

22.6.66

Premises

Illinois  
 Institute of  
 Technology  
**N - M - R**  
 Newsletter

No. 92  
 MAY, 1966

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

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NOTA BENE: DO NOT REFER IN PRINT TO THIS NEWSLETTER BY NAME. A RECENT VIOLATION OF THIS RULE - WHICH IS GENERALLY WELL-KNOWN AND RESPECTED - REQUIRES THAT THIS CONDITION BE SPELLED OUT AGAIN.

B.L.S.



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

IN REPLY REFER TO: 313.01

15 April 1966

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

POSITIVE INDICATION OF PHASE OUTAGE IN A V-2100B POWER SUPPLY

Dear Barry:

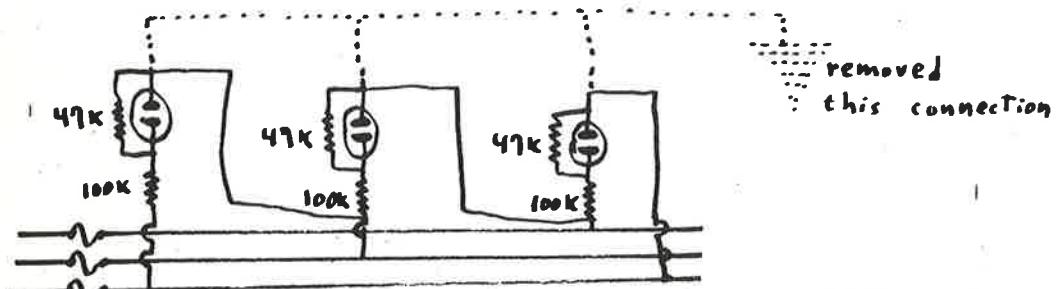
We have had trouble at various times in the V-2100 power supply which turned out to be due to a blown fuse or to failure of one of the 3-phase lines. These faults have not always been immediately apparent, since the three neon lights provided on the back will continue to operate on voltage fed back through one of the transformer primaries.

The diagram below shows the modification that we have made so that the neon lights are connected line-to-line. Now an open 3-phase line causes one (or two) pilot lights to go out, thus indicating the trouble immediately and without fail. We have at the same time added NE-2 bulbs, in series with 100 K resistors, across each of the fuses in the lines from the autotransformer secondary to the high voltage transformer. In case one of these fuses blows, the neon light indicates the trouble at once.

Yours very truly,

*Jam*

Thomas C. Farrar  
Inorganic Chemistry Section



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INSTITUTE FOR APPLIED TECHNOLOGY

INSTITUTE FOR MATERIALS RESEARCH  
CENTRAL RADIO PROPAGATION LABORATORY

## UNIVERSITY OF KENTUCKY

LEXINGTON, KENTUCKY 40506

COLLEGE OF ARTS AND SCIENCES  
DEPARTMENT OF CHEMISTRY

April 18, 1966

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

Dear Dr. Shapiro:

Title: Structure of the Oxidized Pyruvate NAD Adduct

The ternary unreactive complex of NAD, Pyruvate, and the "H<sub>4</sub>" Isozyme of Bovine Heart Lactate Dehydrogenase (LDH) has been postulated as a kinetically significant intermediate in the overall reaction mechanism of LDH. As a model for studying conformational changes of coenzyme and substrate when bound to the enzyme, the pyruvate adduct of NAD was prepared.<sup>1</sup>

The signals in the NMR spectrum of the pyruvate NAD adduct are characteristic of the pattern observed for NAD.<sup>2</sup> For the protons on the ribose moiety, the pattern is unchanged from that found in NAD itself. The adenine protons (C<sub>2</sub>, C<sub>8</sub>) appear as a broad band at ca. 8.37 ppm. From the nicotinamide moiety, the protons appear as a one-proton singlet at 9.67 ppm and a one-proton doublet at 9.09 ppm (*J* = 5.2 cps.), corresponding to the protons on C<sub>2</sub> and C<sub>6</sub> respectively. That the signal from the proton on C<sub>5</sub> is buried underneath the peaks of adenine is established by a spin-decoupling experiment.

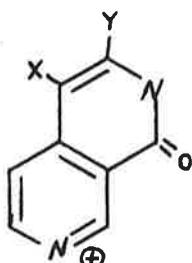
The most significant features of the spectrum are the absence of a peak corresponding to the proton on C<sub>4</sub> of nicotinamide, the presence of a one-proton singlet at 7.27 ppm characteristic of an olefinic proton and the absence of any signals from aliphatic protons which would result from normal addition of pyruvic acid to C<sub>4</sub>. These observations firmly establish that addition has taken place at C<sub>4</sub> of nicotinamide and that the singlet at 7.27 ppm is due to a proton from the pyruvate moiety. The NMR spectra of the fluoropyruvate and acetone adducts are similar except for the absence of the olefinic signal in the fluoropyruvate adduct.

- 2 -

Relative to NAD, the signal from the proton on C<sub>2</sub> of nicotinamide in the pyruvate adduct is shifted downfield 0.21 ppm, suggesting that the amide group of nicotinamide is cyclized with the pyruvate moiety. In such a cyclized adduct the carbonyl group of nicotinamide would no longer be free to rotate and the diamagnetic anisotropy of the carbonyl group would cause a downfield shift in the resonance of the proton on C<sub>2</sub>.

To rule out the possibility of cyclization of the pyruvic acid into the adenine ring, the NMR spectrum of the enzymatically cleaved (with snake venom diesterase) pyruvate NAD adduct was obtained. This spectrum is identical in every respect with the spectrum of the pyruvate NAD adduct except for the absence of the signals arising from the adenine and one of the ribose rings.

The data given above are consistent with the proposed structure for the pyruvate NAD adduct (Figure 1). A similar structure has been proposed for the acetone NAD adduct<sup>3</sup> where a tautomer of la. is present at neutral pH. That no signal from the aliphatic protons which would result from this tautomer was observed in any of the spectra eliminates this as a possibility. Except for an upfield shift of all signals, the NMR spectrum of the pyruvate NAD adduct in base ( $D_2O + Na_2O_2$ ) is identical to the spectrum in  $D_2O$ . The proposed structure in Figure 1b. is consistent with the NMR data and with the shift to longer wavelength observed from the UV spectrum (from 370  $\mu\text{m}$  at pH 7 to 415  $\mu\text{m}$  at pH 9).

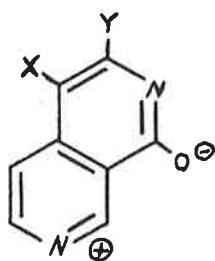


la. Neutral pH

Pyruvate NAD

Fluoropyruvate NAD

Acetone NAD



lb. Basic pH

X = H      Y = CO<sub>2</sub>H

X = F      Y = CO<sub>2</sub>H

X = H      Y = CH<sub>3</sub>

Figure 1. Proposed structure of the pyruvate NAD adduct.

- 3 -

References

1. This work was done with H. A. Lee and A. D. Winer of the Department of Biochemistry.
2. O. Jardetzky, N. G. Wade and J. J. Fischer, Nature, 197, 183 (1963).
3. M. I. Dolin and K. B. Jacobson, J. Biological Chem., 239, 3007 (1964).

Sincerely yours,

*Richard H Cox      Stanford L Smith*

Richard H. Cox and Stanford L. Smith

SLS:dd

Chemical shifts relative to DSS (*in ppm*)

	NICOTINAMIDE				OLEFIN		ADENINE		CH <sub>3</sub>
	C <sub>2</sub>	C <sub>6</sub>	C <sub>4</sub>	C <sub>5</sub>	C <sub>8</sub>	C <sub>2</sub>			
NAD	9.46	9.33	8.99	8.36			8.60	8.46	
P-NAD	9.67	9.09			8.37	7.27			8.37
Fluoro-P-NAD	9.70	9.18			8.44	6.65	8.50	8.30	
Acetone-NAD	9.48	8.89			8.01	6.65	8.53	8.40	2.46
P-NAD in base	9.51	8.88			8.03	7.04	8.22	7.91	
Cleaved P-NAD	9.68	9.08			8.33	7.36			

THE JOHNS HOPKINS UNIVERSITY  
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DEPARTMENT OF PHYSIOLOGICAL CHEMISTRY

TELEPHONE 955-5000  
AREA CODE 301

April 18, 1966

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

NMR Activities at Johns Hopkins Medical School

Dear Barry,

Since we last contributed to the Newsletter our HA-100 Spectrometer and C-1024 have been installed and put in good working order. Our work has required a great deal of C-1024 use and we have been quite pleased with its performance having had no failures thus far. Our HA-100 has performed well in terms of resolution, stability and sensitivity but the Mars transmission has not stood up under extensive time-averaging. We are on our third one in about ten months, the previous two having jammed.

The HA-100 has been a great help in our further work on the structure of the alkyl side chain in heme A, allowing us to obtain more accurate estimates on the proton counts using small amounts of sample available (~3 mg). That this side chain is unsaturated seems definitely established by the NMR and by chemical data obtained earlier by J. L. York and more recently by Sue McCoy of these laboratories. If any readers are particularly interested, we will have pre-prints available soon.

Other projects in progress included a study of substrate binding by dehydrogenase enzymes and using the A-60 a study of the magnetic susceptibilities of a number of metalloporphyrins with a view to learning more about the relationships between ligand structure, porphyrin substituents, and metal spin states to the susceptibility.

I hope these few remarks are in time to continue our valued subscription uninterrupted. Sorry we had to be reminded.

Best regards,

D. P. Hollis

W. S. Caughey

*Don Hollis*

*W. S. Caughey*

## University of Bristol

## School of Chemistry

Professor F. G. A. Stone

Telephone: Bristol 24161 Ext. 311

Department of Inorganic Chemistry

Bristol 8.

England

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

18th April, 1966.

Dear Barry,

<sup>19</sup>F N.M.R. of Pentafluorophenylthio- and  
Pentafluorophenyl-metal Complexes.

Our N.M.R. facilities are presently disrupted by the transfer of the spectrometers from the old buildings to our new chemistry laboratories. However, very shortly we expect to have our Perkin Elmer R10, Varian A60 and HA100 instruments functioning well in their new environments.

As our contribution to I.I.T.N.M.R. Newsletter we would like to report some further <sup>19</sup>F studies. Firstly we have been investigating the C<sub>6</sub>F<sub>5</sub>S group as a ligand to transition metals and have prepared the complexes C<sub>6</sub>F<sub>5</sub>SFe(CO)<sub>2</sub>( $\pi$ -C<sub>5</sub>H<sub>5</sub>) and [C<sub>6</sub>F<sub>5</sub>SFe(CO)<sub>3</sub>]<sub>2</sub>. The latter compound would be expected to have a bent iron-iron bond. The analogue with C<sub>2</sub>H<sub>5</sub>S groups bridging the iron atoms has been studied by X-ray crystallography by Dahl and Wei (Inorg.Chem., 2, 328(1963)) who showed that the two ethyl groups are anti to each other. It was of some interest to determine the relative orientation of the two pentafluorophenylthio groups in [C<sub>6</sub>F<sub>5</sub>SFe(CO)<sub>3</sub>]<sub>2</sub>; i.e. whether the Fe-Fe bond leads to syn- or anti- configurations for the C<sub>6</sub>F<sub>5</sub>S bridging groups. The <sup>19</sup>F spectrum of our product showed groups of bands corresponding to two C<sub>6</sub>F<sub>5</sub> groups in different environments, as expected for the asymmetric anti-structure. The structure is thus similar to that found for [C<sub>2</sub>H<sub>5</sub>SFe(CO)<sub>3</sub>]<sub>2</sub> from the crystallographic study. Interestingly, there is no evidence for the presence of any of the syn-isomer in our samples of [C<sub>6</sub>F<sub>5</sub>SFe(CO)<sub>3</sub>]<sub>2</sub>. This is in contrast to R.B. King's [J.Am.Chem.Soc., 84, 2460(1962)] proton n.m.r. studies on [CH<sub>3</sub>SFe(CO)<sub>3</sub>]<sub>2</sub> which revealed the existence of both stable syn- and anti- isomers for this CH<sub>3</sub>S complex. More recently King [Inorg.Chem., 4, 482(1965)] has shown by proton n.m.r. studies that

Continued Page 2/

- 2 -

Professor Shapiro

18th April, 1966.

$[(\text{CH}_3)_3\text{CSFe}(\text{CO})_3]_2$  is isolated only as the anti-isomer. Some  $^{19}\text{F}$  chemical shifts and coupling constants are given in Tables I and II. The increasing shift to low-field of the ortho-fluorine atoms of the  $\text{C}_6\text{F}_5\text{S}$  group as the substituents on sulphur vary from H to  $\text{Fe}(\text{CO})_2(\pi\text{-C}_5\text{H}_5)$  and  $[\text{Fe}(\text{CO})_3]_2$  is noteworthy. It is apparent that the metal atom, although not directly bonded to the  $\text{C}_6\text{F}_5$  group still contributes to the deshielding of the ortho-fluorine nuclei (see Bourn, Gillies and Randall, Proc. Chem. Soc., 200(1963)).

Secondly we have been continuing our studies on platinum complexes  $\text{trans-(Et}_3\text{P)}_2\text{Pt}(\text{C}_6\text{F}_5)\text{X}$  [see I.I.T.N.M.R. Newsletter 82-10] and wish to report some corrected and some additional  $J_{\text{Pt-F}}$  <sup>ortho</sup> values dependent on the group X as follows:  $\text{ONO}_2$  (472 c/s),  $\text{Cl}(463)$ ,  $\text{Br}(461)$ ,  $\text{NCS}(443)$ ,  $\text{I}(441)$ ,  $\text{NO}_2$  (385),  $\text{CN}(348)$ , and  $\text{CH}_3$  (282). The results show that the lower the ligand X stands in the trans directing series for square planar platinum complexes the higher is the magnitude of  $J_{\text{Pt-F}}$  <sup>19</sup>F <sup>ortho</sup>, from which can be inferred an increasing Pt-C( $\text{C}_6\text{F}_5$ ) bond strength. This is in accord with expectation since groups which are highly trans directing (e.g. CN or  $\text{CH}_3$ ) are considered to weaken the platinum to ligand bond in the trans position and  $J_{\text{Pt-F}}$  coupling should thus be reduced. We believe that the magnitude of platinum-ortho-fluorine coupling constants in  $\text{trans-(Et}_3\text{P)}_2\text{Pt}(\text{C}_6\text{F}_5)\text{X}$  compounds represent a sensitive probe of the ligand field strength of the group X.

Yours sincerely,

F. J. Hopton

F. Hopton

Gordon

F.G.A. Stone

J. Cooke

Miss J. Cooke

A.J. Rest

A.J. Rest

TABLE I.       $^{19}\text{F}$  CHEMICAL SHIFTS<sup>†</sup>

COMPOUND	$\delta_o$	$\delta_p$	$\delta_m$
$\text{C}_6\text{F}_5\text{SH}$	139.9	161.5	164.6
$\text{C}_6\text{F}_5\text{SFe}(\text{CO})_2\text{Cp}^*$	132.2	159.3	164.5
$[\text{C}_6\text{F}_5\text{SFe}(\text{CO})_3]_2$	126.7 127.3	152.1 152.9	159.4 160.2

<sup>†</sup>Chemical shift in p.p.m. from  $\text{CCl}_3\text{F}$ (0 p.p.m.) as internal reference.

