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Newsletter

No. 83
AUGUST, 1965

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

UNIVERSITY OF KENT AT CANTERBURY

UNIVERSITY PHYSICS LABORATORIES,
CANTERBURY, KENT.

TELEPHONE
CANTERBURY 66761

RF/GH

20th July, 1965.

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

Dear Dr. Shapiro,

May I take advantage of the widespread circulation of
I.I.T. NMR newsletter and ask if anyone could suggest a source of
supply of perdeuteriochlorobenzene (C_6D_5Cl). I need only a few ml.

Yours sincerely,



R. FIGGINS

PURDUE UNIVERSITY

DEPARTMENT OF CHEMISTRY

LAFAYETTE, INDIANA 47907

July 21, 1965

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

Dear Barry:

What Causes Proton Chemical Shifts in Saturated Unstrained Hydrocarbons?

You will recognize in this question a variation on the title of the recent letter from Dr. R. F. Zürcher. The origin of proton chemical shifts remains perhaps the one most intriguing puzzle in NMR, and I have long felt that unstrained, saturated hydrocarbons are materials of unusual significance in this regard, since here one expects only a few key parameters to be involved, and the tricky influences of strongly polar groups, or aromatic ring currents, or pi bonds, all are absent.

Bill Tosch and I called attention to some very puzzling features of hydrocarbon shifts in J. Chem. Phys., 37, 1167 (1962), but at the time we could not assign accurate numerical values to many of the shifts encountered because of the complexity of the spectra we had. Since then, Dick Hughes has looked at a number of additional compounds, and through the courtesy of the Varian people at Pittsburgh he has obtained several 100 Mcps spectra. In particular, the spectrum of cis-1,3,5-trimethylcyclohexane is beautifully simplified at 100 Mcps and yields numerical data which allow at least one major anomaly to be brought very sharply into focus.

The following table gives the shifts we found, with values for conformationally fixed cyclohexane, from J. Phys. Chem., 68, 2026 (1964) for comparison:

<u>Proton Type</u>	<u>Shift in p.p.m. from Tetramethylsilane</u>	
	<u>cis-1,3,5-trimethylcyclohexane</u>	<u>cyclohexane</u>
Methyl	-0.86	-----
Axial methine	-1.40	-----
Axial methylene	-0.43	-1.19
Equatorial methylene	-1.66	-1.66

One fact jumps out at you: Introduction of the equatorial methyl groups has no effect on the shift of the adjacent equatorial methylene proton, but a large effect on the adjacent axial methylene proton. This is seen also when one looks at other cycloalkanes with cis-1,3-dimethyl substitution.

Professor B. L. Shapiro
July 21, 1965
Page 2

To try to account for this, we can play with the various "effects" that people have invented over the past decade or so. But there is an obvious joker: Each methylene group has a local symmetry plane, bisecting the H-C-H angle and perpendicular to the plane of the H-C-H group. When methyl groups are introduced on the adjacent carbon atoms, the newly formed C-C bonds lie in this local symmetry plane. In other words, the two methylene protons are symmetrically disposed with respect to the entering methyl substituents, and no matter which "effect" is tried out, they ought to suffer the same perturbation.

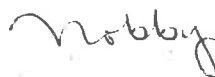
I do not know how to resolve this anomaly.

Every now and then you see something in print which implies that on the basis of one or more "effects" we are really in a rather good position to understand and predict chemical shifts. On the basis of data like those presented here, it seems hard to escape the conclusion that any simple theory of the origin of chemical shifts must be wrong. At any rate, my attitude on these publications is neatly expressed in the refrain of one of my number-one daughter's favorite folk songs:

And as I look around me I'm very apt to smile
To see so many people putting on the style.

With the best of wishes,

Sincerely,



Norbert Muller

NM/kd



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/CH

20th July, 1965

Dr. Bernard L. Shapiro,
Department of Chemistry,
Illinois Institute of Technology ~~Research Institute~~,
10, West 35th Street,
Chicago,
Illinois,
U.S.A.

Dear Dr. Shapiro,

1. Cooling System for A-60

Some readers may be interested in the details given below of the home-made closed-circuit water system we use for cooling our A-60 spectrometer. The help of Mr. J. Whitley is gratefully acknowledged.

Heat Exchanger

24in. x 14 in. diam. cylindrical copper tank with flanged top (material £12). Cooling tubes: three coils in parallel, each consisting of 25ft. of 3/8in. O.D. soft copper tubing wound on a 4 1/2 in. diam. former.

Reservoir

25in. x 3in. diam.

Pump

Monopump 'B'15 with Crane type 2 (RH) BR.191 mechanical seal fitted. (Monopumps Ltd., Selforde Street, London, E.C.1., England; £30).

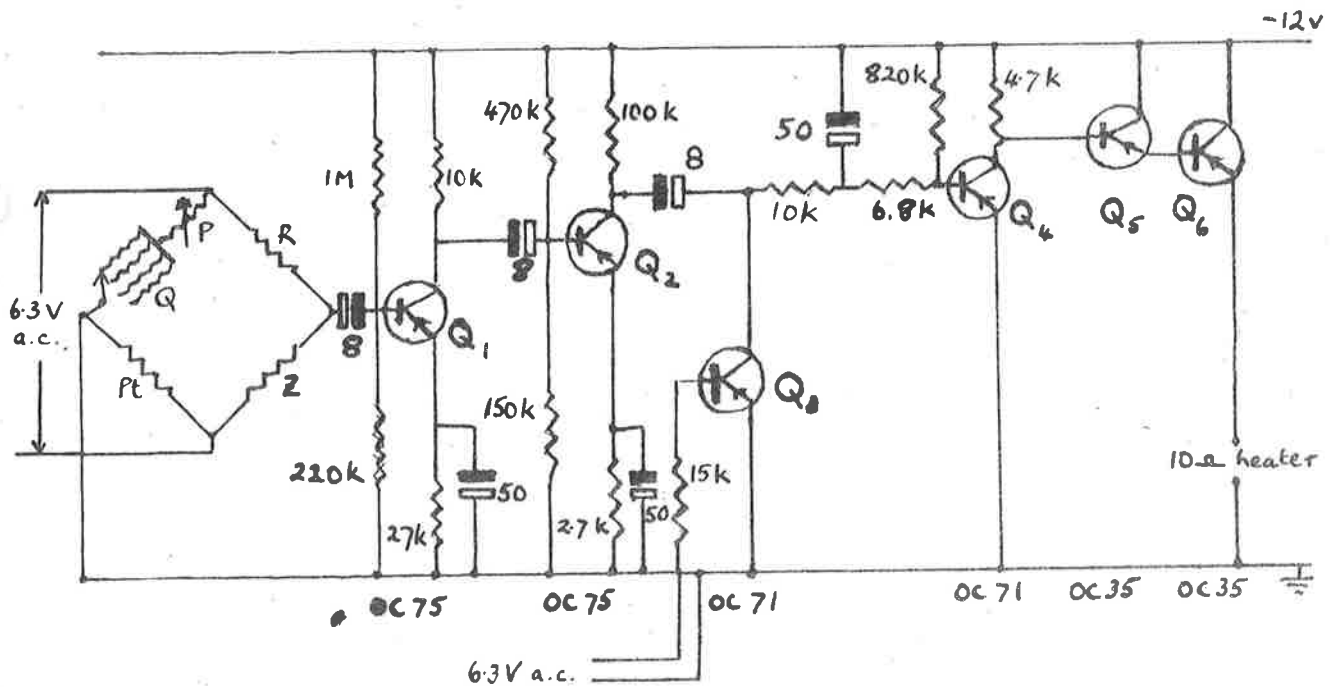
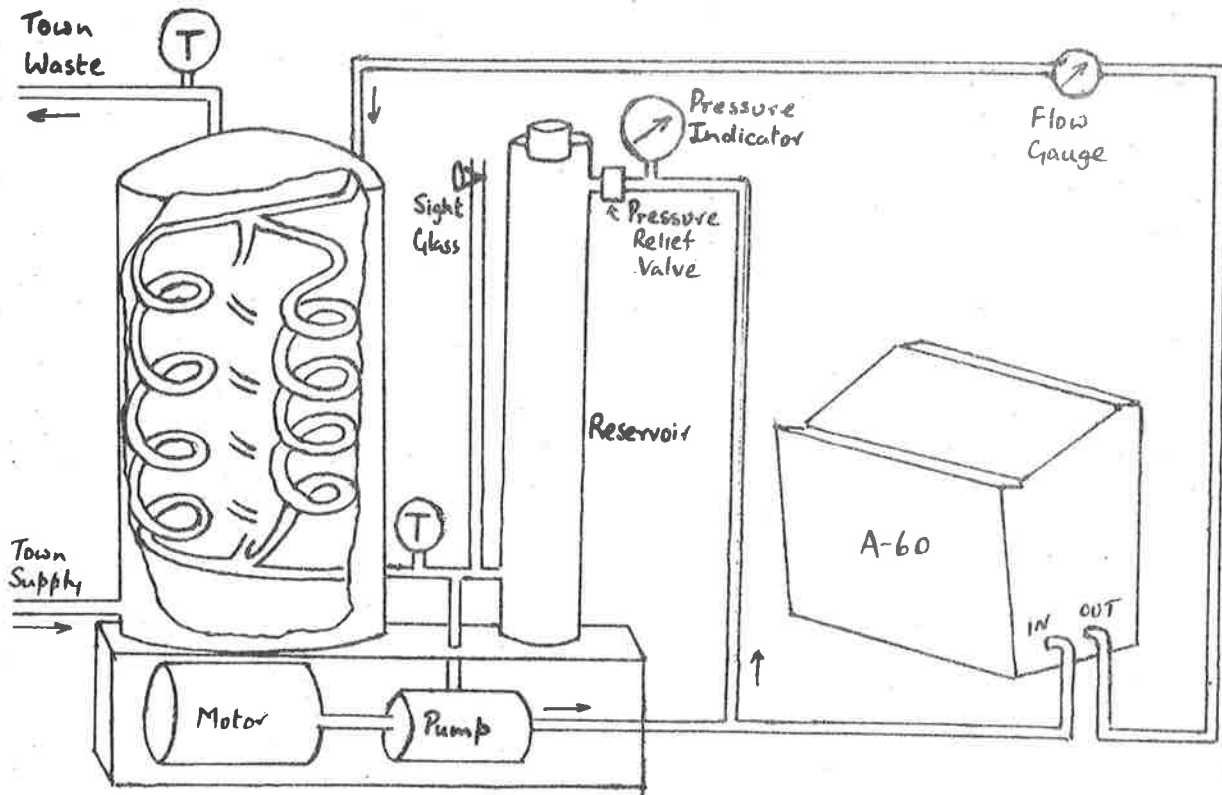
Motor

A.E.I. 1/4 h.p. electric motor, 1425 r.p.m., 240 v. (Cat. No. 35425), sleeve bearings, resilient mounting (A.E.I. Ltd. Trafford Park, Manchester, 17, England; £10).

Motor and pump are set within a frame 18 x 18 x 10 1/2 in., which supports the heat exchange and reservoir.

Thermometers (T) 30-110°F. 4in. dial (Cambridge Instrument Co., Cambridge England).

Continued.....



-2-

Flow Gauge Platon water Gasmeter A-10 14.5/HS (0.3-3.0 litres/min.) with brass end-blocks.

Closed-circuit System All copper piping is $\frac{1}{2}$ in. diam. The deionised water contains about 5 p.p.m. 'Panacide' (B.D.H., Poole, Dorset, England).

2. Sample Thermostat for Broad-line Spectrometer

In order to keep steady the temperature of a solid sample cooled by heat leaking down a brass rod from a refrigerant container, Mr. J. E. Pearson has fixed a small platinum resistance coil, labelled Pt, between the probe (linked to the spectrometer from below) and a 10ohm heater. The platinum coil consists of about four feet of 46 SWG wire and forms one arm of an a.c. bridge, the output of which is caused by the transistor circuit shown to actuate the above-mentioned heater when the temperature drops. (Reversal of the relative phase of the two a.c. sources will cause the heater to operate when the temperature has risen slightly!) Several temperature ranges can be selected by choice of Q.

Resistance P is a 1 kohm potentiometer and resistance R is chosen to have about the mid-range value of the Pt resistance, which is about 27 ohms. If a and b are the minimum and maximum values of Pt for a given range setting of Q, then the other bridge elements are given by:

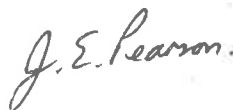
$$R = \frac{aP}{b-a} = 2.5 \text{ kohm for a } 100^{\circ}\text{C. range}$$

$Q = \frac{aP}{b-a}$; in our case, the four values 0.536, 1.52, 2.50, 3.48 kohms for Q give overlapping temperature ranges to cover +200 to -200°C.

Yours sincerely,



D. W. Jones



J. E. Pearson

A simple method of obtaining a 6DB improvement in S/N ratio in
A-60's

Without becoming involved in quoting relative noise figures of various devices I would like to pass on what I consider to be a significant improvement in signal to noise ratio of our A-60 here at C.S.I.R.O. Canberra. It is easily ascertained that the first stage of the receiver is the main source of electrical noise. On looking around I found that as far back as July 1960 Electronics (P73) Radio Corporation of America claimed a 2-4DB improvement in S/N for their 6CW4 nuvistors. Without further ado two were mounted on a noval plug and the 6922 at V201 was directly replaced. Realignment of the receiver and probe was necessary. A check of characteristics showed that the operating conditions of V201 Cascode first stage would be quite satisfactory without any circuit value alterations.

Although Varian quote 5:1 signal to noise we had been able to obtain 7 or 8:1. With this simple replacement we were able to obtain 15 or 16:1 on the quartet of 1% ethyl benzene without any degradation of the .3 cps resolution with acetaldehyde over the last month.

I don't think this matter should rest here as there is still room for improvement possibly with Field effect transistors or tunnel diodes although this would require a lot more work to install. I am sure that this could have other possible applications in other instruments. Just at the moment I cannot see any advantage in incorporating this in the control receiver.

William P.A. Pascoe
27.VII.65

W. R. GRACE & CO.
RESEARCH DIVISION



Washington Research Center, Clarksville, Maryland 21029

July 13, 1965

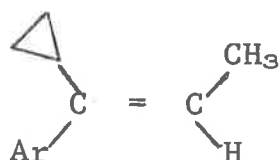
Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry,

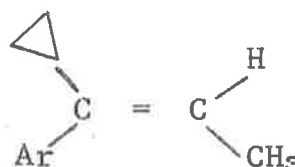
An Unusual Chemical Shift in Propenyl-Benzene Derivates

In initiating our subscription to IIT NMR Newsletter, I would like to report an unusual chemical shift.

We have examined compounds of the type Ia and Ib by PMR.



Ia



Ib

The aryl groups were varied as shown in the table below.

We attempted to assign the geometrical isomer structure to the compounds on hand by assuming that the olefinic proton in Ia would be shifted downfield relative to the olefinic proton in Ib due to the nearby aromatic ring current. In every case, assigning structure in this way gave the same assignment as that arrived at by other chemical and physical methods, i.e., retention times and pyrolysis rates to known products.

The isomeric shift of the methyl group is a phenomenon that is surprising. If the assignments shown below are correct, then the methyl group in compound Ib is upfield from the methyl group in Ia by 0.4 ppm.

W. R. GRACE & CO.
Research Division
 WASHINGTON RESEARCH CENTER
 CONTINUATION

- 2 -

The methyl proton resonance in cis-and trans-propenyl benzene shows no large shift.

Furthermore, the methyl proton shift in Ia is normal for such systems while the shift in Ib is at higher fields than is normally found in the propenyl benzenes.

We feel that this shift is due to cyclopropyl-double bond conjugation, in Ib, which is absent in Ia. We are testing this idea by studying similar compounds. IR and uv data do not show differences, however, that give weight to this hypothesis.

