

Joseph B. Lambert

Illinois
Institute of
Technology
N-M-R
Newsletter

No. 109
OCTOBER, 1967

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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". Reference to the IIT NMR Newsletter by name in the open literature is strictly forbidden.

These restrictions apply equally to both the actual Newsletter participant-recipients and to all others who are allowed access to the Newsletter issues. Strict adherence to this policy is considered essential to the successful continuation of the Newsletter as an informal medium of exchange of NMR information.

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Deadline Dates: No. 110: 6 November 1967
No. 111: 6 December 1967

Reminder: For the period August 10, 1967 to August 15, 1968 inclusive, all Newsletter contributions, enquiries, etc., should be addressed as follows:

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

-continued on outside back cover



Your Reference:

Our Reference: JF/JC

5th September, 1967.

Dear Barry,

Measurement of CCH Coupling Constants in Allyl Bromide

Recently Peter Pauwels (of Kodak Research Laboratories, Kirkby, Liverpool) and myself have been using triple resonance measurements to determine CCH coupling constants in allyl bromide.

Figure 1 illustrates the nature of the triple resonance experiments on the ^{12}CH Spectrum: by strong irradiation at the allylic CH_2 (not shown) we obtain an AMX spectrum on which tickling experiments can be carried out very easily. In Figure 2 are shown the results of the same triple resonance experiments carried out for the ^{13}CH lines in the molecules $\text{CH}_2 = ^{13}\text{CHCH}_2\text{Br}$ in natural abundance: the tickling measurements in this case locate the hidden lines from ^{13}CCH coupling. By this method one obtains the coupling constants

$$J_{^{13}\text{C}-\text{C}-\text{H}_\text{A}} = 1.0 \pm 0.3 \text{ Hz} \text{ and } J_{^{13}\text{C}-\text{C}-\text{H}_\text{M}} = 2.9 \pm 0.2 \text{ Hz}$$

A C-1024 spectrum accumulator was used to improve the signal-to-noise ratio of the ^{13}CH satellite spectrum.

Yours sincerely,

Jim Feeney

J. Feeney

Professor B.L. Shapiro,
Department of Chemistry,
Stanford University
Stanford,
CALIFORNIA 94305.

FIG. 1

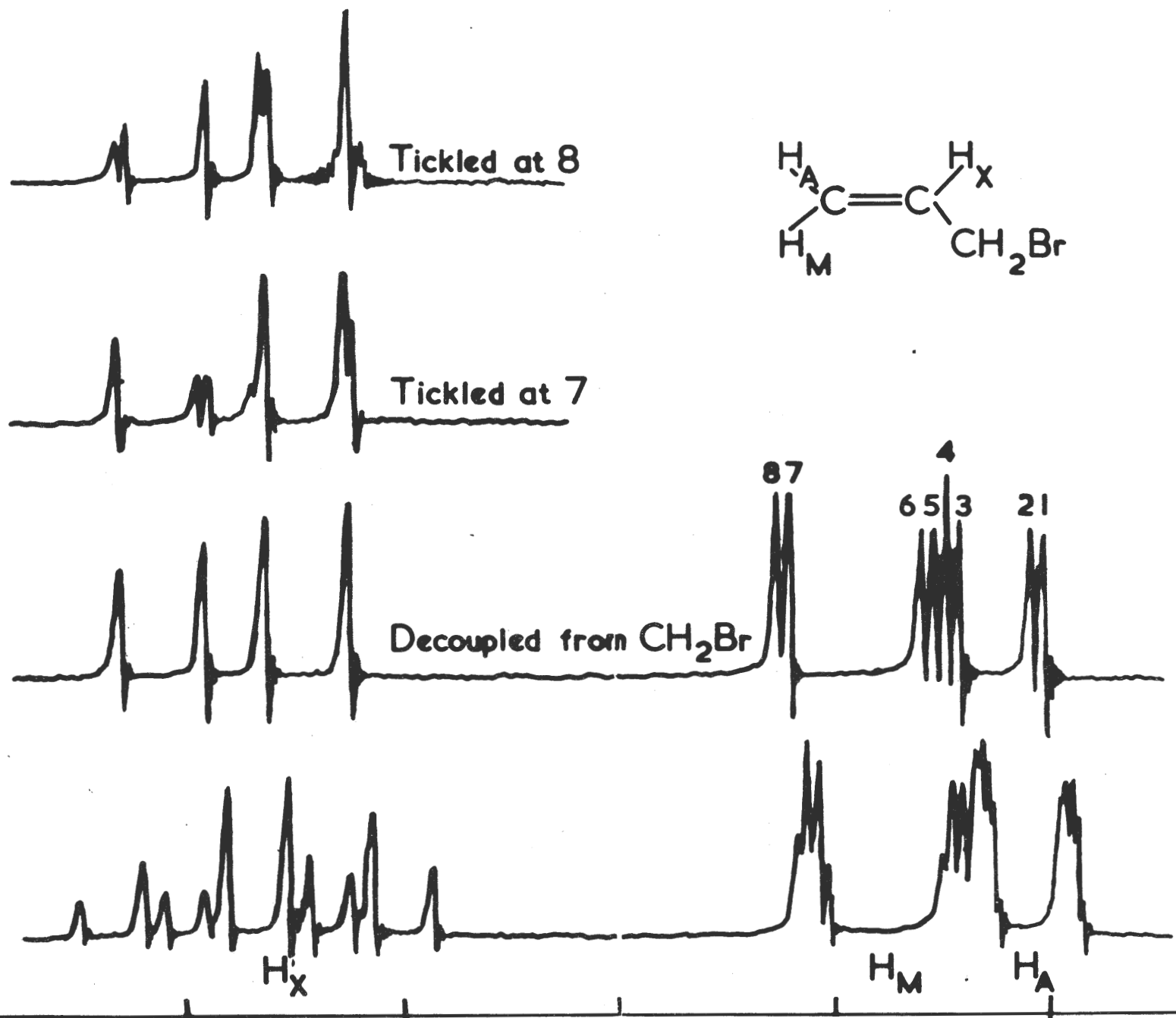
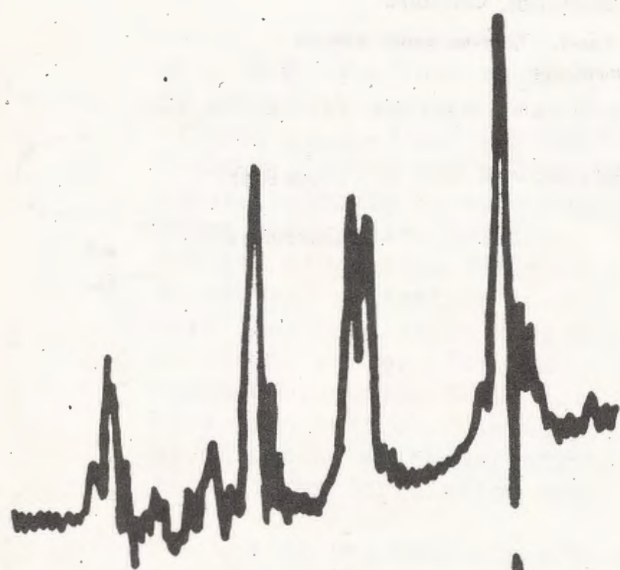
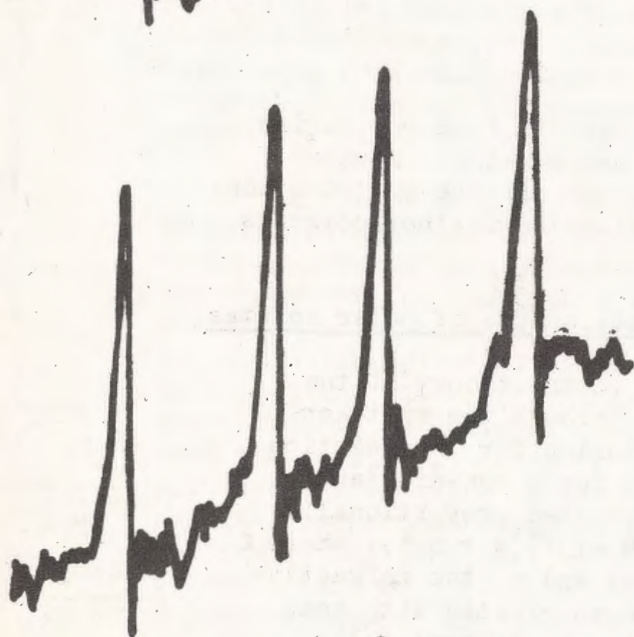


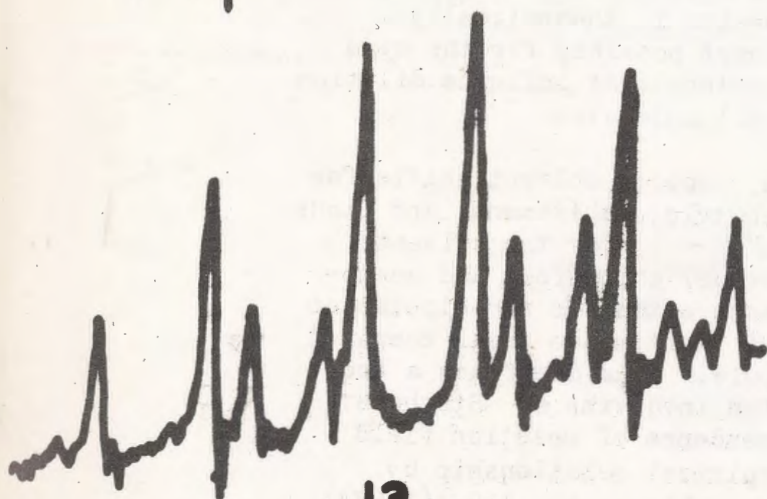
FIG. 2.



