 0,7 cps

2) La nature du substituant est en général faible, avec une exception pour un substituant azoté, qui donne des couplages voisins de ceux obtenus dans les homologues tétracoordinés



- R = F, OCH_3 OC_6H_5

$J_{P-H_{ax}}$ = 2,8 cps

$J_{P-H_{eq}}$ = 11,8 cps

- R = $N(CH_3)_2$ comparable à $\phi O(O)P$

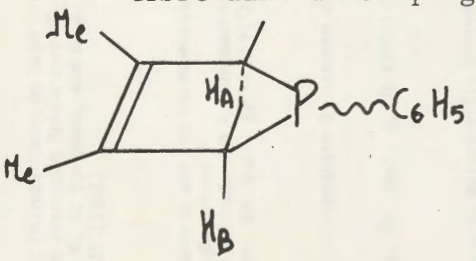
$J_{P-H_{ax}}$	= 4 cps	= 4 cps
$J_{P-H_{eq}}$	= 20 cps	= 22 cps

D. GAGNAIRE J.B. ROBERT J. VERRIER

Ref. 1 : D. GAGNAIRE, J.B. ROBERT, J. VERRIER, Bull. Soc. chim. Fr. à paraître.

Ref. 2 : E.J. BOROS, K.J. COSKRAN, R.W. KING et J.G. VERKADE, J. amer. chem. Soc., 88, 1140 (1966).

P.S. : Plus remarquable encore est l'influence de l'orientation du doublet libre dans un couplage $^2J_{P-C-H}$ dans l'exemple suivant :



J_{P-H_A} = ± 25

J_{P-H_B} = ∓ 6

Noter le changement de signe et de grandeur !

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