\*Cp =  $\pi\text{-C}_5\text{H}_5$

TABLE II       $^{19}\text{F} - ^{19}\text{F}$  COUPLING CONSTANTS (cps)

COMPOUND	$J_{13}(=J_{35})$	$J_{23}(=J_{34})$	$J_{12}(=J_{45})$	$J_{25}(=J_{14})$	$J_{15}$	$J_{24}$
$\text{C}_6\text{F}_5\text{SH}$	1.4	$\pm$ 20.1	$\pm$ 23.1	$\mp$ 8.0	$\pm$ 3.2	$\pm$ 1.4
$\text{C}_6\text{F}_5\text{SFe}(\text{CO})_2\text{Cp}$	0	$\pm$ 21.2	$\pm$ 25.0	$\mp$ 8.2	$\pm$ 1.4	$\pm$ 1.4
$[\text{C}_6\text{F}_5\text{SFe}(\text{CO})_3]_2$	2.4	$\pm$ 21.2	$\pm$ 24.1	$\mp$ 8.2	$\pm$ 3.6	$\pm$ 3.6



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**SPACE AND DEFENSE SYSTEMS**

A DIVISION OF FAIRCHILD CAMERA  
AND INSTRUMENT CORPORATION

21 April 1966

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

Waiting for Varian Equipment

We have very little to report in N.M.R. at this point. We are waiting for our old DP unit to be modified by Varian into a DA internal lock system with Y-axis autoshim control and for delivery of a C1024. All of this is scheduled to take place next month. We expect to start work on our dyes just as soon as the apparatus arrives and is installed.

I apologize for such a sparce news note, however, I hope to make it up in my next letter.

Sincerely yours,

James E. LuValle  
Director Basic Research

JEL/mi

# TECHNISCHE HOGESCHOOL EINDHOVEN

(TECHNOLOGICAL UNIVERSITY, EINDHOVEN, NETHERLANDS)

LABORATORIUM VOOR  
INSTRUMENTELE ANALYSE

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

Uw ref. (your ref.):

Eindhoven, April 13, 1966

Onze ref. (our ref.):

R/S

Postbus 513

Onderwerp (subject):

Medium effects in gases and liquids Tel. (04900) 68000

Dear Dr. Shapiro,

In a previous letter (1) two additions to the gas theory of medium effects (2) were proposed, see also (3). Applying this modified model to liquids (i.e. calculating the gas-to-liquid shift) good agreement was obtained (1,4) between experimental and calculated gas-to-liquid shifts of isotropic, non-polar molecular (i.e. molecules where only the van der Waals term  $\sigma_w$  is operative). This of course would open the door to the determination of precise values for the neighbour anisotropy effect  $\sigma_a$  and the polar effects  $\sigma_E$ . Before jumping to this we first of all want to further establish (or modify) the basic principle of our approach. To this end the following experiments are under way on this side of the ocean;

- 1)  $C_2H_6$ ,  $C_2H_4$  and  $C(CH_3)_4$  vapour are being studied for the pressure - and temperature dependence of  $\sigma_w$ . This should tell us whether the proposed repulsion effect factor  $(1-(r_o/r)^6$  is necessary and sufficient. The measurements are also extended to very high pressures to see whether or not there is a discontinuity in  $\sigma_w$  at the vapour-liquid transition (5). At any rate the non-unity of the slope of the calculated versus experimental gas-to-liquid shifts (1,4) needs explanation.
- 2) We also found that protons from different sites (such as methyl- and methylene protons in  $Si(CH_2CH_3)_4$ ) had different gas-to-liquid shifts but the differences were not in quantitative agreement with the calculated values. This is probably because for such large molecules a single force center potential is no longer adequate and should be replaced by a multi force center potential. The theoretical work is being assisted by experimental work on five  $X(CH_2CH_3)_4$  compounds.

## TECHNISCHE HOOGESCHOOL EINDHOVEN (Technological University, Eindhoven)

Ref.: R/S

Date: April 13, 1966

Page: 2

- 1) I.I.T-NMR Newsletter 82-14
- 2) W.T. Raynes, A.D. Buckingham, H.J. Bernstein, J.C.P. 36 (1962) 3481
- 3) F.H.A. Rummens, H.J. Bernstein, J.C.P. 43 (1965) 2971
- 4) F.H.A. Rummens, H.J. Bernstein, Copenhagen Congress, August 1965
- 5) S. Gordon, B.P. Dailey J.C.P. 34 (1961) 1084.

*H.V. Bernstein*

Yours sincerely,



(F.H.A. Rummens)

92-12

DIVISION OF PURE CHEMISTRY  
DIVISION DE CHIMIE PURE



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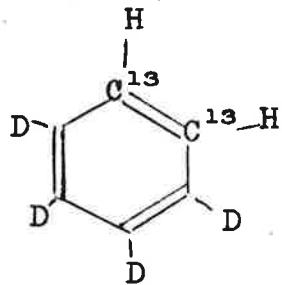
NATIONAL RESEARCH COUNCIL  
CONSEIL NATIONAL DE RECHERCHES  
CANADA

OTTAWA 2, 19 April, 1966

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

Dear Barry,

Roger Reavill has been working here on  
the orthobenzene shown below:



The analysis of the NMR spin multiplets  
gave (in c/s):-

$$J(C_{13}H) = 161.5 \quad J(CCH) = 1.0$$

$$|J(C_{13}C_{13})| = 57.0 \quad |J(HCCH)| = 7.7$$

I believe these values are accurate to  
about 0.1 c/s and are of interest to some of your readers.

Best regards,

Yours sincerely

H. J. Bernstein.

HJB/mn



RESEARCH LABORATORIES  
GENERAL MOTORS CORPORATION

April 21, 1966

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

Subject: Wide Line NMR Computer Programs

Dear Barry:

My contribution this time is very brief. It just consists of an announcement that I have available a report describing computer programs for wide line NMR computations. The report describes nine programs - three programs for a G.E. Basic system and six (Fortran II) for an IBM 7094. The programs described perform the following functions: 1) computation of second moments from experimental derivative line tracings; 2) calculation of theoretical rigid lattice second moments; 3) derivation of  $T_1$  from experimental decay or growth curves; 4) analysis of  $T_1$  data in terms of BPP theory; and 5) analysis of line narrowing curves in terms of the BPP theory. More than one version of a line narrowing program is given, and the G.E. basic programs are little more than adaptations of the 7094 programs.

A limited number of copies of the report are available upon request.

Yours truly,

George W. Smith  
Physics Department

ms  
Enclosure

CHEMISCHES LABORATORIUM  
DER UNIVERSITÄT MÜNCHEN  
INSTITUT FÜR ORGANISCHE CHEMIE

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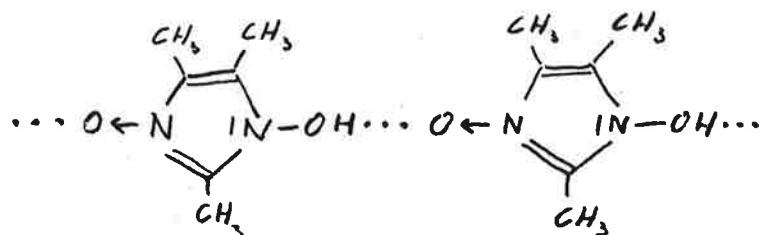
Prof. Dr. Herbert Zimmermann

Herrn  
Prof. Dr. Bernhard L. Shapiro  
Department of Chemistry  
Illinois Institute of Technology  
Chicago 16, Illinois  
USA

Sehr geehrter Herr Dr. Shapiro!

NMR-spektroskopische Untersuchung der Wasserstoffbrückenbindung von 1-Hydroxy-2.4.5-trimethyl-imidazol-N-oxyd

In den letzten Jahren haben wir die Wasserstoffbrückenbindung des Imidazols mit verschiedenen spektroskopischen Methoden untersucht. Die Arbeiten wurden nun auf ein Derivat des Imidazols, 1-Hydroxy-2.4.5-trimethyl-imidazol-N-oxyd, ausgedehnt. Die Substanz bildet intermolekulare Wasserstoffbrückenbindungen des Typs



Im IR-Spektrum sind die Wasserstoffbrückenbindungen durch extrem langwellig verschobene, stark verbreiterte OH-Valenzschwingungsbanden charakterisiert (langwellige Verschiebung ca.  $2500 \text{ cm}^{-1}$ ).

Die NMR-Spektren wurden in Chloroform im Konzentrationsbereich von 0,024 bis 1,75 Mol/l gemessen. Die Lage des OH-signals ist stark konzentrationsabhängig. Für hohe Konzentra-

tionen strebt das Signal dem Grenzwert  $\tau = -7,3$  ppm zu (bezogen auf TMS = +10 ppm). Der stark langwellig verschobenen OH-Valenzschwingungsbande entspricht demnach ein OH-Signal der Wasserstoffbrückenbindung bei extrem niedrigem Feld.

Die Viskosität der Lösung nimmt mit steigender Konzentration zu. Daraus kann geschlossen werden, daß unter geeigneten Versuchsbedingungen hochmolekulare kettenförmige Assoziate ausgebildet werden.

Mit freundlichen Grüßen

Ihr

*Gerd Henning*  
Gerd Henning

*H. Zimmermann*  
Herbert Zimmermann

DUQUESNE UNIVERSITY  
PITTSBURGH, PENNSYLVANIA  
15219

April 25, 1966

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

Dear Professor Shapiro:

Re: NMR study of ternary complexes

For the purpose of quickly deciding whether a ternary complex is formed on mixing a metal salt and two ligands, A and B, we have carried out experiments on the  $ZnCl_2$ -imidazole (A)-cytosine (B) and  $ZnCl_2$ -imidazole (A)-purine (B) systems.

Fig. 1(1) shows the proton magnetic resonance spectra of 0.1 M imidazole in dimethyl sulfoxide (DMSO). In the presence of equimolar  $ZnCl_2$ , Fig. 1(2), the C-2 proton signal moves further downfield than the C-4,5 protons showing that the coordination site in imidazole is the "pyridine" nitrogen. The equal downfield shifts of the C-5 and C-6 proton signals in cytosine, in the absence and presence of  $ZnCl_2$  as shown in Fig. 1(3) and (4) indicate the formation of Zn-cytosine complex with coordination site at the N<sub>3</sub> position. The positions of the signals of imidazole and cytosine in the absence of  $ZnCl_2$ , Fig. 1(5), as compared with Fig. (1) and (3), indicate that there is no interaction between imidazole and cytosine in DMSO. Fig. 1(6) shows that in an equimolar mixture of  $ZnCl_2$ , imidazole and cytosine, both the imidazole and cytosine signals are in the same positions as those of imidazole and cytosine, each separately in the

-2-

presence of  $ZnCl_2$ . This clearly demonstrates that a ternary complex is formed in which imidazole is bonded to Zn, which in turn is bonded to cytosine.

Fig. 2(3) and (4) show that purine is weakly bonded to  $ZnCl_2$ . A mixture of purine and imidazole gives a spectrum in which the imidazole signals are displaced downfield, indicating that there is hydrogen-bonding between imidazole and purine. In Fig. 2(6), for equimolar mixtures of  $ZnCl_2$ , imidazole and purine, the imidazole proton signals are shifted further downfield while the purine proton signals are displaced upfield from those in the presence of  $ZnCl_2$  only. The spectrum gives qualitative evidence that a ternary complex is formed, in which Zn is bonded to imidazole, which is hydrogen-bonded to purine.

We have given here some qualitative NMR evidence of the formation of ternary complexes, and have shown how NMR spectra can be used to demonstrate whether the ternary complex is of the type A-M-B or M-A-B, for systems in which A is imidazole and B is cytosine or purine. NMR studies on other ternary complexes, qualitative and quantitative, are in progress.