We welcome any suggestion as to the cause of this chemical shift.

Sincerely yours,

A. J. Berlin
 A. J. Berlin

TABLE I

Chemical SHifts of Olefinic and Methyl Protons in
 Various Compounds of the Type Ia and Ib

<u>Compound</u>	<u>τ, CCl_4, 5%</u>	
	<u>Olefinic Proton</u>	<u>Propenyl Methyl Protons</u>
Ia, Ar= p-methoxyphenyl	4.40	8.12
Ib, Ar = p-methoxyphenyl	4.58	8.53
Ia, Ar = phenyl	4.29	8.12
Ib, Ar = phenyl	4.52	8.52
Ia, Ar = p-fluorophenyl	4.33	8.12
Ib, Ar = p-fluorophenyl	4.52	8.52

Glaxo Research Limited

Greenford · Middlesex

TELEPHONE: BYRON 3434 TELEGRAMS: Glaxotha, London, Telex CODE: New Standard, Bentleys

Enclosures as supplied:

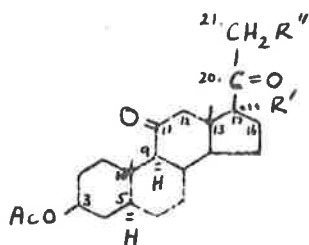
13th July, 1965.

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

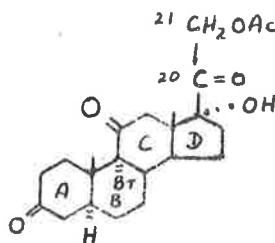
Dear Professor Shapiro,

Proton Resonance Spectra of 11-Oxosteroids

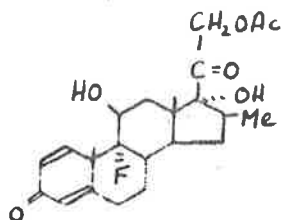
We have recently recorded the p.m.r. spectra of a wide range of 11-oxosteroids and have examined the effect of various ring-C and ring-D substituents on the proton resonances of the 12-methylene, 10-methyl, and 13-methyl groups. Since the spectra of the simpler 5 α -androstan-11-ones have been the subject of several recent papers,^{1,2} a summary of our work, which is being submitted for publication in the Journal of the Chemical Society might be of interest.



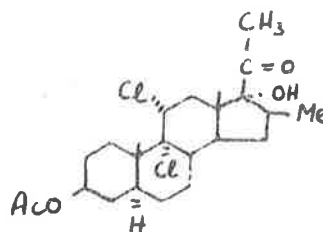
(I)



(II)



(III)



(IV)

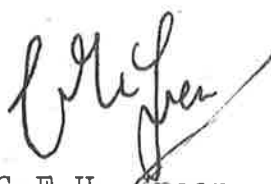
The 12-methylene protons of simple androstan-11-ones, pregnane-11,20-diones (e.g., I; $R' = R'' = H$), and 11-oxo-isosapogenins show a two-proton singlet in $CDCl_3$ solution, but an AB quartet in pyridine or benzene solution, whereas the 12-methylene protons of ergostan-11-ones and 21-acetoxypregnane-11,20-diones (e.g., I; $R' = H$, $R'' = OAc$) form an AB quartet in $CDCl_3$, benzene and pyridine solution. Provided the steroid does not have a 17α -substituent, the doublet for the equatorial 12β -proton appears at lower field than the axial 12α -proton. In 17α -hydroxypregnane-11,20-diones (e.g., I; $R' = OH$, $R'' = OAc$), the 17α -hydroxyl deshields the 12α -proton and causes the 12α -proton peaks to appear at lower field than those for the 12β -proton. Introduction of a 9α -halogen (e.g., II) deshields the 12α -proton still further, the extent of the downfield shift depending on the size of the halogen atom.

The 12-methylene protons of 9α -halogeno-11 β ,17 α -dihydroxy- (e.g., III) and 9α ,11 β -dihalogeno-17 α -hydroxy-pregnane-20-ones (e.g., IV) also give an AB quartet, the doublet for the axial 12α -proton appearing at lower field than that for the equatorial 12β -proton.

Yours sincerely,



J.E. Page



G.F.H. Green



S.E. Staniforth

- 1.) N.S. Bhacca and D.H. Williams, "Applications of N.M.R. Spectroscopy in Organic Chemistry". Holden-Day, San Francisco, 1964.
- 2.) E.R.H. Jones and D.A. Wilson, J. Chem. Soc., 1965, 2933.

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

July 28, 1965

AIRMAIL

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

Dear Barry:

Audio-Frequency, Voltage-Swept Generator for a Field-Frequency Lock

We are now in the process of completing an internal field-frequency lock of the Anderson-Freeman type for our HR-100. The only unusual feature of the system is the frequency-sweep generator. Since one of the main reasons for our putting this system together was a need to use the 100 with a CAT, we decided that the sweep generator should not employ any mechanical elements such as a motor-driven potentiometer or capacitor. We therefore turned to a solid-state voltage-to-frequency converter which could be driven by the ramp output of an integrator of the type which we described in our last two letters.

We are using a specially modified Vidar (Mountain View, Calif.) Model 241 Converter. This unit provides a pulse-rate output proportional to the DC input voltage with a normal range of 0 to 10 Kc. for an input change of 0 to 10 V. Our unit was ordered with an extra "flip-flop" circuit board which divides the output frequency by two and provides a square wave rather than a pulse output. In addition we specified a special span and zero input frequency that has since been modified so as to give 1000 to 4000 cps (square wave) for an input variation of 0 to +10 V with a full scale linearity of better than 0.025% and a short-term stability (1 record averaging time) of the order of 2 to 3 ppm.

Since a true square wave contains no second harmonic component, it is possible to use a low pass filter with a sharp cutoff characteristic to obtain a uniform sine wave output over somewhat less than a 2.5:1 frequency range. We increased the frequency of our integrator to 2.5 Kc so it would lie somewhat within a 2 Kc continuous sweep (from about 2 to 4 Kc) which should be more than ample for most 100 Mcps. work. We also have provided a second range which will let us

sweep down from 2.5 Kc to 1.0 Kc with only a little distortion at the lowest frequencies so that we can use the lock on our 56.4 Mcps system in either the field-or the frequency-sweep mode without having to compensate for the severe eddy-current losses in the thicker-walled probe that begin to set in above 2 Kc.

Our locking system utilizes two Varian integrators. One is tuned to 2.5 Kc and contains at the input a very narrow-band, 2.5 Kc notch filter employing a transistor amplifier and twin-tee network that can be switched into the system when we are decoupling in the frequency-sweep mode to prevent the decoupling frequency from disturbing the lock. The second integrator has been extensively modified so that it can be driven by the output of the variable frequency generator. It's amplitude response is flat to within about 5% over either sweep range, and there is little detectable change in the phase of the signal. A means has also been provided for obtaining, for counting purposes, the frequency representing the difference between the fixed and variable frequencies. This unit is used to provide the locking signal in the field-swept mode and the spectrum in the frequency-swept case. We would be glad to supply details to anyone interested.

Sincerely yours,



R. E. Lundin, Principal Chemist



R. H. Elsken, Electronics Engineer
Molecular Structure Investigations
Wool and Mohair Laboratory



Title: A56/60 Spectra of $\text{CF}_3\text{CF}_2\text{CF}_2\text{H}$

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

July 29, 1965

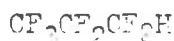
Dr. B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois

Dear Barry,

I had better pay my dues to your magazine or else my credit rating will be in jeopardy from some IIT collection agency.

We have very recently received our A-56/60 spectrometer and are learning how to use it and all its idiosyncrasies. Thus far this instrument seems to be considerably more stable than our old A-60 and to have about 3-4 times the signal to noise figure of the latter. However, to obtain good resolution is still a tricky problem so far and the ^{19}F frequency offset oscillators seem to draft rather more than one would wish.

Enclosed are some ^{19}F spectra of a sample of $\text{CF}_3\text{CF}_2\text{CF}_2\text{H}$ which I cooked up several years ago. These spectra reveal the magnitudes of two small coupling constants which were not observed by my colleague Dan Elleman back in the late 1950's when he first looked at the n.m.r. spectra of this molecule with the spectrometer he built in his thesis work. Below are listed the observed first order coupling constants derived from measurements of both the ^1H and ^{19}F spectra recorded with our A-56/60 spectrometer with calibrated sweep widths.



3 2 1

$$J_{F_1H_1} = +51.96 \pm 0.04 \text{ cps}$$

$$J_{F_2H_1} = +4.57 \pm 0.04 \text{ cps}$$

$$J_{F_3H_1} = | 0.88 \pm 0.02 | \text{ cps}$$

$$J_{F_1F_2} = -4.50 \pm 0.04 \text{ cps}$$

$$J_{F_1F_3} = | 7.23 \pm 0.05 | \text{ cps}$$

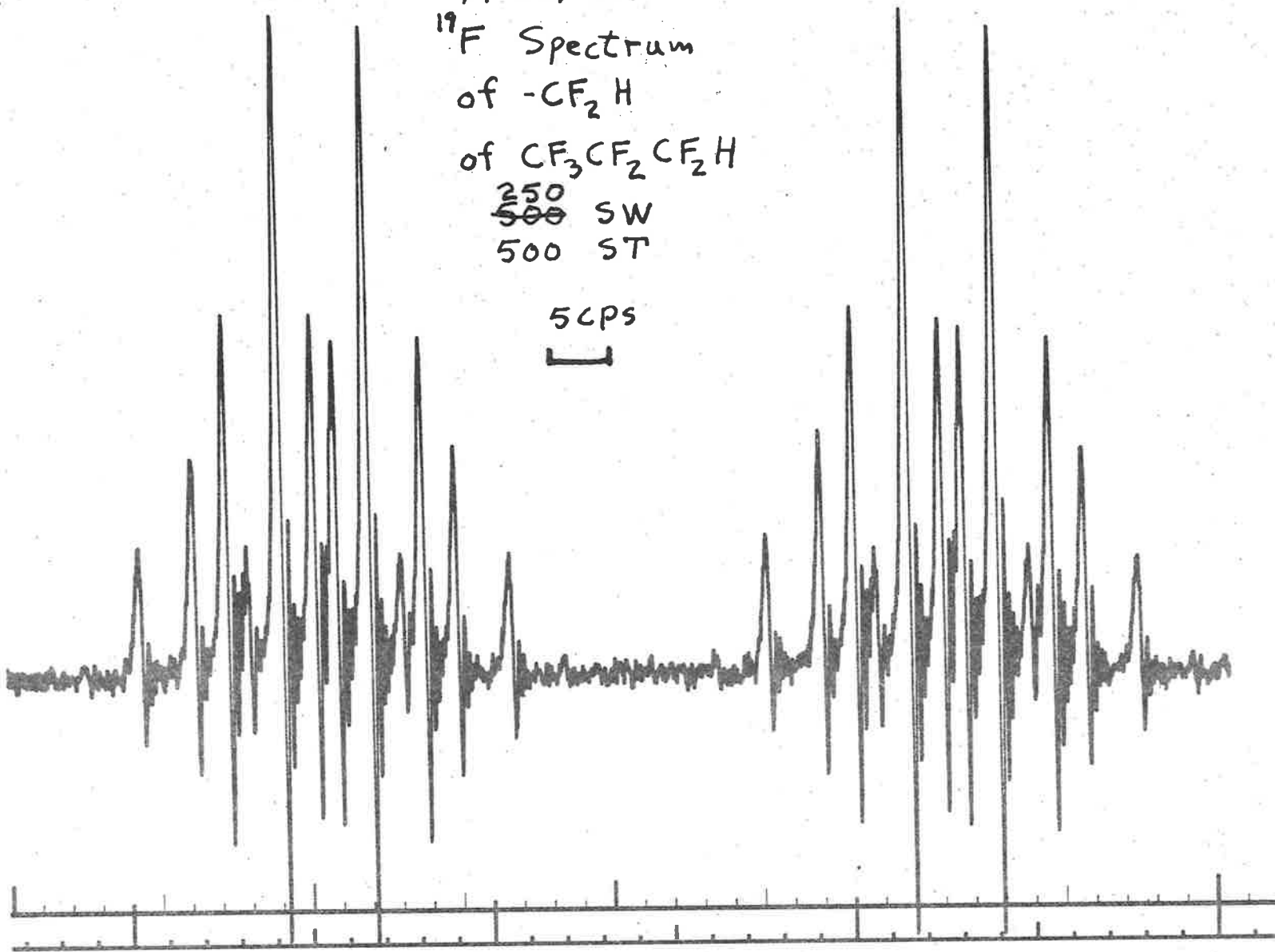
(probably +)

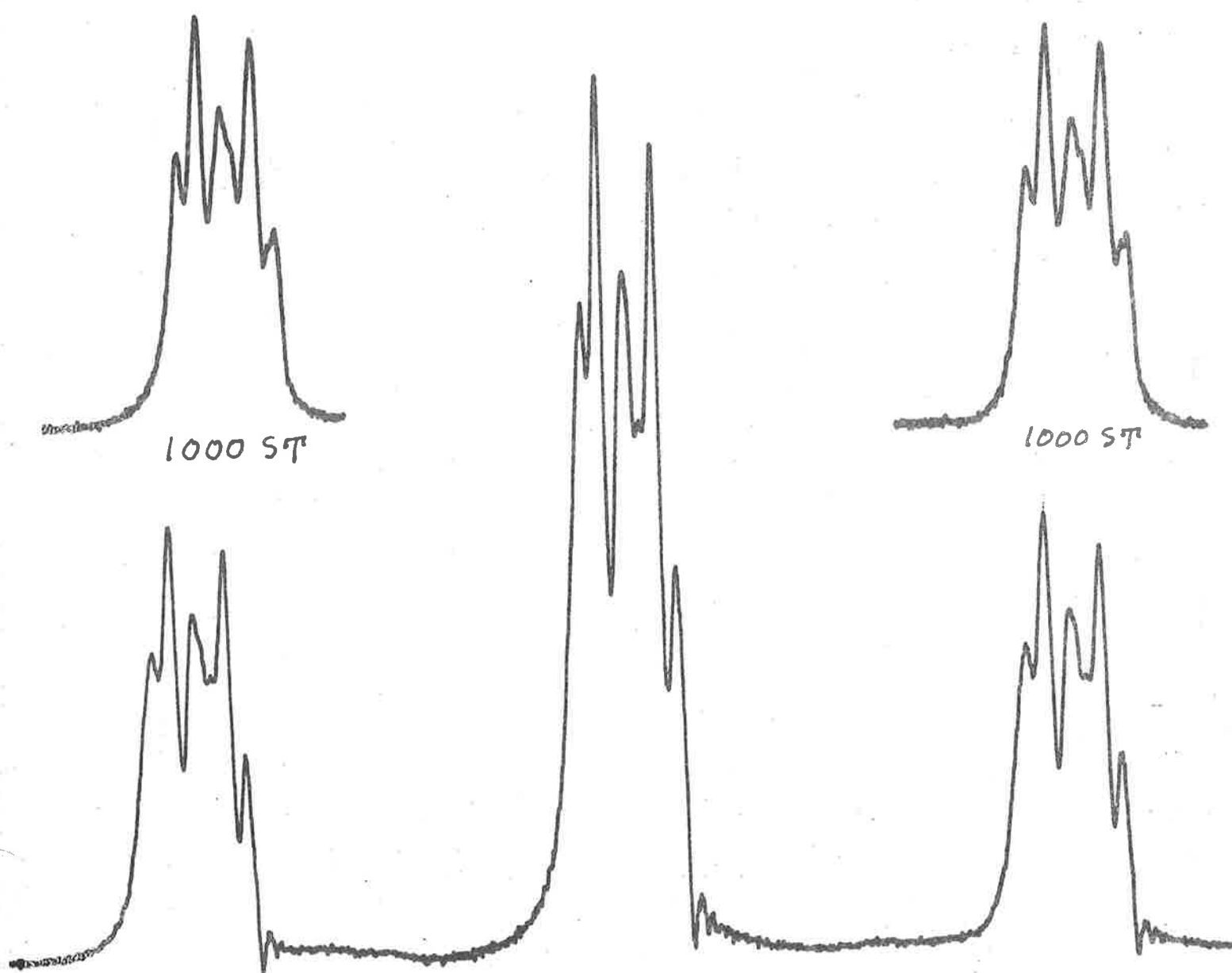
$$J_{F_2F_3} = | 0.35 \pm 0.02 | \text{ cps}$$

Now that we know the small couplings are there we will try to determine some signs in frequency - sweep experiments with our HR instrument. The signs given above are from our previous work on this and other fluorocarbon molecules involving field-sweep ^{19}F - [^{19}F], ^{19}F - [^1H] and ^1H - [^{19}F] experiments.

