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Monthly Ecumenical Letters from Laboratories Of N-M-R

No. 62

Mailed:
29 November 1963

DEADLINE FOR NEXT ISSUE

23 December 1963

MELLON INSTITUTE
4400 FIFTH AVENUE
PITTSBURGH 13, PA.

October 31, 1963

Dr. B.L. Shapiro
Mellon Institute
4400 Fifth Ave.
Pittsburgh 13, Pa.

Dear Barry,

In my haste to beat last month's deadline I allowed a bad formulation to remain in my letter (MELLONMR#61, p. 45). The correction equation should be written:
ϕ^*(corrected) = ϕ^*(predicted) - Σcorrection factors.
The correction factor sum is a function of each of the ortho-meta pairs adjacent to the F atom in question. I apologize to those interested people who may have become confused through my error.

Very truly yours,

John

John J. Burke

CYANAMID

AMERICAN CYANAMID COMPANY

STAMFORD RESEARCH LABORATORIES

1937 WEST MAIN STREET, STAMFORD, CONN.

FIRESIDE 8-7331

October 31, 1963

Dr. B. L. Shapiro
Mellon Institute
Pittsburgh 13, Pennsylvania

Dear Barry:

The accompanying spectra show a double resonance determination of the relative signs of J_{PCH} and J_{PCCH} in the tetraethyl phosphonium cation; similar to the procedure used for the thallium diethyl cation[1].

Spectrum A is the normal proton spectrum of a D_2O solution of $Et_4P^+I^-$ at 56.4 mc. The two triplets and two quartets of this A_2B_3X system can be identified in spite of some second-order appearance; their centers are noted as shifts (in cycles/sec) from the internal standard sodium 2,2-dimethyl-2-silopentane-5-sulfonate.

The Johnson field-sweep decoupling technique was used, with the irradiating r.f. field intensity carefully adjusted to be sufficiently selective. Spectrum B is obtained when the difference between the irradiating and observing frequencies is set approximately equal to the apparent chemical shift of the outer quartet-triplet pair; in spectrum C, the frequency difference corresponds to the apparent chemical shift of the inner quartet-triplet pair. Since the low-field quartet is perturbed when the upfield triplet is irradiated, J_{PCH} and J_{PCCH} have unlike signs.

Two recent publications[2] have summarized the magnitudes and possible absolute signs of the P-H couplings in phosphines. J_{PCCH} appears to be positive, and J_{PCH} appears to be smaller in magnitude and also positive. In triethyl phosphine, however, J_{PCH} becomes negative[3], (-0.5 cps.), possibly due to the increase of s-character in the P-C bonds as the bulkiness of the substituents is increased. Quite likely J_{PCCH} is also positive in the cation, and therefore J_{PCH} has taken on a large negative value. In view of the trend observed in the phosphines with increased substitution, this is perhaps not unexpected with the attainment of tetrahedral hybridization.

The numerical values are J_{PCH} = -13.1 cps, J_{PCCH} = +18.3 cps. $|J_{HH}|$ is $\sim$ 7.8 cps.

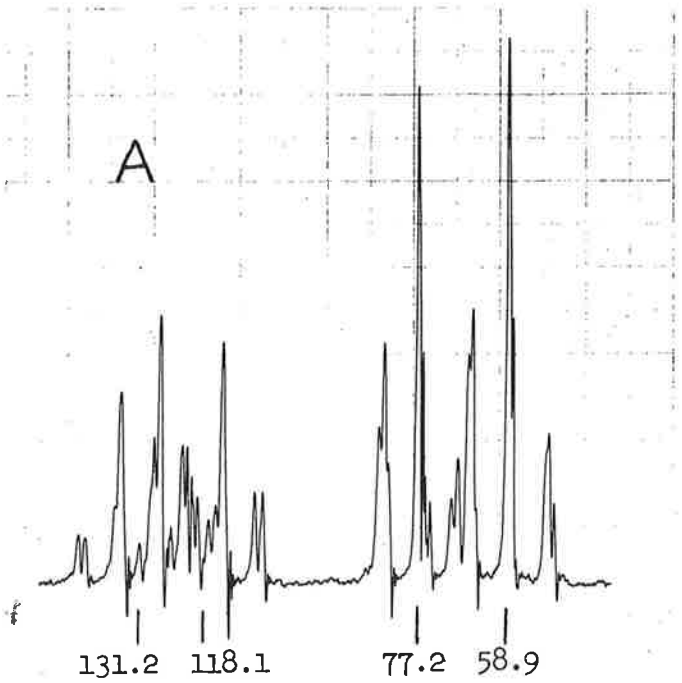
Very truly yours,

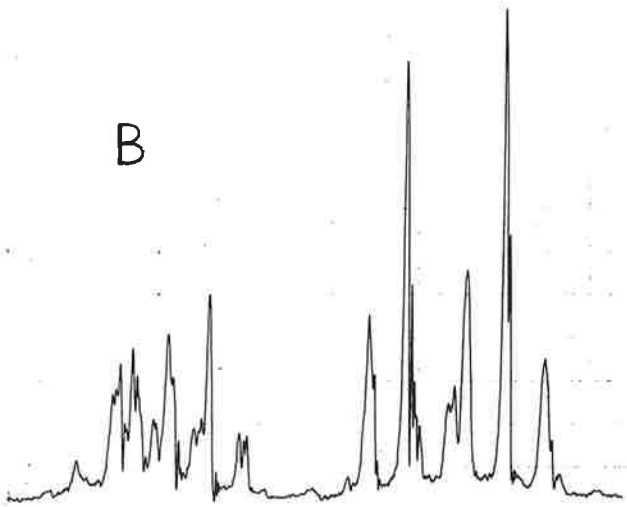
John E. Lancaster

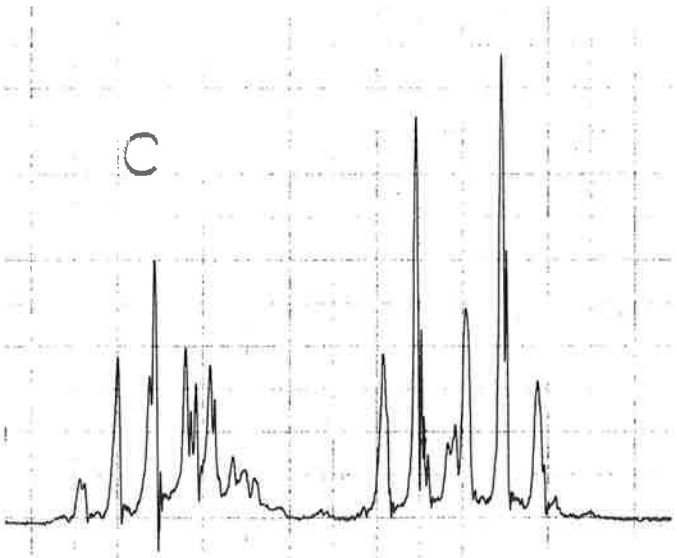
John E. Lancaster, Group Leader
Magnetic Resonance Group
Research Service Department

td
Enclosure

References
1. Maher and Evans, Proc. Chem. Soc., 208 (1961).
2. Manatt, Juvinall and Elleman; and Whitesides, Beauchamp and Roberts, J.A.C.S. 85, 2664-2666 (1963).
3. Narasimhan and Rogers, J. Chem. Phys. 34, 1049.


A
131.2
118.1
77.2
58.9


B


C

62-4

RIJKSUNIVERSITEIT-GENT

LABORATORIUM
voor
ORGANISCHE CHEMIE

Dir.: Prof. Dr. F. GOVAERT

GENT, October 30th 1963.

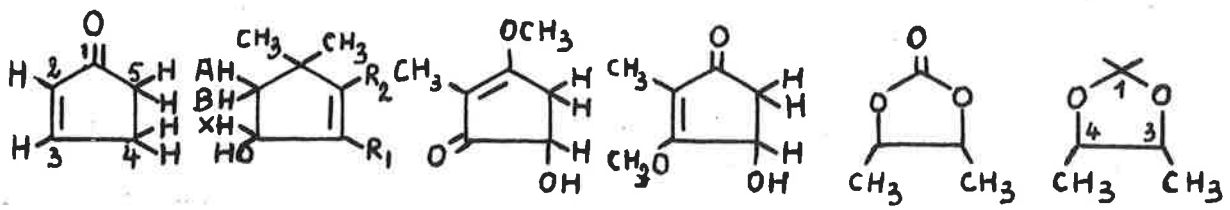
J. Plateaustraat, 22 Tel. 25.26.02

Dr. B. L. SHAPIRO,
MELLON INSTITUTE,
4400 Fifth Avenue,
PITTSBURGH 13, PA.
U. S. A.

Concerning : Cis- and trans coupling in (planar) pentacyclic compounds.

Dear Dr. Shapiro,

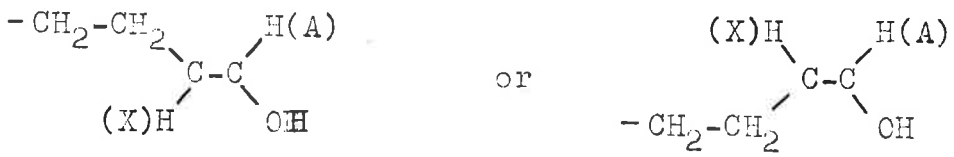
We wish to make some remarks about the J coupling in pentacyclic compounds for which Ch. De Puy and C. Lyons[1] accept J^{4-5}_{cis} = 2,5 cps and J^{4-5}_{trans} = 6,4 in cyclopentenone (I) and derivatives.



I II III IV V VI

A smaller J_{cis} and a larger J_{trans} is for example the case in the dioxolane VI where trans coupling of the hydrogens on carbon 3 and 4 can be studied in the dl isomer and reaches 8,35 cps, while cis coupling in the meso dioxolane has the J value 5,85 cps[2].

In the pentacyclic carbonates V (meso and dl forms) there is no difference in trans and cis coupling constant[2] and presumably this is because the sp2 carbonyl carbon makes the ring more planar, the dihedral angle being perhaps about 25°[3]. This effect is still more pronounced in cyclopentenone derivatives where three sp_2 carbon atoms are in the ring. Analysis of the NMR spectra of such cyclopentenone derivatives, obtained from humulone where a fixed AX pattern



.../...

is observed, show that for the two possibilities, the proton cis (H(A)) to the C-C bond has the higher shift[4]. This is consistent with calculations according to Ramsey[5]. The same effect is observed for the proton cis to an OH group. This permits correct assignment of the cis and trans protons, and it was found that the cis coupling is the higher. A second example then is compound II synthetised in our laboratory[6] where J_{cis} is circa 7,4 cps and J_{trans} only ≈ 4,6 when $R_1 = R_2 =$ Br and where J_{cis} is 7,3 cps and J_{trans} = 4,8 cps when $R_1 = R_2 =$ H.
The cis and trans hydrogen NMR signals are again recognised in these ABX patterns, because the hydrogen cis to the hydroxyl group (trans to the secondary alcohol hydrogen on C_4) is shifted upfield in comparison to the trans hydrogen. The same situation exists in the compound for which either structure III or IV can be accepted[7] : the cis proton can be recognised because of its higher shift and it has again the lower J value.

We therefore think that the assignment of De Puy mentioned at the beginning of this letter should be reversed.