Sincerely yours,

Sung-Nan Wang  
Sung N. Wang

Norman C. Li  
Norman C. Li

Fig. 1

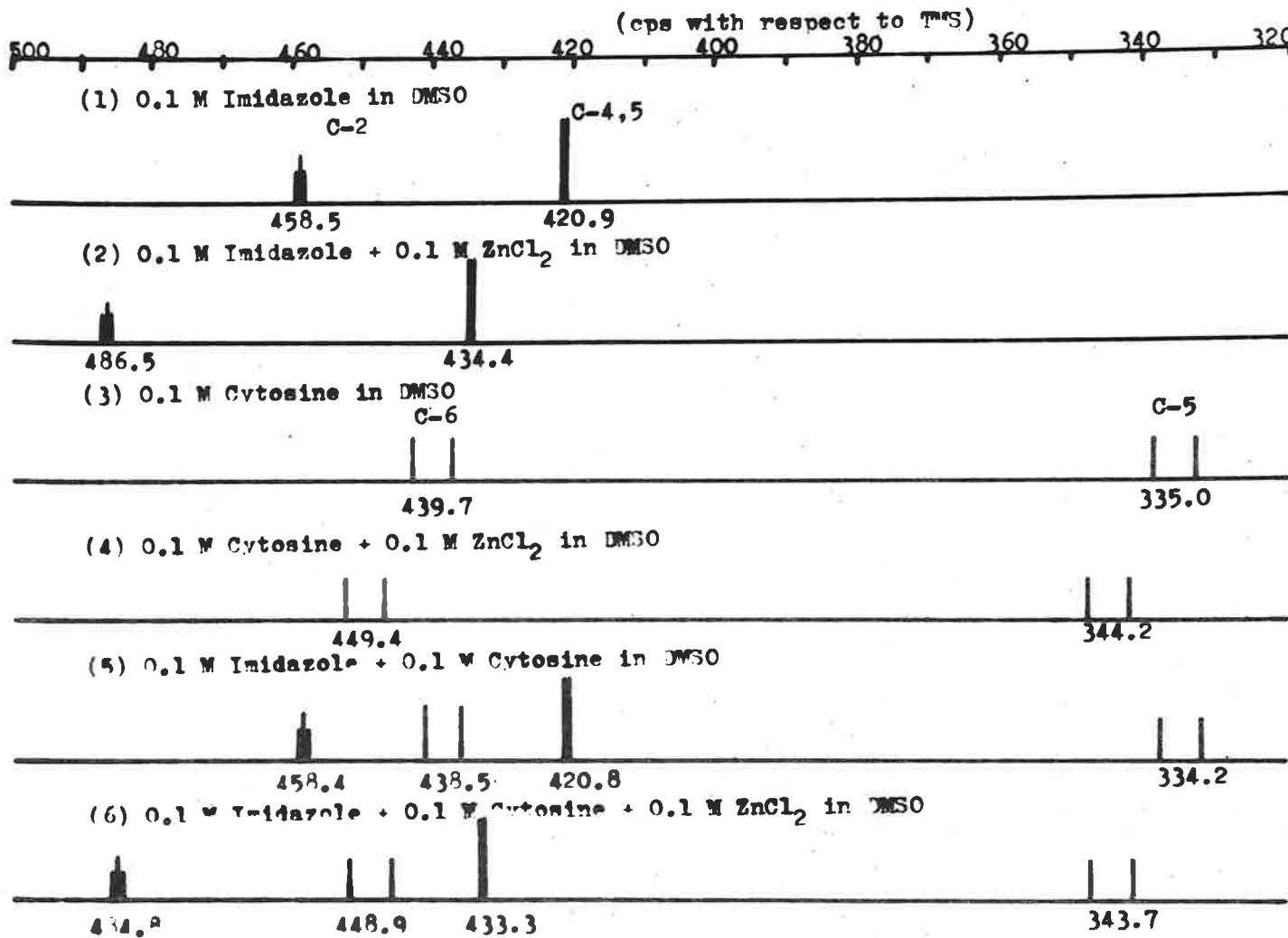
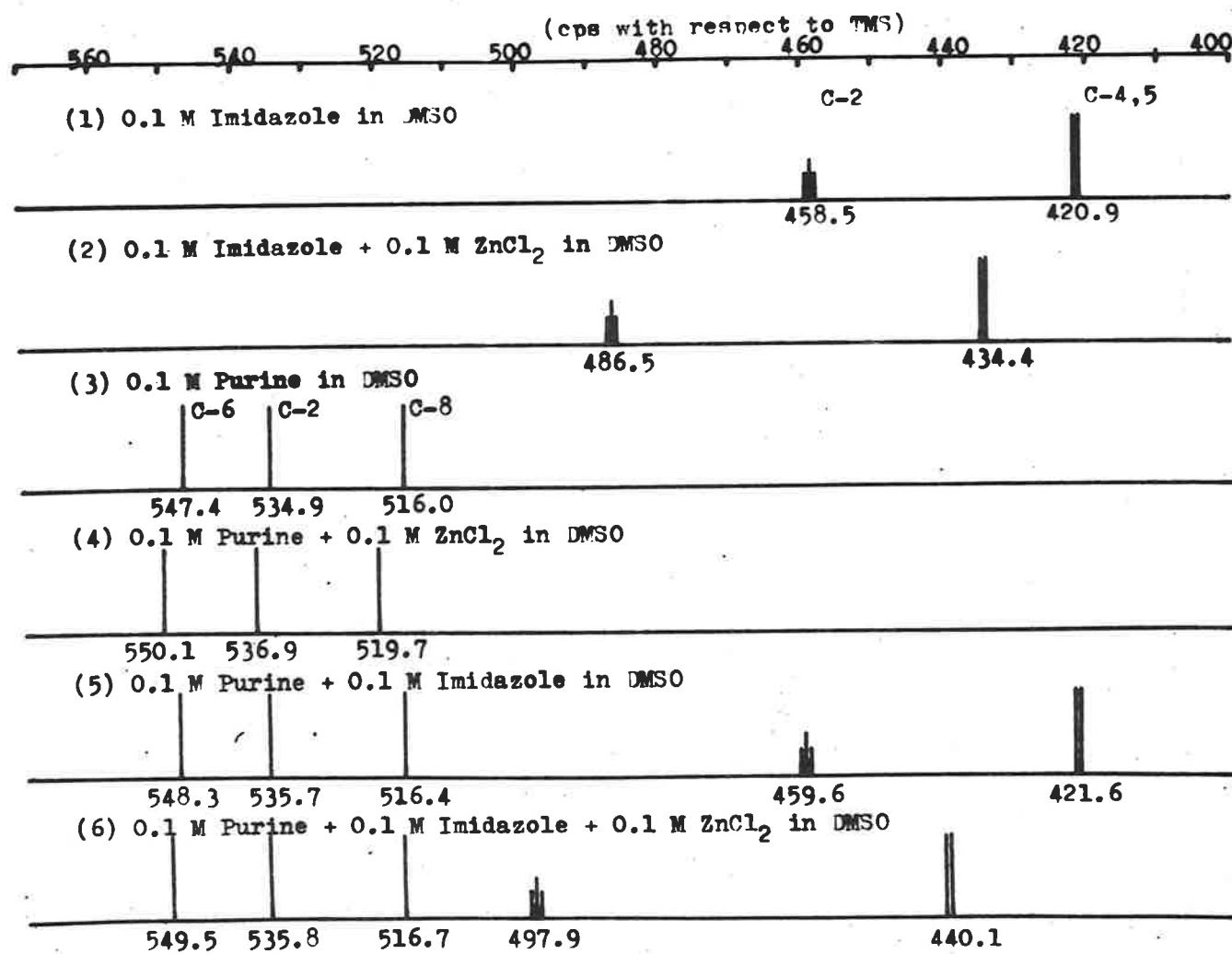


Fig. 2



THE UNIVERSITY OF WISCONSIN  
MADISON 53706

DEPARTMENT OF CHEMISTRY

April 26, 1966

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

Dear Barry:

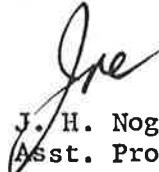
As a down payment on a subscription to IIT newsletters I would like to submit the following.

In cooperation with Dr. Paul Treichel of this department I have been doing the  $^{55}\text{Mn}$  NMR of a number of diamagnetic manganese complexes. The range of chemical shifts which we have so far observed spans 2500 ppm. The preliminary results are indicated on the enclosed table.

These results have suggested several trends which we are currently investigating - and won't discuss until they are investigated. The most curious result is the linewidth of the Manganese Carbonyl dimer. The observed linewidth is possibly the instrumental resolution. If the structure in solution is that determined by X-ray crystallography\* one would expect a healthy field gradient at the nucleus and, due to quadrupole relaxation, the very broad lines found in the other compounds.

The resonance in  $\text{KMnO}_4$  is narrow, as expected, because of the cubic symmetry about the Manganese. We observed this resonance but its position could not be measured accurately due to poor signal to noise. The table therefore gives the value calculated from the published magnetogyric ratio of "Manganese". (Cf. Sheriff and Williams, Phys. Rev. 82 651 (1951)). The large chemical shifts indicate that the compilers of tables of magnetic moments should indicate the compound to which the data applies.

Yours very truly,

  
J. H. Noggle  
Asst. Professor

JHN/jke

Enclosure

\*L. F. Dahl and R. E. Rundle, Acta Cryst., 16, 419 (1963).

<u>Substance</u>	<u>Solvent</u>	<u>Resonant Field At 4.33470 Mc.</u>	<u>Full Width At <math>\frac{1}{2}</math> Max. Intensity</u>
MnO <sub>4</sub> <sup>-</sup> *	Aqueous	4107.5 Gauss	~ 100 cps.
(C <sub>2</sub> H <sub>5</sub> ) <sub>4</sub> N <sup>+</sup> Mn(CO) <sub>4</sub> Br <sub>2</sub> <sup>-</sup> (cis)	CH <sub>3</sub> NO <sub>2</sub> (3.9%)	4108.9 Gauss	5100 ± 200 cps.
Mn(CO) <sub>5</sub> Br	CH <sub>2</sub> Cl <sub>2</sub> (2%)	4111.7 Gauss	700 ± 100 cps.
CF <sub>3</sub> Mn(CO) <sub>5</sub>	CH <sub>2</sub> Cl <sub>2</sub> (7.5%)	4114.2 Gauss	2900 ± 200 cps.
CH <sub>3</sub> Mn(CO) <sub>5</sub>	CH <sub>2</sub> Cl <sub>2</sub> (7%)	4116.0 Gauss	2800 ± 200 cps.
C <sub>5</sub> H <sub>5</sub> Mn(CO) <sub>3</sub>	CH <sub>2</sub> Cl <sub>2</sub> (22%)	4116.0 Gauss	13000 ± 1000 cps.
Mn <sub>2</sub> (CO) <sub>10</sub>	CH <sub>2</sub> Cl <sub>2</sub>	4116.3 Gauss	≤ 130 cps.
HMn(CO) <sub>5</sub>	neat (wet)	4117.2 Gauss	4500 ± 100
NaMn(CO) <sub>5</sub>	THF	4118. Gauss	9000 ± 400 <sup>†</sup>

\* Calculated from Varian Tables of Nuclear Moments.

† Uncertain due to poor signal to noise and possible impurities.

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

I Berlin 12, den April 26, 1966  
Straße des 17. Juni Nr. 115 (Chemiegeb.)  
Fernruf: 32 51 81, App. 252 Prof. B/Ma

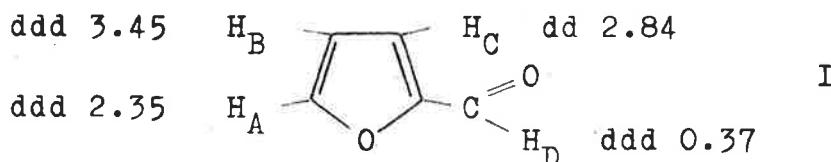
Professor Bernard L. Shapiro  
Illinois Institute of  
Technology

Chicago, 60616

U. S. A.

Dear Professor Shapiro:

In connection with structure determination work we have measured some furane compounds which may be of some interest. The main point was to study the influence of a carbonyl group in different distances in conjugated systems. As shown in the table the effects are small but easily to recognize. The HA 100 spectrum of furfural must be interpreted in the following way (in the Varian catalog there is a mistake).



$$J_{AB} = 1.7; J_{AC} = 0.8; J_{AD} = 0.5; \quad J_{BC} = 3.5; J_{BD} = 0.4 \text{ cps.}$$

Perhaps a little bit surprising is the clear difference in the shielding of H<sub>B</sub> in IX/X and XI/XII.

Yours sincerely,

F. Bohlmann

**Organisch-Chemisches Institut**

der

**Technischen Universität Berlin**

- 2 -

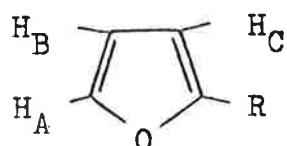
Direktor: Prof. Dr. F. Bohlmann

Professor Bernard L. Shapiro  
Illinois Institute of  
Technology

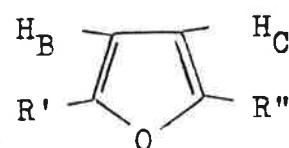
1 Berlin 12, den April 26, 1966

Straße des 17. Juni Nr. 115 (Chemiegeb.)

Fernruf: 32 51 81, App. 252



	R	H <sub>A</sub>	H <sub>B</sub>	H <sub>C</sub>
I	-CHO	dd 2.35	dd 3.45	dd 2.84
II	-CH=CHCHO	dd 2.49	dd 3.53	dd 3.30
III	-COCH <sub>3</sub>	dd 2.49	dd 3.53	dd 2.94
IV	-CH=CHCOCH <sub>3</sub>	dd 2.56	dd 3.58	dd 3.42
V	-CO-CH=CH-	dd 2.46	dd 3.50	dd 2.83
VI	-CO(CH=CH) <sub>2</sub>	dd 2.47	dd 3.53	dd 2.86
VII	-CH=CHCO-	dd 2.55	dd 3.57	dd 3.36
VIII	-(CH=CH) <sub>2</sub> CO-	dd 2.63	m 3.63	



	R''	H <sub>B</sub>	H <sub>C</sub>	R'
IX	-CHO	dt 3.48	d 2.80	HC≡C-CH <sub>2</sub> -
X	-CH=CH-CHO	dt 3.66	d 3.37	HC≡C-CH <sub>2</sub> -
XI	-CHO	dt 3.55	d 2.88	CH <sub>3</sub> [C≡C] <sub>2</sub> CH <sub>2</sub>
XII	-CH=CH-CHO	dt 3.60	d 3.25	CH <sub>3</sub> [C≡C] <sub>2</sub> CH <sub>2</sub>

**KYOTO UNIVERSITY**  
 DEPARTMENT OF CHEMISTRY, FACULTY OF SCIENCE  
 KYOTO, JAPAN

April 27, 1966

Effects of Excitation Energies on Spin Coupling

Dear Professor Shapiro:

In calculating nuclear spin coupling constants by making a mean triplet excitation energy approximation in perturbation theory, it is usually assumed that variations of the excitation energies  $\Delta E$  in a series of closely related molecules are negligible. There has been, however, some doubt as to the validity of this presumption, and recently Muller<sup>1</sup> questioned openly whether  $\Delta E$  is the same for the  $^{13}\text{C}-\text{H}$  coupling in methane and methyl halides.

We have examined the question by explicitly taking into account excited states, according to the method of Pople.<sup>2</sup> Given in Table 1 are the reduced coupling constants thus calculated together with those obtained with the mean energy approximation. The table shows that relative orders of magnitude of the experimental values are better reproduced when the 'same  $\Delta E$ ' approximation is discarded. The variations of the excitation energies may be seen in Fig. 1, where the excitations that make dominant contributions are indicated by arrows.

In spite of many a rough, unjustified approximations involved, the above results clearly indicate the importance of considering variations of the excitation energies in discussing nuclear spin coupling constants. Thus it may be hazardous to deduce hybridization, ionic character and bond angles from coupling constant data, taking account of the numerator alone in the second-order perturbation energy expression for spin coupling.

<sup>1</sup> N. Muller, J. Chem. Phys. 42, 4309 (1965).

<sup>2</sup> J. A. Pople and D. P. Santry, Mol Phys. 8, 1 (1964).

TABLE 1.

Molecule	$10^{-20} K_{\text{CH}} (\text{cm}^{-3})$ Mean Energy Approx.	Expt.
$\text{CH}_4$	33.1	41.4
$\text{CH}_3\text{F}$	46.1	49.3
$\text{CH}_3\text{Cl}$	40.7	49.7
$\text{CH}_3\text{Br}$	40.3	50.3
$\text{CH}_3\text{I}$	39.7	50.0

Yours sincerely,  
 A. SAIKA

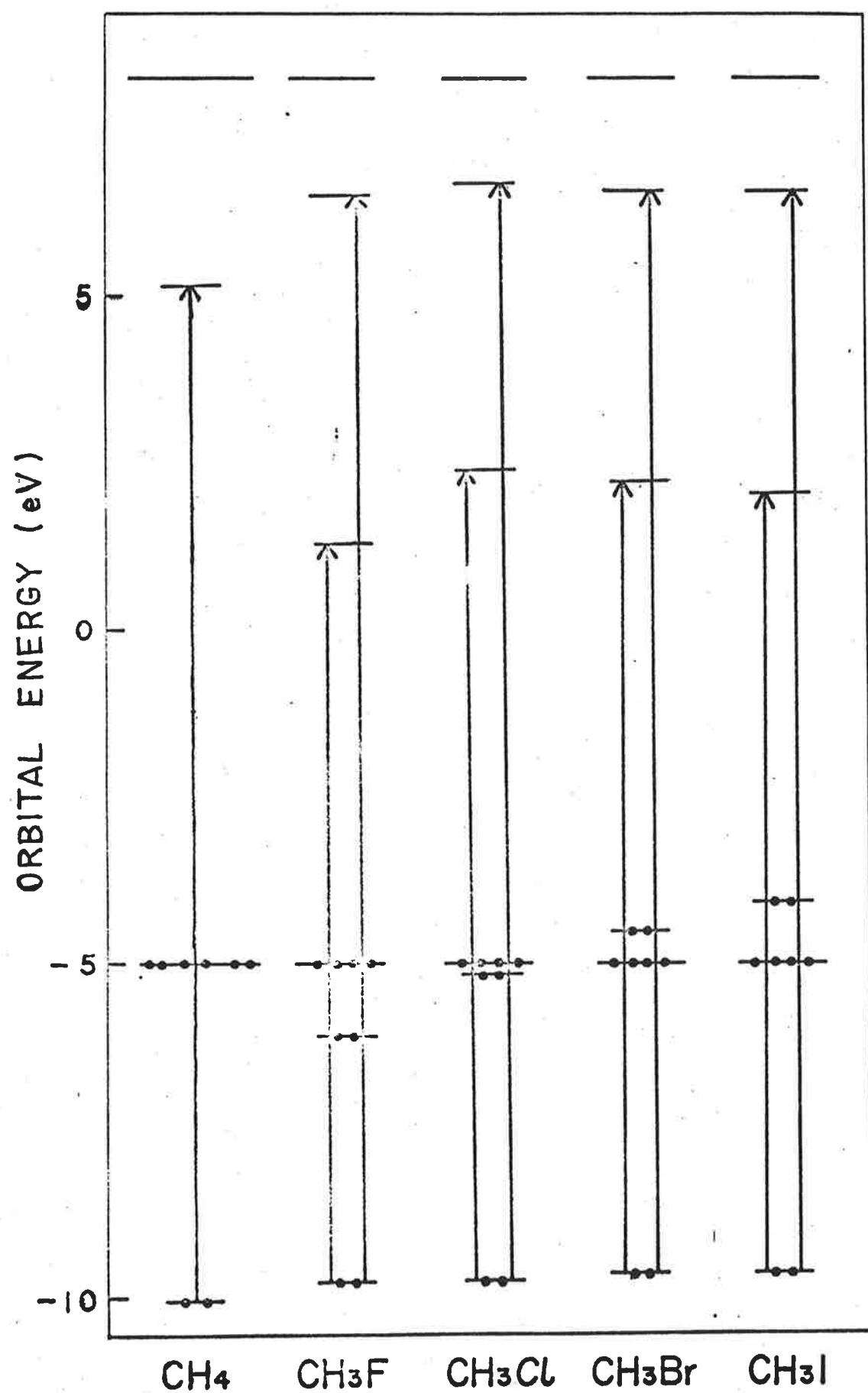


FIG. 1.