A 56/60
 ^{19}F Spectrum
of $-\text{CF}_2\text{H}$
of $\text{CF}_3\text{CF}_2\text{CF}_2\text{H}$
250 SW
~~500~~ ST

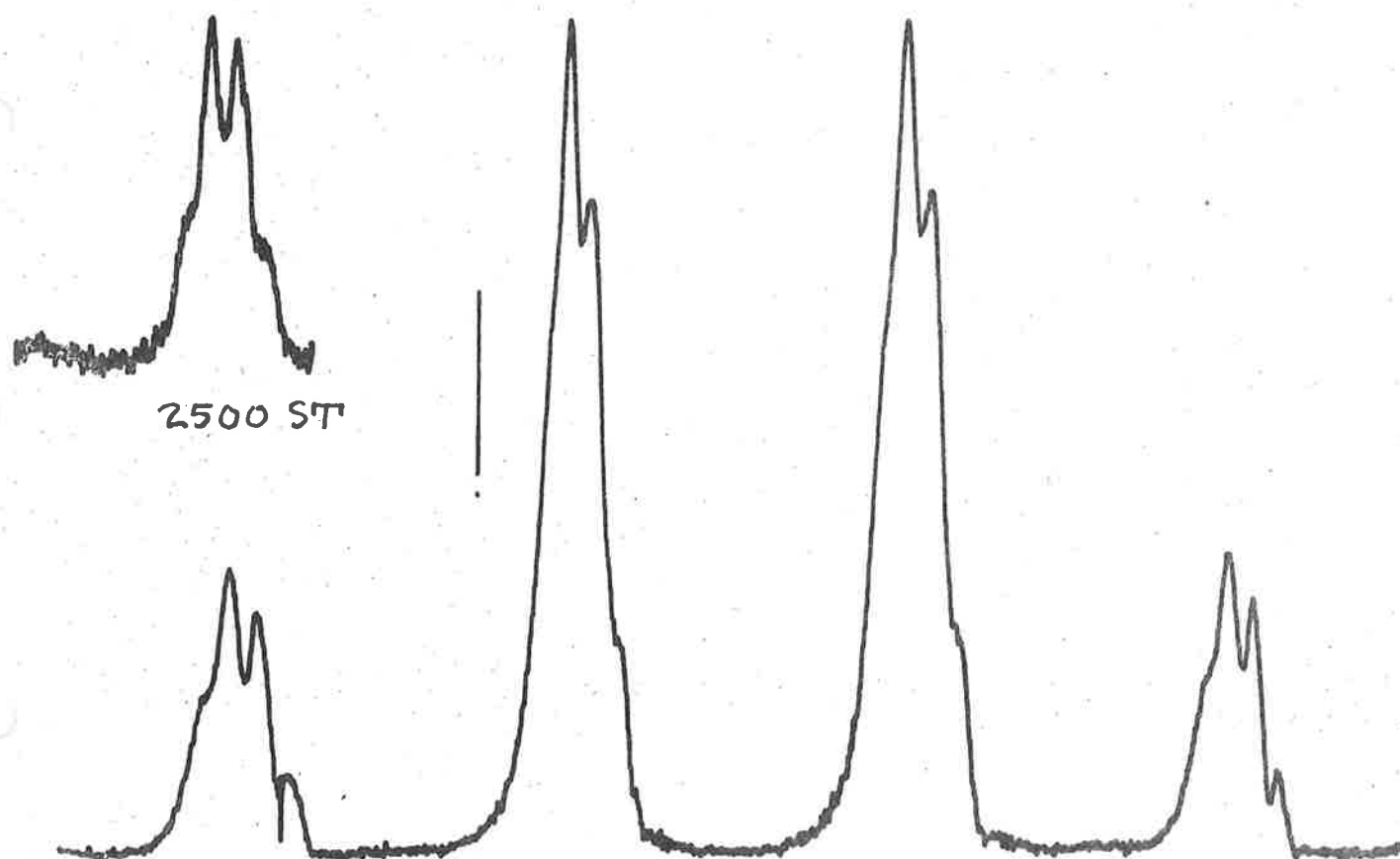
5 cps





A 56/60 ^{19}F Spectrum of CF_3^- of $\text{CF}_3\text{CF}_2\text{CF}_2\text{H}$
50 SW 500 ST





A 56/60 ^{19}F Spectrum of $-\text{CF}_2-$ of $\text{CF}_3\text{CF}_2\text{CF}_2\text{H}$
50 SW 500 ST

האוניברסיטה העברית בירושלים
THE HEBREW UNIVERSITY OF JERUSALEM

DEPARTMENT OF PHYSICAL CHEMISTRY

המחלקה לכימיה פיסיקלית

July 27, 1965

Professor B.L. Shapiro, Editor
IITNMR
Illinois Institute of Technology
Chicago, Illinois
U.S.A.

Dear Barry:

From my summer travels, the abstract of a paper I have recently submitted to J.Chem.Phys., which might be of interest to your readers.

"On the Magnetic Susceptibility of Aromatic
Hydrocarbons and Ring Currents".

It is shown that the anisotropic magnetic susceptibility (and chemical shift) of aromatic hydrocarbons generally attributed to π -electron "ring currents" can be correctly represented as the sum of contributions from localized electrons of both π - and σ -character. The "delocalization" of the electronic distribution plays no part whatever in the effect and is only an artifact of London's approximate calculation. Anisotropic Pascal's constants for aromatic carbon atoms are determined empirically and excellent agreement with the experimental anisotropic susceptibilities is obtained.

I will be pleased to send a Xerox copy of this to anyone who is desperately interested when I return to Rockefeller Institute in September.

The implications of this work for NMR are (1) π -electrons in aromatic rings are to be treated no differently than any other anisotropically distributed electrons, such as the σ -electrons in benzene or in cyclohexane; and (2) a definition of aromaticity based on "ring currents" does not really make sense. My feeling on aromaticity of large monocyclic rings is that all bonds should be of equal length so that a regular two-dimensional geometry is obtained for a molecule to be called aromatic. The bond alternation discussed by Longuet-Higgins and Salem, plus an angle alternation occurs in the annulenes: by any non-ring current argument these molecules cannot be considered aromatic.

With best regards.

Yours sincerely,

Jeremy

Jeremy I. Musher

JIM:sn

Reprint
and
28.9.65

Battelle Memorial Institute • COLUMBUS LABORATORIES

505 KING AVENUE COLUMBUS, OHIO 43201 • AREA CODE 614, TELEPHONE 299-3151 • CABLE ADDRESS: BATMIN

July 29, 1965

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

Dear Barry:

In IITNMR #73, Page 4, we mentioned a cabinet which we built to improve the field stability of our HR60 and promised to send our plans. We have had the drawings completed for some time but have been derelict in our duty to send them in for general circulation.

The need for the various access doors shown in the attached plans is for the most part obvious except possibly those denoted "E" and "G". These doors were installed simply to enable us to uncable and recable the magnet completely without removing the cabinet. To do high-temperature work, we simply open doors "A" and "C". The leads connecting the super stabilizer and the sweep coils are passed through the small hole above door "C".

We particularly recommend that anyone who builds a cabinet based on the enclosed design recheck the gross dimensions for his particular system. Our magnet was removed from the wooden pallet during installation and was set on an "I" beam frame to distribute the floor load. For this reason, it is probably atypical.

We built this cabinet ourselves. However, Scientific Advances, Incorporated, (SAI) 1400 Holly Avenue, Columbus, Ohio, 43212, has indicated that they might be willing to quote on and build a "cabinet kit" for anyone who is interested. Bob Watkins of SAI can be contacted by writing to the above address or by calling him at (614) 294-5436.

Best personal regards,

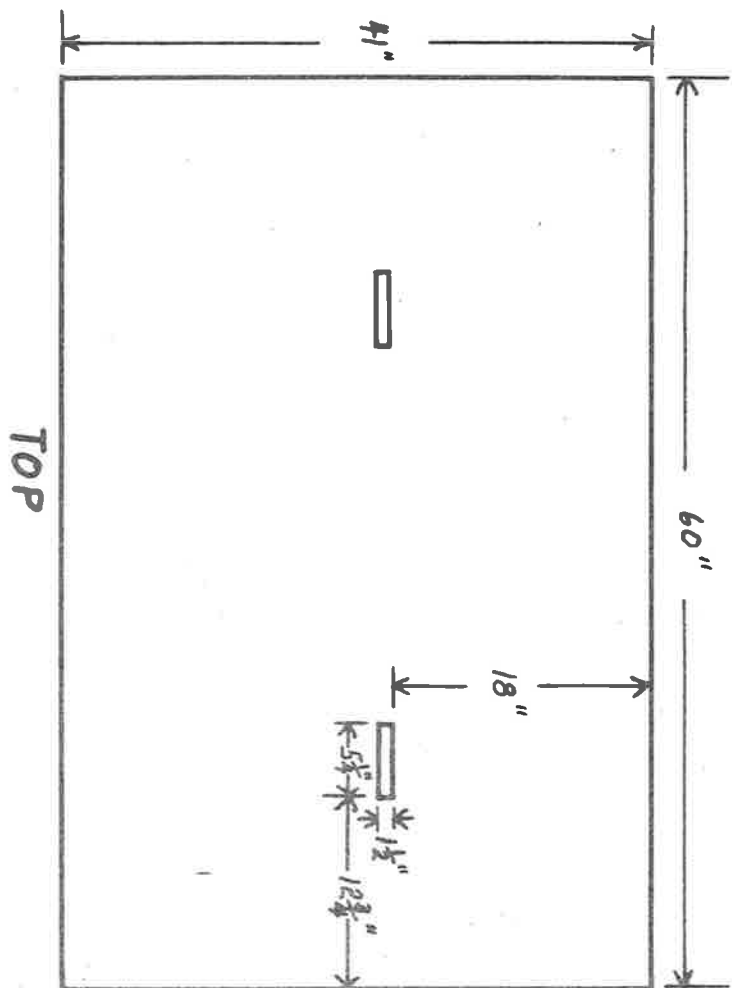
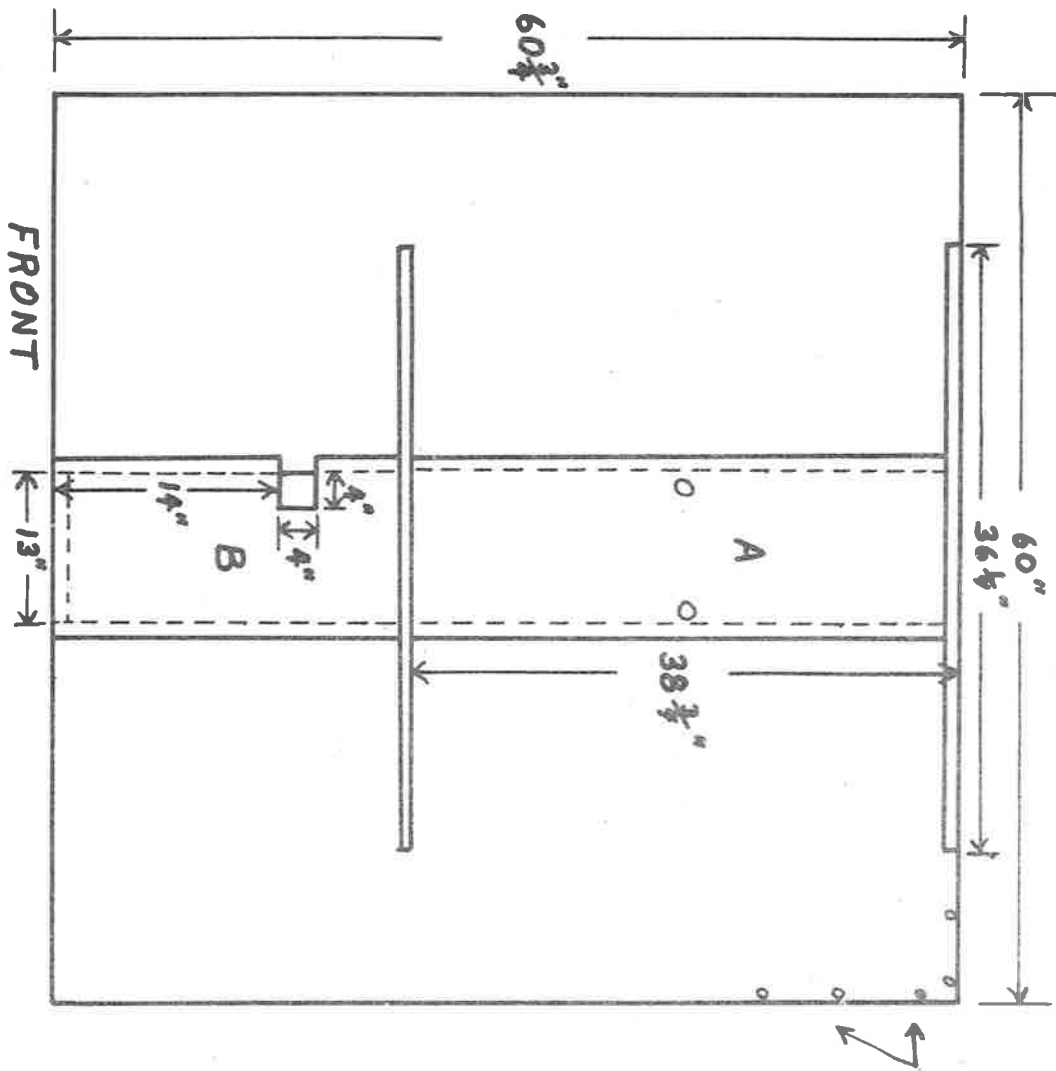
Tom

Thomas F. Page, Jr.