---

1 C. De Puy, C.Lyons; MELLONNR 58-38 (1963).
2 F. Anet; J.Am.Chem.Soc., 84, 747 (1962).
3 C. Brown; Acta Cryst., 7, 92 (1954).
4 F. Alderweireldt, M. Anteunis, to be published.
5 N.F. Ramsey; Phys.Rev., 86, 243 (1952).
6 M. Schamp and coll., unpublished results.
7 Unpublished results; cfo. H.Orchin, L.Butz; J.Am.Chem. Soc., 65, 2296 (1943).

Sincerely yours,

Prof. Dr. F. Alderweireldt.

Dr. M. Anteunis,
Associated Prof.

62-6

THE MANCHESTER COLLEGE OF SCIENCE AND TECHNOLOGY

(Faculty of Technology in the University of Manchester.)

MANCHESTER 1 · TELEPHONE CENTRAL 3266

Department of Chemistry Dr. J. Lee

7th November, 1963.

Dr. B. L. Shapiro,
Mellon Institute,
4400, Fifth Avenue,
Pittsburgh, 13,
Pennsylvania,
U. S. A.

Dear Barry,

Erratum

My attention has been drawn to some errors of calculation in Table 1 of our recent communication to MELLONMR (no. 60, page 44). The values of a (the ratio of coupling constants $^{117}Sn/^{119}Sn$) are in error. In row order, the corrected values should be

0.950, 0.963, 0.948, 0.969, 0.963, 0.959.

In view of these corrections, I feel we ought to withdraw our remark regarding a coupling constant isotope effect (B2 on page 43).

Yours sincerely,

Joe

CABLE ADDRESS "RESEARCH"

IN YOUR REPLY PLEASE QUOTE

FILE No. .....

NATIONAL RESEARCH COUNCIL

CANADA

IVISION OF APPLIED CHEMISTRY

OTTAWA 2,

November 8th, 1963.

Dr. B. L. Shapiro,
Mellon Institute,
4400 Fifth Avenue,
Pittsburgh 13, Pa.,
U. S. A.

Dear Barry:

I have never been dunned with as pleasant a letter before. It is bound to bring prompt results. We have been studying the system CH_3OH-BF_3 in liquid sulfur dioxide as solvent. At room temperature some rather complex reactions slowly occur. Provided the samples are kept frozen, except when their spectra are obtained, there is no difficulty in studying several of the rapid equilibria. With an excess of BF_3 the two reactions which may be observed are:

$$BF_3^* + CH_3OH.BF_3 \rightleftharpoons BF_3 + CH_3OH.BF_3^*$$

$$CH_3OH.BF_3 \rightleftharpoons CH_3OBF_3^- + H^+$$

The first reaction occurs by a bimolecular displacement, as shown, rather than by dissociation and recombination of the CH_3OH-BF_3 complex. The enthalpy of activation is -5.1 ± 0.2 k.cal./mole and the Arrhenius factor is $10^{8.0}$. Chemical shifts of free and complexed BF_3 are 123.3 and 148.1 p.p.m. to high field of $CFCl_3$.

For the second reaction the lifetime between exchange processes is found to be independent of the concentration of complex. We intepret this to mean that the exchange occurs by dissociation of the complex and recombination with another proton.

.... 2

If anyone believes this conclusion to be erroneous I would appreciate comments since I have not yet convinced myself that this is correct. The lifetimes are obtained by collapse of the CH_3 and OH spin multiplets, where J_{H-CH_3} = 4.11 cycles/sec. The enthalpy of activation is -12.3 ± 0.1 k.cal./mole and the Arrhenius factor is $10^{11.6}$.

When more than one equivalent of methanol is present the following equilibrium occurs.

$$CH_3OH + CH_3OH.BF_3 \rightleftharpoons (CH_3OH)_2BF_3$$

At sufficiently low temperatures a separate methyl peak can be observed for the 1:1 complex. From the relative areas of the methyl peaks K2:1 = 4.1 and 3.5 at 208 and 218°K respectively. The temperature dependance of the chemical shift for the OH proton in CH_3OH and $CH_3OH.BF_3$ are shown in the Figures. That for $(CH_3OH)_2BF_3$ was calculated from the observed shift (an average of the rapidly exchanging species) and the equilibrium constants. The different points are for two compositions.

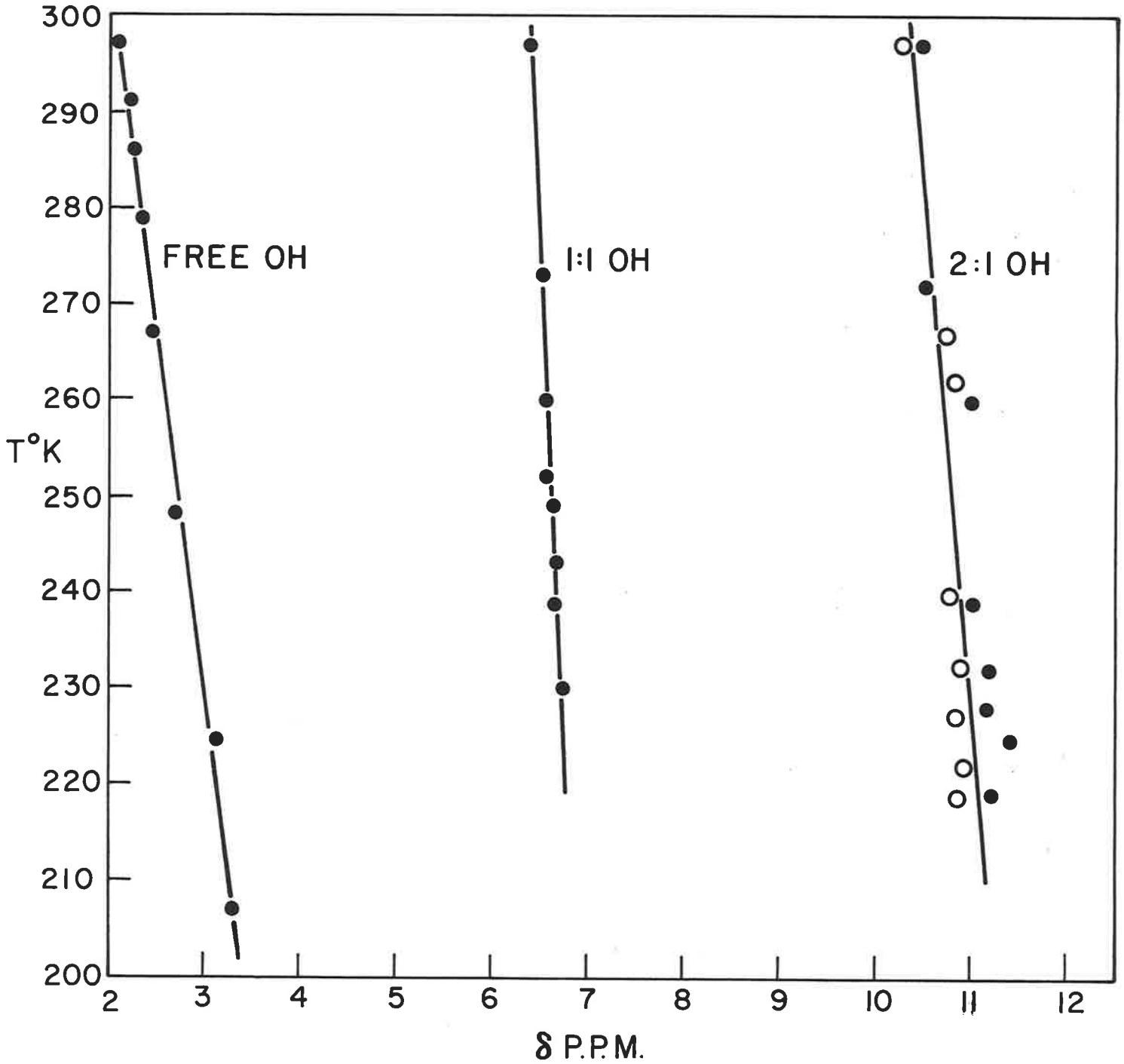
We are looking for various simple alkyl fluorides. If any readers are aware of a source of supply the information would be appreciated.

Yours truly,

Syd Brownstein

S. Brownstein

SB:cc


300
290
280
270
260
T°K
250
240
230
220
210
200
2
3
4
5
6
7
8
9
10
11
12
δ P.P.M.
FREE OH
1:1 OH
2:1 OH

62-10

THE PROCTER & GAMBLE COMPANY

MIAMI VALLEY LABORATORIES

P. O. BOX 39175
CINCINNATI 39, OHIO

November 11, 1963

Dr. Bernard L. Shapiro
Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Pennsylvania

Dear Barry:

Thank you for the subscription reminder. As my contribution I would like to describe an automatic cycling device for the Varian HR spectrometer system. In our experience this device has considerably reduced the time to achieve a homogeneous field because the cycling process is reproducible and does not depend on how fast one turns the fine current dial. The principle of the device is to shunt one of the reference resistors in the power supply. The amount of current increase is governed by the value of the variable shunting resistor. Since the time spent at increased current is also important, we have incorporated a timing device as well. In this way the optimum settings for a particular magnetic field can be measured once and used repeatedly without change.

Our cycling procedure for 14,100 gauss is as follows: First we reverse the field for a couple of minutes to erase previous cycling history. Then we find an nmr signal. Next we punch the cycling button which raises the current from 1.58 amps to 1.62 amps for one minute. One of these cycles is usually enough--however the button may be punched again if the field is not sufficiently flat.

Some readers might be interested in a source of instability in the V-4100B power supply on which we spent a lot of time because the difficulty seemingly comes from the Super Stabilizer. It appears as an oscillation of the galvanometer when the SS is locked in. One finds that the oscillation disappears when the mag reg control in the back is turned down. Then about a week later, the galvanometer oscillates again, to disappear when the mag reg is turned down. This goes on until there is practically no coupling between the SS and the magnet power supply. One immediately suspects the Super Stabilizer and, indeed cleaning of the copper-copper contacts in the pickup system restores a

measure of stability (which quickly disappears). However, we found that the symptoms vanish when the OC3 voltage reference tubes in the chopper amplifier of the V-4100B power supply are replaced. The old tubes test OK on a checker; evidently their condition is extremely critical for proper operation.

Best wishes.