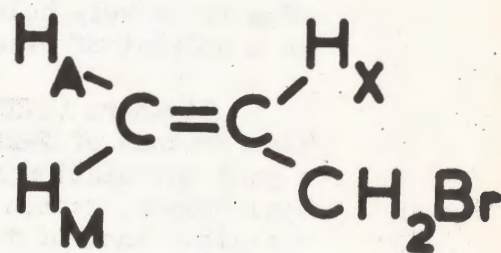
Tickled at 8'



$^{13}\text{C}\text{H}_2\text{Br}$ irradiated



$^{13}\text{C}\text{H}_x$ 25 Scans





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7th September, 1967.

Your Ref:

Our Ref: JKP/CF

Dr. B. L. Shapiro,
Chemistry Department,
Stanford University,
Stanford,
California 94305,
U.S.A.

Dear Barry,

We hope soon to send some results from our Varian 220 Mc/s spectrometer, which is now working. However, prompt action is needed to renew our overdue contribution, and as an interim measure I am offering further comments on:

Reaction field effects on chemical shifts of polar solutes

Much confusion has arisen in the theory of the reaction field effect in n.m.r. through the mistaken application of the Onsager expression for the reaction field, which is appropriate only for a non-dipolar medium. Deviations from the predicted proportionality of the reaction field shift to $(\epsilon - 1)/(2\epsilon + n_0^2)$, where ϵ is the permittivity of the medium and n_0 the refractive index of the pure solute, have been greeted with some surprise. In fact this expression is theoretically invalid for dipolar media, except possibly for the case of a relatively bulky solute molecule at infinite dilution in a solvent of relatively small molecules.

Stothers (IITNMR 106-54) reports solvent shifts for the α protons of 2-chloro-4-*t*-butylcyclohexanone, and finds a good correlation with $(\epsilon - 1)/(\epsilon + 1)$ for the solvents cyclohexane, carbon tetrachloride, chloroform and acetonitrile. Each of these solvents either is non-dipolar or has molecules whose moments of inertia are small compared with that of the solute molecule. Thus one finds a good correlation with the expression involving ϵ . Stothers concludes that the linear dependence of reaction field shift on ϵ^2 proposed as an empirical relationship by Kotowycz and Schaefer (Canad. J. Chem., 45, 1093 (1967)) is not generally applicable.

I would go further than this and assert that no expression for the reaction field shift which is a function

FROM: J. K. Becconsall.

CONTINUATION SHEET NO.: 2

TO: Dr. P. L. Shapiro.

DATE: 7th September, 1967.

only of ϵ , the low-frequency permittivity of the medium, can be generally applicable. The main reason for this, as already pointed out (IITNMR 100-14), is that the polarising field acting on the medium in the neighbourhood of a polar solute molecule is time-dependent as a result of the molecule's random tumbling. This does not affect the resulting reaction field in a non-dipolar medium, whose dielectric relaxation time is extremely short compared with the correlation time for random reorientation of the solute molecule. For a dipolar medium, however, the time-averaged reaction field acting on the solute molecule is less than that calculated for a stationary solute molecule by an amount which depends on the ratio of the relaxation time to the correlation time.

A second possible effect which may cause departures from the Onsager expression for the reaction field, especially for small solute molecules with large dipole moments, is dielectric saturation close to the solute molecule; this again applies only for dipolar media.

The use of mixtures of solvent pairs in varying proportions has been advocated by Laszlo and Musher, and again by Kotowycz and Schaefer, to investigate the dependence of the reaction field shift on ϵ . This has the advantage of eliminating complications from effects specific to particular solvents. However, as a technique for investigating which effects are of importance in producing the shifts it is useless, because any plot that one makes begs the question. A smooth curve will always result if the solute chemical shift is plotted against a function of physical properties which vary continuously with the mixture ratio. The real test of generality in any theory of the effect must be to attempt to predict the shift for a polar solute in any solvent, subject to the limitations of choice imposed by known complications such as ring-current effects.

Yours sincerely,

Jack Becconsall

J. K. Becconsall



INTERNATIONAL SYMPOSIUM ON ELECTRON AND NUCLEAR MAGNETIC RESONANCE

SPONSORED BY THE AUSTRALIAN ACADEMY OF SCIENCE AT MONASH UNIVERSITY CLAYTON VICTORIA
11-14 AUGUST 1969.

PLACE: Monash University, Clayton, Victoria, Australia, 3168

The Monash University campus is approximately 13 miles from the centre of Melbourne. Opened in 1961, Monash University now (1967) has 7,000 students.

DATE: 11 - 14 August 1969

Chemists will note that this symposium is being held in the week preceding the IUPAC Congress, Sydney, Australia, 20 - 30 August 1969. Oversea participants may well wish to go on to Sydney via Canberra.

PROGRAMME:

The programme will include papers on nuclear magnetic resonance (high resolution, wide-line, and spin echo); electron spin resonance of solids, liquids and gases; pure quadrupole resonance; other forms of magnetic resonance such as ferromagnetic resonance, and cyclotron resonance. It is hoped to emphasise the more physical aspects of these phenomena.

ACCOMMODATION:

Participants will be accommodated in the modern Halls of Residence on the campus. If they prefer it they may be accommodated in motels which surround the University.

SPONSORSHIP:

The symposium will be under the aegis of the Australian Academy of Science.

CONFERENCE LANGUAGE:

English will be the language of the conference, although papers in other languages may be submitted.

FURTHER INFORMATION:

Should you desire additional information or if you would like to receive extra copies of this notice, and/or the second circular, please complete the attached slip.

ORGANIZING COMMITTEE:

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CSIRO Division of Chemical Physics,
Clayton, Victoria, Australia, 3168

Professor H.S. Gutowsky,
University of Illinois

Dr J.R. Pilbrow (Secretary)
Physics Department, Monash University,
Clayton, Victoria, Australia, 3168

Professor S. Fujiwara,
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Professor R.D. Brown, FAA
Monash University

Professor N. Sheppard, FRS,
University of East Anglia

Professor R. Street,
Monash University



INTERNATIONAL SYMPOSIUM ON ELECTRON AND NUCLEAR MAGNETIC RESONANCE

SPONSORED BY THE AUSTRALIAN ACADEMY OF SCIENCE
AT MONASH UNIVERSITY CLAYTON VICTORIA 11-14 AUGUST 1969

(Block letters please)

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I wish to receive ☐ copies of this notice

I wish to receive the symposium circulars

My interest lies in:

High resolution NMR

Wide-line NMR

Relaxation NMR or ESR

Electron Spin resonance

Other magnetic resonance

Please send to:

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CHARACTERISATION OF AROMATIC PETROLEUM FRACTIONS BY ABSORPTION MODE CARBON-13 NMR

Detailed characterisation of petroleum fractions is both difficult and time-consuming, due to their extreme complexity. For this reason, various analytical schemes have been devised (1, 2) which attempt to describe a particular petroleum fraction in terms of a hypothetical average molecule. Such schemes are useful for examining oils on a comparative basis and in monitoring processes, eg dehydrogenation, where the number of aromatic rings per molecule is likely to increase.

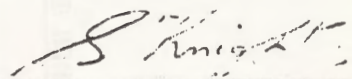
Hydrogen NMR provides a method (2) for the characterisation of aromatic fractions but it does suffer from the disadvantage that the characteristic features of the carbon skeletons have to be calculated from hydrogen type area ratios. In particular it is very difficult to estimate the carbon to hydrogen weight ratio of saturated substituents. Given total carbon content from combustion analysis, carbon-13 NMR enables direct measurement of aromatic and saturate carbon contents. Three characteristic bands may be discerned in a typical carbon-13 NMR spectrum of an aromatic fraction (3). The band at lowest field (A_1) characterises ring junction carbon, substituted ring carbon and one half of the unsubstituted ring carbon. The next low field band (A_2) characterises one half of the unsubstituted ring carbon and the high field band (A_3) characterises the carbon in saturated groups (4). These three areas, normalised to unity, allow the determination of the relative amounts of carbon in aromatic rings, in unsubstituted sites in aromatic rings and in saturated groups.