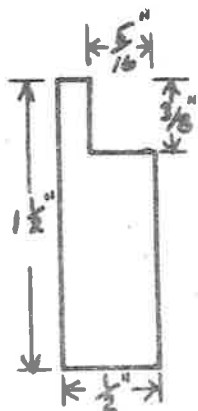
Warren E. Bresler

Warren E. Bresler
Molecular Spectroscopy

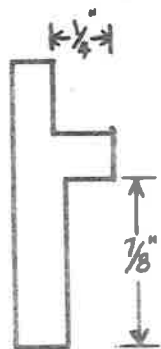
TFP:WEB/gf
Enclosures



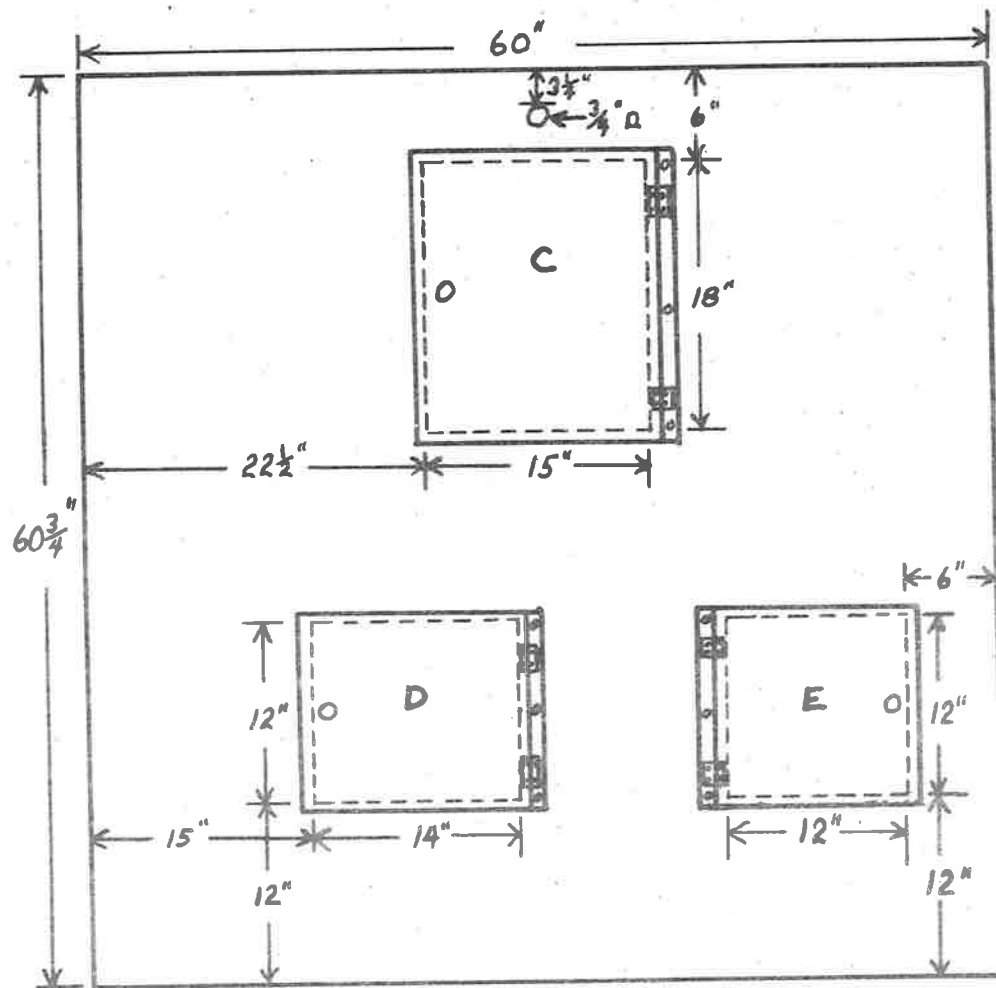
SCREW HOLES AROUND EDGES OF ALL PANELS
APPROX. 5 3/4" BETWEEN CENTERS.
DOORS OVERLAP OPENINGS 1/2".



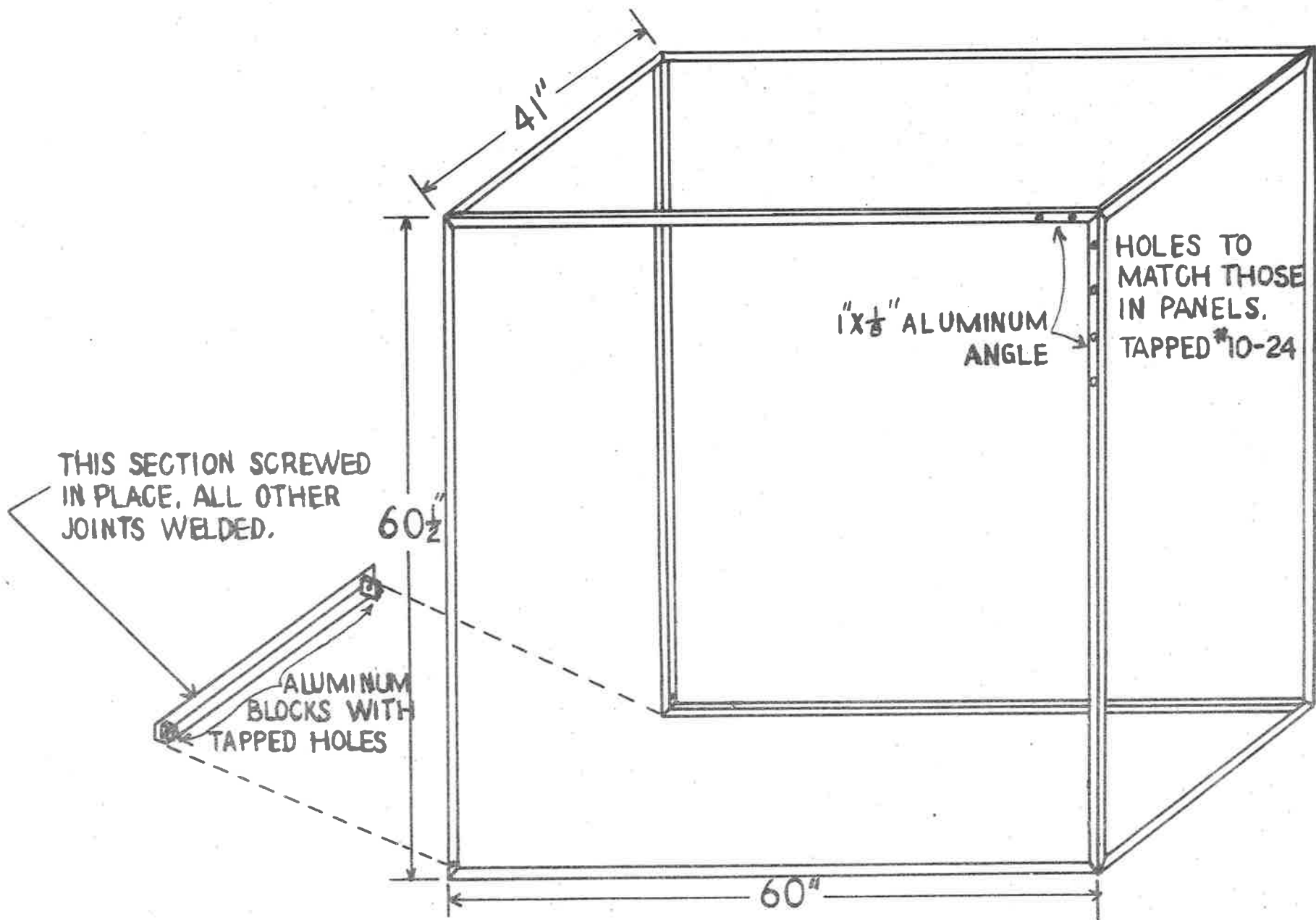
CROSS SECTION OF
SLIDING DOOR
TRACKS



CROSS SECTION OF CENTER
PORTION OF BOTTOM TRACK.
CUT OUT FOR TOP EDGE
OF PANEL B.



BACK



CABINET FRAME

Organisch-Chemisches Institut
der
Technischen Universität Berlin
Direktor: Prof. Dr. F. Bohlmann

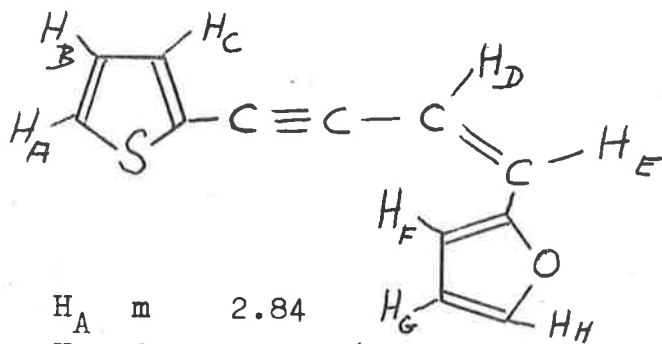
1 Berlin 12, den 30. Juli 1965
Straße des 17. Juni Nr. 115 (Chemiegeb.)
Fernruf: 32 51 81, App. 252 Prof. B/Ma

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

Ch i c a g o , 60616 USA

Dear Dr. Shapiro:

Perhaps the NMR-data of the following compound is of interest to you as an example of long range coupling (H_{EG} and H_{EH}):



H_A	m	2.84	
H_B	dd	3.08	($J = 4.7$ and 4.0)
H_C	m	2.84	
H_D	d	4.36	($J_{DE}=12$)
H_E	ddd	3.42	($J_{DE}=12$; $J_{EG}=0.6$; $J_{EH}=0.6$)
H_F	d	2.99	($J_{FG}=3.4$)
H_G	ddd	3.63	($J_{FG}=3.4$; $J_{GH}=1.8$; $J_{EG}=0.6$)
H_H	ddd	2.69	($J_{GH}=1.8$; $J_{EH}=J_{FH}=0.6$)

We have now finished the rearrangement of our NMR-combination. We have transformed the HR 100 to the HA 100 and combined it with a DP 60 using a second magnet but the same consol. Also the "Cat" is working well with the HA 100. Dr. Arndt perhaps will write down the special requirements when he is returning from holydays.

Yours sincerely,

F. Bohlmann

ircha

Institut
national
de recherche
chimique
appliquée

83-25
établissement public à caractère
industriel et commercial,
12 quai Henri IV, Paris 4
rc Seine 58 b 7806
n° d'entreprise 971 75 104 9051
ccp Paris 9085 96 au nom
de l'agent comptable de l'ircha

Direction générale
Services de Paris
12 quai Henri IV, Paris 4
téléphone : 272 82 70

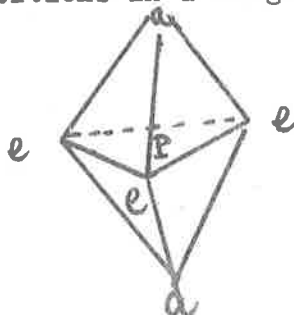
Pr. B.L. SHAPIRO
Dpt. of Chemistry
Illinois Institute of Technology
CHICAGO, Ill. 60616 -
U.S.A.

Paris, 30th of July, 1965

Dear Barry,

I hope I beat the deadline before leaving for Copenhagen. When preparing a review article on NMR studies on phosphorus compounds except P_{31} resonance - I have found that, generally speaking, any accurate interpretation of observed spin-spin couplings and, to a greater extent, chemical shifts is deceiving. Some rough correlations with hybridization, electronegativity, a.s.o., are obtained in restricted series, but they are complex, due to many significant overlapping factors. The only case where it seems possible to improve the description of published data on the same grounds is the following.

F_{19} - P_{31} couplings are observed in a number of pentacoordinated phosphorus compounds (**). With R_2PF_3 compounds, it is possible to distinguish axial and equatorial fluorine atoms, R's occupying generally equatorial positions in a trigonal pyramidal conformation (*** with $d=d_{z^2}$) :



- (*) : To appear in Adv. in NMR spectroscopy (L.H. Sutcliffe ed.), vol.2;
(**) : See R. Schmutzler, Angew. Chem. 77, 530, 1965 for an extensive review of experimental data;
(***) : but exceptions are known : $(Me_2N)_2PF_3$ for example, Muetterties et al. Inorg. Chem. 3, 1298, 1964.

-2-

One observes that $J_{\text{axial}} < J_{\text{equ.}}$ (the ratio ranging from 0.8 to 0.95), axial resonance being downfield relative to equatorial one; these facts being interpreted as a noticeable difference in s character of P F bonds. Our interest is especially in J couplings, as they reflect directly phosphorus hybridization. To study more quantitatively this relation, we describe orbitals in such a pentacoordinated phosphorus by the two following kinds of hybrids (F.A. Cotton, "Chemical applications of group theory", Interscience 1964, p.116 sqq) :

$$\begin{aligned} \text{equatorial} & \quad \frac{1}{\sqrt{3}} (s \sin \alpha - d_{z^2} \cos \alpha) + \frac{2}{\sqrt{6}} P_x \\ \text{axial} & \quad \frac{1}{\sqrt{2}} (s \cos \alpha + d_{z^2} \sin \alpha) + \frac{1}{\sqrt{2}} P_z \end{aligned}$$

α an adjustable parameter. If we assume that the Fermi contact is pre-eminent and that it varies mainly with s content of P orbitals, we have :

$$\begin{aligned} \frac{J_a}{J_e} &= \frac{3}{2} \cot^2 \alpha \\ \text{with : (s character)}_a &= \frac{1}{2} \cos^2 \alpha = \frac{(J_a/J_e)}{3 + 2(J_a/J_e)} \\ (\text{s character})_e &= \frac{1}{3} \sin^2 \alpha = \frac{1}{3 + 2(J_a/J_e)} \end{aligned}$$

The experimental ratios yield an estimate of these characters. To illustrate :

<u>R's</u>	<u>J_a/J_e</u>	<u>s_a</u>	<u>s_e</u>
Me ₂	.804	.174	.217
Me	.840	.179	.213
φ ₂	.864	.182	.211
φ Cl	.924	.190	.206
Cl ₂	.945	.193	.204

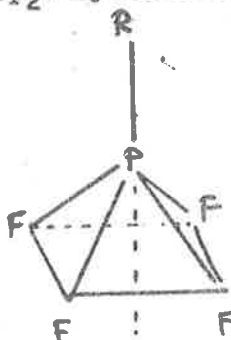
As R's electronegativity increases (F electronegativity being always greater), axial and equatorial bonds tend to an equal s character. This is consistent with WALSH's rule (*) : phosphorus has orbitals of s content with the most electropositive substituents. The following figure illustrates this dependence (G_x = Taft's inductive constant).