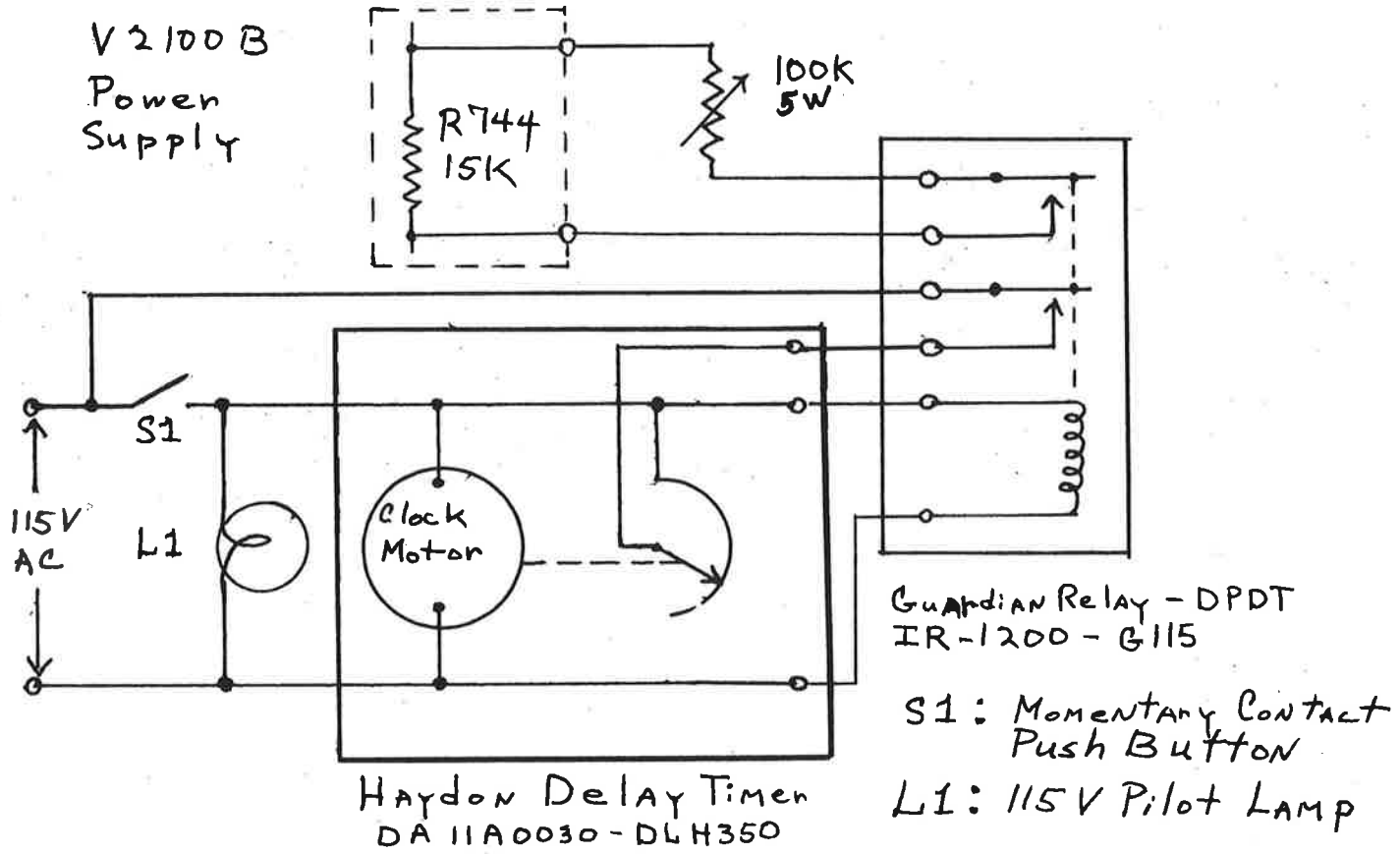
Very truly yours,

THE PROCTER & GAMBLE COMPANY
Research & Development Department

Tom

T. J. Flautt
Research Division

cw


V2100B
Power
Supply
R744
15K
100K
5W
S1
115V
AC
L1
Clock
Motor
Guardian Relay - DPDT
IR-1200 - G115
S1: Momentary Contact
Push Button
L1: 115V Pilot Lamp
Haydon Delay Timer
DA 11A0030 - DLH350

MELLON INSTITUTE
4400 FIFTH AVENUE
PITTSBURGH 13, PA.

12 November 1963

N.M.R. Studies of Compounds Related to Tetramethylbiphosphine

Dr. B. L. Shapiro
Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Pa.

Dear Barry:

Thank you for the reminder that my subscription to MELLONMR is due. I am grateful because I have been intending to send the details of the following work somewhere for about a year now. Along with Dr. R. G. Hayter, who is now with the Shell Development Company in Emeryville, California, I have been examining the proton N.M.R. spectra of tetramethylbiphosphine and related compounds. The first one we looked at, $Me_2P\,P\,Me_2$, gave an interesting $X_6AA'X_6'$ spectrum, from which it was possible to obtain by direct measurement (using equations related to those of Anet[1] and of Bothner-By and Naar-Colin[2]) all the nuclear magnetic parameters including some of the relative signs of the coupling constants. We could not, however, distinguish between the long- and short-range (H,P) coupling constants. Tetramethylbiphosphine itself gives a deceptively simple spectrum[3], so only limited information could be obtained. The monosulphide, $Me_2P\,P\,Me_2$ (S) gave a number of values for (H,P) coupling constants, but this time it was not possible to distinguish between $^2J_{HP}V$ and $^3J_{HP}III$ nor between $^2J_{HP}III$ and $^3J_{HP}V$. The width of lines in the ^{31}P resonance provide a clue to the assignments, but double resonance experiments in the field-sweep mode recently carried out for us by Dr. J. Stothers at the University of Western Ontario have given spectra that are not as simple to interpret as we had hoped. Relative signs of the coupling constants are at the moment still unknown. Our results on these and related compounds (mostly in $CDCl_3$ solution) are summarized in the table. Comparisons may be made with other results in the literature. The most interesting point is perhaps the variation in J_{PP} from tetramethylbiphosphine, where it presumably has a value rather similar to that in diphosphine itself,[4]

to the disulphide. Incidentally, it is worth noting that resonances of methyl protons attached to arsenic have linewidths greater than expected presumably due to incomplete decoupling of the (As,H) coupling via the arsenic quadrupole moment. We hope this work will not take another year to publish in a 'regular' journal.

With best wishes,

Robin

R. K. Harris

References

1. Anet, J.A.C.S. 84, 747 (1962)
2. Bothner-By and Naar-Colin, J.A.C.S. 84, 743 (1962).
3. Abraham and Bernstein, Can. J. Chem. 39, 216 (1961).
4. Lynden-Bell, Trans. Far. Soc., 57, 888 (1961).
5. Whitesides, Beauchamp and Roberts, J.A.C.S. 85, 2665 (1963).
6. Manatt, Juvinall and Elleman, J.A.C.S. 85, 2664 (1963).

COMPOUND	$^2J_{HP^{III}}$	$^3J_{HP^{III}}$	$^2J_{HP^{V}}$	$^3J_{HP^{V}}$	$^1J_{PP}$	$\tau(MeP^{III})$	$\tau(MeP^{V})$	$\tau(MeAs^{III})$
$Me_2P.PMe_2$	$\lvert {}^2J_{HP^{III}} + {}^3J_{HP^{III}} \rvert = 14.1$				$\approx 108^{\dagger}$	8.94		
$Me_2P(=S).PMe_2$*	4.09^a	5.84^b	12.02^b	17.61^a	≈ 220	8.79	8.24	
$Me_2P(=S).P(=S)Me_2$*			$\pm 12.74^c$	$\mp 7.26^c$	18.7		8.04	
$Me_2P(=S).AsMe_2$			11.83	15.55			8.19	8.81
$Me_2As.AsMe_2$								8.89
Me_3PS			13.10				8.22	
Me_3P [5]	2.7					9.06		
Me_2PCl	8.31					8.35		
Et_3P [6]	∓ 0.5	± 13.7						

* Assignments uncertain — the two coupling constants marked with a superscript 'a' may be interchanged, as may those marked 'b' and those marked 'c'.

† Value in P_2H_4, from reference 4.

FAIRCHILD
SPACE AND DEFENSE SYSTEMS
A DIVISION OF FAIRCHILD CAMERA AND INSTRUMENT CORPORATION
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November 13, 1963

Dr. Bernard L. Shapiro
Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Pennsylvania

Dear Barry:

We have been examining the spectra of some cyanine dyes dissolved in 50% $CDCl_3$ - 50% $(CF_2Cl)_2CO$ as the solvent. Unfortunately, at the dye concentrations necessary to obtain a reasonable N. M. R. signal, the dyes have already started to form micelles. The data obtained to the present are summarized in terms of chemical shifts from tetramethylsilane taken as zero. The shift of the CH_3- and CH_2- groups attached to the partially quaternized nitrogen appear to be a function of the substituents in the heterocyclic ring. The methine (-CH=) peak appears superimposed on the aromatic (=CH-) peaks. The aromatic (-C=) shift just indicates the approximate center of the aromatic peaks. We have now prepared pure samples of the parent quaternary heterocyclic salts and hope to obtain additional data on the effect of substituents in the heterocyclic ring on the shifts of the alkyl groups attached to the quaternary nitrogen and also on the degree of quaternization of the nitrogen. The data are summarized in the enclosed table. I hope this will renew my subscription.

Sincerely yours,

Jim

James E. LuValle
Director of Basic Research

JL:jc

CHEMICAL SHIFT

	CH_3 of 3-Et	CH_2 of 3-Et	CH (Aliph)	CH_2 of Ring	CH (Arom)	(9-Me)	CH_3 of 11-Et	CH_3 of toluene sulfonate	CH_2 of 11-Et
[I] Monomethine Cyanine Iodide	1.56	4.45	7.63		7.60±0.05				
[I] Trimethine Cyanine Iodide	1.50	4.18	7.45		7.45				
[I] -9-Methyl-Trimethine Cyanine Iodide	1.59	4.40	7.55		7.55	2.64			
[I] Pentamethine Cyanine Iodide	1.52	4.25	7.45		7.45				
[I]-10-Bromo Pentamethine Cyanine Iodide	1.54	4.29	7.52		7.52				
[I] Heptamethine Cyanine Iodide	1.49	?	7.49		7.49				
[II] Pentamethine Cyanine Iodide	1.50	4.26	7.48		7.48				
[III]-9-Methyl Trimethine Cyanine Iodide	1.53	4.15	7.43		7.43	2.67			
[III] Heptamethine Cyanine Iodide	1.51	3.59	7.45		7.45				
[IV]1-Ethyl Trimethine Cyanine Chloride	1.47	4.47	7.80		7.20		2.87		4.47?
[V]-1-Methyl Trimethine Cyanine Chloride		3-Me 4.42	7.84		7.84		11-Me 2.66		
[V]-1-Phenyl Trimethine p-Toluene Sulfonate		3-Me 4.38	7.70		7.70			2.34	
[IV] Pentamethine Cyanine Iodide	1.81	4.65	7.72		7.72				
[VI] Pentamethine Cyanine Iodide	1.57	4.16	7.79		7.79				
[VII] Pentamethine Cyanine Iodide	1.37	4.20	7.38		7.38				
[VIII] Heptamethine Cyanine Iodide	1.32	3.95(4.86,	7.28?)	3.39					
[VIII] Pentamethine Cyanine Iodide	1.29	4.00	4.87 7.28? 2.24?	3.50					
[VIII] Trimethine Cyanine Iodide	1.29	4.02	7.53? 2.25?	3.47					

[I] [2-Bis (3-Ethylbenzothiazolyl)]
[II] [2-Bis (3-Ethylbenzoselenazolyl)]
[III][2-Bis (3-Ethylbenzoxazolyl)]
[IV][2-Bis (3-Ethyl-β-Naphthothiazolyl)]
[V] [2-Bis (3-Methyl-β-Naphthothiazolyl)]
[VI] [2-Bis (3-Ethyl-α-Naphthoxazolyl)]
[VII][2-Bis (3-Ethyl-4, 5-Diphenyl Thiazolyl)]
[VIII] [2-Bis (3-Ethyl Thiazolinyl)]

מכון ויצמן למדע
THE WEIZMANN INSTITUTE OF SCIENCE

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P.O.B. 26 • PHONE 951721-7

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ISOTOPE DEPARTMENT

מחלקת האיזוטופים

Ref. No. 8614

November 11, 1963

Dr. B. L. Shapiro
Mellon Institute
4400 Fifth Ave.
Pittsburgh 13, Pa.