Similarly, three characteristic bands may be identified in the hydrogen NMR of an aromatic petroleum fraction. These three areas give respectively, the relative amounts of hydrogen on aromatic rings (a_1), on carbon atoms α to aromatic rings (a_2) and other saturate carbon (a_3), (2).

The six relative areas A_1 , A_2 , A_3 , a_1 , a_2 , a_3 together with other analytical data in the form of weight per cent carbon (C), hydrogen (H), sulphur (S) and the molecular weight (M) enables a particularly comprehensive carbon type analysis of the aromatic petroleum fraction to be made. Some typical results are shown in Table 1.

An analysis of this type enables fractions from different crude sources or different wells in the same source to be critically compared with one another whilst the uncertainties introduced in earlier type analyses, because of the difficulty in estimating the naphthenicity (number of alicyclic rings) of saturate groups, are eliminated. A more extensive discussion of this work will be published shortly.

S.A. Knight



References

- (1) K. Van Nes and H.A. Van Westen. "Aspects of the Constitution of Mineral Oils" Elsevier Pub., Co. Inc. Amsterdam, 1951.
- (2) R.B. Williams. "Symposium on Composition of Petroleum Oils, Determination and Evaluation" ASTM Special Technical Publication No. 224, p 168-184, (1958).
- (3) IITNMR 98-62
- (4) P.C. Lauterbur, J. Am. Chem. Soc. 1961, 83, 1838.

Acknowledgement

The absorption mode carbon-13 NMR spectra were obtained with the kind co-operation of Perkin-Elmer Ltd, Beaconsfield, Buckinghamshire, England using a computerised R10 spectrometer operating at 15.1 MHz.

TABLE 1

CARBON TYPE ANALYSIS OF (A+S) FRACTIONS

Sample	C _A [*]	C _A	n	C ₁ ^S	C ₁ ^U	C ₁	%AS	T. No. C _A	T. No. C ₁	T. R _A	NAG	C _S	R _N	M
African 1	55.9	49.7	1.95	20.1	25.8	45.9	43.8	7.46	6.88	1.29	3.01	39.2	0.92	180
African 2	51.8	46.1	2.14	20.0	22.7	42.7	46.8	8.07	7.48	1.30	3.50	42.8	1.44	210
African 3	56.1	48.7	2.30	16.6	22.6	39.2	42.3	9.34	7.51	1.92	3.17	38.1	1.24	230
North African Blend	36.1	31.7	3.96	14.2	15.5	29.7	47.7	8.19	7.68	1.26	3.66	56.1	2.38	310
African Blend	45.5	39.5	3.23	14.7	16.3	31.0	47.5	10.54	8.25	2.15	3.92	47.4	1.49	320

Glossary

C_A^{*} Fraction per cent of carbon atoms in aromatic rings
 C_A Weight per cent carbon in aromatic rings
 n Average number of carbon atoms per saturated substituent
 C₁^S Substituted aromatic ring carbon, weight per cent
 C₁^U Unsubstituted aromatic ring carbon, weight per cent
 C₁ Total substitutable aromatic ring carbon, weight per cent
 %AS Per cent substitution of saturated groups on substitutable aromatic ring carbon atoms.

T. No. C_A Number of aromatic ring carbons atoms per molecule
 T. No. C₁ Number of substitutable aromatic ring carbons atoms per molecule
 T. R_A Number of aromatic rings per molecule
 NAG Number of saturated substituents per molecule
 C_S Weight per cent carbon in saturated groups
 R_N Number of naphthene (alicyclic) rings per molecule
 M Molecular weight


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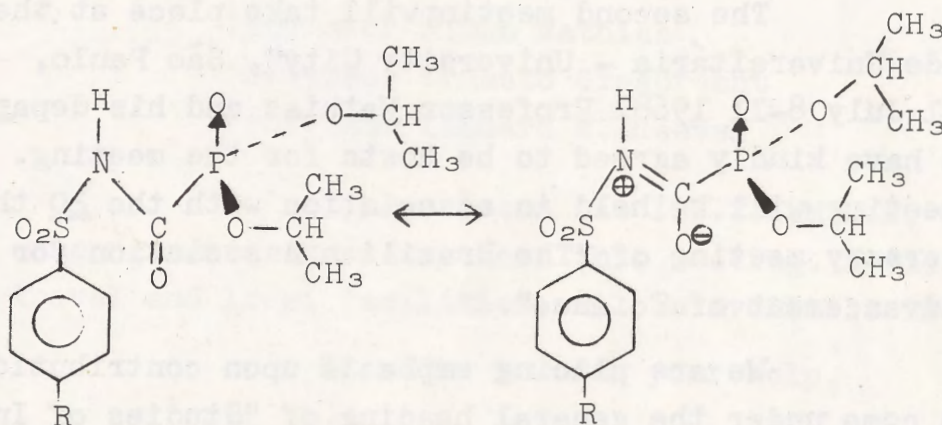
74074

September 12, 1967

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

Dear Dr. Shapiro:

An interesting example of nonequivalence of methyl protons in the carbamoyl phosphonate has been found for I. Two overlapping



Doublets (P-O-C-H splitting) occur at $\delta 1.32$ and $\delta 1.27$ are observed. From other results just published in the literature we assume here that as a result of increased double bond character in the C-N bond, rotation around the P-O bond is restricted so that the methyl groups are nonequivalent. Details of the work will be published shortly.

Sincerely yours,

K D Berlin

K. D. Berlin
 Professor

KDB/caf

UNIVERSIDADE DE SÃO PAULO
FACULDADE DE FILOSOFIA, CIÊNCIAS E LETRAS
CAIXA POSTAL 8105 — BRASIL, SP.

September 8, 1967

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

Dear Barry:

I am using the medium of I.I.T.N.M.R.N. to announce the second international symposium on nuclear magnetic resonance following the first meeting Sept. 1-3 1965 arranged by Professor Fujiwara in Tokyo.

The second meeting will take place at the "Cidade Universitária - University City", São Paulo, - Brazil July 8-11 1968. Professor Mathias and his department have kindly agreed to be hosts for the meeting. The meeting will be held in association with the 20 th anniversary meeting of "The Brazilian Association for the Advancement of Science".

We are placing emphasis upon contributions which come under the general heading of "Studies of Intermolecular Forces". This topic includes not only all phases of matter but all parameters measured with NMR - instruments. We shall be contacting many people through our mailing system but this letter will serve to invite contributions from any readers of I.I.T.N.M.R.N.

We should like to have tentative titles as soon as possible so that we can begin to arrange the sessions. We have invited and will further invite leading

speakers to act as chairman of these sessions and to give major contributions.

The conference will be published as a - collection of papers under the auspices of the "Brazilian Association from the Advancement of Science". The funds for this purpose have been generously supplied by the "Fundação de Amparo à Pesquisa do Estado de São Paulo". We are anxious that abstracts are available at the meeting and in order to provide this service we need them by April 20 - 1968. They should be sent to either of the following three persons at the above address:

Professor Simão Mathias
Professor Ernesto Giesbrecht
Professor Leonard W. Reeves

I hope I can use I.I.T.N.M.R.N. for further announcements and news about this meeting. Advice regarding travel and local facilities will be available in due time.

Thanking you for your help.

Sincerely

Leonard W. Reeves.

Leonard W. Reeves
Visiting Professor

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

1 Berlin 12, den September 8, 1967
Straße des 17. Juni Nr. 115 (Chemiegeb.)
Fernruf: 85701, App. 252 Prof. B/Ma
31 07 81

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

Chicago 60616 / Ill.