Despite its roughness (assuming all equatorial orbitals equivalent underestimates s_e and surestimates s_a), this model seems of some interest. Of course, similar data on PF₅, RPF₄ would be of

(*) : (See H.A.BENT, Chem. Revs. 60,275, 1960 and other references)

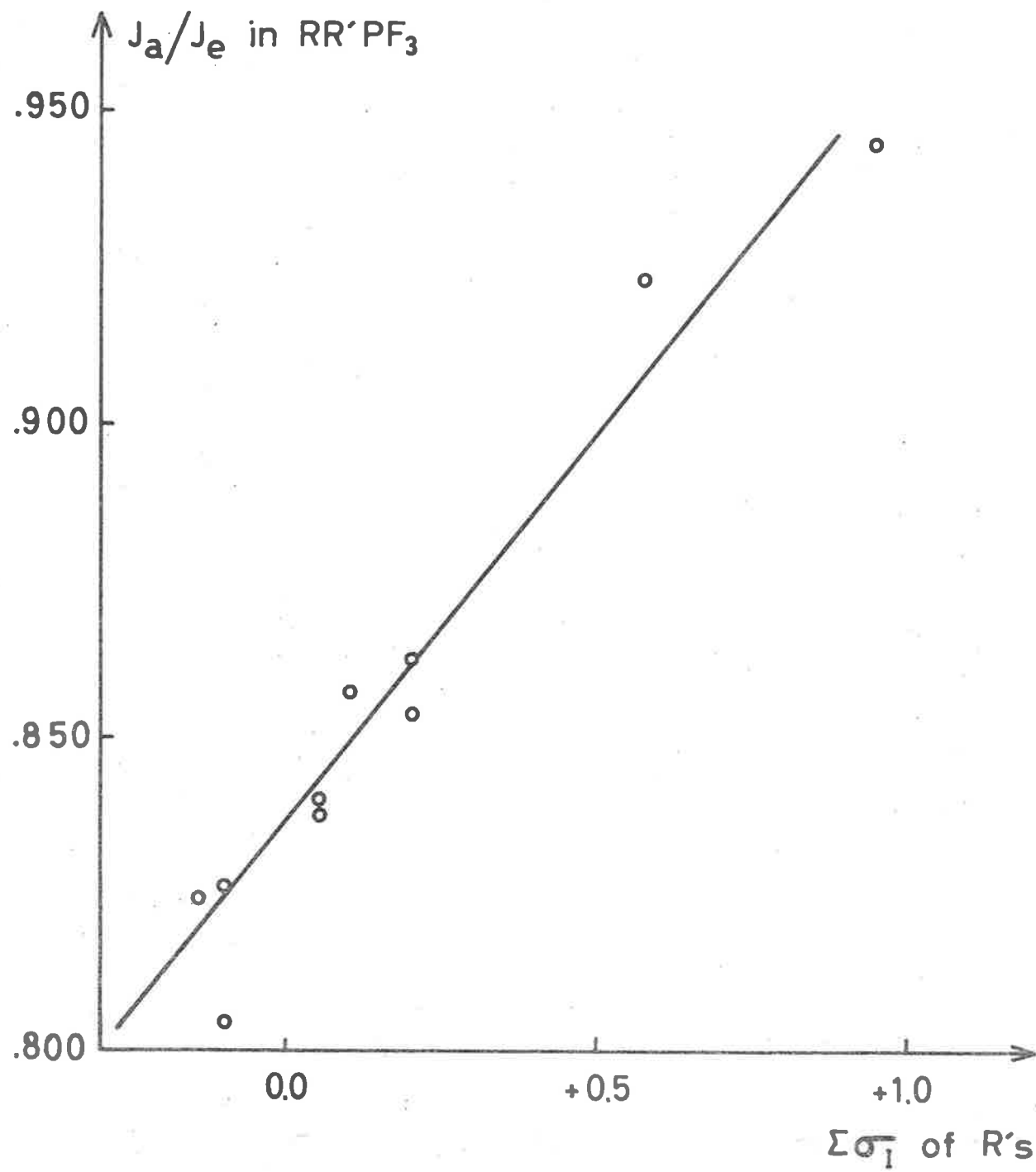
-3-

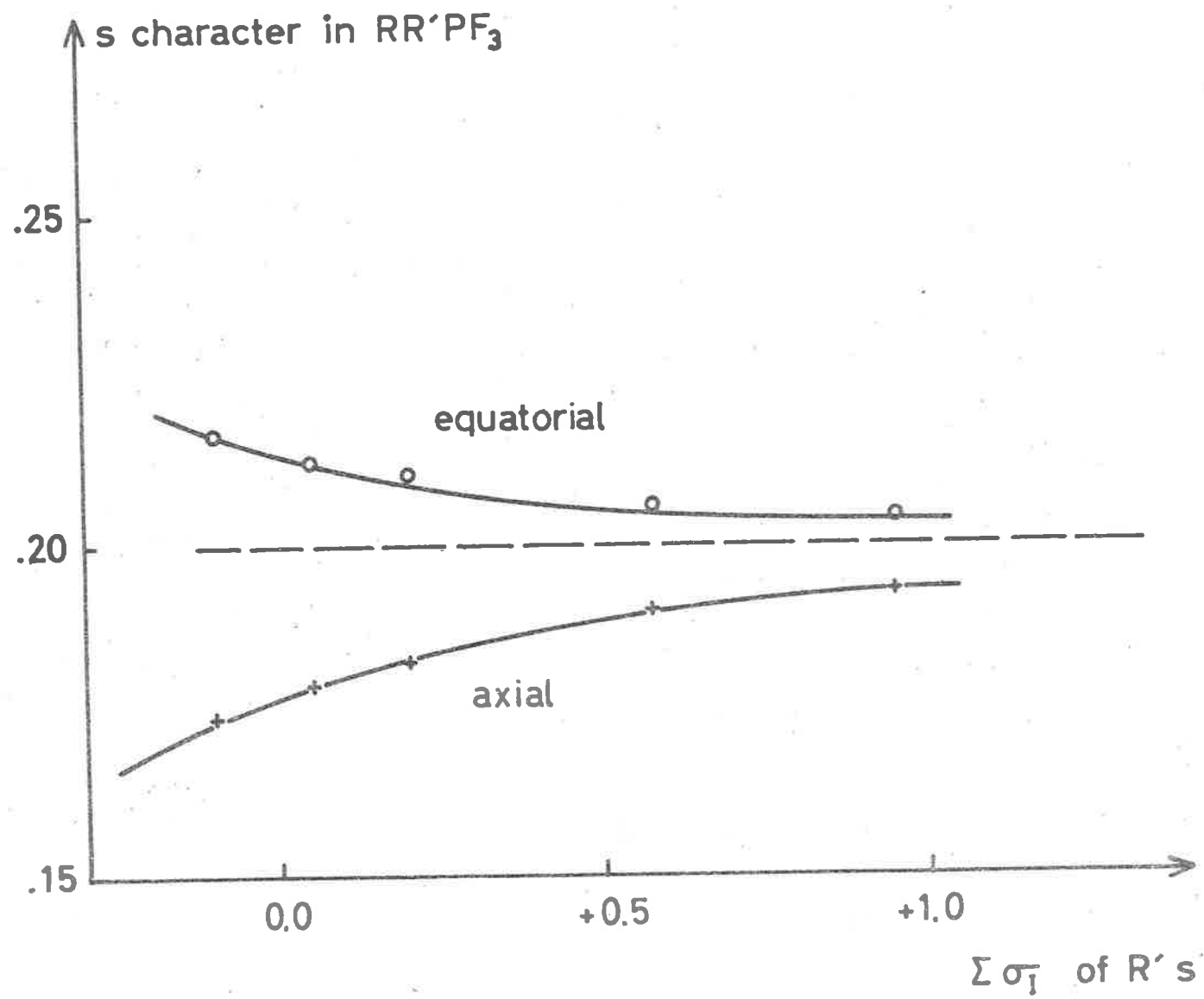
considerable interest, but fluorine intramolecular scrambling occurs (at room temperature at least) in PF_5 and perhaps in RPF_4 . For the latter compounds, another explanation for fluorine equivalence is the possibility of another sp_2d hybridization (with $d = d_{x^2-y^2}$). (See E.L. Muetterties et al., Inorg. Chem. 2, 615, 1963).



With my very best regards,

G. MAVEL.







2 August 1965

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

Dear Barry:

SPITTOON

An accurate method of measuring ^{13}C transition frequencies is to monitor the splitting that occurs on a ^{13}C satellite line in the proton NMR spectrum when a weak rf field (H_2 at ω_2) is swept through a connected ^{13}C transition. Baker has called this the INDOR experiment [J. Chem. Phys. 37, 911 (1962)]. The resulting spectra consist of a series of negative-going peaks that correspond in general to certain ^{13}C subspectra, which may be combined to form an (inverted) replica of the ^{13}C spectrum, but with the benefit of the higher sensitivity of proton NMR. Although this is perhaps not self-evident, the statistical weights of the lines in an INDOR multiplet are the same as those of the corresponding ^{13}C multiplet (i.e., 1:3:3:1 for a quartet). The resemblance to ENDOR is rather superficial; the experiment is basically spin tickling observed in the frequency sweep (ω_2) mode. Although theoretically it may be complicated by population redistributions, these are usually negligibly small because of the lower magnetogyric ratio and much longer relaxation times of ^{13}C .

Unfortunately ^{13}C satellites are themselves rather difficult to find in proton spectra, particularly those that correspond to the weak long-range ^{13}CH couplings, because they may be hidden in the wings of the strong lines from ^{12}C molecules. It is even harder to monitor their peak heights effectively in the presence of a very strong sloping background signal and the residual instability of the field-frequency control.

We have managed to circumvent these two problems by imposing a slow pulse modulation on H_2 and synchronously detecting the modulation appearing in the proton spectrum. For a sufficiently slow modulation frequency (in practice 0.25 cps) one obtains simply the difference between the steady-state signals that are observed with H_2 off and with H_2 on. If ω_1 is held at the exact resonance frequency of a ^{13}C satellite all components of the strong ^{12}C proton signals cancel and the output is at a null until ω_2 sweeps through a connected ^{13}C transition, when a "burst" of modulation is received. We have therefore chosen to represent the responses as positive-going peaks (in contrast with conventional INDOR), for example the figure illustrates the pulsed INDOR responses of 1,3,5 trichlorobenzene (^{13}C at position 2) corresponding to the two ^{13}C subspectra generated by the directly bonded proton. This illustrates a general result for systems of three or more non-equivalent spins: the relationship between the particular ^{13}C satellite that is monitored and the subspectrum obtained indicates the relative signs of the relevant spin coupling constants. In this case, $J(\text{HH}, \text{meta})$ and $J(^{13}\text{CH})$ are found to have like signs.

Readers may wish to suggest a suitable name for this technique to prevent us from dubbing it CUSPIDOR (Carbon-13 Unravelling by Slowly Pulsed Internuclear DOuble Resonance). Preprints available.

Best wishes,

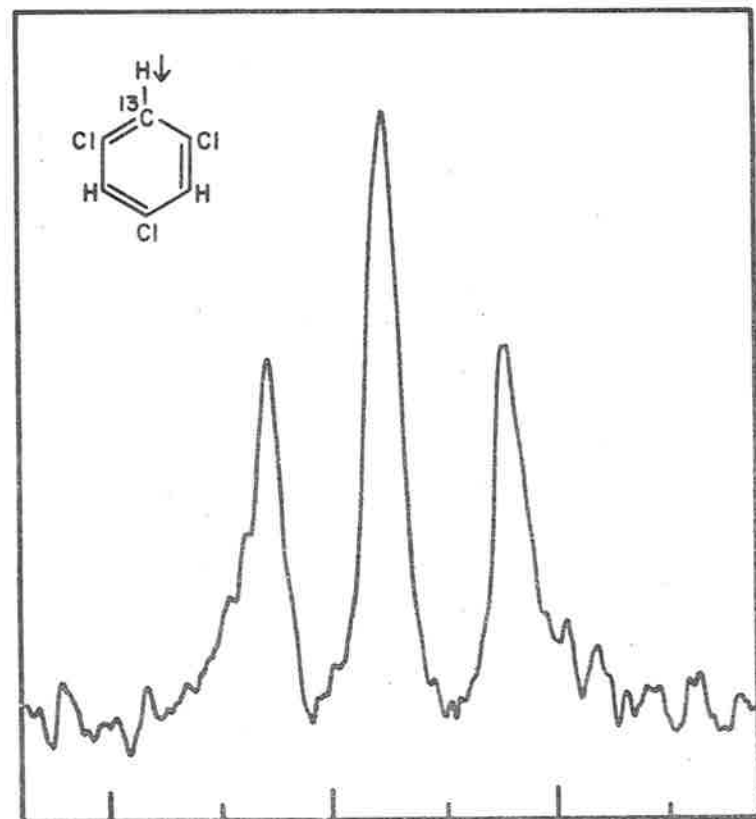
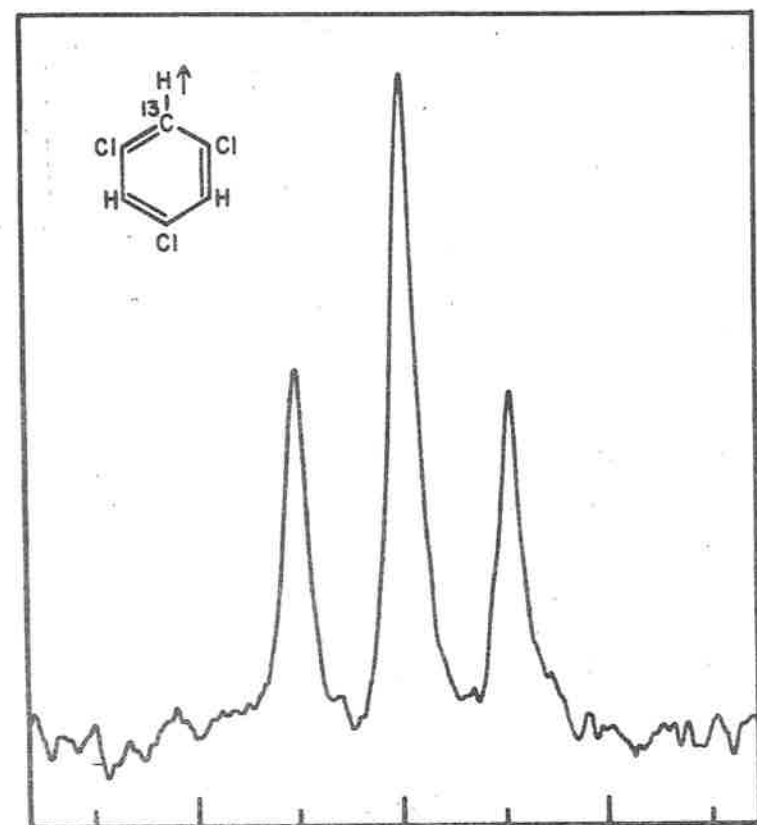
Ray

Ray Freeman

Analytical Instrument Research

RF:iw

PULSE MODULATED DOUBLE RESONANCE OF SYM-TRICHLOROBENZENE



15 088 580 590 600

750 760 770 CPS

^{13}C IRRADIATION FREQUENCY $\omega_2 / 2\pi$

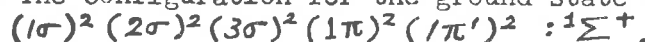
Department of Chemistry
Kobe University
Kobe, Japan

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

M.O. Calculation of the Coupling Constant in HF

Dear Professor Shapiro:

Pople and Santry¹ showed by elegant arguments that some of the coupling constants between directly bonded nuclei could be negative by explicitly considering excited states. In their treatment, two-center integrals were neglected, and only the lowest unoccupied molecular orbital was taken into account for excitation. Dr. Kato and I have attempted to extend their calculations by 1) including two-center integrals, 2) taking into account higher unoccupied M.O.'s, and 3) employing energywise better M.O. wave functions. As an example we have chosen hydrogen fluoride for which several functions are available and among them used wave functions by Ransil,² Nesbet,³ and Karo and Allen.⁴ The configuration for the ground state of HF is



Most of the two-center integrals can be evaluated by the Barnett-Coulson method, but some need the help of Gegenbauer's function.

The results obtained so far have revealed the following features. Excitations from (1σ) to $(n\sigma)$ can be neglected. In general the main contribution comes from $(3\sigma) \rightarrow (4\sigma)$ and $(2\sigma) \rightarrow (4\sigma)$, giving rise to negative coupling in agreement with Pople's suggestion. For Nesbet's function, contributions from $(3\sigma) \rightarrow (n\sigma)$ and from $(2\sigma) \rightarrow (n\sigma)$ almost cancel each other, when $n = 4, 5, 6$ and 7 , although they are, of course, very sensitive to their respective excitation energies. For $n = 8$ and 9 , however, contributions from $(3\sigma) \rightarrow (n\sigma)$ and $(2\sigma) \rightarrow (n\sigma)$ are large and do not cancel each other, finally giving rise to positive coupling. This situation may arise from the fact that these SCF M.O.'s are for the ground state of the molecule and higher unoccupied orbitals are particularly inappropriate for describing excited states, as may be noticed from unusually large coefficients for some basis functions of these higher orbitals. Thus the excitation may better be truncated at (7σ) with Nesbet's function. Whether the sign of coupling is positive or negative experiment. The fact that Karo's function gives the seemingly best result and Nesbet's the poorest, while they are energetically equally good, is

can be determined by

very interesting. The two functions are representative of the two approaches to the construction of molecular function, namely use of 'best atomic' orbitals and use of as 'many' basis orbitals as to describe deformation of atomic orbitals on formation of the molecule. The Fermi term is dependent on the electron density only at the nuclei, and that the former function gives a better result may suggest that the density at the nuclei is little affected on molecular formation. In any event, coupling appears to be very sensitive to several parameters to be used, but agreement with experiment could be obtained by arbitrary choice of them, irrespective of whether it may be the proper choice or not. We also plan to make variation-al calculation.