Dear Barry:

We have just looked at the O^{17} NMR spectrum of xenon oxide tetrafluoride ($XeOF_4$) and found a remarkably large spin-spin coupling constant between O^{17} and Xe^{129}. Xenon oxyfluoride-O^{17} labelled with 11.5 atom % was prepared by Dr. Jacob Shamir of the Hebrew University by the reaction of XeF_6 with the stoichiometric amount of O^{17} enriched water. The derivative of the absorption mode of the liquid mixture of oxyfluoride and HF was measured in a Kel-F tube using a DP 60 Varian NMR spectrometer operating at 8.13 Mc. The spectrum appeared as a strong central peak with two symmetrically disposed satellites (as is seen in the enclosed figure). A similar picture was found by Brown et al (J. Chem. Phys. 18, 3029, 1963) in the F^{19} NMR spectrum of $XeOF_4$. The O^{17} chemical shift using water as an external reference was - 313.5 ± 1 ppm.

The spacing between the two satellites is 692 ± 10 c.p.s. Evidence that these satellites are due to spin-spin coupling with Xe^{129} (spin 1/2) is provided by the fact that the ratio of the area of the main peak to one of the satellites is 5.6 (determined by weighing the paper under the peak). The calculated ratio from the abundance of Xe^{129} (26.24 %) is 5.62.

The other stable isotopes of xenon have spin zero except for Xe^{131} with a spin of 3/2. Apparently the coupling in this case is much weaker.

Smith, who first reported (Science 140, 899) (1963) the synthesis and infra-red spectral properties of xenon oxyfluoride has suggested that the oxygen is double bonded to xenon. The large spin-spin coupling observed lends support to the suggestion that xenon d-orbitals are involved in this bond.

... 2

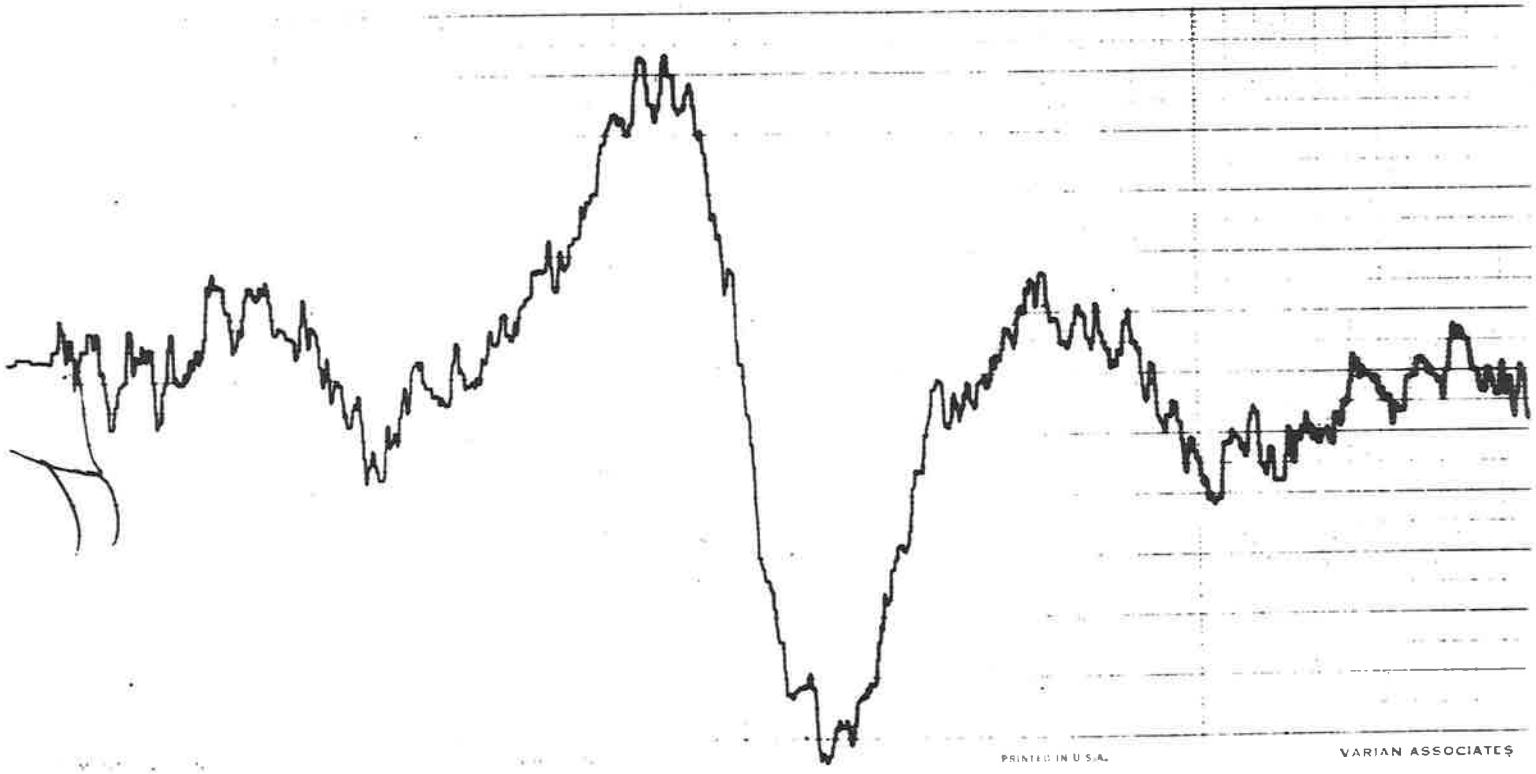
It is interesting to note that Xe^{129} was included amongst the 13 nuclei with spin 1/2 and natural abundance greater than 20% listed by Christ and Diehl recently (Helv. Phys. Acta 36, 170 (1963) which should exhibit spin-spin coupling when bound to O^{17}. So far coupling has been observed with H, P^{31} and Xe^{129}.

Yours sincerely,

J. Reuben

David Samuel

DS:lg
encl.



62-19

62-20 LABORATOIRE DE SPECTROSCOPIE HERTZIENNE
ANNEXE DU
LABORATOIRE DE RECHERCHES PHYSIQUES A LA SORBONNE
1, RUE VICTOR COUSIN, PARIS V°

PARIS, LE 12 novembre 1963
ODÉon : 24-13 POSTE 263

1322/161/RF/MNF

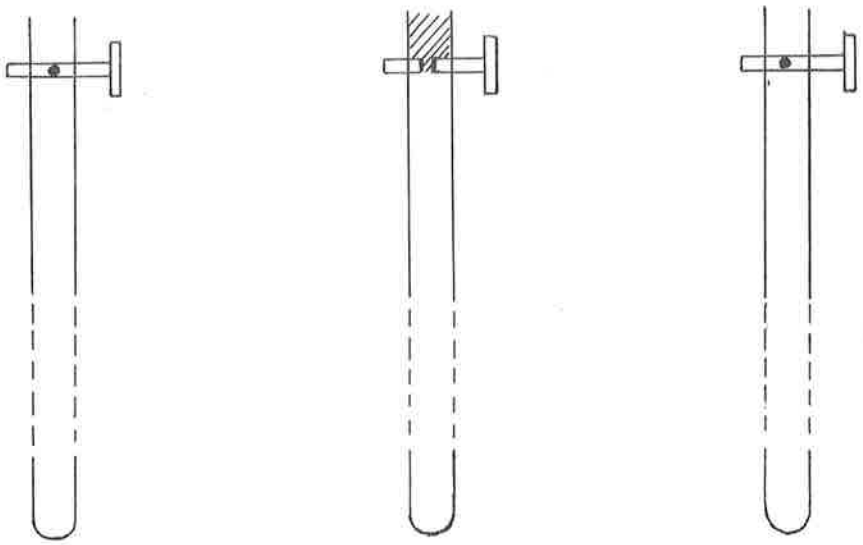
Dr. B. L. SHAPIRO
Mellon Institute
4400 Fifth Avenue
PITTSBURGH 13, Pa.
U. S. A.

Dear Dr. Shapiro,

I am sending you, herewith, some results achieved by Messrs Dayan, Jouve and Widenlocher at my laboratory. This concerns developments in the same field as the paper previously published in MELLONMR concerning N. M. R. study of gases with a Varian A 60 spectrometer : ''compressed gases under fairly high pressures'' (1).

Due to the apparatus described below, Messrs Dayan, Jouve and Widenlocher enlarged the method to gases and vapors under low pressure. These techniques allow therefore to compare the compounds as liquid pure (or in dilute solution) to the same compounds as vapor, at room temperature. In both cases, tetramethylsilane (T. M. S.) was used as internal reference : either liquid with liquid compounds or vapor with vapor compounds.

Apparatus used : The sample is a common tube supplied by Varian Associates, capped with a pyrex micro-tap (or valve). The following schemas explain the process to obtain both vapors of T. M. S. and compound.



vacuum in the sample about 7mm Hg

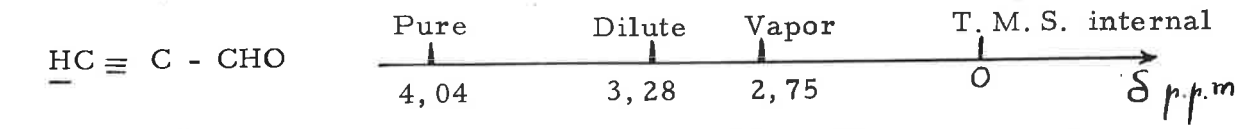
compound to study and T. M. S. (5 %) vaporized at room temperature

compound and T. M. S. as vapors

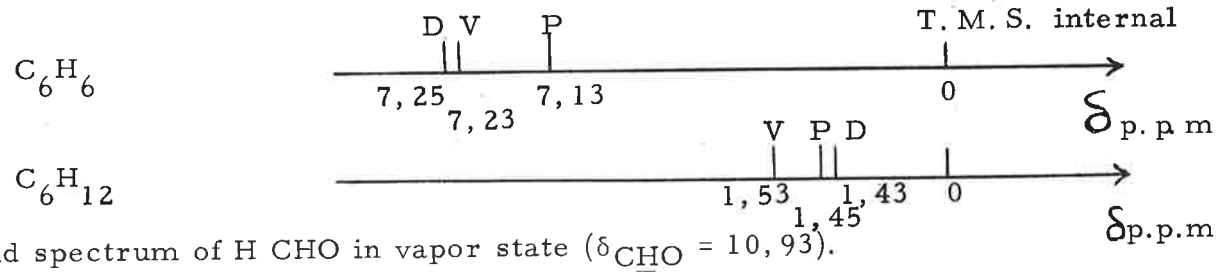
The vapor pressure of the compound must be at least of 100 mm Hg at room temperature, in order to detect the resonance signal with a Varian A 60.

The magnetic susceptibility of vapor T. M. S. being not well known but liquid T. M. S. being corrected for bulk diamagnetic susceptibility, we suppose, at first sight, that positions of T. M. S. liquid and vapor are identical (2). Work is continuing to determine exactly chemical shift between T. M. S. liquid and vapor.

Results : In most cases, the transition from liquid state to vapor state corresponds to a shift towards increasing magnetic field, as below :



Nevertheless, we point out some interesting compounds as :



and spectrum of H CHO in vapor state ($\delta_{\underline{CH}O}$ = 10, 93).

The coupling between NH and $\underline{CH_3}$ in $NH(CH_3)_2$ was observed in vapor state and measured : J = 4, 5 Hz.

Sincerely yours,

R. FREYMANN

(1) R. Freymann - MELLONMR n°58
(2) P. Jouve, G. Widenlocher, E. Dayan - C.R. Ac. Sc. France 257, 1079(1963)

62-22

UNIVERSITY OF CALIFORNIA

LAWRENCE RADIATION LABORATORY
BERKELEY 4, CALIFORNIA

Inorganic Materials Research Division
Latimer Hall

November 12, 1963

Dr. B. L. Shapiro
Mellon Institute
4400 Fifth Avenue
Pittsburg, Penn.