U S A

NMR-spectra of Monodeuteromethyl compounds

Dear Professor Shapiro,

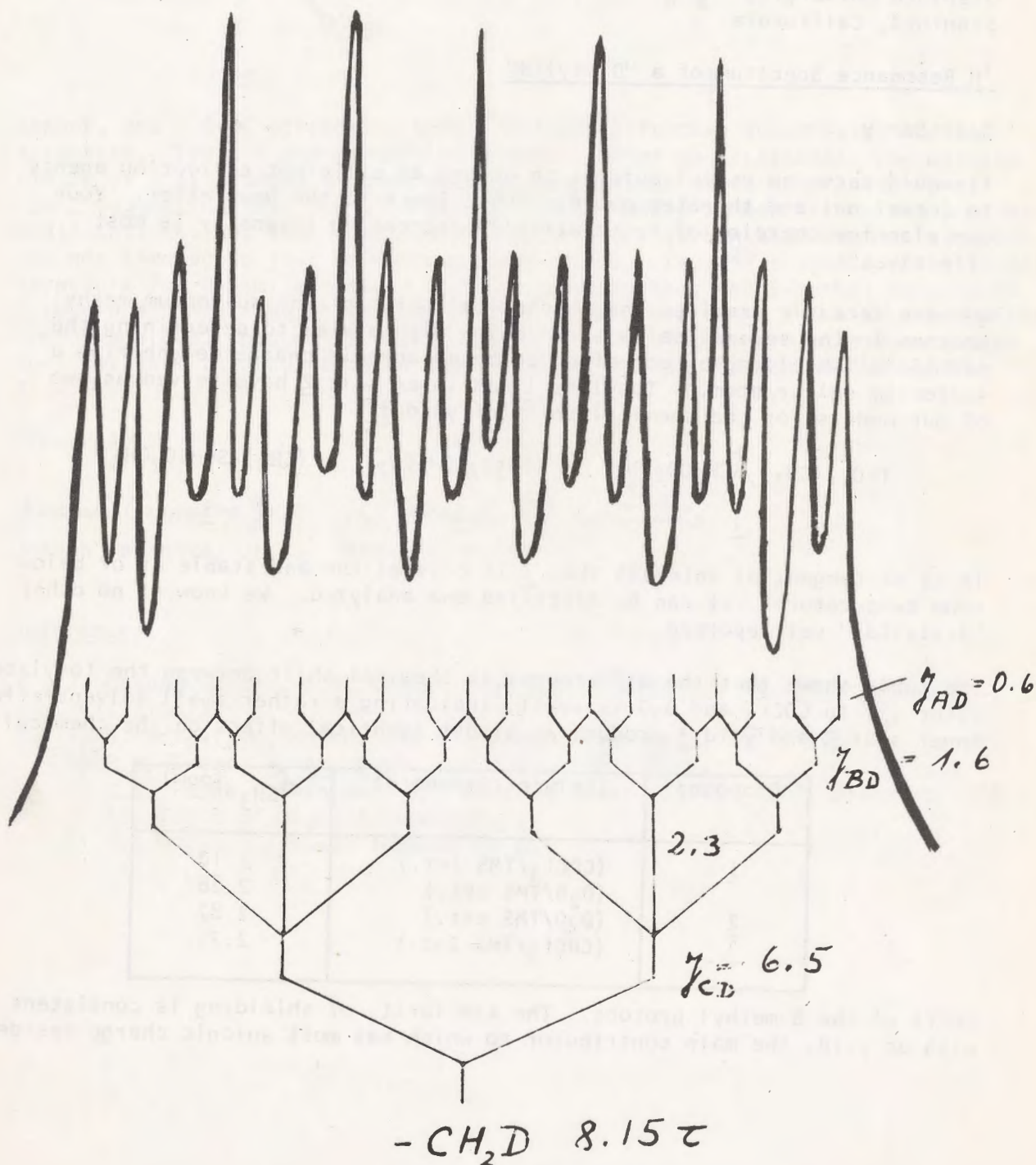
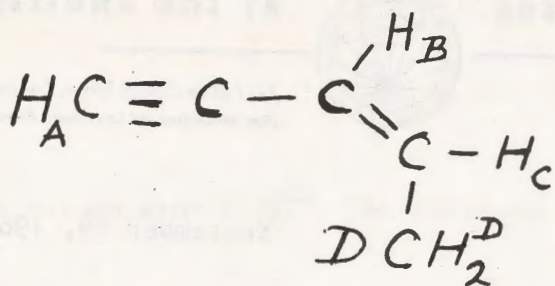
In connection with mass spectroscopy of polyynes we have prepared a number of deuterated compounds. Perhaps the NMR-data are of interest to some of your readers. In part they are very complicated as the deuterium gives rise to a splitting of the methyl signal as already recognized by G. Triers for Monodeuterotoluene (J. chem. Physics 29, 963 (1958)). For example the methyl group in cis-pentenin gives a nicely separated nineteen lines signal (see picture).

The spectra of the other compounds are not so complicated.

DCH_2CHO	td 7.95 (J = 2 and 2.7)
$\text{DCH}_2\text{CHOH}_2\text{C}\equiv\text{CH}$ OH	td 8.78 (J = 1.9 and 6.1)
$\text{DCH}_2\text{CH}=\text{CH}[\text{C}\equiv\text{C}]_2\text{CH}=\text{CHCH}_2\text{OH}$	tdd 8.20 (J = 2.3, 6.8 and 1.6)
$\text{DCH}_2\text{CH}=\text{CH}[\text{C}\equiv\text{C}]_2\text{CH}=\text{CHCHO}$	tdd 8.14 (J = 2.3, 6.8 and 1.8).

Yours sincerely,

F. Bohlmann





5151 State College Drive, Los Angeles, California 90032
(San Bernardino and Long Beach Freeways Interchange)

Department of Chemistry

September 14, 1967

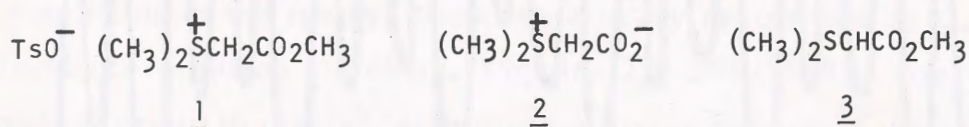
Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California

^1H Resonance Spectrum of a "Distylid"

Dear Barry:

It would serve no useful purpose to engage an efficient collection agency to ferret out and threaten overdue subscribers to the Newsletter. Your own plan for coercion of systematically increasing intensity is most effective.

We have recently examined the ^1H chemical shift of the sulfonium methyl protons in the several salts shown below with a view to determining the additional shielding effect afforded by an anionic charge neighboring a sulfonium sulfur atom.¹ Tosylate 1 and inner salt 2 have served as two of our models for the chemical shift of ylid 3:



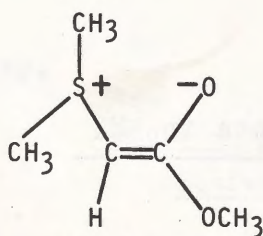
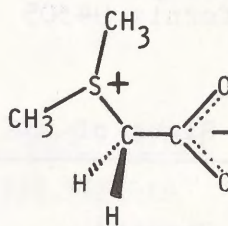
It is of tangential interest that 3 is crystalline and stable at or below room temperature. It can be distilled and analyzed. We know of no other "distylids" yet reported.

The table shows that the differences in chemical shift between the tosylate ester (1) in CDCl_3 and D_2O is small, indicating a rather small solvent effect. Inner salt 2 and ylid 3 produce an almost identical effect on the chemical

Compound	(Solvent/Standard)	δ_{CH_3} (ppm)
<u>1</u>	(CDCl_3 /TMS int.)	3.10
	(D_2O /TMS ext.)	2.86
<u>2</u>	(D_2O /TMS ext.)	2.87
<u>3</u>	(CDCl_3 /TMS int.)	2.76

shift of the S-methyl protons. The similarity of shielding is consistent with an ylid, the main contributor to which has most anionic charge resident

on the oxygen atom (2a)^{2a}. The infrared carbonyl frequency of 2 is

2a3

absent, and a $C=C$ stretching band (1621 cm^{-1}) further supports an enolic structure. Trost^{2b} and Payne^{2c} have made similar observations. The methine proton of 2 is a broad singlet at room temperature which becomes sharp at -30° , suggesting that the ylid exists predominantly as one of the two possible modifications, and that rotation of the carbon-carbon bond becomes slow on the nmr time scale just below room temperature. Trost^{2b} suggests the cis (2a) structure for a similar ylid. It is noteworthy that the S-methyl protons of 2 remain a sharp singlet throughout the temperature range in which the methine proton undergoes change. However, due to d-orbital geometry this observation does not permit speculation on the nature of $d\pi-p\pi$ carbon-sulfur bonding in 2.

Sincerely,

Joseph Casanova, Jr.

Joseph Casanova, Jr.

David A. Rutolo

David A. Rutolo

References

1. J. Casanova, Jr., N.D. Werner, and H.R. Kiefer, *J. Am. Chem. Soc.*, **89**, 2411 (1967).
2. a) H. Nozaki, K. Kondo, and M. Takaku, *Tet. Let.*, 251 (1965);
 b) B.M. Trost, *J. Am. Chem. Soc.*, **89**, 138 (1967);
 c) G.B. Payne, 154th Nat. ACS Meeting, Sept. 11-15, 1967, Paper No. 158 (Division of Organic Chemistry);
 d) G.B. Payne, private communication.

KYOTO UNIVERSITY

DEPARTMENT OF CHEMISTRY, FACULTY OF SCIENCE

KYOTO, JAPAN

Professor B.L.Shapiro,
Department of Chemistry,
Stanford University,
Stanford, California 94305

Signs of the coupling constants in NH_3

Dear Professor Shapiro,

Although convergence of the perturbation expansion for calculation of the coupling constant in HF^1 could not be confirmed, Loève and Salem² obtained a negative geminal coupling constant in CH_4 by truncated expansion. In this event, we attempted in the same approximation to calculate the coupling constants (Fermi term only) in NH_3 . The results are summarized in the following table.