Yours sincerely,

A. Saika

A. Saika

- 1) Pople and Santry, Mol. Phys. 8, 1 (1964).
- 2) Ransil, Rev. Mod. Phys. 32, 245 (1960).
- 3) Nesbet, J. Chem. Phys. 36, 1518 (1962).
- 4) Karo and Allen, J. Chem. Phys. 31, 968 (1959).

TC/PS



Please address any reply to
THE DIRECTOR

and quote: BP 5/7/01

Your reference:

Ministry of Technology

NATIONAL PHYSICAL LABORATORY

TEDDINGTON, Middlesex

Telex: 262344 Telegrams: Physics, Teddington, Telex

Telephone: TEDDINGTON Lock 3222, ext.

BASIC PHYSICS DIVISION

5th August 1965

Dear Dr. Shapiro,

We thought readers of the ITNMR newsletter might be interested in some modifications which we have made to our HA 100 spectrometer when used in conjunction with a C 1024 Time Averaging computer. As supplied, this system has certain undesirable features for those wishing to sweep small portions of a spectrum repetitively with the object of measuring splittings in the observed spectrum. An example of such a use is the determination of the temperature dependence of splitting in ^{13}C sidebands of symmetrically 1,2 disubstituted ethanes. (T.M. Connor and K. A. McLauchlan, J. Phys. Chem., 69, 1888, 1965). The main snags arising are the following:

1. It is not possible to sweep a width of less than 50 cps, which if the spectrum of interest is ~ 10 cps wide leads to considerable waste of time and channels when sweeping repetitively.
2. More seriously, the read-in time for the computer is not always the same as the read-out time for identical settings of the appropriate sweeps. We have found the read-in time to be stable, whereas the read-out time varies considerably depending on the temperature of the apparatus, i.e. on how long the computer has been switched on, how many windows or doors are open, etc. In order to preserve the chart calibration it is essential that the read-in and read-out times are the same, and whilst it is possible to adjust the latter using trimmers in the computer, the high temperature coefficient makes accurate calibration difficult for those not working in temperature controlled rooms.

The first of these points is easily coped with by connecting a second microswitch across the right-hand limit switch in the recorder. This is actuated by the bar joining the two halves of the pen carriage, and is mounted on a slotted bar on the underside of the recorder. This movable limit switch can be placed in any desired position along the bar so that ~~placed in~~ repetitive sweeps < 50 cps are possible. In fact the number of possible positions is limited by the available recorder and computer sweep speeds.

As a cure rather than prevention of the second difficulty we have made provision for putting calibration marks on the spectrum which appear on the recorder chart and are fed into the computer memory simultaneously. This is done by feeding a small voltage onto the lead connecting the integrator/decoupler to the computer (Pin P; J009). This voltage is provided by the circuit shown, the microswitches being actuated by a suitable attachment to the above mentioned bar joining the halves of the pen carriage. These two microswitches are also mounted on the slotted bar, one being attached to the same mounting plate as the movable limit switch. The size of the mark is controlled by the

/two

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



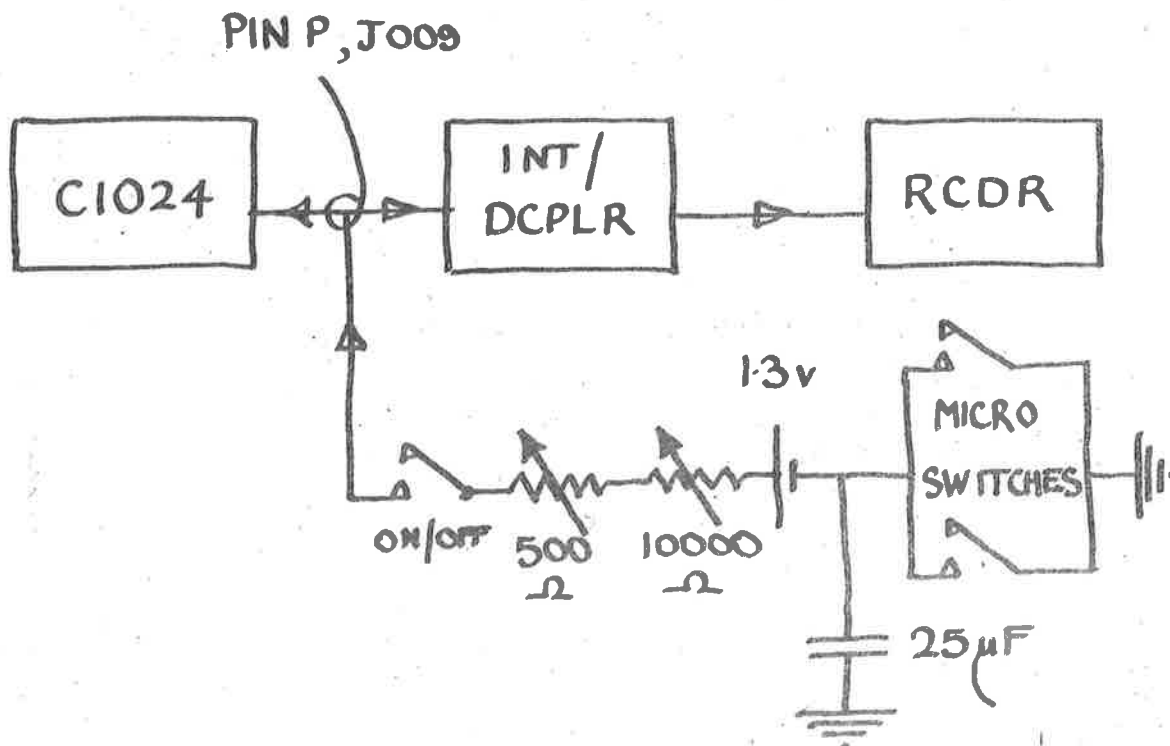
two potentiometers and also depends on the position of the coarse and fine output controls on the integrator/decoupler. The switch and potentiometers are mounted below the gain and damping controls on the recorder control panel and when not required, the movable limit switch can be placed at the right hand end of the slotted bar so that the right hand limit switch again comes into play.

We have found that this device produces precise and coincident marks on repetitive sweeps, and does not interfere with the spectrometer when in normal use.

Yours sincerely,

Tom Connor

TOM CONNOR



TATA INSTITUTE OF FUNDAMENTAL RESEARCH

National Centre of the Government of India for Nuclear Science and Mathematics

Telegrams: ZETESIS

COLABA, BOMBAY 5

Telephone: 213141

August 3, 1965

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

Dear Professor Shapiro,

Thank you for your letter of July 23, 1965 addressed to Professor Dharmatti. I regret to say that Professor Dharmatti passed away on May 2, 1965. He had been keeping indifferent health for two years after he had suffered an heart attack. Inspite of this he was taking keen interest in the research programme of the Nuclear and Electron Magnetism Group of the Tata Institute of Fundamental Research which he helped to build after his return from Stanford in 1953. The Group at present has about twentyfive research people working under five disciplines: Wideline NMR, High Resolution NMR, Magnetic Susceptibility, Electron Paramagnetic Resonance, Mössbauer Effect and Microwave Spectroscopy of Gases. I do not want to enumerate here the important contributions Professor Dharmatti made during his lifetime since they are well known to the readers of the Newsletter. I would appreciate the insertion of the news of his death, which, I am sure, will be mourned by the workers in NMR. I am sending, in a separate letter, the activities of our Group.

Yours sincerely,

R. Vijayaraghavan
R. Vijayaraghavan

TATA INSTITUTE OF FUNDAMENTAL RESEARCH

National Centre of the Government of India for Nuclear Science and Mathematics

Telegrams: ZETESIS

COLABA, BOMBAY 5

Telephone: 213141

August 3, 1965

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

Dear Professor Shapiro:

The following problems are currently being studied:

1. Temperature effects on the n.m.r. lines in borates
2. Proton and deuteron resonances in glycine and its addition compounds at various temperatures
3. Fluorine resonance in uranium fluoride compounds
4. Study of ^{119}Sn resonance in rare-earth tin alloys
5. Chemical and Knight shift studies in CdSe , SnSe and InBi semiconductors.

Yours sincerely,

R. Vijayaraghavan
R. Vijayaraghavan

UNIVERSITY OF CALIFORNIA, SAN DIEGO

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



SANTA BARBARA • SANTA CRUZ

SCHOOL OF SCIENCE AND ENGINEERING

P. O. BOX 109
LA JOLLA, CALIFORNIA 92038

August 2, 1965

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

Dear Professor Shapiro:

During the past year we have been calculating coupling constants according to the theory developed by Pople and Santry [Mol. Phys., 8, 1 (1964)]. We are using the extended Hückel theory [R. Hoffmann, J. Chem. Phys., 39, 1397 (1963)] including all overlaps to generate one-electron wave functions and energies. In addition to the dominant terms involving one-center integrals (see Pople and Santry) we include a number of contributions involving the product of a one- with a two-center integral. The additional contributions can have a significant effect on the final result, especially for couplings through one and two bonds. The specific details of the calculations will not be given here, but it should be emphasized that none of the parameters, other than structural, were varied from molecule to molecule. We will gladly provide the details to anyone who is interested.

Selected results are given in the accompanying table. The corresponding experimental results have been omitted to save space.

Calculated values of the directly bonded ^{13}CH couplings for a variety of hydrocarbons correlate well with observed couplings, but the magnitudes in all molecules are low by a factor of 1.5. For long range couplings agreement between theory and experiment ranges from excellent (methane, ethane, cyclohexane) to miserable (gem coupling in ethylene and the ^{13}CCH coupling in acetylene). For the ortho, meta and para couplings in benzene, we obtain 5.6, -0.5 and 1.2 c.p.s., respectively, as compared to 7.7, 2.5 and 1.8 calculated by Gil and Murrell (IITNMR NL, No. 81, 6).

Sincerely,

Robert C. Fahey Gary C. Graham Robert L. Piccioni
Robert C. Fahey Gary C. Graham Robert L. Piccioni

TABLE. Calculated values of coupling constants (in c.p.s.)

Molecule	$^{13}\text{CH}^a$	$^{13}\text{CH}^b$	gem^a	gem^b	vicinal^b	$^{13}\text{CCH}^b$
Methane	66	83	-17.8	-16.5		
Ethane	69	84	-20.5	-16.7		-4.0
$\phi = 0^\circ$					7.8	
$= 30^\circ$					5.7	
$= 60^\circ$					1.8	
$= 90^\circ$					0.2	
$= 120^\circ$					3.4	
$= 150^\circ$					9.2	
$= 180^\circ$					11.9	
Cyclohexane ^c	73	85	-22.5	-17.0	11.0 ax-ax 1.8 ax-eq 1.9 eq-eq	-4.1 ax -4.2 eq
Cyclopentane ^d	73	85	-23.1	-17.0	7.8 cis 3.8 trans	-4.2
Cyclobutane ^d		93		-18.1	5.4 cis 5.1 trans	-3.3
Cyclopropane		107		-19.3	3.7 cis 5.6 trans	-2.8
Ethylene	89	107	-21.4	-15.2	5.9 cis 16.8 trans	-8.0
Benzene	88	100				-6.9
Acetylene	142	169			8.5	-5.9

^aIncludes one-center integrals only^bIncludes terms containing a product of a one- with a two-center integral^cChair form^dPlanar



RESEARCH LABORATORIES

GENERAL MOTORS CORPORATION

July 30, 1965

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

Dear Barry:

Our Laboratory's contribution to IITNMRN consists of the attached description of "A Convenient Power Source for the Varian G-14 Event Marker," which was designed and built by Mr. B. W. Joseph.

Current research interests in our Lab are tending toward wide line NMR and we are making a beginning in NQR.

Some recent reprints which are available consist of the following:

"Modulation Effects in Magnetic Resonance: Widths and Amplitudes for Lorentzian and Gaussian Lines,"
J. App. Phys. 35, 1217 (1964).

"Proton Magnetic Resonance Studies of Compounds with the Structure $(CH_3)_4X$," in Liquids: Structure, Properties, Solid Interactions, T. J. Hughel, editor, Elsevier Publishing Co., Amsterdam (1965), p. 219.

"Measurement of Brittleness Temperature of Neoprene by Proton Magnetic Resonance - The Effect of Plasticizers," J. App. Polymer Sci. 9, 1553 (1965).

"Proton Magnetic Resonance Studies of Solid Tetramethyl Silicon, Germanium, Tin, and Lead," J. Chem. Phys. 42, 4229 (1965).

"Comment on the Letter 'Measurement of Molecular Rotation by N^{14} Nuclear Quadrupole Resonance Relaxation Times,'" J. Chem. Phys. 42, 3341 (1965).

Dr. B. L. Shapiro
page two
July 30, 1965

"Nuclear Spin-Spin Coupling of the Type J_{X-C-H} "
J. Chem. Phys. 42, 435 (1965).

"Nuclear Spin-Spin Coupling, Hyperfine Coupling, and
Effective Nuclear Charge for Outer s-Electrons,"
General Motors Research Publication GMR-444,
October 24, 1964.

Yours very truly,



George W. Smith
Physics Department

GWS:ms

Encl.

A CONVENIENT POWER SOURCE FOR THE VARIAN G-14 EVENT MARKER

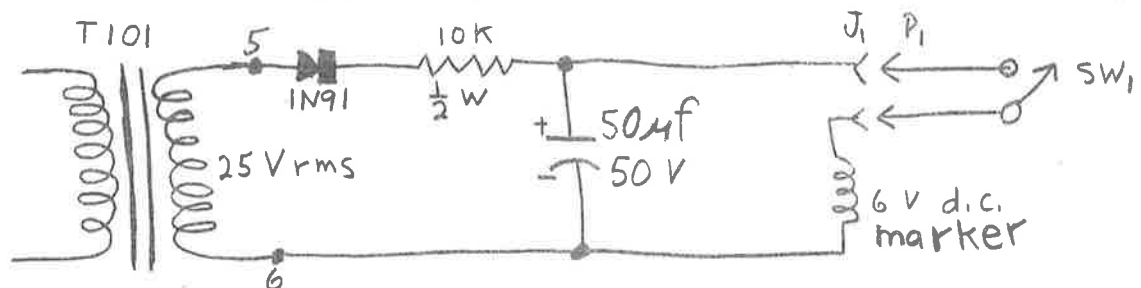
B. W. Joseph

Research Laboratories, General Motors Corporation, Warren, Mich.

Varian Associates sell an Event Marker Kit as an accessory for their G-14 recorder. The marker can be obtained with a variety of actuating voltages from 6V d.c. to 115V a.c. In all cases the marker requires an external power source for actuation.

In many cases the 115V a.c. model is the most convenient: no external batteries or transformers are required, only a line cord and switch. However, we have recently developed a modification which is even more convenient and has the added advantages of safety and neat installation.

The modification consists of a capacitor-discharge system to actuate a 6V d.c. marker. The capacitor charging current is obtained from the power transformer T-101 in the recorder. The simple circuit is shown below.



The diode, resistor and electrolytic capacitor are mounted on a small piece of phenolic circuit board. The board in turn is mounted with a single screw to one of the tapped posts on the side plate of the recorder. Each side of the recorder has two spare posts, and there is more than enough room to mount two circuit boards when two markers are installed.

J_1 is an Amphenol 80-PC2F two-pin receptacle which is mounted in a 5/8 inch hole about 2 inches toward the rear from, and on the same side as the chart roll-out knob. P_1 is an Amphenol 80-MC2M two-pin plug with a convenient length of cable to the push-button switch SW_1 . We can use either hand or foot switches as expediency dictates. Again, there is more than enough room to mount a pair of receptables on either or both sides of the recorder when two markers are installed.