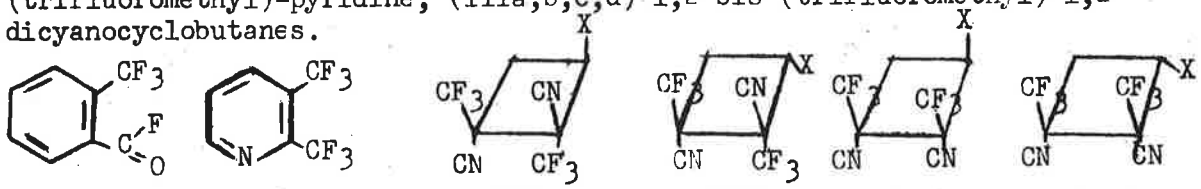
Dear Barry:

Fluorine-Fluorine Coupling Constants

We believe we have unravelled the subtle nature of fluorine-fluorine coupling constants. We explain the pattern of these coupling constants on the basis of two spin coupling mechanisms: the "through-bond" and the "through-space" mechanisms. In the former, the interaction proceeds via the electronic structure in the intervening bonds. This is the primary mechanism for proton-proton interactions. It is highly dependent on the electron-withdrawing power of the substituents on the carbon skeleton. This contribution to the coupling constant goes to zero when the sum ofthe electronegativities of the substituents becomes sufficiently high. The latter mechanism is operative when two fluorine atoms are sufficiently close in space so that there is appreciable overlap of their electronic clouds. This "through-space" interaction proceeds via an electronic structure where there is no bond per se.

Geminal[1] and vicinal fluorine coupling constants vary widely in magnitude and are dependent on the electronegativity of the other substituents attached to the carbon skeleton. This suggests that highly electronegative substituents withdraw the excited triplet or π-state electrons, which are responsible for the transmission of nuclear spin information, from the bonds between the interacting nuclei, thereby reducing the coupling constants. Indeed, for a series of halogen substituted ethanes the observed coupling constants are directly proportional to the sum of the Pauling electronegativities of the substituents. The vicinal fluorine coupling constants in halogen substituted propanes also bear the same relationship if an electronegativity of 3 is assigned to groups like CF_3, CF_2Cl, CF_2Br, and CF_2H. It is to be noted that on the basis of this argument, vicinal fluorine atoms attached to nearly perfluorinated carbon skeletons would be expected to show a zero coupling constant, as is observed in perfluoroethyl groups. This would also predict a zero coupling constant through the bonds between fluorine atoms separated by more than three bonds.

The "through-space" mechanism was postulated by Petrakis and Sederholm to come about as a result of the overlap of the electronic clouds of the fluorine atoms whenever the geometry of the molecules offers such opportunity[2]. To test this we have studied the NMR spectra of several compounds: (I) o-trifluoromethyl benzoyl fluoride, (II) 2,3-bis-(trifluoromethyl)-pyridine, (IIIa,b,c,d) 1,2-bis-(trifluoromethyl)-1,2-dicyanocyclobutanes.



I II IIIa IIIb IIIc IIId

X = OCH_2CH_3, $SC(CH_3)_3$, p-CH_3OCoH_4

Compounds I and II show coupling constants of 10.9 and 12.8 cps, respectively. These values may be compared with those in trans-2-chloroheptafluorobutene-2[3], in which the coupling constant between the two CF_3 groups is 1.3 cps. We regard this magnitude as normal for coupling through four saturated and one olefinic bonds. Whereas in this butene compound there can be no direct overlap of the electronic clouds of the fluorine atoms of the trans-oriented CF_3 groups, the geometry of molecules I and II allows such overlap due to the proximity of the fluorine atoms. The large coupling constants in I and II must be due almost entirely to through-space coupling. The fluourine NMR spectra of compounds IIIa,b,c,d have been reported[4]: IIIa and IIIb exhibit "singlet resonances" for the trans-oriented CF_3 groups whereas IIIc and IIId show substantial coupling between the cis-oriented CF_3 groups. One of the authors, S. Proskow[5] has provided us with the coupling constants of IIIc and IIId, differently substituted in X. They range from 10 to 12 cps. No doubt the cis-oriented CF_3 groups in IIIc and IIId are sufficiently close together to engage in overlap of the electronic clouds, thereby effecting through-space coupling. As the fluorine atoms are separated by five saturated bonds, it is not expected that through-bond coupling is operative. It is obvious that the compounds considered here fully justify the through-space coupling mechanism.

This work in greater detail is being submitted to the Journal of Chemical Physics for publication. Preprints are available upon request. } accep. 7.6.64

Sincerely,

C. H. Sederholm

C. H. Sederholm

Soon Ng

Soon Ng

CHS/SN:ch

References:

(1) J. Dyer, Proc. Chem. Soc., 275, (1963)
(2) L. Petrakis and C. H. Sederholm, J. Chem. Phys., 35, 1243 (1961)
(3) H. M. Beisner, L. C. Brown, and D. Williams, J. Mol. Spectr. 7, 385 (1961)
(4) S. Proskow, H. E. Simmons, T. L. Cairns, J. Am. Soc. 85, 2341 (1963)
(5) S. Proskow, Private Communication

62-24

BP RESEARCH CENTRE

SUNBURY-ON-THAMES

MIDDLESEX

ENGLAND

STRUCTURE OF DIPROPYLENE GLYCOL ISOMERS

We would like to describe the elucidation of the structure of three isomeric dipropylene glycols (DPG) as an example of a very simple technique which we have used in our laboratories on several occasions for distinguishing between isomers. The three isomeric dipropylene glycols, I, II and III (R = H) are formed by the 'tail to tail', 'head to head' and 'head to tail' coupling of two propylene oxide molecules during hydrolysis. The NMR spectra of the three isomeric diols (Figure 1) cannot be interpreted unambiguously because of the small chemical shift between the CH_2 and CH resonances. Now if chemical groups possessing large magnetic anisotropies are attached to the molecules, through the OH groups, then the chemical shifts between the CH_2 and CH resonances are increased. This enables the structure of the derivative, and hence the original glycol, to be deduced unambiguously from the NMR spectrum. In this particular case p-$NO_2C_6H_4CO$ O- groups are easily attached to the molecule by condensation of the respective acid chlorides with the OH groups, they have the required large magnetic anisotropy and give no bands in the 4 to 9τ region. The prepared nitrobenzoates of isomers A, B and C were examined in $CDCl_3$ solution and these derivatives gave spectra (Figures 2, 3 and 4 respectively) that could be easily and unambiguously interpreted.

In Figure 2 the multiplets at 6.28τ and 4.69τ characterize the CH_2 and CH groups respectively, the latter because of their low field position must be vicinal to the nitrobenzoate residue. Thus this isomer of DPG must have the structure I. (R = p-$NO_2C_6H_4CO$-).

In Figure 3 the CH multiplet (5.98τ) now occurs to high field of the CH_2 doublet (5.63τ) thus it must be the latter which is vicinal to the nitrobenzoate residue. Hence this isomer of DPG has the structure II (R = p-$NO_2C_6H_4CO$-).

In Figure 4 the two sets of doublets and multiplets indicate that all the CH and CH_2 groups in the molecule are in different environments. The Figure 4 must be the spectrum of the 'mixed' isomer (III, R = p-$NO_2C_6H_4CO$-) of DPG. In all the three above cases assignments were confirmed by integrated spectra.

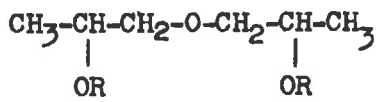
Thus the spectra of isomers A, B and C (Figure 1) are those of the structures I, II and III (R = H) respectively.

It may be noted that the resonance of CH_2 groups vicinal to the nitrobenzoate residue is not shifted to low field to the same extent as the resonance of the CH groups in the same invironment. Also the -C<u>H</u>-O- multiplet of isomer II occurs at a lower field (5.98τ) strength than the corresponding multiplet (6.07τ) for the hydrogen atom in the 'mixed' isomer III. These differences are probably due to the different preferred conformations that these derivatives of DPG isomers take up in solution.

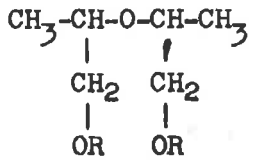
The glycols and their derivatives were prepared by Mr C. Simpson.