J_{NH}	J_{HH}	Used MO
+10.20	+1.63	Slater MO (Duncan ³)
+8.96	+1.70	H-F MO (Kaplan ⁴)
+22.10	+10.00	Slater MO with $\alpha = 1.2$ (Kaldor-Shavitt ⁵)
+31.25	+7.23	Double-Zeta MO (Kaldor-Shavitt ⁵)
43.6	10.35	Experiment (Bernheim et al ⁶)

The two estimates by use of Kaldor-Shavitt's MO's appear to be in fair agreement with experiment in absolute magnitude. Although our results give positive signs for both J_{NH} and J_{HH} , in contrast to the case of methane, our results are not decisive, because of no reliable knowledge of contributions from higher excitations and electron correlation. Only if these neglected effects happen to cancel each other, our estimates would be adequate. It is more probable that J_{NH} is positive, and it is desirable to determine the sign of J_{HH} by double resonance on the assumption that J_{NH} is positive.

Sincerely yours,

A. Saika
A. Saika

1. Kato and Saika, J.Chem.Phys. 46, 1975 (67).
2. Loève and Salem, ibid. 43, 3402 (65).
3. Duncan, ibid. 27, 423 (57), 39, 240 (63).
4. Kaplan, ibid. 26, 1704 (57).
5. Kaldor and Shavitt, ibid. 45, 888 (66).
6. Bernheim, ibid. 40, 3446 (64).

From Professor N. Sheppard F.R.S.

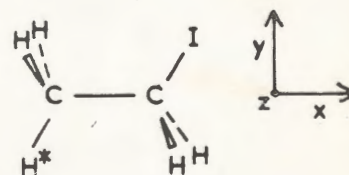
School of Chemical Sciences
University Plain
Norwich NOR 85C
Telephone Norwich 52651

Dear Barry,

Liquid-Crystal Spectrum of Ethyl Iodide;
Equivalence in anisotropic spectra

The remarks by Bulthuis, Hilbers and MacLean in the July number concerning sub-spectral analysis and liquid-crystal spectroscopy prompt us to remark that we have been looking at the spectrum of ethyl iodide in the nematic phase (see figure), which is a particularly good example of the possibilities of this technique. Assuming rapid internal rotation of the CH_3 group the methyl protons (and the methylene protons) in ethyl iodide are fully equivalent (i.e. they are magnetically equivalent, with all inter-group coupling constants equal, and there are equal coupling constants within each group). The spectrum may therefore be analysed, using the composite particle method, as a superposition of QT, QS, DT and DS subspectra. The QS and DS subspectra, taken together, constitute an \underline{a}_3 subspectrum, which is simply a 1:2:1 triplet with spacing $\frac{1}{2}D_{AA}$ (marked * in figure), and the DT and DS subspectra, taken together, form an \underline{ab}_2 sub-spectrum (marked †). In fact, in this case, where one coupling constant (D_{BB}) is very large, the \underline{ab}_2 sub-spectrum may be analysed approximately as \underline{ax}_2 , because the X resonance occurs in the wings of the spectrum, far removed from the A region in the centre. The relative signs of D_{AA} , D_{BB} and D_{AB} were easily determined from a more thorough investigation of the QT lines, and the sign of J_{AB} relative to the D's was determined conclusively from a least-squares fit using a modified version of LAOCOON. The parameters obtained at 76° in 4,4'-di-n-hexyloxyazoxybenzene were as follows:

$$\begin{array}{ll} D_{AA} = +1188 \text{ c/s} & c_{3z^2-r^2} = -0.1460 \\ D_{BB} = +2781 \text{ c/s} & c_{x^2-y^2} = +0.0597 \\ D_{AB} = -265 \text{ c/s} & c_{xy} = +0.1586 \\ J_{AB} = +7.16 \text{ c/s (sign and magnitude assumed)} \end{array}$$



The most favoured orientation is therefore with H_o more or less parallel to the longest 'geometrical axis' of the molecule ($H^* \text{---} I$).

As far as we know, no-one has yet considered the problem of notation for spin-systems in anisotropic spectra. There are three forms of equivalence involved - chemical, magnetic and full (defined above) - and although magnetic equivalence is strictly irrelevant in the analysis of anisotropic spectra, we are reluctant to ignore it altogether, because, on principle, it is preferable to have a single notation applicable to both isotropic and anisotropic spectra. We therefore propose that the notation usually attributed to Corio be modified, so that A_n now represents a fully equivalent group, and a magnetically equivalent group containing p fully equivalent groups of m nuclei is written $A_{m \times p}$. In this notation, benzene is $A_{1 \times 6}$, allene is $A_{2 \times 2}$, ethane is $A_{3 \times 2}$ and SF_6 is $A_{2 \times 3}$, while propane is $A_{3 \times 2} B_2$, and S_2F_{10} is $AB_{2 \times 2} B_{2 \times 2} 'A'$. To obtain the normal notation (which can of course still be used for isotropic analysis), the subscripts are simply multiplied together.

We have also been considering the possibilities of studying conformational problems by liquid-crystal spectroscopy. However, it can be proved that the anisotropic coupling constants (averaged over all conformations) at a single temperature can never determine the isomeric ratio except within very broad limits. Assuming that all the anisotropic coupling constants can be obtained from the spectrum, and that the geometry of each conformer is known fairly accurately, it is only possible to determine the quantities $\alpha_i P_i$, where α_i is the proportion of conformer i , and P_i a motional constant for this conformer. These quantities do not enable the α_i and P_i to be determined separately when more than one conformer is present. However, where it can be shown, e.g. by other spectroscopic methods, that a molecule exists only in one dominant form, liquid-crystal N.M.R. spectroscopy should be a very powerful tool for determining which isomer this is, and much quicker than the alternative method of X-ray diffraction of the crystalline solid.

Yours sincerely,

Norman

Chris Woodman

N. Sheppard
C.M. Woodman

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

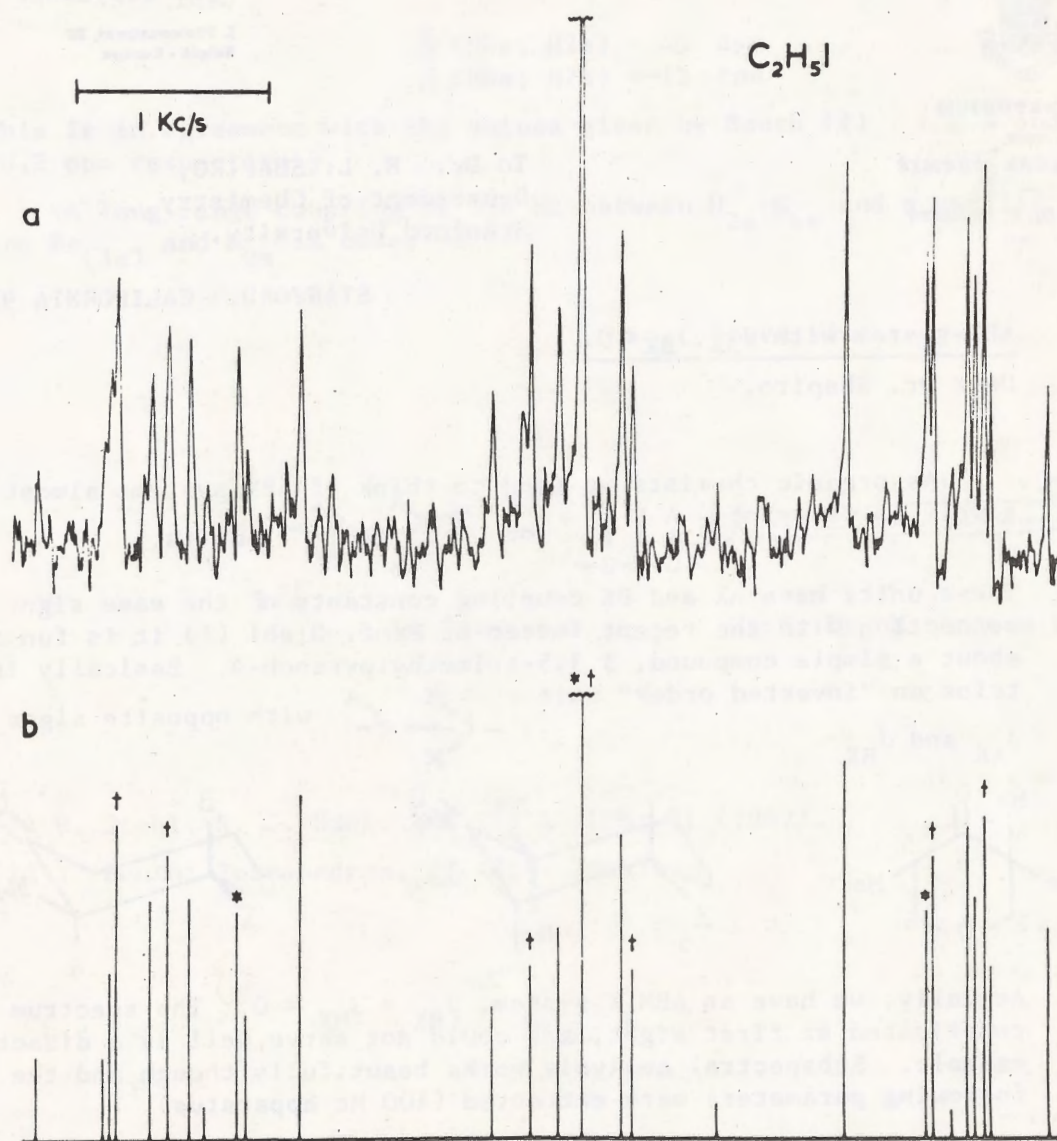


Fig. 1. The spectrum of ethyl iodide in PAHB at 71° : (a) observed spectrum, (b) calculated A_3B_2 spectrum with D_{AB} and J_{AB} of opposite sign. DT transitions are marked \dagger , and QS transitions are marked $*$.