*Fysiska Institutionen, Uppsala*

Professor B.L. Shapiro  
 Department of Chemistry  
 Illinois Inst. of Techn.  
 Technology Center  
 CHICAGO, Illinois 60615  
 U.S.A.

Dear Barry,

Subjects: (a) Small talk; (b) A review of NMR; (c) More about signs of coupling constants.

During the past we have been collaborating intensely and commuted frequently between Uppsala and Stockholm. The NMR group in Stockholm has acquired a HA 100 spectrometer which has been in continuous operation for about six months by now. A few examples of its performance will be presented in connection with our second letter. In Uppsala a HA 60 - II is about to be installed. It is high time that the vintage 1957 steam engine is relieved of its high resolution duties to pass on its old days as a wide-line spectrometer.

One of us (R.A.H.) will spend the academic year 1966/67 at M.I.T. with John Waugh.

A good deal of our efforts during the past academic year have been concentrated on the shaping of a review of (and as introduction to) the subject of high resolution nuclear magnetic double and multiple resonance. This article will appear as Chapter 2 in the first volume of the Pergamon Press series "Progress in NMR Spectroscopy" (almost unpaid advertisement!)

Now, that sign determinations of coupling constants are hardly highest fashion any more, we may release the news that a general proof has given that the spectrum of a weakly coupled group X in any spin system is reflected in  $\chi$  if all spin coupling constants  $J_{KX}$  between X and other groups K are reversed in sign.

A corollary of this theorem is that the signs of  $J_{KX}$  with respect to some other coupling may be obtained provided that at least one pair of spins in the system are strongly coupled and provided that a non-symmetrical appearance of the X spectrum is permitted. We anticipate that strict symmetry of

the X part spectrum will as a rule only occur in single resonance spectra of the  $AEXn$  type. The fact that the spectra of weakly coupled groups are not in general symmetrical was emphasized by Lee (MELLON-M-R 50, 18) but his requirement that the secular equations should not be factorizable into  $2 \times 2$  subdeterminants was too narrow because it excludes spectra of the  $AB_2X$ -type (Diehl and Grünacher, J. Chem. Phys., 34, 1846 (1961)) or the  $ABEX$ -type (Lee and Sutcliffe, Trans. Far. Soc. 54, 308 (1958); B.D.N. Rao and P. Venkateswarlu, Proc. Indian. Acad. Sci. 54, 305 (1960)).

Most double resonance methods employed for sign determination in weakly coupled spin systems may be regarded as means of introducing assymetries.

Please credit this letter to R.A.H.

Best regards

*Ragnar A. Hoffman*  
/Ragnar A. Hoffman/

*Sture Forsén*  
/Sture Forsén/

P.S. We have been doing a few experiments also. Results will be given in a forthcoming letter.

Telephone: Coventry 24011

From : Dr. J.A.S. Smith

School of Molecular Sciences  
University of Warwick, Coventry

19th April, 1966.

Dear Professor Shapiro,

We think that it may be of interest to note that  $^{13}\text{C}$ - $^1\text{H}$  spin-spin coupling constants can be measured in the  $\beta$  - diketone chelate complexes of certain elements. High solubility is important, and with the high gains required to see the satellites, spinning sidebands may be troublesome so that the spectra have to be recorded at several spinning speeds. The general procedure was to record the entire spectrum at high gain on the 500 c/s sweep width, and by varying the spinning speed, identify the  $^{13}\text{C}$  satellites. The positions of these satellites were then accurately measured on the 100 c/s sweep width to  $\pm 0.02$  c/s. The spectrum of the two sets of  $^{13}\text{C}$ - $^1\text{H}$  satellites of the two types of  $\text{CH}_3$  group in  $\text{Be}(\text{CH}_3\text{COC}(\text{CH}_3)\text{COCH}_3)_2$  is enclosed. The sample was a saturated solution in  $\text{CDCl}_3$ .

These satellites have now been observed in over eleven  $\beta$  - diketone complexes; values for acetylacetone (enol) and five of its complexes are given in the table.

Cont'd.....

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. 2 .

Table  $J(^{13}\text{C}-^1\text{H})$  values (c/s) for some acetylacetone chelates

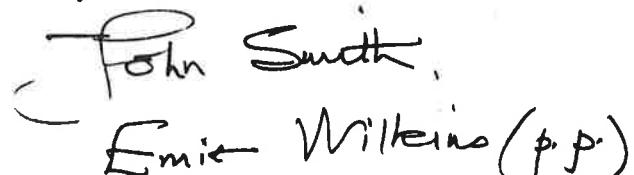
<u>Compound</u>	<u>Solvent</u>	$J(^{13}\text{C} \text{ in } \text{CH}_3)$	$J(^{13}\text{C} \text{ in CH})$
Acetylacetone (enol)	Neat Liquid	127.8	165.6
$\text{B}(\text{acac})\text{F}_2$	$\text{CDCl}_3$	130.2	174.5 <sup>(a)</sup>
$\text{Si}(\text{acac})_3\text{ClHCl}$	$\text{CDCl}_3$	129.6	-
$\text{Be}(\text{acac})_2$	$\text{CDCl}_3$	127.2	163 <sup>(b)</sup>
$\text{Al}(\text{acac})_3$	$\text{CDCl}_3$	127.5	-
$\text{Ga}(\text{acac})_3$	$\text{CDCl}_3$	127.6	-

(a) Determined on a molten sample of the solid (m.p. 38-40° C).

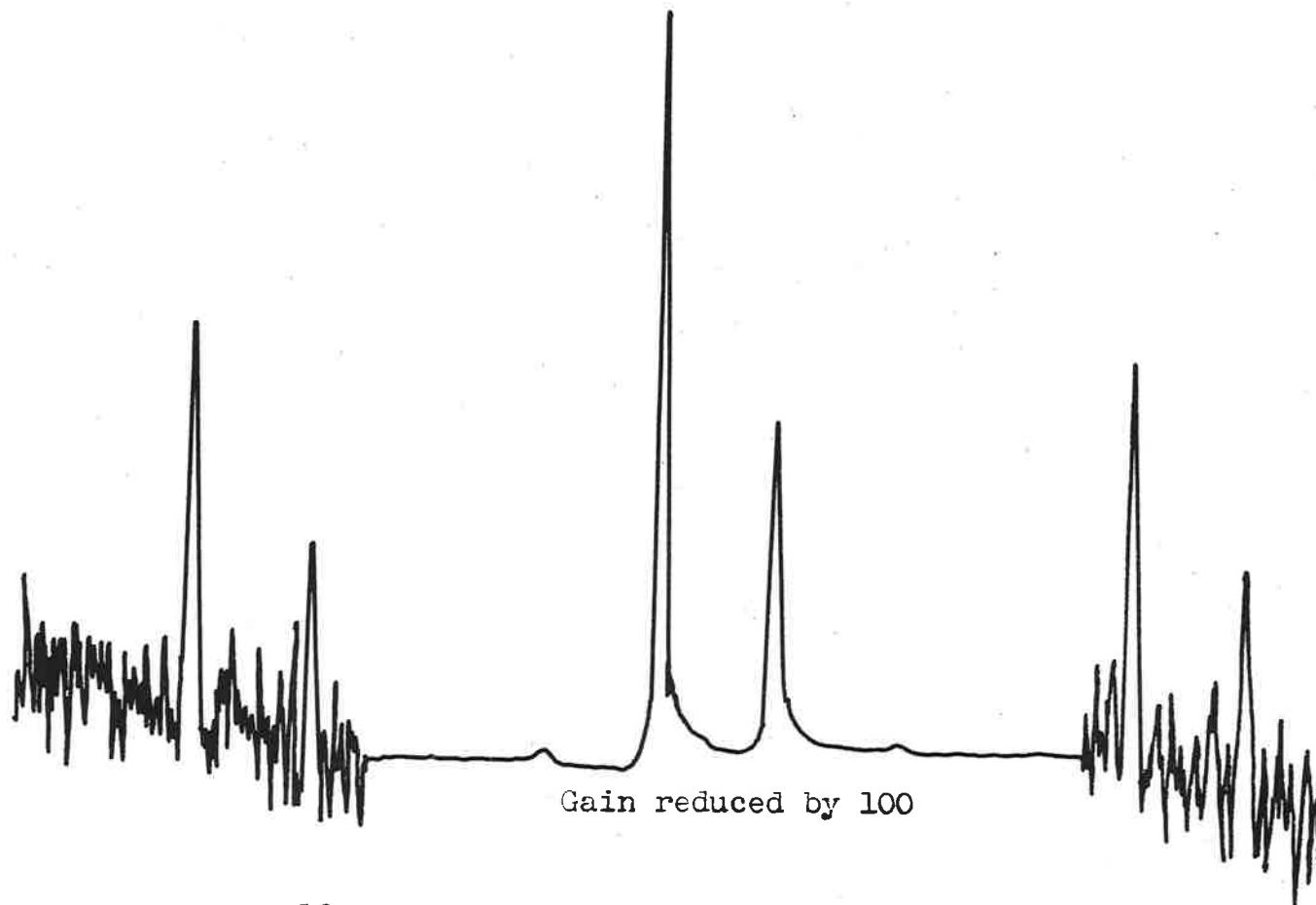
(b) Weak; determined on 500 c/s sweep.

At the moment, we are attributing the small differences between the complexed and free ligand to small variations in the electronegativity of the ring carbon atom.

Yours sincerely,



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



$^{13}\text{C} - ^1\text{H}$  coupling in  $\text{Be}(\text{Meacac})_2$  in  $\text{CDCl}_3$ .

Université Libre de Bruxelles



FACULTE DES SCIENCES  
Avenue F.D. Roosevelt, 50

Bruxelles 5, le April 20, 1966.

STRUCTURE OF THE CYCLIC DIONES FORMED BY  
CONDENSATION OF DIETHYL MALONYL CHLORIDE  
WITH POLYNUCLEAR AROMATIC HYDROCARBONS  
AND ALUMINIUM CHLORIDE.

Service: Organic Chemistry  
Dir.: Prof. R.H. MARTIN

(A rappeler dans la réponse)

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

Dear Dr. Shapiro,

The results of an earlier NMR investigation of cyclic aromatic ketones<sup>1)</sup> have been used to elucidate the structure of nine cyclic diones (I - IX) formed by condensation, at room temperature in CS<sub>2</sub>, of diethyl malonyl chloride with naphthalene, phenanthrene, pyrene and dibenzo[g,p]chrysene in the presence of aluminium chloride.

The NMR spectra of the diones, separated either by crystallization or by column, plate or vapor phase chromatography, are represented diagrammatically in Fig. 1 (c/s at 60 Mc, TMS = 0, solvent CDCl<sub>3</sub>).

- Naphthalene. We have fully confirmed the three structures I, II (ortho-condensations) and III (peri-condensation) attributed by FREUD<sup>2)</sup> on the basis of oxidative degradations of two of them [I (m.p. 81-82°) and II (m.p. 120-122°)].
- Phenanthrene. Three ortho-condensation products [IV (m.p. 126-127°), V (m.p. 149-150°) and VI (m.p. 88-88,5°)] have been fully characterized. Compound V (m.p. 149-150°) is probably the non identified dione (m.p. 137-139°) previously isolated by FREUD<sup>2)</sup>.
- Pyrene. This reaction gave two isomeric diones : an ortho-condensation product (VII) and a peri-condensation product [VIII (m.p. 164-165°)]. VII was not isolated in a pure state, but its spectrum

was easily deduced from the spectrum of a 50:50 mixture with VIII.

- Dibenzo[*g,p*]chrysene. The only isolated dione is the ortho-condensation product IX (m.p. 264-265°). The NMR spectrum of IX clearly shows the singlets of H<sub>13</sub> (H- $\alpha$ 3) and H<sub>17</sub> (H- $\alpha$ 4) :  $\delta_{H_{13}} = 5$  -  $\delta_{H_{17}} = 2$  c/s in CDCl<sub>3</sub> and 4,7 c/s in C<sub>6</sub>D<sub>6</sub><sup>3)</sup>.

- CH<sub>2</sub>--. In a given series, the CH<sub>2</sub> (quadruplet) of the peri-condensation product are deshielded (7-9 c/s) compared to the CH<sub>2</sub> of the indanediones (III/I, II - VIII/VII).

	$\delta_{CH_2}$	$\delta_{CH_3}$	J
I	116	44	$J_{4,5} = 8,2$
II	116	44	
III	125	47	$J_{4,5} = 7,1; J_{4,6} = 1,4; J_{5,6} = 8,2$
IV	117	45	$J_{4,5} = 9,0; J_{10,11} = 8,4$
V	117	45	$J_{7,11} = 0,7$
VI	120	47	$J_{4,5} = 8,0$
VII	122	47	$J_{10,11} = 9,0$
VIII	130	50	$J_{1,2} = 8,3$
IX	118	47	

- 1) R.H.MARTIN, N.DEFAY and F.GEERTS-EVRARD, Tetrahedron 20, 1505 (1964).
- 2) M.FREUD and K.FLEISCHER, Ann. 373, 291 (1910) and 422, 231 (1921).
- 3) R.H.MARTIN, N.DEFAY, F.GEERTS-EVRARD and S.DELAVARENNE, Bull.Soc. Chim.Belg. 73, 189 (1964).