S.A. Knight

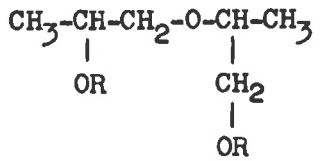
R.L. Erskine



I



II



III

Pages 1 - 2
Figures 1 - 4 (Serial Nos. 63/1637 - 1640 incl)
CMW

SERIAL No. 63/1637/BP

SPECTRA OF DPG ISOMERS
80 Mc/sec

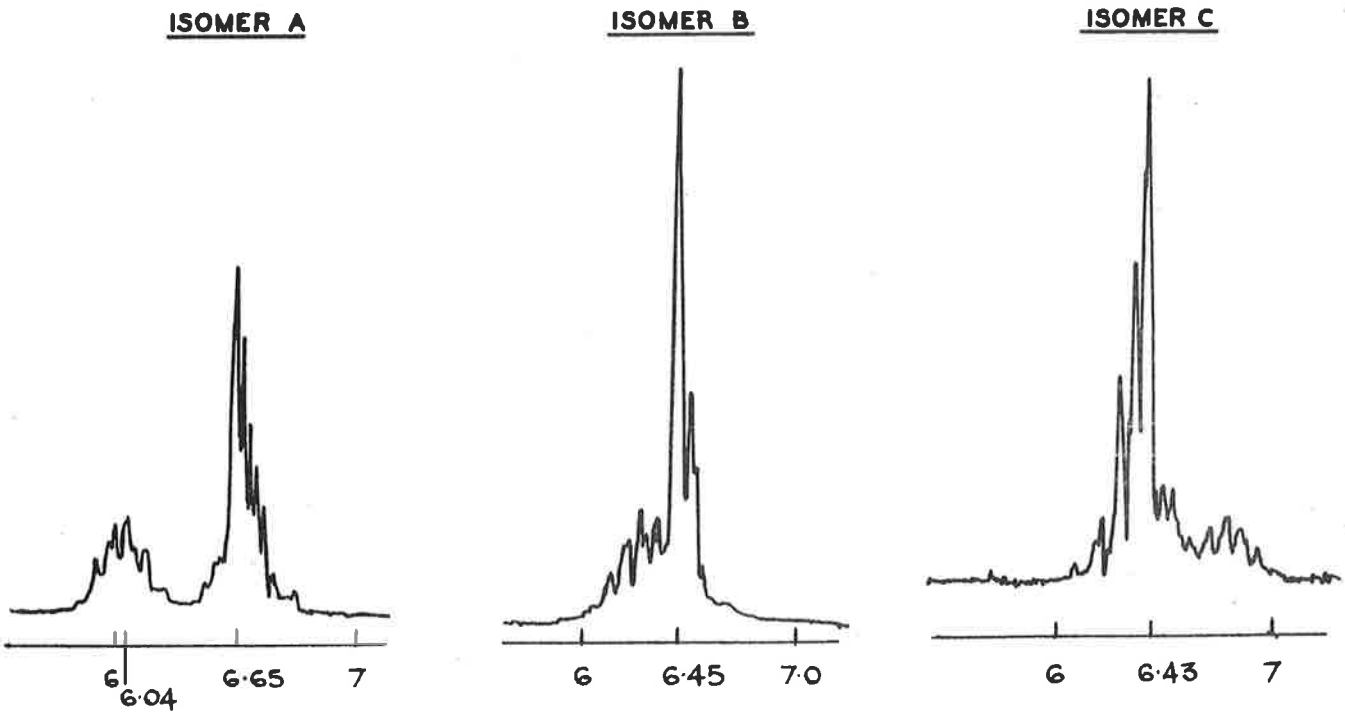


FIG 1

SERIAL No. 63/1638/KC

NITROBENZOATE OF DPG ISOMER A

$CDCl_3$, 80 Mc/sec

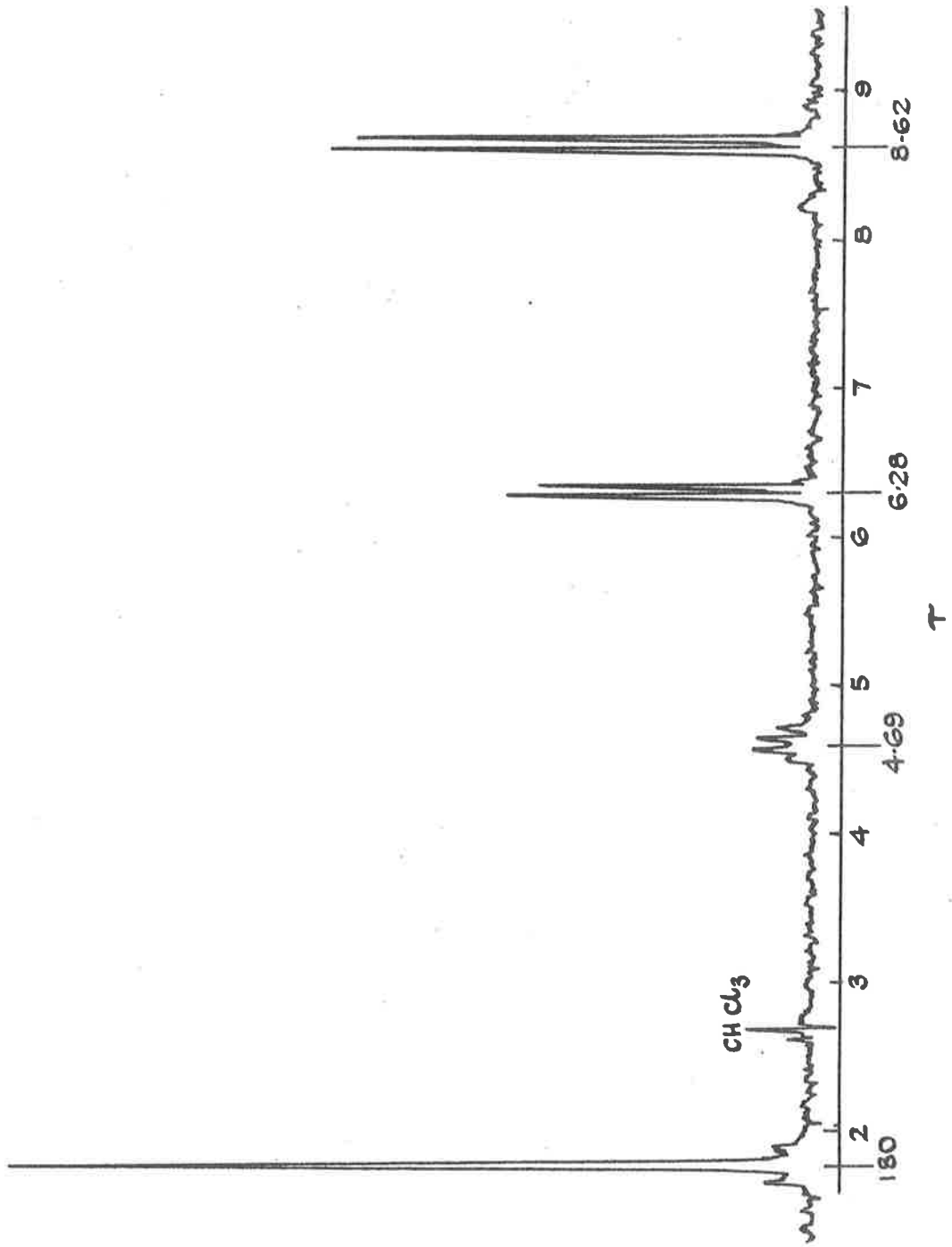


FIG 2

62-28

SERIAL No. 63/1639/KC

NITROBENZOATE OF DPG ISOMER B

$CDCL_3$, 80 Mc/sec

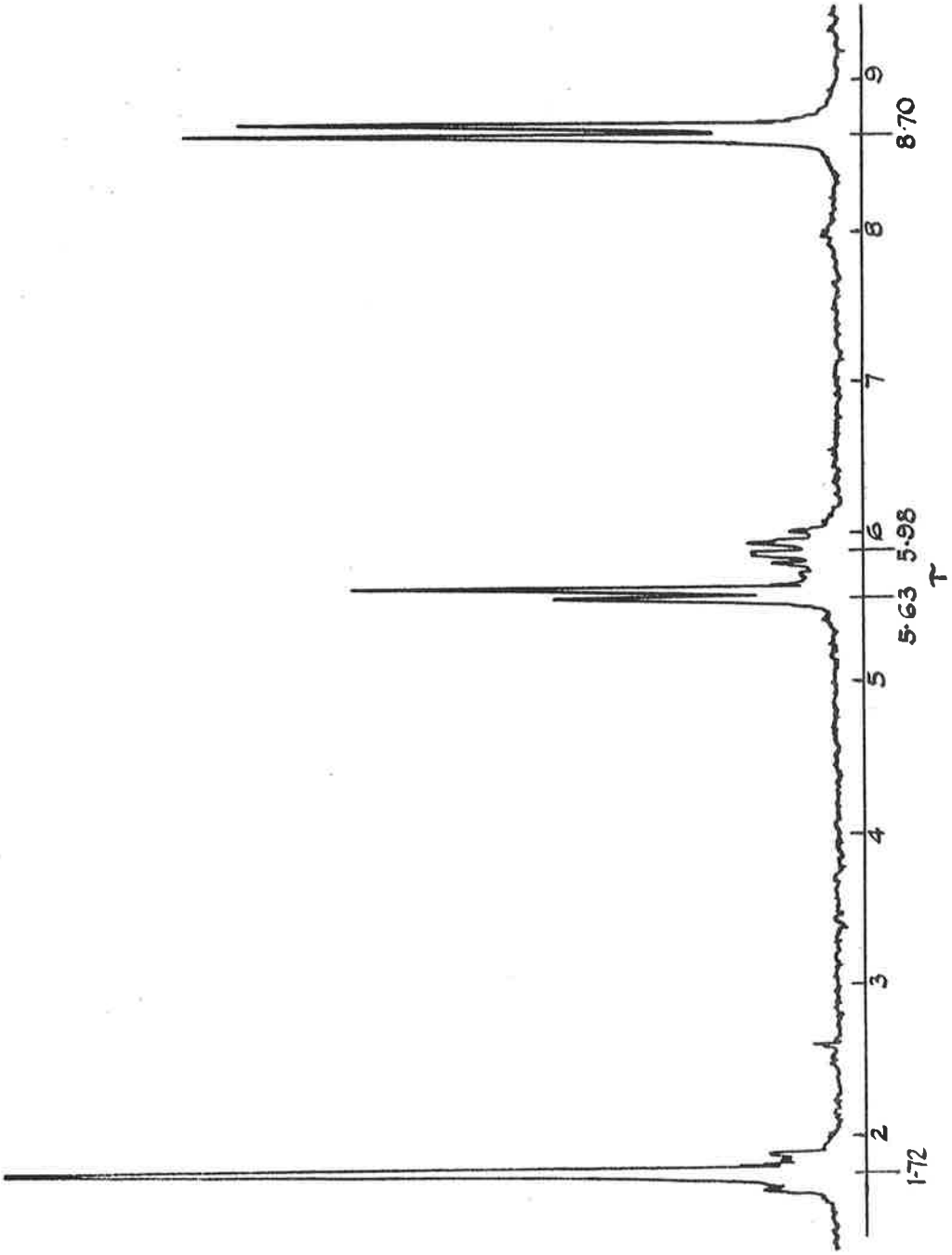


FIG 3

SERIAL No. 63/1640/BP

NITROBENZOATE OF DPG ISOMER C
$CDCL_3$, 80 Mc/sec

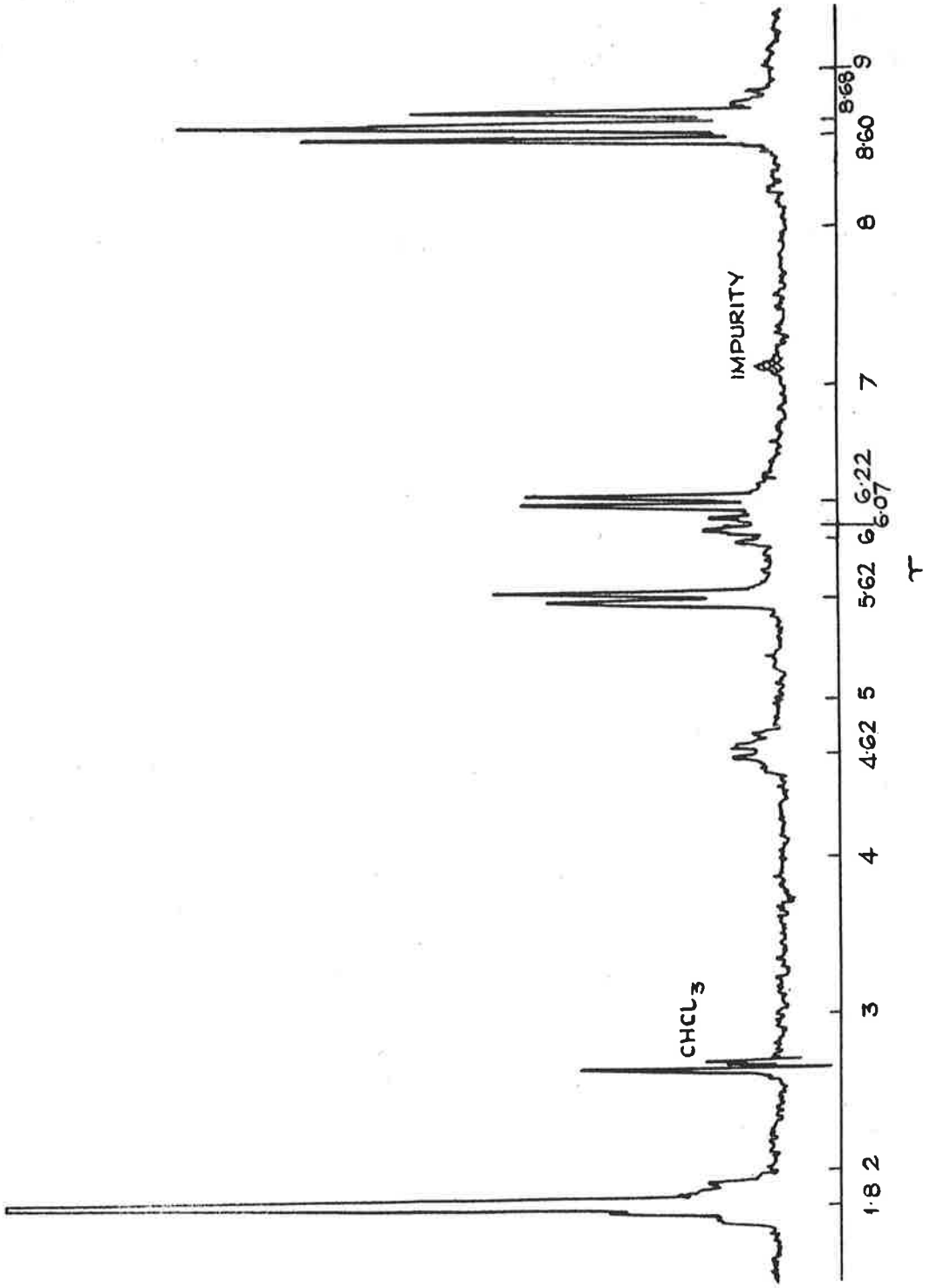


FIG 4

SHELL DEVELOPMENT COMPANY
A DIVISION OF SHELL OIL COMPANY
EMERYVILLE, CALIFORNIA

November 19, 1963

Dr. B. S. Shapiro
Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Pennsylvania

Dear Barry,

I know that the renewal of my subscription to MELLONMR is not due for several months yet, but I thought I would set a good example for those who tend to be late (myself included) by being early for a change.