LABORATORIUM
VOOR
ORGANISCHE CHEMIE
—
Dir.: Prof. Dr. F. GOVAERT
—

GENT, September 20th 1967.

J. Plateastraat, 22
België - Europa

Tel. 23.38.21

To Dr. B. L. SHAPIRO,
Department of Chemistry
Stanford University,

STANFORD. CALIFORNIA 94305.

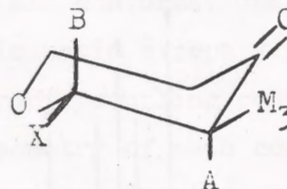
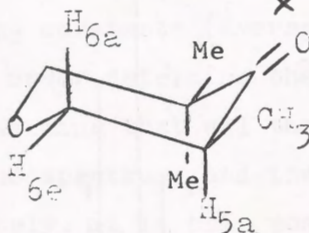
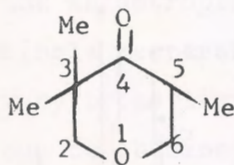
ABX-system with $J_{AX} \cdot J_{BX} < 0$.

Dear Dr. Shapiro,

As organic chemists we tend to think of ABX systems almost exclusively in terms of $\begin{smallmatrix} A & B & X \\ | & | & | \\ -C-C-C- \end{smallmatrix}$ or $\begin{smallmatrix} A & X \\ | & / \\ -C- & C- \\ | & \backslash \\ B & \end{smallmatrix}$ units.

These units have AX and BX coupling constants of the same sign. In connection with the recent letter of Prof. Diehl (1) it is fun to tell about a simple compound, 3,3,5-trimethylpyranon-4. Basically it contains an "inverted order" unit $\begin{smallmatrix} B & A \\ | & / \\ -C- & C- \\ | & \backslash \\ X & \end{smallmatrix}$ with opposite signs of

J_{AX} and J_{BX} .



Actually, we have an ABM_3X system, $J_{BX} = J_{MX} = 0$. The spectrum looks complicated at first sight, and could not serve well as a didactic example. Subspectral analysis works beautifully though, and the following parameters were extracted (100 Mc apparatus).

$J_{5a6a} = J_{AB} = 11,4$ cps	
$J_{6e5a} = J_{AX} = 7,0$ cps	} opposite signs
$J_{6e5a} = J_{BX} = -10,8$ cps	
$J_{5a-Me} = J_{AM} = 6,2$ cps	
$\delta(H6e) = 4.13$	$\delta(H2e) = 3.68$
$\delta(H6a) = 3.16$	$\delta(H2a) = 3.29$
$\delta(H5a) = 2.84$	

Comparison of the shifts of the H2 and H6 atoms yields a nice illustration of the effect of an axial methyl group on the adjacent hydrogens.

.../...

.../...

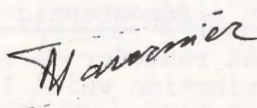
$$\Delta (H_{6e}; H_{2e}) = 45 \text{ cps}$$

$$\Delta (H_{6a}; H_{2a}) = -13 \text{ cps}$$

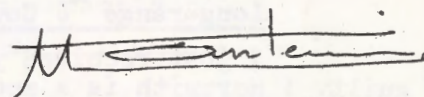
This is in agreement with the values given by Booth (2) : + 0,4 and -0,2 ppm respectively.

A long-range coupling of 1,8 Hz between H_{2e} - H_{6e} and a smaller one $Me_{(3a)}$ and H_{2a} is observed.

Yours sincerely,



D. Tavernier



Prof. Dr. M. Anteunis.

(1) P. Diehl, R. C. Huck, NMR IIT n° 106, 31 (1967).

(2) J. Booth; Tetrahedron, 22, 615 (1966).

FACULTÉ DES SCIENCES DE MARSEILLE - SAINT-JÉROME

TRAVERSE DE LA BARASSE - MARSEILLE (13^e)

TÉLÉPHONE : 64.31.61

DÉPARTEMENT DE CHIMIE ORGANIQUE

LABATOIRE DE CHIMIE M.P.C.

J. C. MAIRE : MAITRE DE CONFÉRENCES.

19 .9.1967

Prof.B.L.SHAPIRO
 Department of Chemistry
 Stanford University
STANFORD, Calif. 94305

Dear Prof.Shapiro,

Long-range 4J Couplings in organostannic compounds :

I just received your second reminder of September 7 th. I'm pleading guilty ! Herewith is a modest contribution which I hope will suffice to remove me from the black-list.

We have recently observed long-range couplings in two completely different types of organotin compounds :

First, in di-tert-butylstannane 1 : A 0,7 cps has been found between the methyl protons and the hydride protons, the signal of which ($\tau = 4,7$) is a multiplet with 13 lines resolved. Details will be published in the next issue of J. of Organometal. Chem.

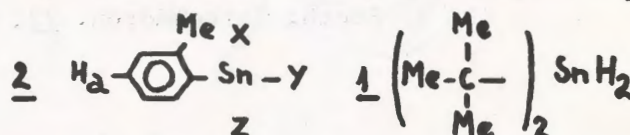
More recently we examined the spectra of some o-tolyltin compounds 2.

a X = Y = Z = o-tolyl

b X = Y = o-tolyl , Z = I

c X = o-tolyl, Y = Z = Br

d X = o-tolyl, Y = Z = I



I must mention that these compounds have been kindly sent to us by T.N.SRIVASTAVA (University of Lucknow). Methyl protons are in allylic position relative to the tin atom, and the ring proton H_a . We were planning to study the CH_3 - H_a coupling, and the influence on it of the substituents attached to the tin atom. In fact, in all cases, the CH_3 signal looks like a broad triplet ($\tau = 7,6$), but the side peaks were too small for the signal to be really a triplet, and were better interpreted as satellite-lines due to the 117 and 119 tin isotopes (7,67 and 8,68 % abundance). Further evidence was obtained by double resonance experiment. The ring protons signal is an ABCD spectrum which could not be analysed. We decided to irradiate it "a priori" with a fairly strong irradiating field. Moving the position of irradiation cps by cps, from one side to the other, we didn't observe any change in the shape of the methyl signal. So that we must admit that a 4J Sn-H coupling does exist with a constant equal to $3 + 0,5$ cps. A similar coupling has been observed in p-tolyl lead compounds (W.KITCHING, V.G.KUMARDAS, P.R.WELLS, Chem.Com. 1967, 7, 356), but could not be observed in (p-tolyl) $_4$ Sn.

Sincerely yours.


 J.C. MAIRE

500 South Ridgeway Avenue
Glenolden, Pennsylvania 19036
Telephone 215 586 4700



Research Division
Research & Development—Philadelphia

File: 001-3-1-1

September 21, 1967

Prof. B. L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

Dear Barry:

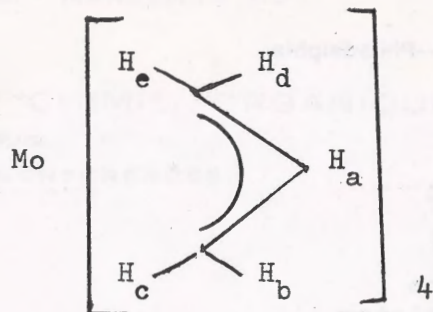
Subject: Tetraallyl Molybdenum: Example of
Asymmetric- π -Bonding

Some of our current work on organometallic systems may be of interest.

Recent reports(1-4) have shown that n.m.r. may be utilized to classify allyl metal complexes of type $M(\text{allyl})_N$, with respect to symmetry and bonding. Three distinct types have been proposed: (i) σ , indicated by an $ABCX_2$ spectrum, (ii) symmetrical- π , indicated by an AM_2X_2 , and (iii) dynamic, indicated by an AX_4 spectrum.

A fourth type of metal-allyl bond, asymmetric- π -allyl, has been suggested(3,5,6) for some substituted allyl compounds. The corresponding n.m.r. spectra are of the AGMPX type, which indicates that the two terminal carbon atoms of the allyl group are bonded unequally to the metal atom. Further evidence for this asymmetric- π -allylic bond was obtained from an n.m.r. study of tetraallyl molybdenum.

The 100 MHz spectrum of $Mo(\text{allyl})_4$ yielded five resonances with an area ratio of 1:1:1:1:1 (Figure 1). Two interpretations are consistent with the chemical shift and area data: (i) two sets of symmetrical- π -bonded allyl groups, indicating that two of the allyl groups are bonded differently than the other two, or (ii) four equivalent asymmetric- π -bonded allyl groups. The small coupling constants $J_{bd} = 1.6$ and $J_{de} = 0.8$ Hz which were confirmed by spin-decoupling (Figure 1-b) indicate that the former is impossible. They are, however, consistent with the latter interpretation.