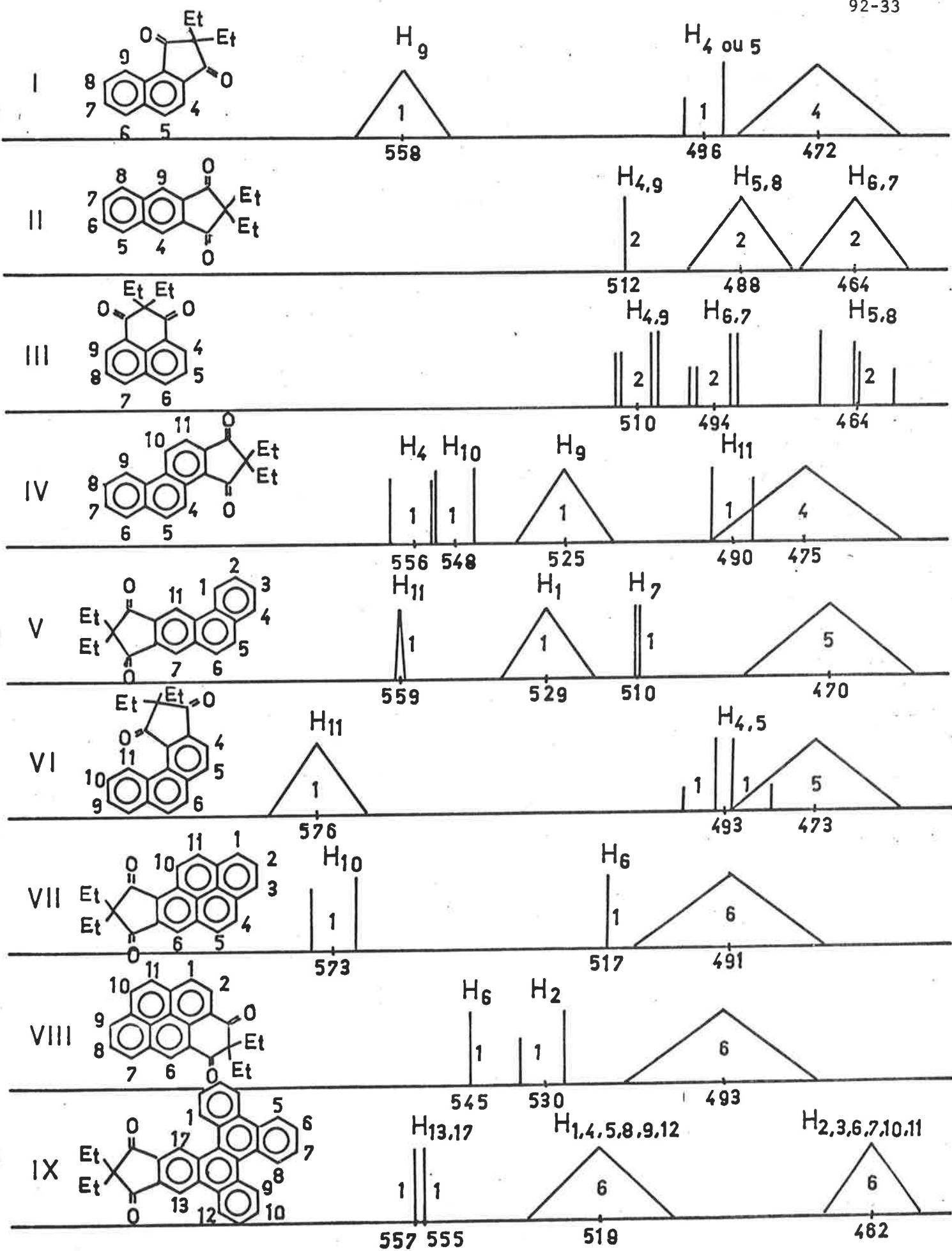
With best regards,

Yours sincerely,

M.DEBLECKER.

N.DEFAY.

R.H.MARTIN.



*Universidad de Buenos Aires*  
*Facultad de Ciencias Exactas*  
*y Naturales*

Buenos Aires, May 2nd., 1966.

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

Dear Prof. Shapiro:

We wish this time to comment on two notes which appeared in the 83 issue of the IITNMR Newsletter, the first by Drs. Ray Freeman and J.A.Ferretti on Transient Nutations in NMR Double Resonance, and the second by Dr. J.S.Martin on Long Range Coupling Values in Benzaldehydes.

1) The method of transient nutations has been in use in this laboratory as an aid in the interpretation of complex NMR spectra, instead of the "tickling" procedure, with very good results.

For the present purposes, the experiment is best performed with much less R.F. power than it was by Drs. Freeman and Ferretti, avoiding in this way the wiggles which may overlap with the other lines. To illustrate this, we include a copy of the first figure of a paper to be published in the Journal of Molecular Spectroscopy in which both techniques, "tickling" and proton INDOR, are used in an AB case. The Proton INDOR method can be considered a kind of "tickling" with the following features: a) Only the transitions which are related to the one which is being irradiated, are recorded. b) There is no possibility of uncertainty about the "progressive" or "regressive" character of the transition.

The advantages of the method were shown when studying some 3-substituted pyridines. We have been able to determine  $J_{26}$  in spite of the quadrupolar broadening of the signals of protons 2 and 6 by  $N^{14}$ . The result for 3-acetyl pyridine is:  $J_{26} = -0.21$  Hz. Preliminary experiments with other 3-pyridines (the procedure is rather lengthy since it requires more than one hundred records) also gives negative values for  $J_{26}$ .

*Universidad de Buenos Aires  
 Facultad de Ciencias Exactas  
 y Naturales*

This result should be compared with the theoretical prediction for pyridine by Drs. Gil and Murrel:  $J_{26} = +0.4$  Hz. (IITNMR Newsletter 81, 6)

2) We agree with Dr. Martin. The values of the Long range couplings in benzaldehydes as given by us were obtained from field swept spectra at 60 MHz. With our present frequency swept, field/frequency locked spectrometer, our precision borders the 0.01 Hz. Nevertheless it seems interesting to observe that in these four (or more) spin systems, the aldehyde signal cannot be accounted for (at 60 MHz) by simple first order theory. This is shown in the 2-methoxybenzaldehyde spectra: The 100MHz spectrum, which was taken by one of us at Varian in Zürich, shows a nice quadruplet, or rather two doublets, equally spaced. The 60 MHz. spectrum, on the other hand, shows two different doublets. So, it is quite natural, that the 60 MHz. published values should be revised, specially when looking for conformational effects.

Incidentally, this case could probably pass as a record in resolution at 100 MHz, since the smallest splitting is only 0.15 Hz!

This, probably shows that the ultimate in resolution is not so much due to modern instruments as to the linewidth of the sample.

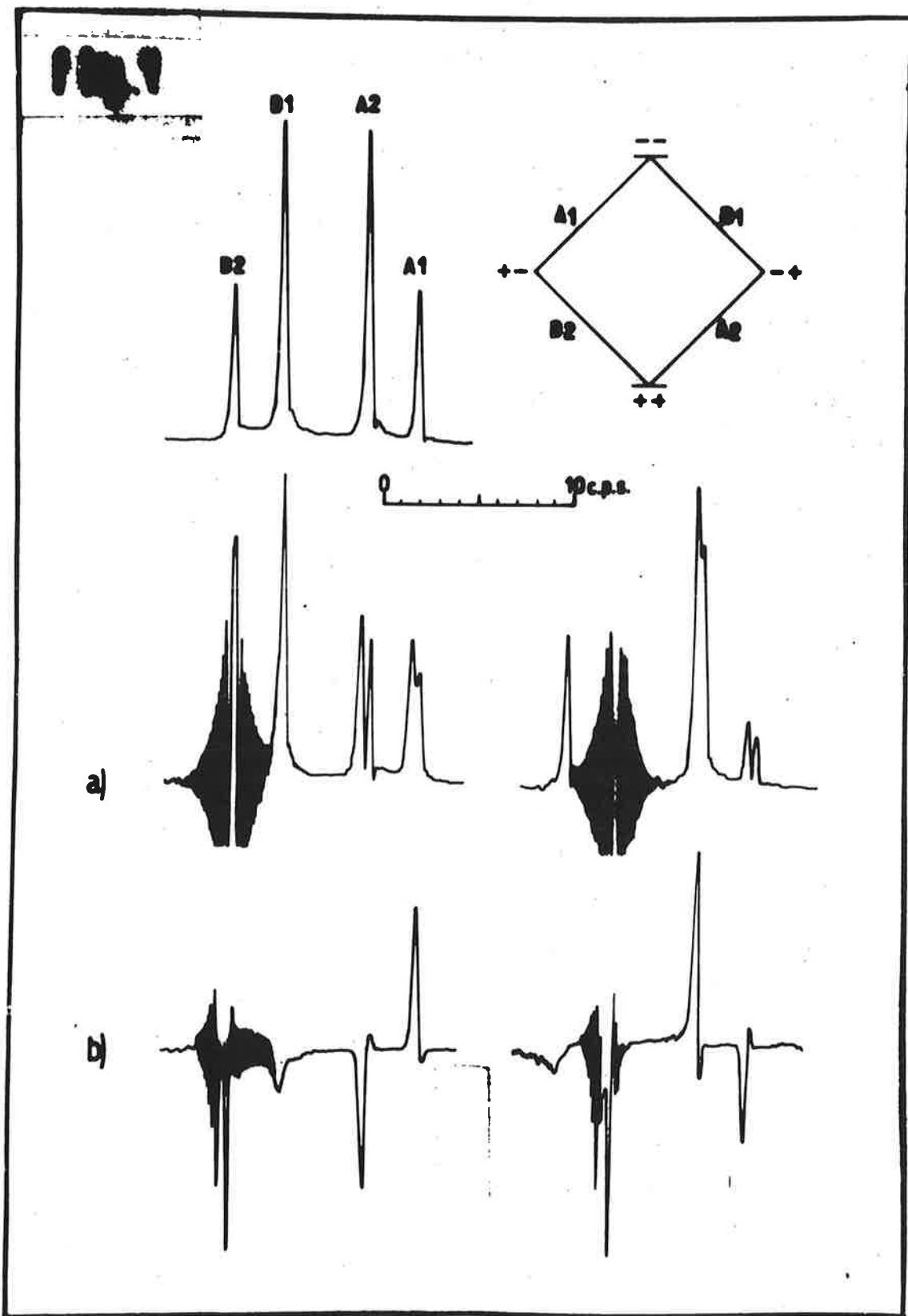
Sincerely yours,

*D.G. de Kowalewski*

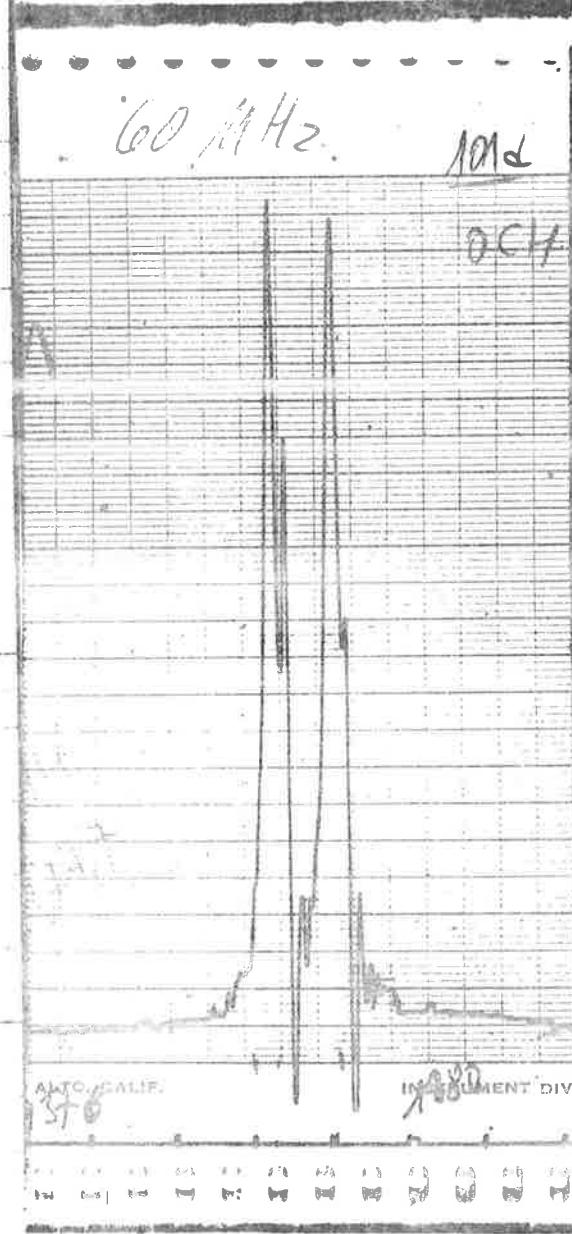
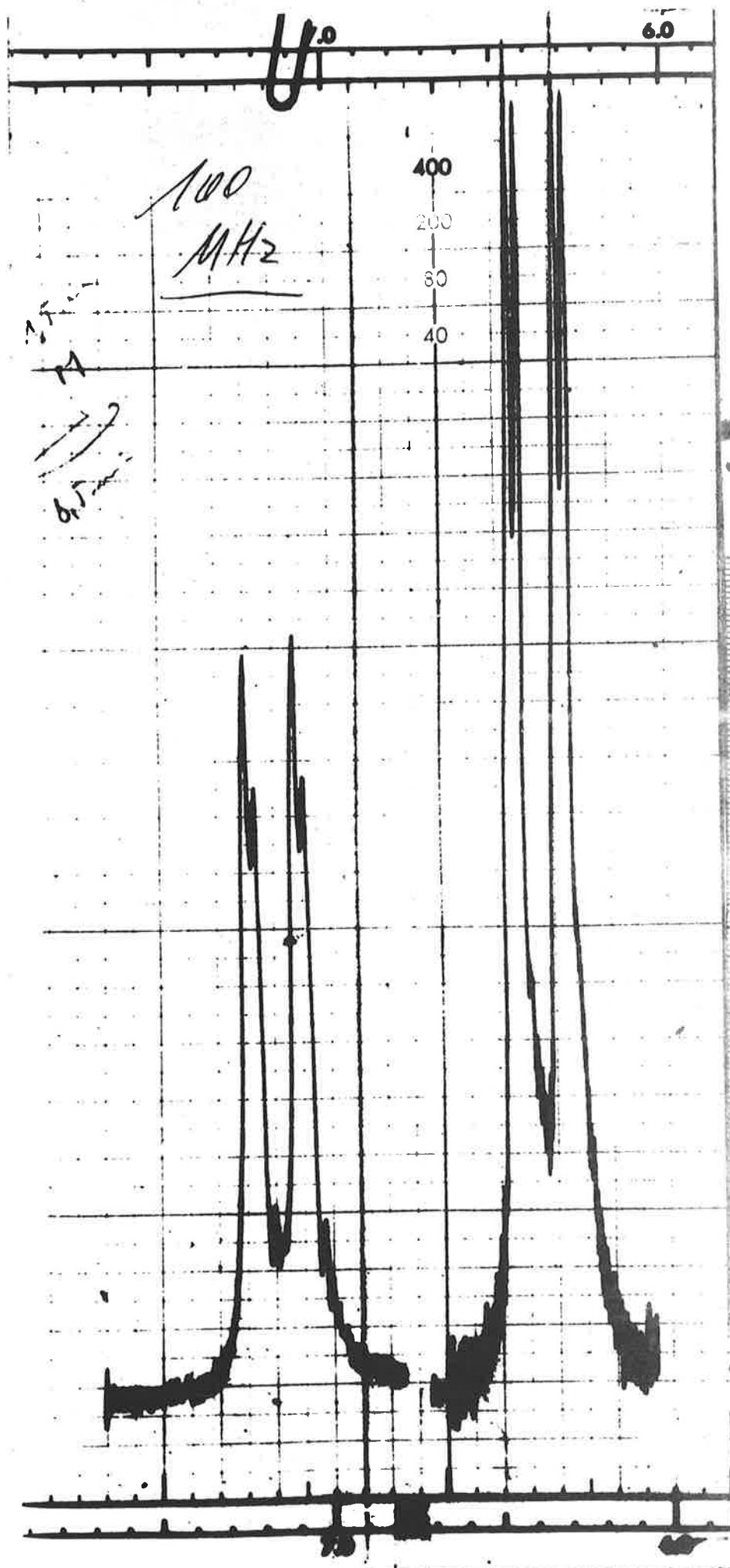
D.G. de Kowalewski

*V.J. Kowalewski*

Suggested title: Proton INDOR experiments: a negative ring coupling.  
 Second order effects in benzaldehydes.