I got a listing of BEQUIV from A. Douglas as offered in MELLONMR No. 59. This subroutine works beautifully except that it does not maintain the coupling between magnetically equivalent protons at zero. I have added the 6 statements in the box below to accomplish this:

```
12    NN = N - 1
      DO 14 I = 1, NN
      JI = I + 1
      DO 14 J = JI, N
      IF(HB(I,1) - HB(J,1)) 14, 13, 14
13    AA(I,J) = 0.0
14    CONTINUE
      DO 10 I = 1, NN
          etc.
```

If any of the readers of MELLONMR know of anyone who has adapted the NMRIT and/or NMREN programs for other than the IBM 7090 or 7094 computers, I would appreciate hearing about it.

Sincerely yours,

Charlie

C. A. Reilly

CAR:jel

BRYN MAWR COLLEGE
BRYN MAWR, PENNSYLVANIA
19010

DEPARTMENT OF CHEMISTRY

18 November 1963

Dr. Barry Shapiro
Mellon Institute
Pittsburgh, Penna.

Dear Dr. Shapiro:

I should like to report some work done at Harvard University in the hope that it will be both suitable material for circulation in MELLONMR as well as down payment for a subscription to the Monthly...Letters.

The nuclear magnetic double resonance (NMDR) of ammonia ($N^{14}H_3^1$) has been described in terms of the time-dependent perturbation introduced by the second r.f. field.[1] In addition, this description[1] included the width of the proton lines due to relaxation through the N^{14} quadrupole moment.[1,2] Recent experiments indicate that this simple description is adequate for low magnitudes of the second r.f. field.[3,4] Figure 1 shows observed and calculated NMDR spectra of $N^{14}H_3^1$ recorded by sweeping the magnetic field. Proton resonance is observed at 40 Mc., with the second r.f. frequency at the N^{14} resonance frequency ($\pm$0.5 cps.), and of strength 11.5 $\pm$ 1.0 cps; J_{NH} = 43.9 $\pm$ 0.3 cps.

However, if the second r.f. field is sufficiently strong, and the nature of the relaxation mechanism appropriate, the effect on the populations and lifetimes of the states is significant. Figure 2 shows an observed NMDR spectrum with second r.f. field at 35 $\pm$ 1 cps., and other parameters unchanged from Figure 1. The calculation is based on the simple description outlined above. The failure of this description is obvious. Preliminary calculations based on a solution of the equation of motion of the density matrix for the spin system following the Bloch-Wangsness-Redfield development[3,4,5] indicate that the effect of changes of the lifetimes of states is very important in the NMDR of NH_3. The broadening due to trace amounts of water present in the ammonia obscured the effects of the second r.f. field on lineshap somewhat and make analysis difficult.

The display of the calculated spectrum shown in Figure 1 was made on the on-line oscilloscope of the IBM 7090 installation used. The calculations were performed at the M. I. T. Computation Center, and the research was supported by the N. I. H. and the N. S. F.

References:

1. J. D. Baldeschwieler, J. Chem. Phys. 34, 718 (1961).

2. J. M. Anderson and J. D. Baldeschwieler, J. Chem. Phys. (to be published).

3. J. M. Anderson, "Studies in Nuclear Magnetic Double Resonance," Thesis, Harvard University, Department of Chemistry, August, 1963.

4. J. M. Anderson and J. D. Baldeschwieler, J. Chem. Phys. (to be published).

5. C. P. Slichter, Principles of Magnetic Resonance (New York: Harper and Row, 1963), pp. 142ff., and references contained therein.

Sincerely yours,

Jay Martin Anderson

Jay Martin Anderson

Encl: Figures

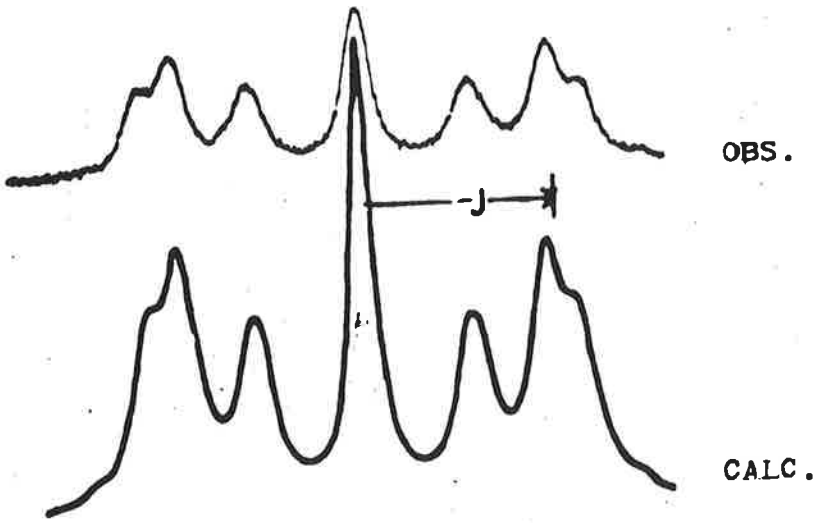


Figure 1. NMDR spectrum of NH_3, low second r.f. field.

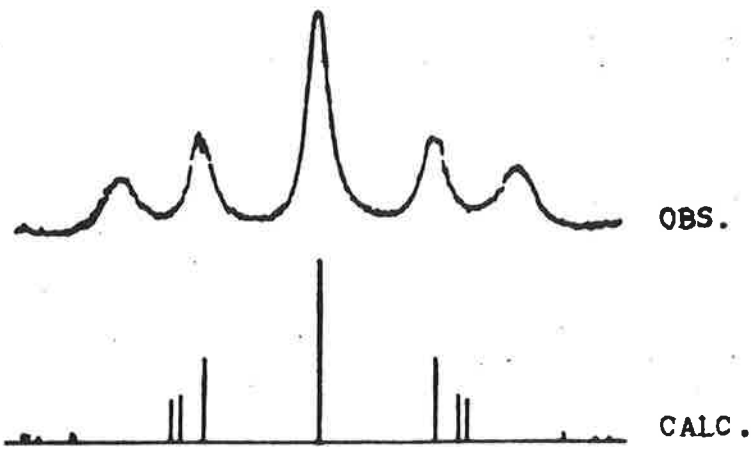


Figure 2. NMDR spectrum of NH_3, high second r.f. field

Central Research Laboratories, Minnesota Mining and Manufacturing Co., St. Paul 19, Minnesota

Tritium NMR Spectroscopy. I. Observation of High-Resolution Signals from the Methyl and Methylene Groups of Ethylbenzene. The Non-Radiochemical Use of Tritium as a Tracer.

G. V. D.Tiers, C. A. Brown, R. A. Jackson, and T. N. Lahr

High-resolution tritium NMR spectroscopy is here reported for the first time. Our sample of neat ethylbenzene contained overall 32.1 curies per ml. by conventional radioassay, which corresponds to about 1 atom percent tritium.

The tritium signal was weak but readily located by manual sweep from the proton region (40 mcps), and was self-identifying as it cannot be produced by any other material[1]. The spectrum consisted of two first-

(1) Tritium has spin 1/2, and its NMR resonance frequency is the highest known for any nucleus, namely 45.414 mcps at 10,000 gauss. (a) H. L. Anderson and A. Novick, Phys. Rev., 71, 372 (1947). (b) F. Bloch, A. C. Graves, M. Packard and R. W. Spence, ibid., 71, 551 (1947).

order multiplets, the higher-field one due to the $-CH_2T$ group being apparently a triplet of triplets. The splittings, taken as coupling constants, were measured as: J(TCH) = 13.6 ± 0.1 cps, and J(TCCH) = 7.90 ± 0.06 cps. Perhaps as a result of the low signal-to-noise ratio, it was not possible to detect the potentially greater complexity of the X-part of an A_2B_2X spectrum[2]. The weaker peak, due to the -CHT- group,

(2) R. E. Richards and T. Schaefer, Proc. Roy. Soc. (Lond.) A246,429(1958).

appeared to be a doublet of quadruplets, as would be expected for X in an A_3BX case. The coupling constants were estimated as: J(TCH) = 14.8 ± 0.1 cps., and J(TCCH) = 7.8 ± 0.1 cps. If due allowance be made for the 6.66% higher magnetic moment of tritium[1b], one would predict J(TCCH) to be 8.16 ± 0.02 cps.[3]. The observed chemical shift between the tritium peaks

(3) Our own measurements, based on an A_3B_2 analysis of the 60 mcps. spectrum of neat ethylbenzene, are J(HCCH) = 7.65 ± 0.02 cps., and Δ = 1.377 ± 0.010 p.p.m., at 38°C.

was 1.355 ± 0.006 p.p.m., in good agreement with the measured proton shift[3]. No signal was found for aryl tritium.

The possibility of using the non-radiochemical NMR properties of tritium in chemical studies has been demonstrated. Tritium is very cheap, about $2 per curie, and is carrier-free. The synthesis of organic compounds having high levels of tritiation, especially at specific sites, is feasible. No special shielding is necessary, the walls of the tube being more than adequate. However, it is of utmost importance that good ventilation and other precautions against inhalation in case of breakage or spills be provided. One-tenth of a milliliter of a 1% tritiated organic compound would normally provide in excess of one curie of radiation, a most severe hazard[4]. A further danger lies in self-radiation-damage leading to

(4) The maximal allowable tritium concentration in air for continuous exposure is given as 2×10^{-7} curies per cubic meter.

breakage from pressure buildup, since a 10-curie sample could produce over 1 ml. of H_2 per day[5].

(5) J. A. Stone and P. J. Dyne, Radiation Research, 3, 353 (1962).

62-36

Max-Planck-Institut für Biochemie
Dr. J. Sonnenbichler

8 München 15, den 7.11.1963
Goethestraße 31
Fernruf 59 42 61/62
Postschließfach 64

Herrn
Dr. B. L. Shapiro
Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Penns. (U.S.A.)

Sehr geehrter Herr Dr. Shapiro !

An unserem Institut wurden in letzter Zeit eine Reihe von Chinolinderivaten vermessen. Durch Vergleich der Signale der kernständigen Protonen dieser Verbindungen läßt sich mit ziemlicher Sicherheit eine Zuordnung durchführen, die für den einen oder anderen Ihrer Leser von Interesse sein mag. Alle Signale sind in ppm-Werten angegeben, bezogen auf Tetramethylsilan = 0 und in Richtung niederes Feld mit positivem Vorzeichen versehen. Gemessen wurden im allgemeinen 0,2 m Lösungen in CCl_4 oder $CDCl_3$.