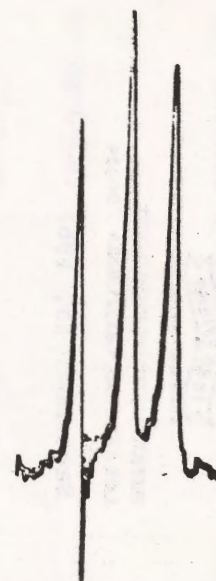
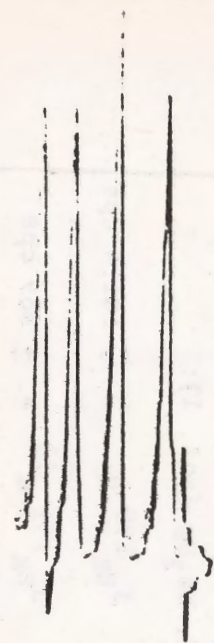
- (1) J.K.Becconsall, B.E.Job and S.O'Brien, J. Chem. Soc.(A), 423 (1967).
- (2) G.Wilke, B.Bogdanovic, P.Hardt, P.Heinbach, W.Keim, M.Kromer, W.Oberkirch, K.Tanaka, E.Steinrucke, D.Walter, and H.Zimmermann, Angew. Chem. Internatl. Edn., 5, 151 (1966)
- (3) G.L.Statton and K.C.Ramey, J. Am. Chem. Soc., 88, 1327 (1966)
- (4) K.Vrieze, C.MacLean, P.Cossee and C.W.Hilbers, Rec. Trav. Chim., 85, 1077 (1966)
- (5) W.B.Wise, D.C.Lini and K.C.Ramey, Chem. Comm., 463 (1967)
- (6) M. McPartlin and R. Mason, Chem. Comm., 16 (1967)

Sincerely yours,

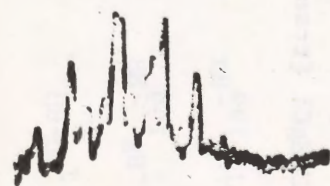
K.C. Ramey : W.B. Wise D.C. Lini
 K. C. Ramey, W.B. Wise, D.C.Lini

KCR:LH

b



a



a

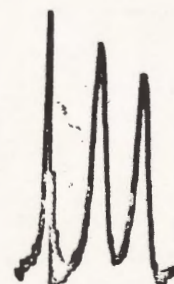


b

c



d



e

6

7

8

9

10
ppm

(a) NMR spectrum of Mo(allyl)_4 in CDCl_3 (room temp. 100 M Hz), (TMS internal stand.)
 (b) decoupled spectrum resulting from irradiation of d proton.

UNIVERSITY OF CALIFORNIA, LOS ANGELES

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SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY
LOS ANGELES, CALIFORNIA 90024

September 15, 1967

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

Signs of Coupling Constants to Hg^{199}

Dear Barry:

We are just writing up some work which Dr. J. L. Sudmeier and I finished some time ago on relative signs of $\text{Hg}^{199}\text{-H}^1$ and $\text{Hg}^{199}\text{-C}^{13}$ coupling constants. McLaughlan, Whiffen, and Reeves¹ have already reported on the relative signs of the coupling constants in $\text{CH}_3\text{Hg}^{199}\text{C}^{13}\text{H}_3$, and our work confirms and extends the conclusions of these workers. Table I shows the compounds which we have investigated.

Table I

EtHgCl:

$$J_{\text{Hg}^{199}\text{-CH}_2} = + 222 \text{ cps}$$

$$J_{\text{Hg}^{199}\text{-CH}_3} = \pm 300 \text{ cps}$$

$$J_{\text{CH}_2\text{-CH}_3} = \pm 8 \text{ cps}$$

MeHgNO₃:

$$J_{\text{Hg}^{199}\text{-H}} = + 258 \text{ cps}$$

$$J_{\text{Hg}^{199}\text{-C}^{13}} = \pm 1800 \text{ cps}$$

$$J_{\text{C}^{13}\text{-H}} = \pm 142 \text{ cps}$$

CHCl=CHHgCl (trans):

$$\begin{matrix} \beta & \alpha \\ J_{\text{Hg}^{199}\text{-H}\alpha} & = \pm 115 \text{ cps} \end{matrix}$$

$$J_{\text{Hg}^{199}\text{-H}\beta} = \pm 160.5 \text{ cps}$$

$$J_{\text{H}\alpha\text{-H}\beta} = \pm 14.6 \text{ cps}$$

cf. Me₂Hg (ref. 1):

$$J_{\text{Hg}^{199}\text{-H}} = + 101.4 \text{ cps}$$

$$J_{\text{Hg}^{199}\text{-C}^{13}} = \pm 689 \text{ cps}$$

$$J_{\text{C}^{13}\text{-H}} = \pm 129.6 \text{ cps}$$

Dr. Bernard L. Shapiro

-2-

September 15, 1967

Me-Hg-C≡CH:

$$J_{\text{CH-CH}_3} \approx 0 \text{ cps}$$

$$J_{\text{Hg}^{199}\text{-CH}_3} = \mp 149 \text{ cps}$$

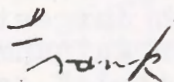
$$J_{\text{Hg}^{199}\text{-CH}} = \pm 69 \text{ cps}$$

Except for the last compound, relative signs were determined by examination of INDOR spectra. Because $J_{\text{CH-CH}_3} \approx 0$ in the last compound, triple resonance experiments were needed for sign determination.² All proton spectra were obtained at 60 Mc/sec.

For each compound, either the top or the bottom combination of signs in Table I is to be taken. Since vicinal H-H coupling constants and C^{13} -H coupling constants are known to be positive from other investigations, it follows that the top combination of signs in Table I is, in fact, the correct one.

1. K. A. McLaughlan, D. H. Whiffen, and L. W. Reeves, Mol. Phys., 10, 12 (1966).
2. A. D. Cohen, R. Freeman, K. A. McLaughlan, and D. H. Whiffen, Mol. Phys., 7, 45 (1963).

Yours sincerely,



Frank A. L. Anet

FALA:efm

Institut für Organische Chemie
der Philipps-Universität Marburg
Bahnhofstraße 7
W. Sauer

355 Marburg/Lahn, den 14.9.67
Fernruf 732202

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

Sehr geehrter Herr Prof. Shapiro!

Die Einstellung eines Kernresonanzspektrometers auf optimale Felddomogenität ist deshalb mühsam, weil nach jeder Korrektur der Feldeinstellung der Schreiber zur Kontrolle der Signalform in die Ausgangsstellung zurückgefahren werden muß. Viele ältere Geräte halten außerdem den Zustand optimaler Homogenität nicht lange, so daß häufig eine Neueinstellung nötig ist. Das folgende einfache Zusatzgerät erlaubt es, eine Feldeinstellung in wenigen Sekunden vorzunehmen.

Die dargestellte Apparatur (siehe Bild) besteht aus einem in eine kreisförmige Nute eingelassenen Widerstandsdraht, einem Abnahmerring und einem sich mit Hilfe eines Synchronmotors um den Kreismittelpunkt drehenden Zeiger, der mit drei Schleifkontakten versehen ist. Am Anfang der Schleifbahn sitzt ein Kontaktpaar, welches die x-Ablenkung eines auf "Single Sweep" eingestellten Oszillographen auslöst. Der Eingang für die y-Ablenkung des Oszillographen ist mit den Buchsen "Signal Output" des Hauptgerätes verbunden. Durch die geschilderte Steuerung der x-Ablenkung des Oszillographen wird gewährleistet, daß die Kernresonanzsignale auf dem Oszillographenschirm deckungsgleich dargestellt werden. Die Höhe der Signale wird wie bei der Aufnahme eines Spektrums mit dem Schreiber bei "Signal Amplitude" geregelt.