92-37



## PHYSIKALISCHES INSTITUT

Professor B.I.Shapiro  
Dept. of Chemistry,  
Illinois Institute  
of Technology,  
Technology Center

Chicago, Ill. 60616  
USA

LEIPZIG C 1, LINNÉSTR. 5 - TEL.: SAMMELNR. 64511

am 27/4/66 Ans: LÖ/Sw.

Dear Professor Shapiro :

Many thanks for your letter of 2nd April, which arrived a few days ago. In addition to my letter of 2nd March containing an outline of our work, I wish to inform you about some results being not yet published in detail.

When looking for effects of a phase change in ferroelectrics at the Curie-point, the application of ESR-signals seems very promising; their splitting depends on the crystalline field. We doped Triglycine-sulfate with Cu<sup>++</sup>, but we could not find any change. On the other side T<sub>1</sub>-measurements of the proton-resonance revealed a change near the Curie-point, which was labelled by the Cu<sup>++</sup>-ions. The relaxation behaviour of the proton spins is much more affected by the paramagnetic ions of low concentration than the ESR-spectrum itself. The explanation for this was found by Dr. Lindsch who made the ESR-measurements. Thus we know that the Cu<sup>++</sup>-ion is forming together with 2 glycine molecules a chelate complex which is rather stable; it could be proofed also in liquid solutions. Such a complex does not show any ferroelectricity; the macroscopic behaviour is only very slightly changed because of the low concentration (1:200) of these complexes. As for the proton spins, their relaxation time is changed by the Cu<sup>++</sup>-ions by spin-diffusion. This mechanism transports the electron relaxation also to remote nuclei. In this way the protons of a special O-H---O-bond which seem to be responsible for the ferroelectricity, are also affected, and the change in T<sub>1</sub> near the Curie-temperature vanishes. In this way the originally unexpected effects may be explained.

We hope to get next more informations from TGS by other paramagnetic impurities.

Yours sincerely,

A. Lösche  
(Prof.Dr.A.Lösche)

- r) We are grateful to Prof. Blinc  
in Lubljana for giving to  
Dr. Grenke from our institute the  
possibility of measuring the  
relaxation time.



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ccp Paris 9065 96 au nom  
de l'agent comptable de l'Ircha

Direction générale  
Services de Paris  
12 quai Henri IV, Paris 4  
téléphone : 2728270  
N° 3085 P. GM 191

Prof. B.L. SHAPIRO  
Department of Chemistry  
Ill. Inst. of Tech.  
CHICAGO, Ill, 60616  
(U.S.A.)

Paris, le 4 Mai 1966

H<sub>1</sub> and P<sub>31</sub> study of conjugation in acetylenic phosphonates.

Dear Barry,

As we begin to know how to use our Perkin-Elmer R 10 spectrometer (with facilities for field- and frequency - sweep decoupling as well as for high resolution B, F, P resonances), we resume our work on organo-phosphorus compounds. We are especially interested in the stereochemistry of vinyl or allyl derivatives, but, for the moment, I will consider closely enough related compounds, i.e.  $\alpha$  acetylenic phosphonates:  $(R'O)_2P(O)C\equiv CR$  with  $R' = C_2H_5$  (or  $C_4H_9$ );  $R = H; CH_3, CH_2OH; C_2H_5, CHClCH_3, CH(OH)CH_3;$   $\pm C_3H_7$  to  $\pm C_6H_{13}$ ; lastly  $C(CH_3) = CH_2$  and  $CH_2O C(CH_3)_3$ . As the interest in PMR of acetylenic phosphorus compounds is increasing, especially over here (<sup>1,2,3</sup>), a time after less sophisticated studies (<sup>4,5</sup>), instead of describing individual spectra, I will discuss only the most intriguing point, for us, that is the possibility of conjugation effects involving the triple bond and the phosphoryl group. From IR, this is likely to occur

.....

$(C_2H_5O)_2 P(O) C \equiv CH$	$\pm C_4H_9C \equiv CH$
$\nu C \equiv C$	2075
$\nu CH$	3295

The simplest PMR data are obtained for

	$\delta_{CH}$	$J_{PCH}$
$(C_2H_5O)_2 P(O) C \equiv CH$	4,0	13,4
$\pm C_4H_9C \equiv CH$	1,72	-
	$\delta_{CH_3}$	$J_{PCH_3}$
$(C_2H_5O)_2 P(O) C \equiv CCH_3$	2,05	4,6
..... $C \equiv CH_3$	1,80	-

For the true acetylenic compound, the comparison is especially clear when using the correlation diagram build-up by DRENTH and LOEWENSTEIN (<sup>2,6</sup>) for various substituted acetylenes. Providing for inductive effects (via the "internal chemical shift" of the parent saturated derivatives), this chart (figure below) points out deviations due to conjugative effects, roughly described as, e.g.,  $HC^- \rightleftharpoons C = N Et_2$ ,  $HC^- = C = O Et$ ,  $HC^- = C = F$ . Evidently, our  $HC^+ = C = P(O^-)(OEt)_2$ . Unfortunately the corresponding phosphine (<sup>1,2</sup>) is also out law in the same direction, but to a lesser extent, and it is rather difficult to write down  $HC^+ = C = \bar{P} Et_2$ .

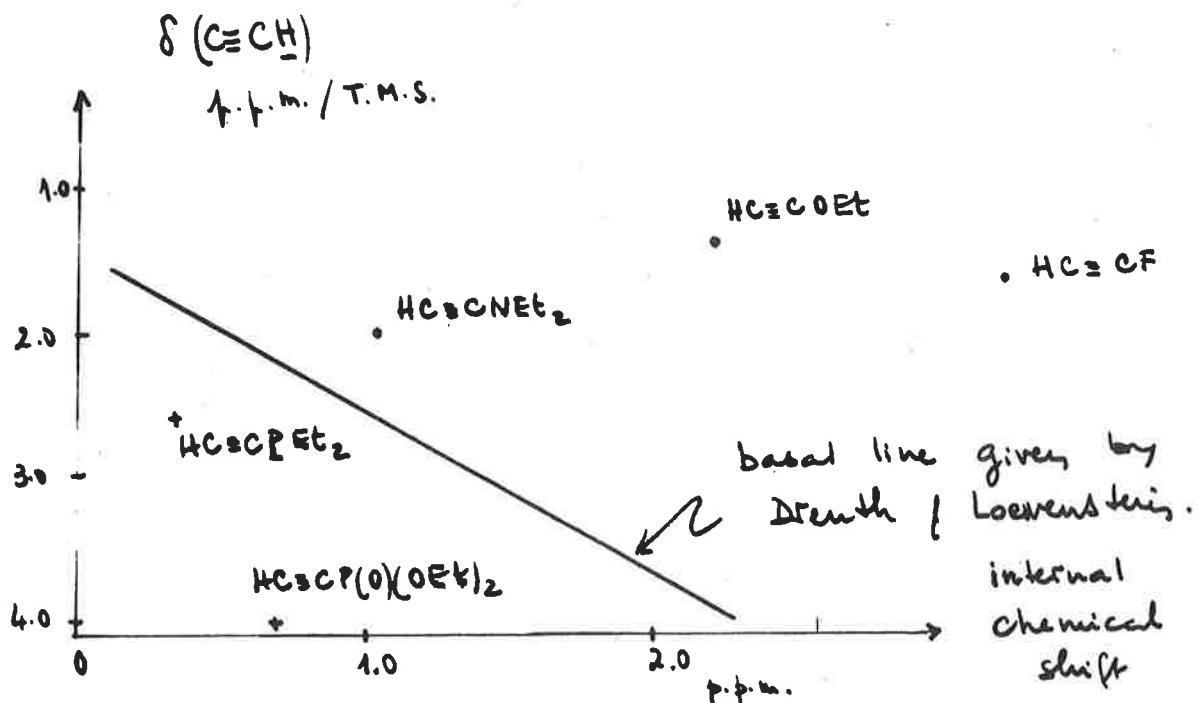
Without trying to present an explanation, we turn to results. All acetylenic phosphonates appear in the range 120.5 to 122.5 up field respect to  $P_4O_6$  (a nice standard indeed for phosphorus resonance) or 8.0-10.0 up field phosphoric acid, when alkyl phosphonates have peaks in the range -28.0 to -32.0<sup>7</sup>. The deviation is actually much greater than for ketophosphonates (-23 against ca. -30)<sup>8</sup> or even for vinyl phosphonates (we observe -10.5 for  $CH_2=CH P(O)(OC_6H_5)_2$  against ca. -24). According to a recent theoretical analysis (<sup>9</sup>), this would indicate some increase in the  $\pi$  bonding around the phosphorus atom. We wait for some UV data to precise the nature of this perturbation -undoubtedly it is a strong one.

With my very best regards

G. MAVEL

\* Thereafter / phosphoric acid

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p. 1002.
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- (6) DRENTH, LOEWENSTEIN, Rec. Trav. Chim., 81, 633, 1962.
- (7) MOEDRTZER, MAIER, GROENWEGHE, J. Chem. Eng. Data 7, 307, 1962; NIELSEN, PUSTINGER, STRÖBEL, ibid. 9, 167, 1964.
- (8) COTTON, SCHUNN, J. Amer. Chem. Soc. 85, 2394, 1963.
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## THE UNIVERSITY OF BIRMINGHAM

TELEPHONE SELLY OAK 1301



Department of Chemistry,

BIRMINGHAM, 15.

10th May 1966.

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

Dear Professor Shapiro,

These values of Fluorine Coupling Constants in Cyclohexanes may be of some interest to readers of the IIT NMR Newsletter. They were obtained by Mr. Alan Peake from the spectra of  $F_1$  and  $F_4$  in  $1CF_3/-C_6F_{11}$ ,  $1CF_3,4H/-C_6F_{10}$ , and  $1CF_3/4H-C_6F_{10}$ , and most of the assignments have been confirmed by double or triple irradiation.

<u>Relative ring position.</u>	Coupling constant (c/sec.)	
	F-F	H-F
1,1	290 - 305	~ 45
1,2 gauche	~ 14	~ 6
1,2 trans	0 - 3	~ 17
1,3 diaxial	~ 27	}
1,3 diequatorial	~ 9	
1,3 axial-equatorial	~ 1	
* $H_{\text{ax}} - F_3?$	= 4.0	; $H_{\text{eq}} - F_3?$ = 3.3 c/sec.

Yours sincerely,

L.F. Thomas.



**BP RESEARCH CENTRE**  
**SUNBURY-ON-THAMES**  
**MIDDLESEX**  
**ENGLAND**

NMR COMPUTER PROGRAM CORRECTIONS

Use of the ALGOL version of FREQUINT IV (Sekuur, IITNMR 67-15, Knight and Bonner IITNMR 82-27) for the calculation of five spin NMR spectra can cause the computer to print out a subscript overflow error message. The error can be eliminated by the inclusion of a suitable IF statement (indicated by an arrow) in the main body of the program. The position of the additional step is given below.

```
-----
I1:=I1+1      J1:=I1+1
END
END'
→ IF NEG(N1)=NNEG THEN CC(I1,I1):=0.5*SW(J2)+.25*SA(J2)'
FOR I:=1 STEP 1 UNTIL NORDER DO
-----
```

Recently we have been able to run the FORTRAN version of NMRIT which we have partly translated into ALGOL. The computer output contained the message - "source program error, execution deleted". After a study of the program, dated 11.14.63, we made some modifications that resulted in the successful execution, and print-out on the correct format, of the test ( $CF_3CCl-CF_2$ ) and other problems. As other contributors to IITNMR may have a copy or card deck of this program, and may like to get it working, a list of the modifications are given below.

NMRIT main program

Delete - IF (NEN) 61,61,60  
 6OWRITE OUTPUT TAPE 6,127,KN,KM,XX,T(JJ),J

SUBROUTINE AMP

Delete - IF (T(KS)-0.1) 7,7,9  
 Insert - IF (T(KS)-0.05) 7,7,9

SUBROUTINE BEQUIV

Delete - IF (AB(I,J,1) 14,13,14  
 Insert - IF (AB(I,J,1)) 14,13,14

SUBROUTINE SPINH

Delete - DIMENSION statement continuation cards 2, 3 and 4  
 Insert new continuation card 2:-

2A(8,8),B(8),DH(256),U(8),SA(64),Y(70)

S.A. Knight

S. Knight

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



Germany  
 Prof. Dr. B.L. Shapiro

Department of Chemistry,  
 Illinois Institute of  
 Technology

Chicago, Illinois 60616

U.S.A.

Ihre Zeichen	Ihre Nachricht vom	Unsere Zeichen	Fernsprecher-Durchwahl	Telex	67 Ludwigshafen
		Dr. Brügel/Fa	(0621) 600 . . .	464 . . .	12. Mai 1966

Betreff Difficulties with the internal standard hexamethyl disiloxane

Sehr geehrter Herr Dr. Shapiro!

Wie ich Ihnen schon schrieb, war es mir infolge längerer Abwesenheit von Ludwigshafen nicht möglich, meine Verpflichtung gegenüber den News Letters rechtzeitig zu erfüllen. Besten Dank für die Fristverlängerung. -

Der weltweit übliche interne Standard Tetramethylsilan (TMS) bringt wegen des sehr niedrigen Siedepunktes dieser Substanz einige Nachteile mit sich, insbesondere wenn man bei höheren Temperaturen arbeitet. Wir haben daher versucht, ihn durch den sogenannten von verschiedenen Autoren empfohlenen inneren Standard Hexamethyldisiloxan (HMDS) zu ersetzen. Dabei haben wir einige Erfahrungen gemacht, die vielleicht auch anderswo von Interesse sind.