One slight disadvantage is that marks cannot be placed on the chart at extremely short time intervals. Due to the 1/2 second time constant of the charging circuit, it is necessary to wait at least 3 seconds between marks. This should not cause any difficulty in most operations, but if it does, the 10K

resistor can be changed to 5K with an attendant halving of the time constant. We do not advise decreasing the capacitance since the energy stored in the 50 μ F capacitor is about the minimum for satisfactory operation of the marker.

We made a number of checks with various input signals and recorder sensitivities as high as 1 millivolt full scale and detected no interaction or interference between marker and signal. With SW₁ closed, the constant current is only 2.5 milliamps. This added drain on T101 is insignificant.

DUQUESNE UNIVERSITY
PITTSBURGH, PENNSYLVANIA
15219

Aug. 12, 1965

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

Dear Professor Shapiro:

NMR Determination of Aggregation Number

Since dinonyl naphthalene sulfonic acid (HD) is an excellent extractant of metal ions into CCl_4 , it was considered of interest to devise an NMR method to determine its aggregation number, x . With this objective, we have measured the SO_3H proton signal of 0.1 F HD in anhydrous CCl_4 , in the absence and presence of various concentrations of dimethylformamide (DMF), and the frequencies are given below:

Concn. of DMF d, M	Table I ν , ppm. downfield from TMS			
	-13.5°	1.5°	35°	61°
0.00	12.47	12.27	11.67	10.32
3.00	9.90	9.60	8.90	8.52
6.45	9.47	9.03	8.53	8.40
10.07	8.96	8.65	8.47	8.33

Since the initial concentration of DMF, d , is much greater than that of HD ($b = 0.1$ F), we need consider only the reaction



If f is the equilibrium concentration in formality of uncomplexed HD, then the equilibrium constant would be given by

$$K = \frac{(b - f)^x}{(f/x) (d - b + f)^x} \quad (2)$$

The observed SO_3H proton frequency may be taken to be

-2-

the weighted average of the characteristic frequencies of the uncomplexed and complexed HD, ν_x and ν_c , respectively, so that

$$\nu_{obs} = \frac{f}{b} \nu_x + \frac{b-f}{b} \nu_c \quad (3)$$

The value of ν_x is the frequency of SO_3H in the absence of DMF. The value of ν_c , 8.33 ppm., is obtained by plotting ν vs. d at the various temperatures and extrapolating to $d = \infty$. The value of f is obtained by rearranging eq. (3) to give

$$f = \frac{\nu_{obs} - \nu_c}{(\nu_x - \nu_c)/b} \quad (4)$$

In order to determine the aggregation number, x , trial values of x from 1.0 through 4.0 in increments of 0.1, were substituted into eq. (2) and the corresponding values of K computed. A portion of the computed results for 35° are shown in Table II. It is seen that the value of x which yields a constant value of K is $x = 2.0$. For $x < 2.0$, the computed values of K increase monotonically when b increases from 3.00 to 10.07 M; whereas for $x > 2.0$, the computed values of K decrease when b increases in the same range.

Table II

Computed Values of K at Various Assumed Values of x , 35° (eq. 2)

	$b = 3.00M$	6.45M	10.07M
$x = 1.6$	0.295	0.440	0.511
1.9	.123	.149	.152
2.0	.091	.102	.102
2.1	.068	.067	.066
2.5	1.95×10^{-3}	1.56×10^{-3}	1.21×10^{-3}

At 35° therefore, HD in CCl₄ is a dimer. This result agrees with the value $x = 2.0$ obtained by Little and Singleterry (J. Phys. Chem. 68, 3453 (1964)) for HD in benzene at 35°, using a vapor pressure method.

The values of x in (HD)_x at -13.5°, 1.5°, 35° and 61°, obtained by the present NMR method, are 3.2, 2.8, 2.0 and 1.4, respectively. A plot of x vs. t° yields a straight line. A simple linear extrapolation of the plot gives $x = 1$ at 78°. The frequency data in Table I, extrapolated to 78°, yields $\nu_M \sim 8.27$ ppm., where ν_M is the characteristic frequency of monomeric HD.

When no DMF is present, the frequency of the SO₃H signal at 35° remains constant at 11.67 ppm., in the range of HD = 0.05 to 0.45 F in CCl₄. This means that x is constant in this concentration range. Kaufman and Singleterry (J. Colloid Sci. 10, 139 (1955)) have estimated the critical range for micelle formation of dinonyl naphthalene sulfonates at 10⁻⁶ to 10⁻⁷ F, and that the aggregation number, from vapor pressure measurements, is independent of concentration. Our NMR result therefore is in agreement with these findings.

It will be noted from Table I that in the absence of DMF, ν decreases by 2.15 ppm., in the temperature range -13.5 to 61°. In the same temperature range, as the concentration of DMF increases, the decrease in ν becomes smaller. This is easily accounted for, because in the absence of DMF, as temperature increases, only the reaction (HD)_x = x HD occurs; whereas in the presence of DMF, eq. (1) needs to be considered. The relative values of ν_x , ν_c and ν_M

-4-

account for the trend of frequencies listed in Table I nicely.

This research was carried out in the laboratory of Professor Norman C. Li at Duquesne University.

Sincerely yours,

William J. Busler

William J. Busler¹

¹ Research Assistant to Brother Edward Doody on an ACS Petroleum Research Foundation grant with Christian Brothers College at Memphis, Tenn.

TEXAS A&M UNIVERSITY
COLLEGE OF ARTS AND SCIENCES
COLLEGE STATION, TEXAS

Department of
CHEMISTRY

August 12, 1965

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

Dear Barry:

Short Title: Variable Temperature of Isobutyl Vinyl Ether and Computer Program

Recently, we began NMR and far infrared studies on a number of ethers and esters. Enclosed are some preliminary temperature studies of isobutyl vinyl ether, taken on our Varian A-60. Clearly, multiplet B arises from the geminal vinyl protons. As the temperature decreases, it can be seen that distinct changes occur in the structure of the B multiplet. Refined measurements on this and related vinyl ethers are now in progress and will be reported in the near future.

The NMR computer program of Stanley, Marquardt and Ferguson^(1, 2) has been converted from its original language of IBM 7040 Fortran IV to that of IBM 7094 Fortran IV for use at our computational facilities.

- (1) R. M. Stanley, D. W. Marquardt, R. C. Ferguson, "Analysis of NMR Spectra", (IBM Share Library, Distribution No. 3165).
- (2) R. C. Ferguson, D. W. Marquardt, J. Chem. Phys., 41 (7), 2087-95 (1964).

Sincerely yours,

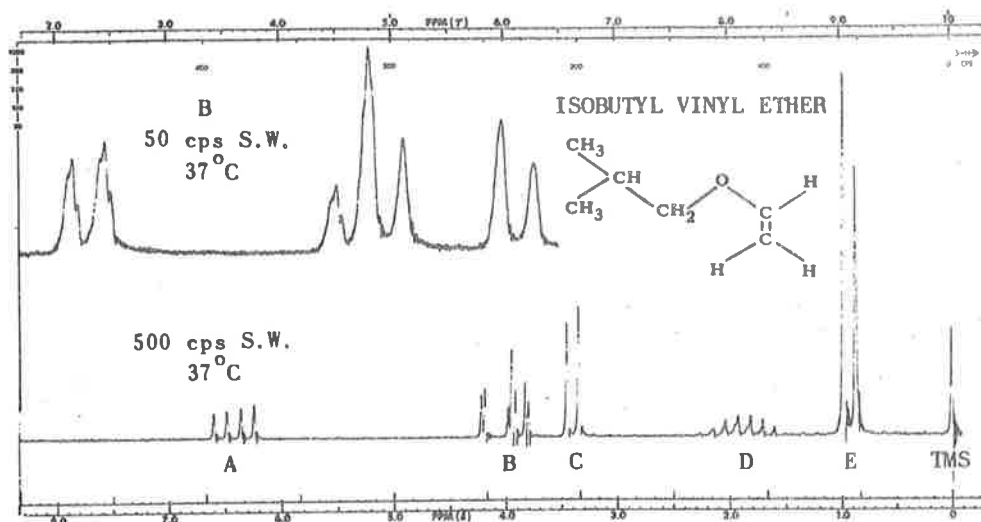
T. Altpeter

A. D. H. Clague

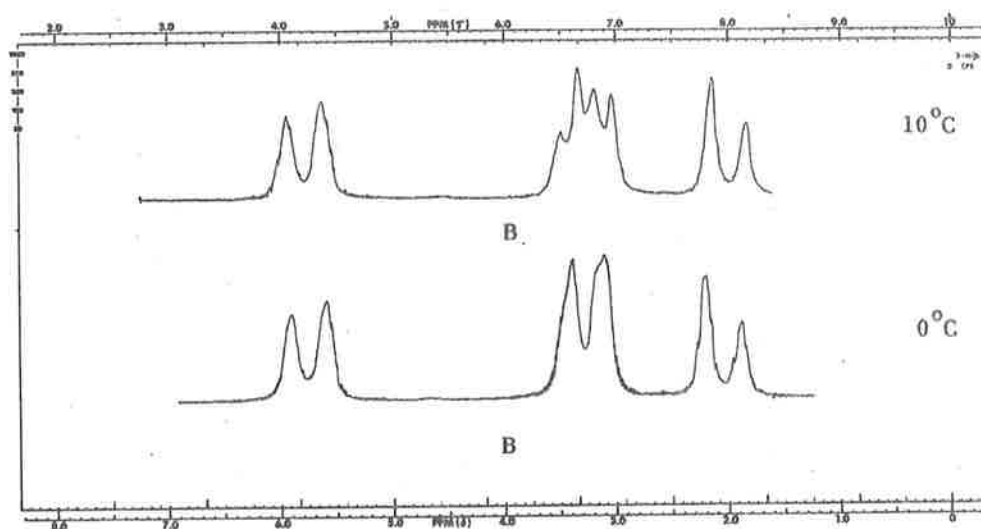
A. Danti

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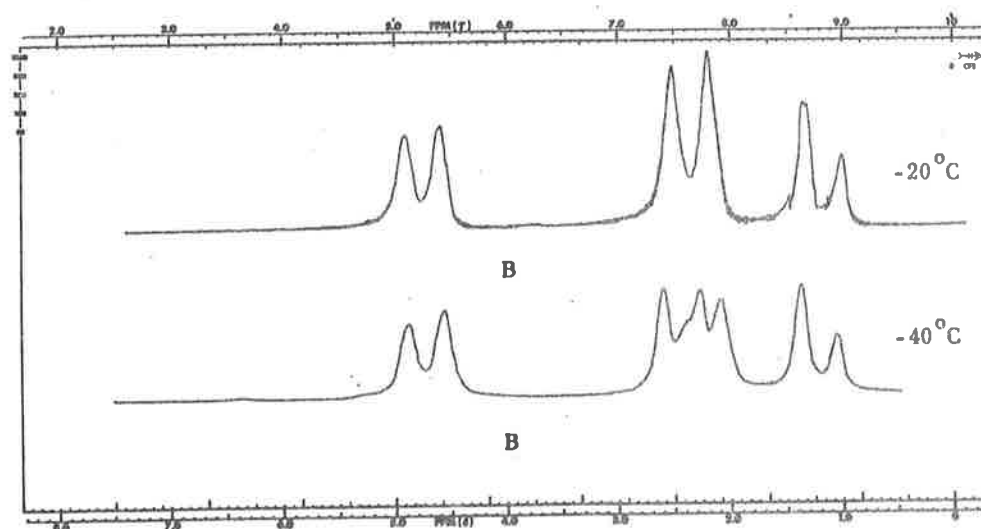
Texas A&M University
Department of Chemistry



DATE: 7/16/65
SAMPLE: IBVE
OPERATOR: A.D.H.C., T.A.
SOLVENT: Neat
R.F. FIELD: 0.04 mG
SPEC. AMPL.: 1.0
FILTER B.W.: 4 cps
SWEEP WIDTH (S.W.): as shown
SWEEP TIME: 500 sec.



SWEEP WIDTH: 50 cps



SWEEP WIDTH: 50 cps

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



Luftpost

Dr. B.L. Shapiro

Department of Chemistry
 Illinois Institute of
 Technology

Chicago, Illinois 60616

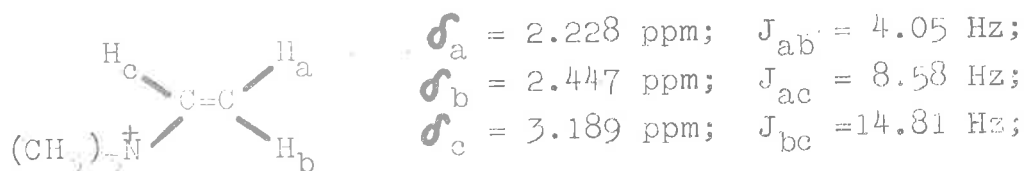
U S A

Ihre Zeichen	Ihre Nachricht vom	Unsere Zeichen	Fernsprecher-Durchwahl	Telex	67 Ludwigshafen
		Dr.Brü/Fa	(06 21) 600 ...	4 64 ...	23. Juli 1965

Betreff Nitrogen-hydrogen-coupling in en-ammonium compounds;
"compensated" NMR-spectra

Sehr geehrter Herr Dr. Shapiro!

Zufällig hatte ich vor kurzem Neurinbromid (Trimethylvinylammoniumbromid) zu untersuchen. Ich erwartete nichts Besonderes, sondern z.B. das übliche Vinylgruppenspektrum möglicherweise mit einer schwachen Aufspaltung der bezüglich des Stickstoffs β -ständigen Protonen gemäß der in Ammonium-Alkyl-Verbindungen beobachteten Kopplung zwischen N und β -ständigen CH-Gruppen (BULLOCK u.a. J.Chem.Phys. 38, 2318, 1963). Überraschenderweise war das 60 MHz-Spektrum viel linienreicher, und die beobachteten Aufspaltungen waren viel größer als erwartet. Insbesondere zeigte auch das Signal der zum Stickstoff α -ständigen Methingruppe eine Aufspaltung durch Kopplung mit N. Zur Sicherung des Befundes bat ich Herrn Dr. Melera, Research Laboratory, Varian AG, Zürich, um ein 100 MHz-Spektrum, wofür hier herzlich gedankt sei (s. Abb.). Die genaue Analyse des Spektrums lieferte folgende Werte der NMR-Parameter:



$$|J_{a,N}| = 5,51 \text{ Hz}; \quad |J_{b,N}| = 2,52 \text{ Hz}; \quad |J_{c,N}| = 3,52 \text{ Hz}.$$

Badische Anilin- & Soda-Fabrik AG

Empfänger

Dr. B.L. Shapiro

Unsere Zeichen

Dr.Brü/Fa

67 Ludwigshafen

23.7.1965

Blatt

2

Betreff

Die δ -Werte sind auf das nicht aufgespaltene Methylsignal bezogen und wachsen nach kleinerem Feld.