Aromatische Protonen einiger Chinolon-(2)-carbonsäure-(4)-ester.

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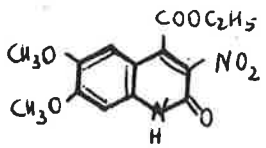
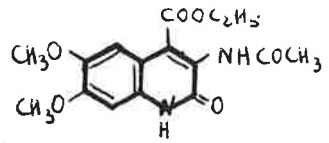
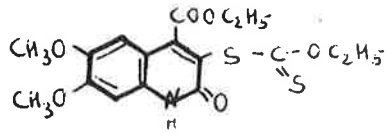
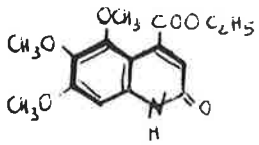
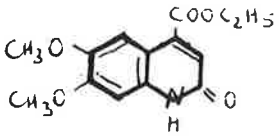
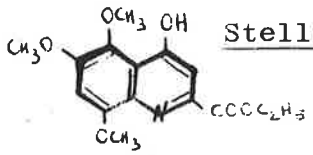
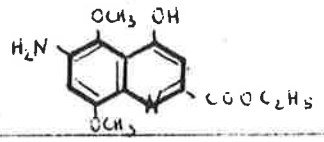
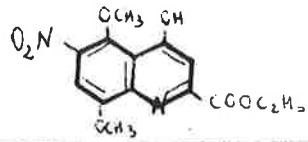
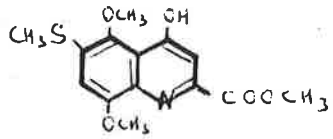
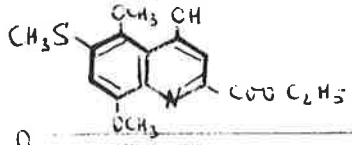
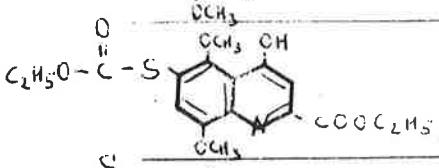
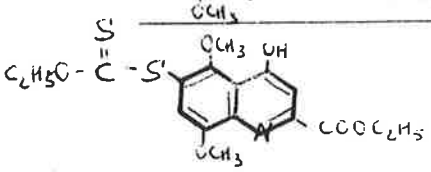
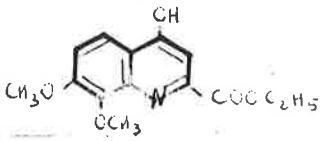
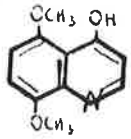
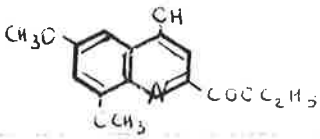
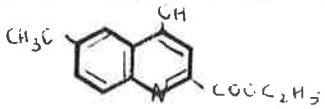
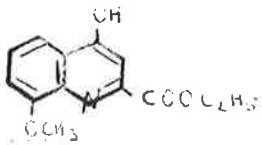
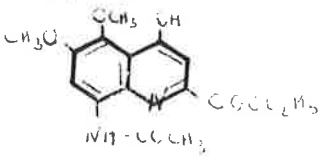
		6.95	7.06
		6.95	6.87
		6.98	6.80
	7.19(br)		7.33
	7.21	6.92	7.95

Aromatische Protonen einiger Chinolon-(4)-carbonsäure-(2)-ester.

62-37

Stellung:	3	5	6	7	8	7	
	6.9 (br.)			6.86			
	6.85 (br.)			6.67			
	6.87 (br.)			7.44			
	6.83 (br.)			6.95			
	6.85 (br.)			6.92			
	6.9			7.22			
	6.92 (br.)			7.12			
	6.92	8.05	7.05				$J_{5:6}$ = 9 Hz
	6.95 (br.)		6,64	6,88			$J_{6:7}$ = 8,5 Hz
	6.93	7.22		6.7			$J_{5:7}$ = 2,5 Hz
	6.75	7.5		7.4	7.95		$J_{7:8}$ = 9 Hz $J_{5:7}$ = 3 Hz
	6.93	7.85	7.26	7.03			$J_{5:6 \text{ und } 6:7}$ = 7,8 Hz $J_{5:7}$ = 1,8 Hz
	7.48			8.75			

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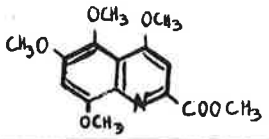
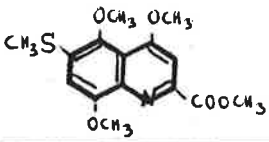
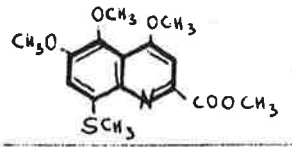
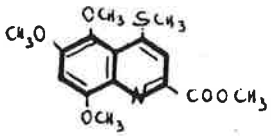
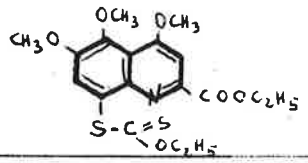
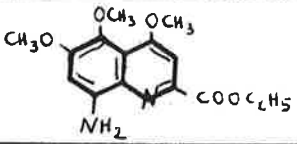
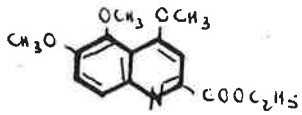
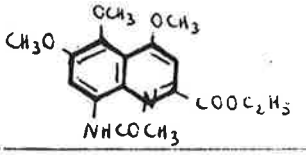
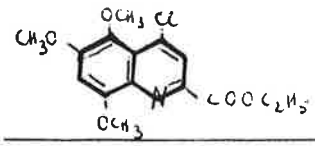
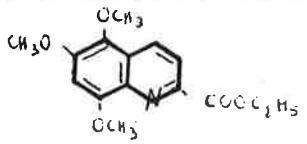
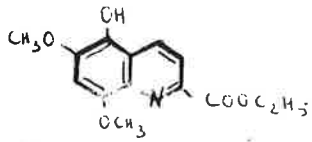
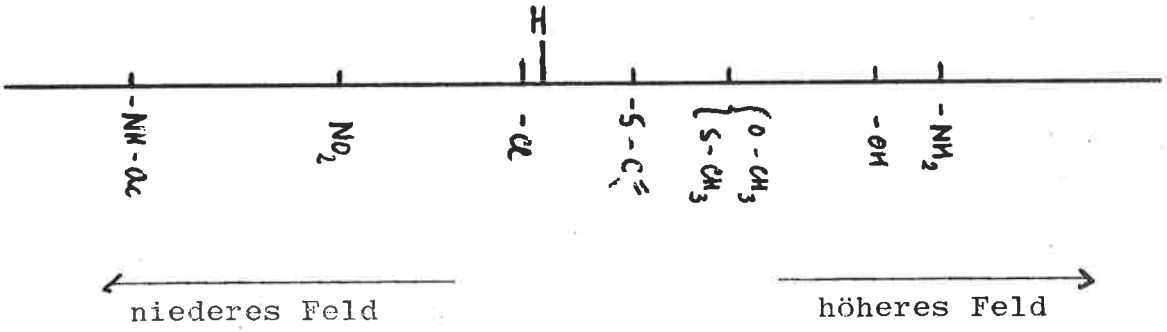
Aromatische Protonen einiger Chinolincarbonsäure(2)ester.

in Stellung:	3	4	7	8		
	7.57		6.9			
	7.65		6.95			
	7.53		7.2			
	7.87		6.89			
	7.7		7.3			
	7.44		6.77		$-NH_2$ 5.59 (br.)	
	7.3		7,4	7.82		$J_{7:8}$ = 9 Hz
	7.46		8.8		H mid 9.9 (br.)	
	8.15		6.92			
	8.15	8.47	6.85			$J_{3:4}$ = 9 Hz
	8.1	8.5	6.86 (br.)			$J_{3:4}$ = 9 Hz

Max-Planck-Institut für Biochemie

An einer größeren Reihe dieser Verbindungen wollen wir versuchen, die Beeinflussung der Kernprotonensignale durch Substituenten systematisch zu erfassen. Vorläufig können wir nur qualitative Aussagen machen:
Deutliche Signalverschiebungen treten ein bei Änderung der o-ständigen Substituenten:



Eine Beeinflussung der meta-Stellung ist kaum festzustellen, dagegen eine starke Wechselwirkung zwischen Stellung 3 und 8, sowie 4 und 7.

Die Verbreiterung des Signals (br) für den C3-Wasserstoff in den Chinolonderivaten ist eine Folge der möglichen Tautomerie. Das ebenfalls breite Signal für den tautomeren Wasserstoff an -NH oder -OH liegt bei den 2-Chinolonen zwischen 10,7 und 13,2 ppm, bei den 4-Chinolonen zwischen 9,15 und 9,8 ppm und ist mit D_2O gut austauschbar. Die Signale der O-CH_3-Gruppen erscheinen zwischen 3,7 und 4,2 ppm, die der S-CH_3-Gruppen um 2,5 ppm.

Alle Substanzen wurden in unserem Institut von Dr. E. Biekert, Dr. E. Härle und Dr. W. Schäfer dargestellt.

With best regards I remain

Sincerely yours

Johann Sonnenbichler

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THE UNIVERSITY OF CONNECTICUT
STORRS, CONNECTICUT

Department of Chemistry

November 20, 1963

Dr. B. L. Shapiro
Mellon Institute
4400 Fifth Avenue
Pittsburgh, Penna.

Dear Barry:

Permit me to use MELLON-M-R as an advertising medium. At present there are no n.m.r. facilities here of any sort. Although I intended to clean up all outstanding research problems at Esso, the variable temperature probe on their A-60 went "poof" about six weeks before I left. This resulted in several pieces of work, all of which involved a temperature dependent n.m.r. study, to remain incomplete. What I should like to inquire into at present is the possibility of gaining access to a n.m.r. spectrometer with variable temperature probe for a few days around the Christmas school vacation. Could I make any such arrangements with someone located within about 200 miles of Storrs? Needless to say such a deal would be very much appreciated!

Sincerely,

Eugene I. Snyder

Eugene I. Snyder

"NMR-Experiments on Solubilization in Soap Micelles"
J. C. Eriksson
Acta Chem. Scand. 17, 1478 (1963)

"Graphic Representation of Proton Chemical Shifts. General Consideration and Methyl and Methylene Groups"
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Y.-R. Naves, P. Ochsner, A. F. Thomas et D. Lamparsky
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A. Lablache-Combier, J. Levisalles, J.-P. Pete et H. Rudler
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V. Delaroff, P. Rathle et M. Legrand
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H. Najer, R. Giudicelli et J. Menin
Bull. Soc. Chim. France 1810 (1963)

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F.-X. Jarreau, Q. Khuong-Huu and R. Goutarel
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Bull. Soc. Chim. France 1899 (1963)

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J. Cancelli, J.-J. Basselier et J. Jacques
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J.-M. Conia et F. Rouessac
Bull. Soc. Chim. France 1925 (1963)

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G. Kersaint, C. Goetschel et C. Mentzer
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C. Tabcik-Wlotzka, M. Mousseron and A. Chafai
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S. Bory, M. Fetizon and P. Laszlo
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M. Truong-Ho, X. Monseur, Q. Khuong-Huu and R. Goutarel
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G. Drillien and C. Viel
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