Das von uns meist benutzte Lösungsmittel war Dimethylsulfoxid (DMSO), zugegebenenfalls völlig deuteriert. Eine lösige Lösung von HMDS in DMSO ist ohne weiteres möglich. Lässt man nun aber

**Badische Anilin- & Soda-Fabrik AG**

<b>Empfänger</b>	<b>Unsere Zeichen</b>	<b>67 Ludwigshafen</b>	<b>Blatt</b>
Dr. B.L. Shapiro	Dr. Brü/Fa	12.Mai 1966	2

**Betreff**

in diesem Gemisch die zu untersuchende Substanz, so zeigte sich, daß HMDS zu einem mehr oder minder großen Teil aus der Lösung verdrängt wurde. Die Folge davon war das Auftreten von zwei Linien des zugesetzten inneren Standards in der Gegend von  $\delta = 0$  ( $\tau = 10$ ) statt der erwünschten einen, die im allgemeinen etwa 3 bis 5 Hz, in Sonderfällen aber bis zu 35 Hz voneinander getrennt waren. Die nähere Untersuchung der Lösung ergab, daß ein Teil des HMDS echt in Lösung geblieben war, während ein anderer Teil in Form ganz feiner Tröpfchen in der Flüssigkeit schwiebte. Diese Erscheinung war besonders ausgeprägt, wenn das DMSO etwas Wasser enthielt (was ja wegen der Hygroskopizität dieser Substanz schwer vermeidbar ist) oder wenn die zu untersuchende Substanz Hydratwasser mitbrachte. Derselbe Effekt wurde auch bei einigen anderen Lösungsmitteln beobachtet. Bei der Verwendung von HMDS als interner Standard scheint mir daher eine gewisse Vorsicht am Platze. Wir verwenden es nur noch bei Hochtemperaturuntersuchungen.

Mit den besten Grüßen

Ihr



(Dr.W.Brügel)

## CARLETON UNIVERSITY

OTTAWA 1, CANADA



DEPARTMENT OF CHEMISTRY

May 12, 1966.

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

Dear Dr. Shapiro:

"The measurement of angles and distances in molecular models".

Here is our first contribution to IITNMR.

We are at present involved in some work on the effects of anisotropic functional groups on methyl resonances in the n.m.r. spectra of steroids and terpenes. We required a precise and reproducible method for locating the methyls from such groups, and have developed a suitable device for this job.

The diagram attached shows the parameters required for location of the methyl group in the case of a double bond as illustrated for 10-Methyl- $\Delta^2$ -octalin. The two angles  $\theta$  and  $\gamma$  and the distance R are readily determined by the use of the device shown in the photograph (I think this will Xerox--see the enclosed Xerox copy tried out here). The reference point for the methyl group is taken as the centre of the circle described by the methyl hydrogens during rotation about the C-CH<sub>3</sub> bond and this is readily determined by a plexiglass template on which this point is marked.

The device itself consists of an extendable locating pointer which pivots at the centre of a Drieding model isolated double bond unit. The centre block of this unit is constructed with two protractors and by means of pointers (A + B on photograph) attached to the main locator the angles  $\theta$  and  $\gamma$  can be read off directly.

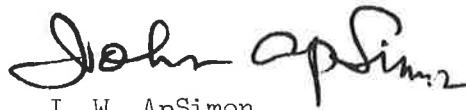
Thus, the whole device can be assembled into a molecular model, the distant group located (a methyl in our cases) and the pivot tightened by means of a screw underneath the pivot point. Direct measurement of the required parameters is then possible.

Our results from the use of such a device are progressing favourably in conjunction with Professor Whalley's group at the School of Pharmacy, University of London.

Copies of the original photograph may be obtained from the undersigned as well as any construction hints. Mr. Raffler of our workshop made our "Ballistic Missiles" while Paul Demarco and Bill Craig made very useful suggestions.

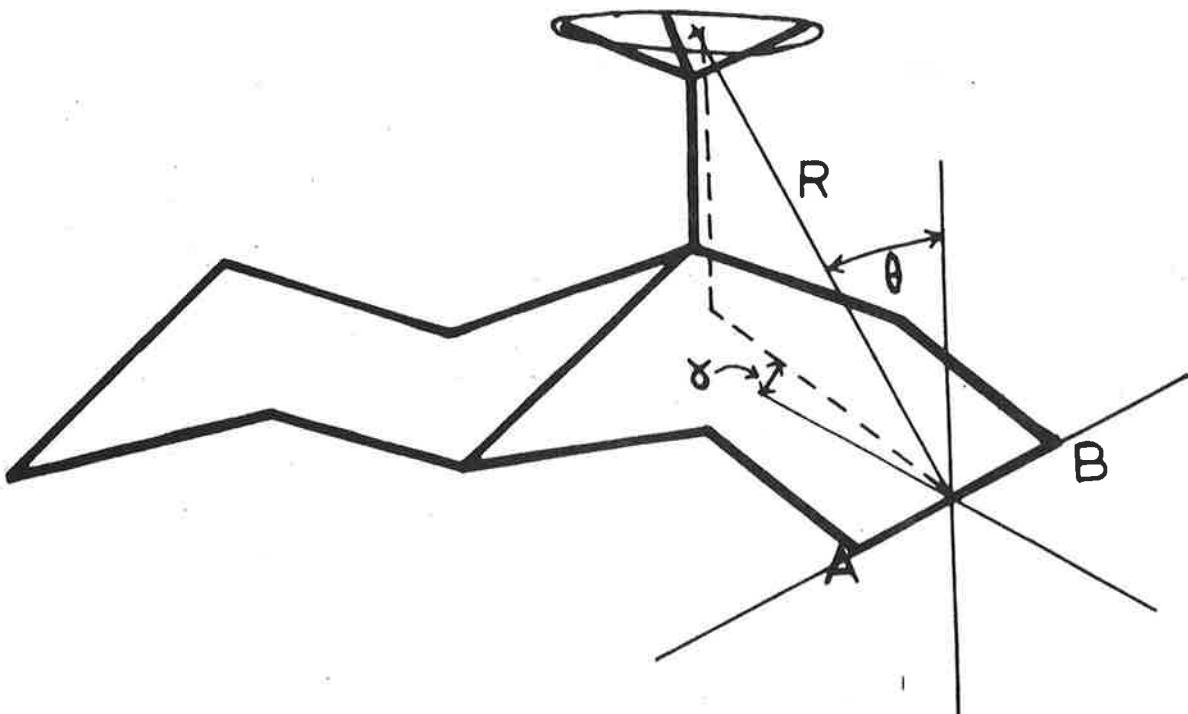
In a lighter vein, you might be interested in an "undergraduate howler" we had in an exam last week describing an instrumental technique as NUCLEAR MAGNETIC RESONANCE -- spectroscopy through rose coloured sample tubes I guess.

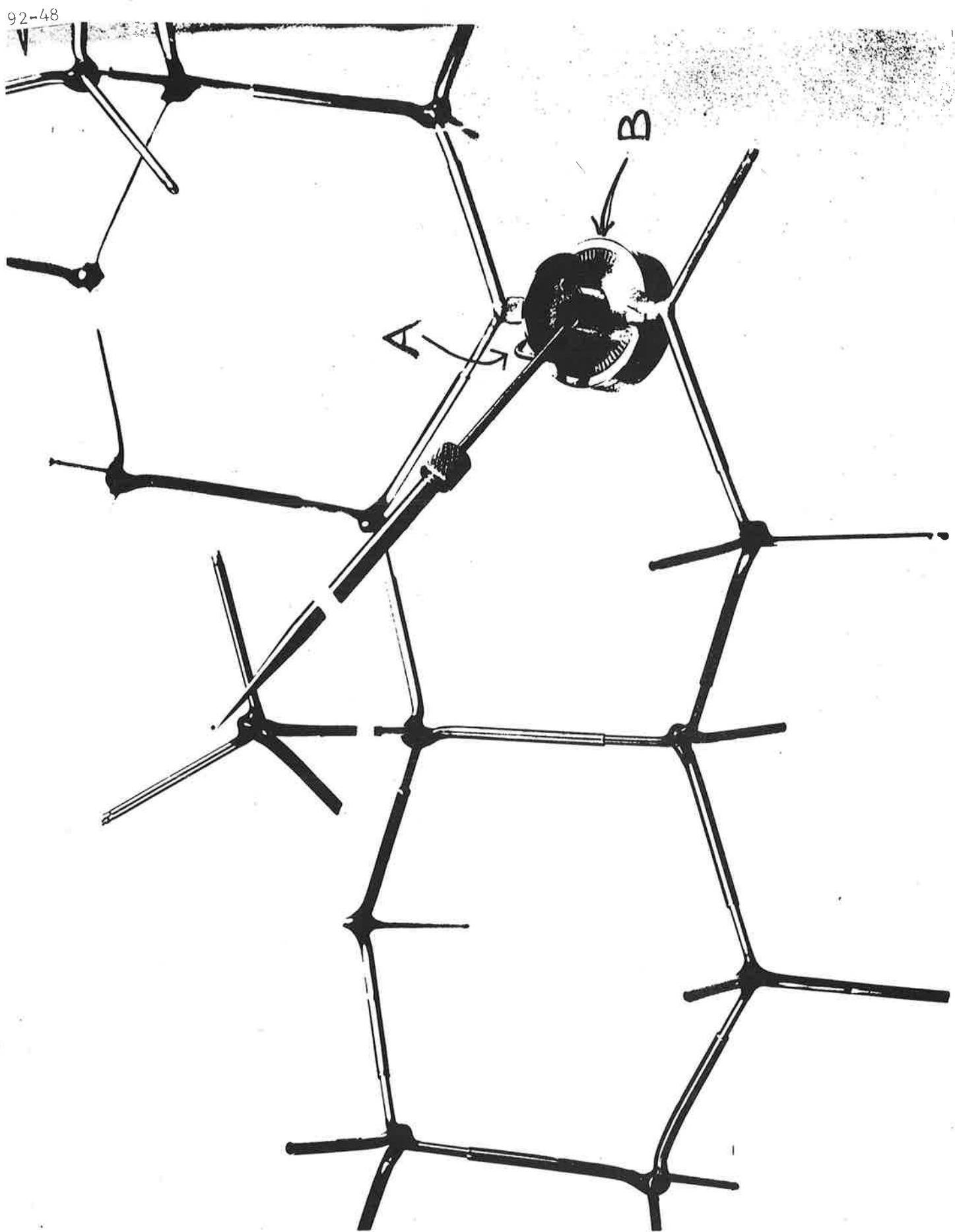
Yours sincerely,



J. W. ApSimon,  
Assistant Professor.

JWA:jf  
Encl.





# Battelle Memorial Institute • COLUMBUS LABORATORIES

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

May 13, 1966

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

Dear Barry:

### Algae Control for 12" Magnet Systems

We have found that for our system the algae growth in the closed loop of the Varian 12" magnet cooling system can be reasonably controlled by adding about 1 or 2 liters of benzyl alcohol to the closed-loop water. We made the original addition of 1 liter about 3 years ago and have added two, one-half liter portions since. We have not found it necessary to change the filter on the back of our 12" magnet since the first benzyl alcohol was added. We still check the filter periodically though just to be on the safe side. The current benzyl alcohol concentration in our system (by NMR of course) is 0.125 mol percent.

### <sup>13</sup>C rf Unit Test Sample

A convenient sample for checking out the rf unit and probe alignment of a <sup>13</sup>C system is an aqueous solution of KMnO<sub>4</sub>. <sup>55</sup>Mn is 100% abundant and comes into resonance only 200 gauss (~300 fine current divisions) above <sup>13</sup>C at 15.085 Mc/sec. Even though the signal is broad--probably due to the spin of 5/2--the KMnO<sub>4</sub> resonance does not saturate readily and therefore gives a strong signal which is easily detected. We have tried a few other manganese compounds also, but with less success.

### Highlights of High Resolution NMR at Battelle

Currently we are looking at the <sup>13</sup>C resonances of polar substituted phenols and are beginning our second year of work on the total analysis of the proton spectra of monosubstituted benzenes.

Best personal regards.

Sincerely,

*Tom*

Thomas F. Page, Jr.  
 Research Spectroscopist  
 Molecular Spectroscopy

TFPjr:KR

# The Goodyear Tire & Rubber Company

Akron, Ohio 44316

RESEARCH DIVISION

142 GOODYEAR BLVD.

May 16, 1966

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

RE: Time Average Spectrum of Polymer Solutions

Dear Dr Shapiro:

We are sorry in delaying to submit our contribution.

Recently, we have been busy in devising a quantitative means to measure small amount of minor micro structure unit in polymers by using time average technique. Dr Field and I have constructed a mechanical "CAT" based on the principle suggested by Crutchfield<sup>(1)</sup>. The timing unit incorporated with the Varian A-60 spectrometer is similar to the one described by Mayo and Goldstein<sup>(2)</sup> and by A W Douglas<sup>(3)</sup>. Besides the necessary hardware for electrical connections, only a multiple cam timer made by Industrial Timer Corp, Pansippany, N J, was used for programming the time sequences of the control switches in the spectrometer. The schematic of connection to the A-60 is given in Figure 1, and a portion of typical time averaged spectrum is shown in Figure 2. We found the technique works well for polymer solutions for the slight loss of resolution due to the discrete intensity has no serious consequence.

In comparing the areas between an intense and a weak peak in quantitative measurements, difficulties sometime arise on keeping the intense peak within the chart paper's range and at the same time to have a measurable intensity on the weak peak. We found that by delaying the pen down switch (1) by a few percent of the integration time, a scaled down profile of the integration lines appeared under the intense lines as shown in Figure 2. The exact scaling factor can readily be found from the spectrum. This modification enables one to compare the areas of a weak peak to an intense one even if the latter is out of recorder chart range.