Kopplung zwischen N und α -ständigen CH-Gruppen wurde, soviel ich weiß, bisher nur im Falle der Isonitrile beobachtet (KUNTZ u.a. J.Chem.Phys. 35, 1533, 1961). Der mitgeteilte Befund dürfte der erste Fall bei einer anderen Konstitution sein. Bemerkenswert ist dabei auch die Größe der Kopplungskonstante zu dem bezüglich des Stickstoffs trans-ständigen Proton und die Größe der geminalen Kopplung in der Vinylgruppe. Über die Gründe für die Stickstoff-Wasserstoff-Kopplung in Ammoniumverbindungen und Isonitrilen ist in den zitierten Arbeiten einiges gesagt. Bei dem hier mitgeteilten Fall spielt für die Größe der Kopplungskonstanten offenbar der ungesättigte Charakter des Stickstoffliganden eine entscheidende Rolle. -

Seit ein paar Wochen verfügt das NMR-Labor der BASF auch über eine "CAT" (time averaging computer C 1024 von Varian). Damit haben wir nicht nur die Möglichkeit, Lösungen mit sehr geringer Konzentration der interessierenden Substanz zu messen, sondern auch so etwas wie "kompensierte NMR-Spektren" (Kompensation im Sinne der optischen Zweistrahl-spektroskopie) herzustellen, ein Verfahren das wir im Bereich der IR-Spektroskopie häufig anwenden und bisher für die NMR-Spektroskopie sehr vermisst haben. Dazu speichern wir zunächst das Spektrum einer Lösung auf Stellung "add" in der CAT und dann in demselben Probenröhrchen und unter peinlicher Einhaltung der Bedingungen das Spektrum des Lösungsmittels auf Stellung "sub". So gelingt es meist recht einfach, die Substanzlinien überlagernden Lösungsmittellinien aus dem Endspektrum zu entfernen. Dieses Verfahren scheint mir wichtig, einmal im Hinblick auf Einsparung der teuren deuterierten Lösungsmittel, zum anderen aber vor allem im Hinblick auf die Reinheitsanforderungen, die an das Lösungsmittel zu stellen sind. Bisher brauchte man sich darum nicht sehr zu kümmern. Jetzt aber, mit der Verfügbarkeit der CAT, kann bei der Untersuchung von Lösungen mit 0,1 % oder weniger der interessierenden Substanz die Reinheitsforderung entscheidend werden. Hier hilft die Kompensation der etwaigen Verunreinigungsbanden häufig schneller und einfacher weiter als eine zeitraubende und teure Extremreinigung der Lösungsmittel - vorausgesetzt natürlich,

Badische Anilin- & Soda-Fabrik AG

Empfänger

Dr. B.L. Shapiro

Unsere Zeichen

Dr.Brü/Fa

67 Ludwigshafen

23.7.1965

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3

Betreff

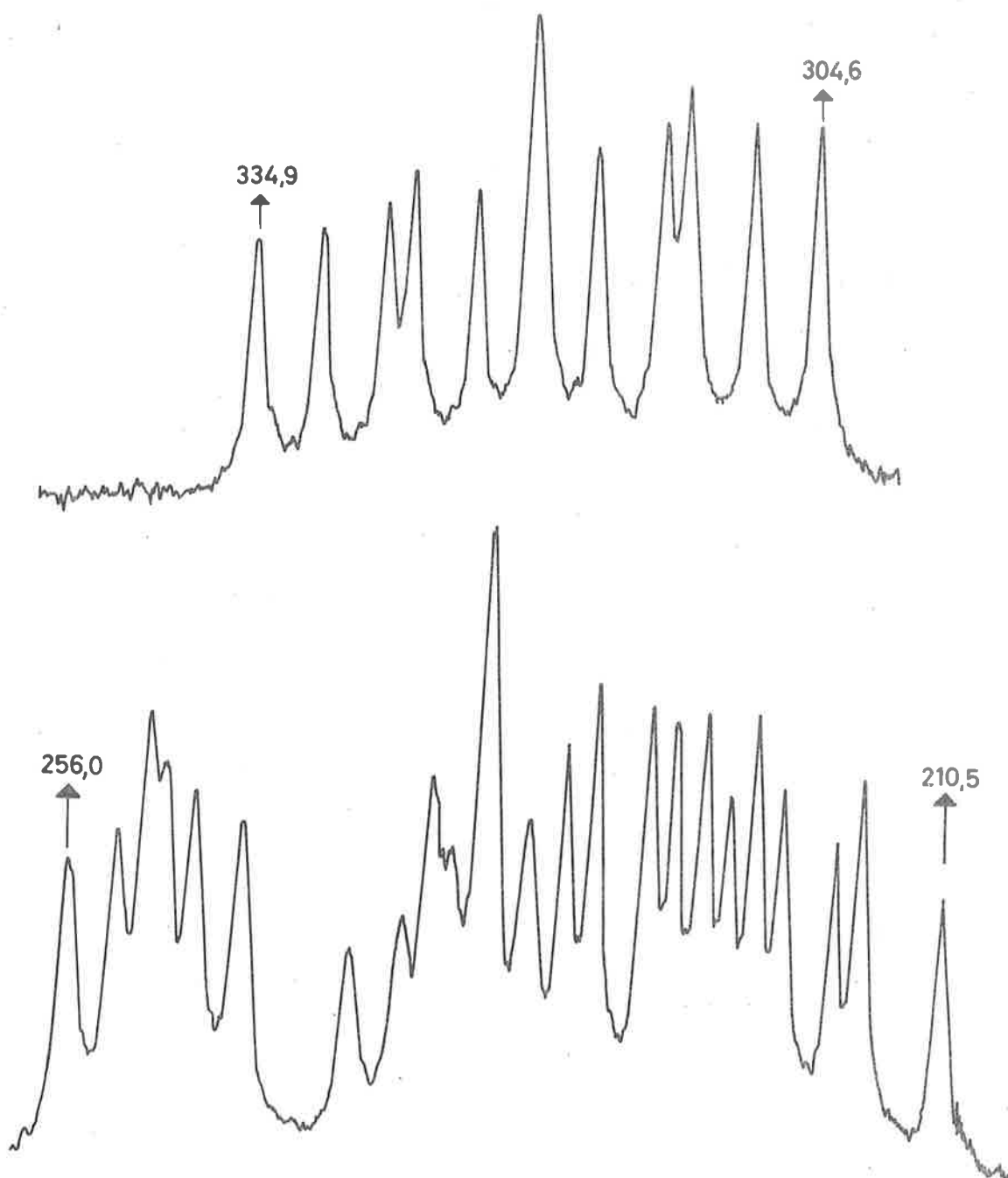
daß ein so teurerer Apparat wie C 1024 vorhanden ist. Ich glaube mit der Ansicht nicht fehlzugehen, daß dieses Kompensationsverfahren bei zunehmender Erfahrung mit der CAT in manchen Fällen Möglichkeiten eröffnet, die uns bisher verschlossen sind.

Mit freundlichen Grüßen

Ihr



(Dr.W. Brügel)



NMR spectrum (100 mc) of the vinyl group of tri-methyl vinyl ammonium bromide (ca 20 % in D₂O).

Top: = CH-group; bottom: = CH₂-group. The figures are cps downfield from the N(CF₃)₃-signal.



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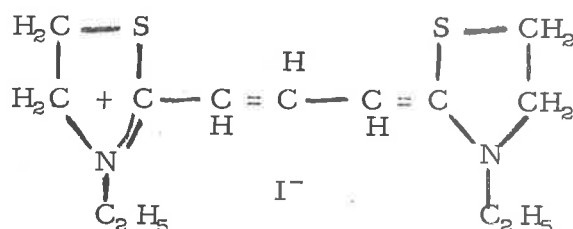
3 August 1965

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

Re: SPECTRA OF CYANINE DYES-EVIDENCE FOR A
DYNAMIC EQUILIBRIUM

Dear Barry:

For some time we have been examining the spectra of cyanine dyes such as [2-bis(3-ethyl thiazolynyl)] trimethine cyanine iodide,



We have had difficulty in obtaining sufficiently concentrated solutions and finally used as solvent a fifty percent mixture of chloroform and tetrafluoro-dichloroacetone-deuterate $[(CF_2Cl)_2/CO \cdot 2.5 D_2O]$. At dye concentrations, (200mg of dye per millimeter of solvent) in which we could obtain reasonable spectra, we were not able to observe and identify the bridge protons. Roy Johnson ran the trimethine for us in a mixed solvent at 2 and 20mg per liter, at 100Mc and with ^{13}C 1024 for 100 traces. We failed to observe the bridge protons. We finally decided to try a run in pure chloroform at 0.1mg of dye per ml., it was run with the C1024 for 820 scans. The expected bridge proton spectra appeared at 614 c. p. s. and 748 c. p. s., apparently in the mixed solvent at the higher concentrations (we couldn't use chloroform alone for the higher concentrations) a dynamic monomer-dimer equilibria exists or the dye and solvent form a complex in a dynamic equilibria which washes out the bridge proton spectra. In support of this idea we found that for 2-methyl thiazoline ethiodide in chloroform, the TMS-chloroform separation of 433 c. p. s. in pure solvent changed to 481.6 c. p. s. when the quaternary

Spectra of Cyanine Dyes -
Evidence for a Dynamic Equilibrium

Page 2

salt was dissolved in the chloroform; apparently the quaternary salt and chloroform interact quite strongly.

Within a vinylogous group of dyes the fine and hyperfine structure disappears as we go from the monomethine to the pentamethine. As the heterocyclic nucleus is varied, the observable hyperfine structure varies. Even the CH_3 and CH_2 groups of the N-ethyl group smear out. It has been known that the dyes either polymerize or form coordinated structures in water and when adsorbed on silver bromide. It now appears that the cyanines tend to polymerize in other solvents as well as water.

The data on the chemical shifts and coupling constants of the thiazolinyl dyes in Table I and Table II were obtained by Asa Leifer before he left. The quaternization of 2-methyl thiazoline caused the 2-methyl group to exhibit a greater chemical shift and changed the ring A_2B_2 group coupling constant to a lower value. However, for the dyes, surprisingly, the chemical shifts of the CH_2 and CH_3 group of the ethyl group attached to the nitrogen and for the ring A_2B_2 group do not vary significantly from dye to dye.

There is, as was expected, a difference between the chemical shift for the quaternary salt CH_2 and CH_3 group of the nitrogen ethyl group and the dyes; however, there was no observable difference in the coupling constants. On the other hand the coupling constant for the ring A_2B_2 group is quite different for the quaternary salt and the dyes. See Table II. The chemical shift of the A_2B_2 groups was much larger for the quaternary salt than for the dyes. However, there was no discernible trend within the vinylogous series of dyes.

Unfortunately the data for other series of dyes than the thiazolinyl were even more difficult to interpret. We will have to modify our DP60 for proton stabilization and obtain a C1024 to continue the dye investigations.

James E. Lu Valle

TABLE I

CHEMICAL SHIFTS AND COUPLING CONSTANTS OF THIAZOLINYL DYES

	2-Methyl		CH ₃ of ethyl group		CH ₂ of ethyl group	
	δ	J _{be}	δ	J	δ	J
			c. p. s.	c. p. s.	c. p. s.	c. p. s.
2-Methyl thiazoline	121.2	1.6	---	---		
2-Methyl thiazoline ethiodide	168.4	---	88.1	7.4	238.2	7.4
2-bis(3-ethyl thiazolinyl) cyanine iodide						
Monomethine			77.4	7.4	214.2	7.4
Trimethine			76.4	7.4	214.3	7.4
7-Methyl trimethine			78.4	7.2	214.3	7.2
Pentamethine			76.9	7.1	214.2	7.1
8-bromopentamethine			80.2	7	214.6	7
Heptamethine			76.3	7.3	214.2	7.3

TABLE II

CHEMICAL SHIFTS AND COUPLING CONSTANTS FOR RING A₂B₂ GROUP

	A Group		B Group		δ_{ab}	J _{ab}	(J/ δ) _{ab}
	δ		δ				
	cps.		cps.		cps.	cps.	
2-Methyl thiazoline	245.8		194.6		51.2	8.35	0.16
2-Methyl thiazoline ethiodide	283.2		232.2		51.0	8.9	0.19
2-bis(3-ethyl thiazolinyl) cyanine iodide							
monomethine	241.8		202.8		39.1	7.4	0.19
trimethine -	244.8		199.8		45.1	7.6	0.17
7-methyl-trimethine	239.3		201.6		37.7	7.7	0.20
Pentamethine	243.0		199.8		43.4	7.5	0.17
8-bromopentamethine	247.2		202.2		45	7	0.16
heptamethine	241.8		199.2		43.0	7.3	0.17

HARVARD UNIVERSITY
DEPARTMENT OF CHEMISTRY

12 Oxford Street
Cambridge 38, Massachusetts, U.S.A.

August 13, 1965

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

Re: Position Wanted

Dear Professor Shapiro:

As the delivery of the new N.M.R. spectrometer to my Prague laboratory is delayed and as my postdoctoral year here at Harvard will finish in February 1966, I would like to join an N. M. R. group for some time.

I am wondering whether there exists such a group with a vacancy ?

Sincerely yours,

Jan Schraml
Jan Schraml

P.S. I am interested mainly in high-resolution N.M.R. work and spent about three years doing applied N.M.R. on organosilicon compounds which meant especially trouble-shooting on a semi-home-made instrument.

MICHIGAN STATE UNIVERSITY EAST LANSING

COLLEGE OF NATURAL SCIENCE • DEPARTMENT OF CHEMISTRY

August 19, 1965

Prof. Barry Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois


Dear Barry:

Several years ago Narasimhan and Rogers (J. Chem. Phys., 34, 1049 (1961); J. Am. Chem. Soc., 82, 34, 5983 (1960)) reported internal chemical shifts and spin coupling constants for a series of compounds of type $M(C_2H_5)_n$, where $M = Hg, Ge, Sn, Pb, Zn$, etc. The internal chemical shifts were shown to fit a modified Dailey-Shoolery relationship between δ and the electronegativity of M ; however, they were determined using pure liquids and the question has always remained whether values obtained at infinite dilution in an inert solvent might differ in an important way.

We have now measured the NMR parameters for a series of these organometallic compounds at infinite dilution in carbon tetrachloride solution. The values do not differ from those reported previously by more than about 6%. Since electronegativity values for the metals are uncertain by more than this the new data do not alter the previous conclusions.

We should also point out an error in the values of $J_{CH_2 - Sn}$ reported previously [J. Chem. Phys. 34, 1049 (1961)]. These should have been $J_{CH_2 - Sn}^{117} = 50.8$ cps and $J_{CH_2 - Sn}^{119} = 52.2$ cps (rather than 30.8 and 32.2 cps). Klose [Ann. Phys. 8, 220 (1961); 9, 262 (1962)] has reported coupling constants in good agreement with the above values.

Yours sincerely,


Max T. Rogers
Roy L. Foley

nh

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B. Gestblom and B. Mathiasson
Acta Chem. Scand. 18, 1905 (1964)

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K. G. Flynn and G. Bergson
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G. Bergson
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"Algal Carotenoids. IV. On the Structure of Fucoxanthin"
Arne Jensen
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"Resource Letter NMR-EPR-1 on Nuclear Magnetic Resonance and Electron Paramagnetic Resonance"
R. E. Norberg
Am. J. Phys. 33, 71 (1965)

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M. Nakejima, A. Hasegawa und F. W. Lichtenthaler
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Y. Tsuda, O. Achmatowicz Jr. und L. Marion
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H. Weber, W. Ring, U. Hochmuth und W. Franke
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H. Machleidt und G. Strehlke
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"Über Neul Hexahydrochinoline"
H. Reinshagen
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"Die Absolute Konfiguration Des Tetramethyl-(--)-Isocatechylchlorids"
K. Weinges und E. Paulus
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"Catechin-Resorcin-Kondensate"
K. Weinges und F. Toribio
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"Umesterungsreaktionen an Podophyllum-Lignanen"
J. Renz, M. Kuhn und A. Wartburg
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"Kleine Kohlenstoff-Ringe, VIII. Valenzisomerisierung von cis- und trans-2-Vinyl-Cyclopropylisocyanat"
E. Vogel, R. Erb, G. Lenz und A. A. Bothner-By
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"Über Alkaloide, V. Inhaltsstoffe von Lespedeza bicolor var. japonica, I. Über Lespedamin, ein Neues Alkaloid"
H. Morimoto und H. Oshio
Ann. Chem. 682, 212 (1965)

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P. R. Wells and W. Kitching
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B. Halperin and L. B. James
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J. R. Surtees
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G. M. Badger, J. A. Elix and G. E. Lewis
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J. W. Clark-Lewis and L. R. Williams
Australian J. Chem. 18, 90 (1965)

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