Sincerely,

*Hung Yu Chen*  
Spectroscopy Section

RESEARCH DIVISION

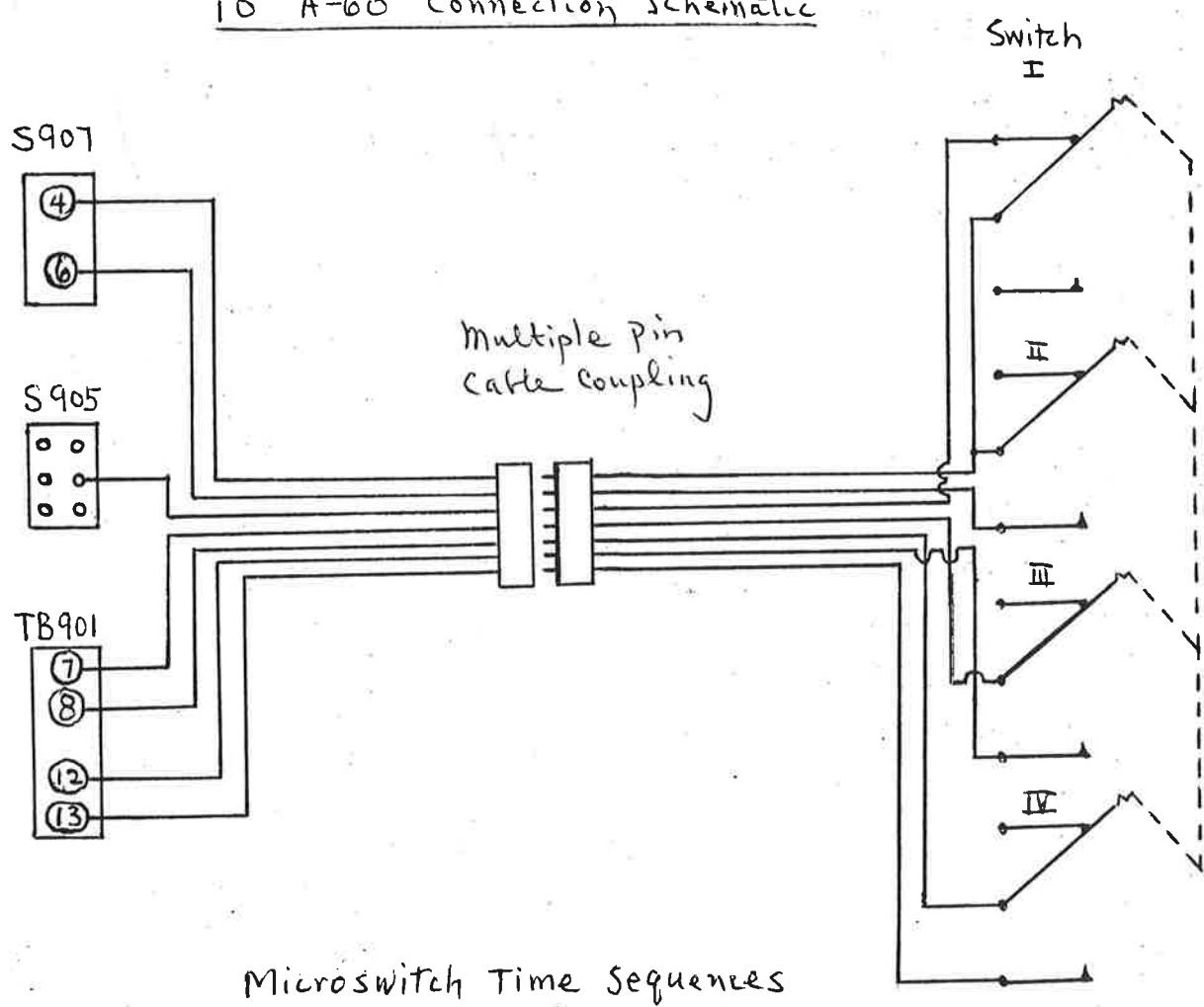
Hung Yu Chen  
bj 5/16/66

(1) M M Crutchfield: 4th OCEAN Conference, Pittsburgh, Pa, Feb 2, 1963.

(2) R E Mayo & J H Goldstein: Rev Sci Inst 35 1231 (1964)

(3) A W Douglas IITNMR 78-17

FIG (I)

TO A-60 Connection schematic

Microswitch Time Sequences

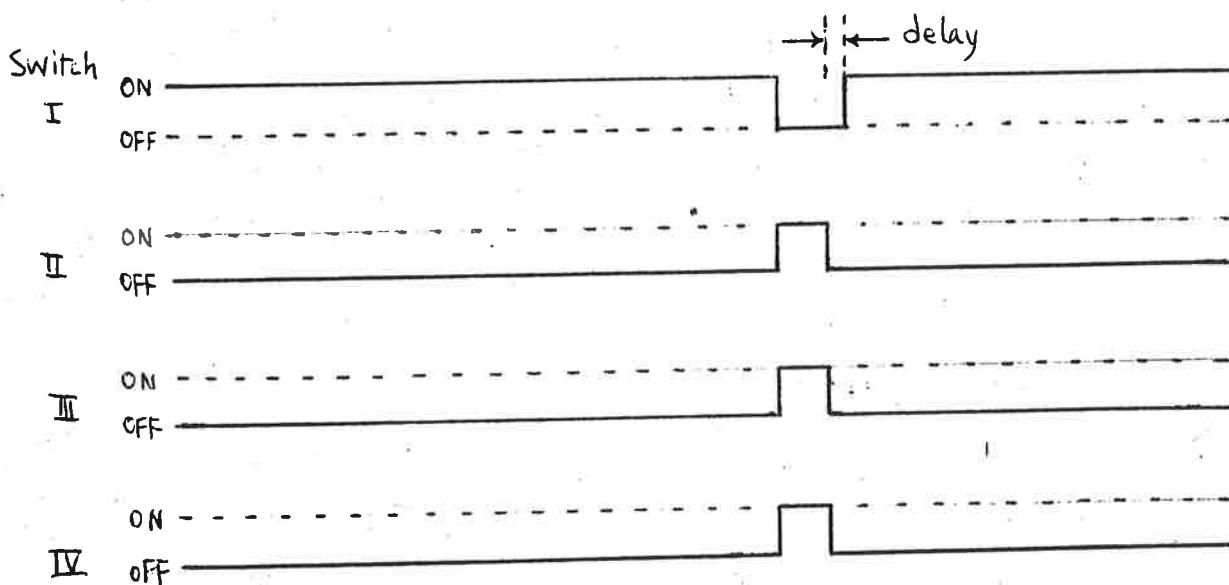
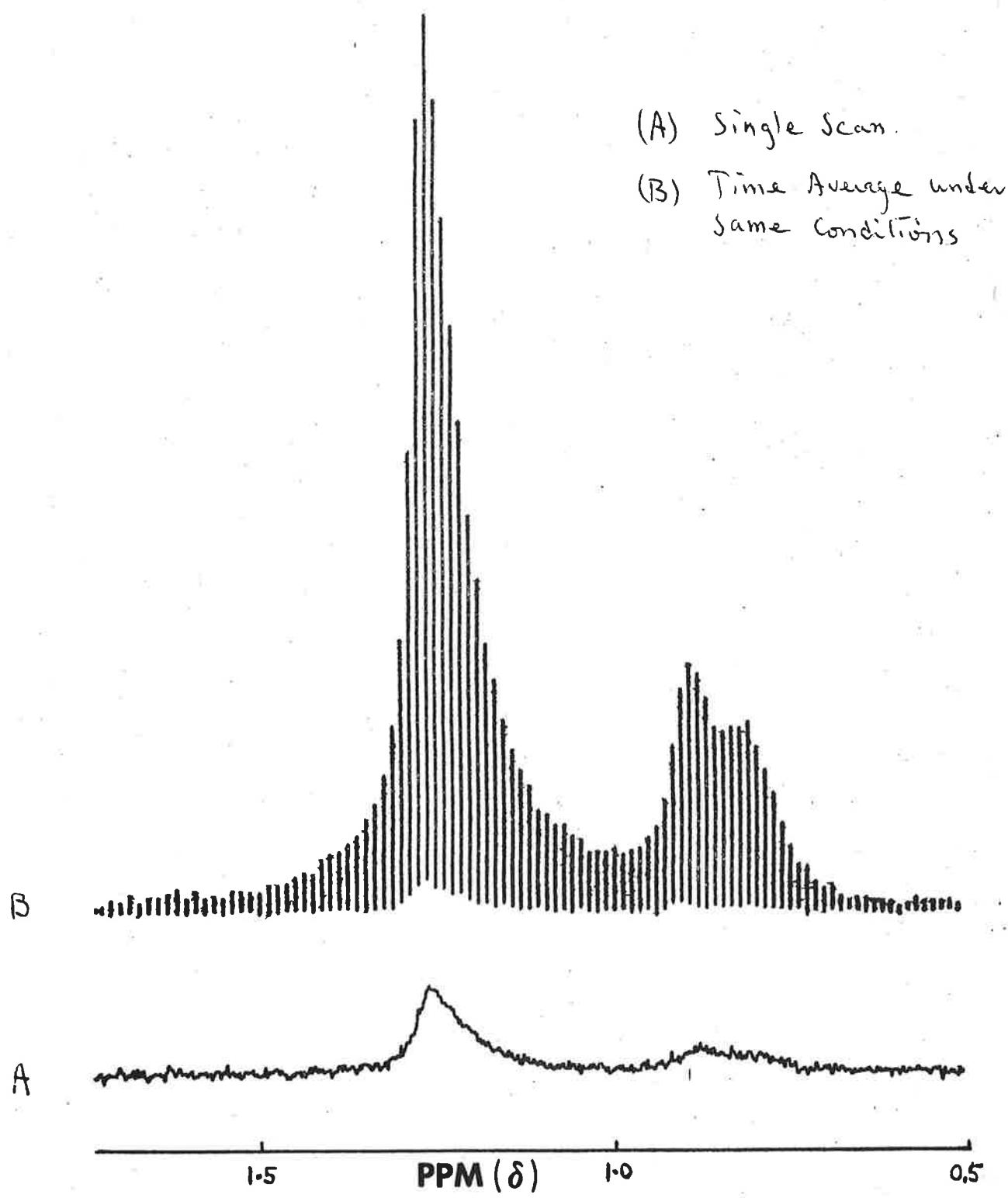


FIG 2 Time Average Spectrum of high M.W. EPR



## STEVENS INSTITUTE OF TECHNOLOGY

HOBOKEN, NEW JERSEY

Department of  
Chemistry and Chemical Engineering

May 16, 1966

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

Dear Professor Shapiro:

1, Magnetic Equivalence Simplification Program  
for an I.B.M. 1620 Computer

2. Proton Shifts of Substituted Ethanes

We have recently rewritten in KINGSTON 16.7 language (for the I.B.M. 1620 computer) the magnetic equivalence factoring program, NMRIT (0), developed by Ferguson and Marquardt (J. Chem. Phys., 41, 2087 (1964)). As presently compiled our program requires a 4K word memory and is limited to the  $A_2B_m$  case. The spectrum of propane was calculated in about 8 minutes. By changing the dimensions it is possible to include three or four different nuclei at the expense of the total number of nuclei. A copy of the source deck is available on request.

In the course of our work on substituent effects we have found that the methyl proton shifts of substituted ethanes  $CH_3-CX'Y'Z'$  are directly additive with respect to the substituents X', Y', and Z' of the  $\beta$  carbon.

Professor B. L. Shapiro  
 Illinois Institute of Technology  
 Chicago, Illinois  
 May 16, 1966

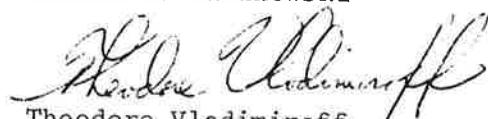
Page 2

Table I lists the parameters computed by a least-square technique and Table II shows a comparison between calculated and observed shifts. All shifts downfield from TMS in units of PPM. Data was observed on compounds in dilute C Cl<sub>4</sub> solutions. The mean deviation between observed and calculated shifts is 0.02 PPM, well within experimental error.

Sincerely yours,

*Edmund R. Malinowski*

Edmund R. Malinowski



Theodore Vladimiroff

lm

TABLE I CHEMICAL SHIFT PARAMETERS AS COMPUTED BY  
 A LEAST SQUARE METHOD FOR COMPOUNDS OF  
 THE TYPE H<sub>3</sub>C-X'Y'Z'

X'	$\eta_{X'}$	X'	$\eta_{X'}$
H	0.282		
Cl	0.900	OH	0.591
CH <sub>3</sub>	0.321	NH <sub>2</sub>	0.422
Br	1.097	C <sub>6</sub> H <sub>5</sub>	0.646
CHO	0.510	SH	0.746
CN	0.735	I	1.285

TABLE II. OBSERVED AND CALCULATED CHEMICAL SHIFT VALUES  
FOR COMPOUNDS OF  
THE TYPE  $\text{H}_3\text{C}-\text{CX}'\text{Y}'\text{Z}'$  (DIRECT ADDITIVITY IN PPM)

<u>Compound</u>	<u>Obs.</u>	<u>Calc.</u>	<u>Diff.</u>	<u>Compound</u>	<u>Obs.</u>	<u>Calc.</u>	<u>Diff.</u>
$\text{CH}_3\text{CH}_3$	0.87 <sup>a</sup>	0.85	-0.02	$\text{CH}_3\text{CH}_2\text{CHO}$	1.09 <sup>a</sup>	1.07	-0.02
$\text{CH}_3\text{CH}_2\text{Cl}$	1.48 <sup>a</sup> , 1.50 <sup>c</sup>	1.46	-0.02, -0.04	$\text{CH}_3\text{CH}(\text{CH}_3)\text{CHO}$	1.10 <sup>a</sup>	1.11	+0.01
$\text{CH}_3\text{CH}_2\text{Br}$	1.65 <sup>b</sup> , 1.67 <sup>c</sup>	1.66	+0.01, -0.01	$\text{CH}_3\text{C}(\text{CH}_3)_2\text{NH}_2$	1.08 <sup>b</sup>	1.06	-0.02
$\text{CH}_3\text{CH}_2\text{I}$	1.86 <sup>b</sup>	1.85	-0.01	$\text{CH}_3\text{CH}(\text{CH}_3)\text{NH}_2$	1.01 <sup>b</sup>	1.03	+0.02
$\text{CH}_3\text{CHCl}_2$	2.05 <sup>a</sup> , 2.24 <sup>c</sup>	2.08	+0.03-0.16	$\text{CH}_3\text{CH}_2\text{CH}_3$	0.89 <sup>a</sup>	0.88	-0.01
$\text{CH}_3\text{CHBr}_2$	2.47 <sup>a</sup>	2.48	+0.01	$\text{CH}_3\text{C}(\text{CH}_3)_2\text{Cl}$	1.58 <sup>b</sup>	1.54	-0.04
$\text{CH}_3\text{CCl}_3$	2.74 <sup>a</sup> , 2.72 <sup>b</sup> , 2.75 <sup>c</sup>	2.70	-0.04, -0.02, -0.05	$\text{CH}_3\text{CH}(\text{CH}_3)(\text{C}_6\text{H}_5)$	1.23 <sup>b</sup>	1.25	+0.02
$\text{CH}_3\text{CH}_2\text{CN}$	1.31 <sup>a</sup>	1.30	-0.01	$\text{CH}_3\text{CH}_2(\text{C}_6\text{H}_5)$	1.20 <sup>b</sup>	1.31	+0.11
$\text{CH}_3\text{CH}(\text{CH}_3)\text{CN}$	1.33 <sup>a</sup>	1.34	+0.01	$\text{CH}_3\text{C}(\text{CH}_3)_2(\text{C}_6\text{H}_5)$	1.32 <sup>b</sup>	1.29	-0.03
$\text{CH}_3\text{CH}_2\text{OH}$	1.17 <sup>b</sup>	1.16	-0.01	$\text{CH}_3\text{CH}_2\text{SH}$	1.31 <sup>b</sup>	1.31	+0.00
$\text{CH}_3\text{C}(\text{CH}_3)_2\text{OH}$	1.22 <sup>b</sup>	1.23	+0.01	$\text{CH}_3\text{C}(\text{CH}_3)_2\text{SH}$	1.39 <sup>b</sup>	1.39	+0.00
$\text{CH}_3\text{CH}(\text{CH}_3)\text{Cl}$	1.54 <sup>b</sup>	1.50	-0.04	$\text{CH}_3\text{C}(\text{CH}_3)_3$	0.94 <sup>b</sup>	0.96	+0.02
$\text{CH}_3\text{CH}(\text{CH}_3)\text{Br}$	1.71 <sup>b</sup>	1.70	-0.01	$\text{CH}_3\text{CH}(\text{CH}_3)_2$	0.89 <sup>b</sup>	0.92	+0.03
$\text{CH}_3\text{C}(\text{CH}_3)\text{Cl}_2$	2.08 <sup>a</sup>	2.12	+0.04	$\text{CH}_3\text{CH}(\text{CH}_3)\text{I}$	1.88 <sup>b</sup>	1.89	+0.01
$\text{CH}_3\text{C}(\text{CH}_3)_2\text{Br}$	1.76 <sup>b</sup>	1.74	-0.02				

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