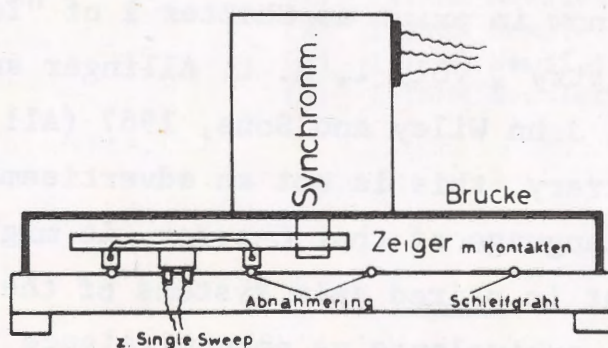
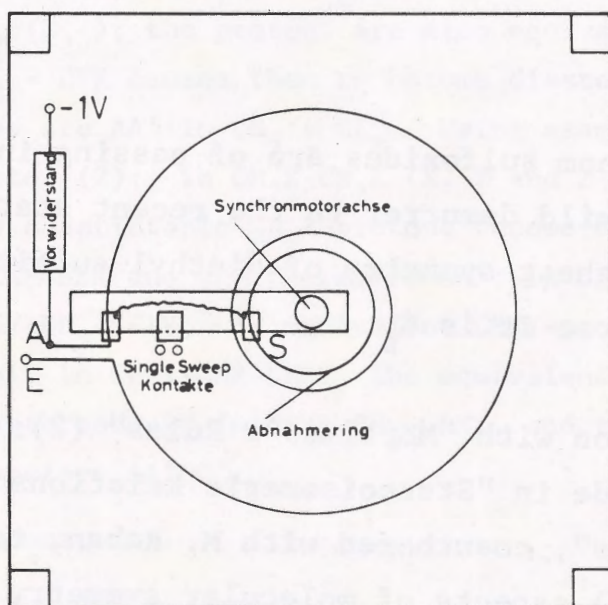
Der Schleifdraht wird über einen Vorwiderstand, an dem die Sweepweite eingestellt werden kann, an die im Hauptgerät erzeugte Sweepspannung von -1V angeschlossen. Mit Hilfe eines am A60-Spektrometer eingebauten Umschalters kann diese Widerstandskombination an Stelle des regulären Sweeppotentiometers in den Meßkreis eingeschaltet werden. Im Abstand von drei Sekunden erscheint das Testsignal auf dem Oszillographenschirm und läßt sich so mühelos auf optimale Form, d.h. auf größte Höhe und exponentielles Abklingen der Wiggles einstellen. In besonderen Fällen, vor allem bei extrem engen Aufspaltungen, ist es sogar möglich, ein Signal selbst als Testsignal zu benutzen, was zweifellos ein optimales Ergebnis garantiert.

Während der Feldeinstellung ist der Schreiber durch Linksdrehen des "Zero"-Potentiometers abgeschaltet. Die Nachleuchtdauer eines üblichen blaugrünschriftenden Oszillographen reicht bei einer Schreibzeit von 3 s durchaus aus, um das neue Signal mit dem vorher Geschriebenen zu vergleichen.

Die Sägezahnspannung, die im vorliegenden Fall mit Hilfe eines Schleifdrahts erzeugt wird, kann selbstverständlich auch leicht mit Hilfe einer vollelektronischen Schaltung hergestellt werden.

Hecklinghoff

W. Sauer



PRINCETON UNIVERSITY
DEPARTMENT OF CHEMISTRY
PRINCETON, NEW JERSEY 08540

Frick Chemical Laboratory

AIRMAIL

September 26, 1967

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

Mortimer's Rules: A Footnote

Dear Barry:

- 1) TEARS (1)
- 2) As one to whom sulfoxides are of passing interest, I register a mild demurrer to the recent statement (2) that the highest symmetry of diethyl sulfite is "None" when of course it is C_s .
- 3) In connection with "Mortimer's Rules" (2): An attempt has been made in "Stereoisomeric Relationships of Groups in Molecules", coauthored with M. Raban, to discuss (inter alia) aspects of molecular symmetry which may be of interest to some practitioners of nmr (3). This article is now in print as Chapter 1 of "Topics in Stereochemistry", Vol. 1, N. L. Allinger and E. L. Eliel, ed., John Wiley and Sons, 1967 (All appearances to the contrary, this is not an advertisement (4)). Using the language of that Chapter, it might be useful to note that in paired spin systems of the type A_2B_2 , A_2X_2 etc., equivalence vs nonequivalence in the spin

Prof. B.L. Shapiro

-2-

September 26, 1967

coupling sense can be easily established using the substitution criterion: if substitution of one of the X's or B's by R in the conformation of highest symmetry changes the relationship of the A's so that they are no longer interconvertible by a symmetry operation, then the A's are nonequivalent in the spin-coupling sense (i.e., AA'), whereas, if substitution results in a change to a species in which the A's are interconvertible by a symmetry operation, then the A's are equivalent in the spin coupling sense (i.e., A₂). For example: in CH₂F₂ (C_{2v}), the protons are stereochemically equivalent and a change to CH₂FR causes them to become enantiotopic; therefore they are A₂ in CH₂F₂. On the other hand, in CH₂ = CF₂ (C_{2v}), the protons are also equivalent but a change to CH₂ = CFR causes them to become diastereotopic; therefore they are AA' in CH₂ = CF₂. Using examples from Reilly's letter (2): in CH₂X-CH₂Z (X, Y and Z are nmr - inactive), the enantiotopic CH₂X-protons become diastereotopic in CH₂X-CHZR and are therefore AA'; in optically active CHXY-CH₂-CHXY, the equivalent CHXY-protons become diastereotopic in CHXY-CHR-CHXY, the equivalent CH₂-protons become diastereotopic in CHXY-CH₂-CRXY, and the spin system is therefore AA'CC'.

Sincerely yours,


 Kurt Mislow

- (1) IITNMR Newsletter 103-20
- (2) IITNMR Newsletter 107-8
- (3) IITNMR Newsletter 94-8
- (4) IITNMR Newsletter 107-66

KM:ec

FACULTE DES SCIENCES
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SERVICE CHIMIE P.C 1

R. JACQUIER
Professeur

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

Tél : (67) 72-14-50

MONTPELLIER, le 27 Septembre 1967

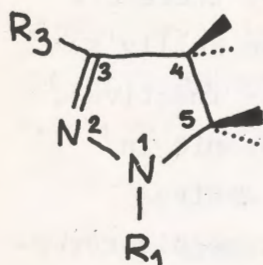
Monsieur B.L. Shapiro
Department of Chemistry
Illinois Institute of Technology
CHICAGO, Illinois 60616

UTILISATION DE L'EQUATION DE McCONNELL
DANS LA SERIE DE LA PYRAZOLINE-2

Cher Professeur Shapiro,

Nous vous prions de nous excuser pour le retard apporté à notre contribution annuelle, d'autant plus que nous tenons beaucoup à recevoir les I.I.T N-M-R Newsletters.

Pour calculer l'effet sur les déplacements chimiques, produit par le remplacement d'un hydrogène en position 4 ou 5 par un méthyle (ou un méthylène),



plusieurs auteurs (1,2,3) ont utilisé la formule de McCONNELL, en supposant $\Delta\chi^{\text{CH}} = 0$; or ApSIMON WHALEY et coll. (4) ont montré que cette hypothèse est fausse, et ont proposé un calcul qui tient compte non seulement de l'anisotropie de la liaison C-H, mais aussi d'un terme correctif en R^{-5} correspondant à un dipôle non ponctuel.

Ayant étudié un nombre élevé de pyrazolines-2 diversement substituées, nous avons pu déterminer les effets moyens suivants , (exprimés en ppm) :

	H cis	H trans
Effet d'un méthyle en position 4 sur	+ 0,42 \pm 0,08	- 0,16 \pm 0,06
Effet d'un méthyle en position 5 sur	+ 0,38 \pm 0,04	- 0,20 \pm 0,06
Valeurs moyennes	+ 0,40	- 0,18

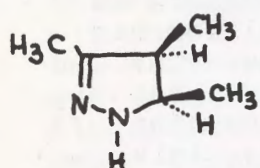
L'effet sur le proton H gem varie dans des limites assez larges (-0,18 à -0,63 ppm), et il ne nous semble pas pouvoir être calculé sans tenir compte pour H_5 des interactions entre R_1 et le méthyle en 5.

On peut essayer de calculer ces déplacements avec les équations d'ApSIMON, WHALLEY et coll. (4), mais c'est laborieux et somme toute les résultats obtenus par les auteurs ne sont pas très satisfaisants.

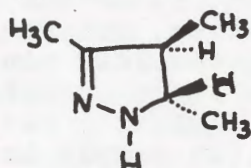
On peut enfin, connaissant le terme géométrique $1-3 \cos^2 \theta / R^3$, essayer de déterminer un paramètre M qui multiplié par le terme géométrique donne l'effet observé : on trouve pour les pyrazolines-2, $M = 15 \cdot 10^{-30} \text{ cm}^3 \text{ molécule}^{-1}$.

M n'a pas de sens physique, mais de la façon dont il est calculé, il correspond au $\Delta\chi^{C-C}$ des auteurs qui prenaient $\Delta\chi^{C-H} = 0$; il est donc intéressant de remarquer qu'il se rapproche de la valeur de REDDY et GOLDSTEIN [16,6. 10^{-30} (7)], plutôt que de celle de MUSHER [8,3. 10^{-30} (8)].

Cette valeur de M nous a permis de déterminer la configuration du couple des triméthyl-3,4,5 pyrazolines-2 isomères :



$$J_{45} = 9 \text{ Hz.}$$



$$J_{45} = 10 \text{ Hz.}$$

les résultats montrent qu'il n'est pas toujours possible d'appliquer le critère généralement admis pour déterminer la stéréochimie des positions 4,5 dans les pyrazolines-2 ($J_{\text{cis}} > J_{\text{trans}}$).

lines-2 ($J_{\text{cis}} > J_{\text{trans}}$).

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

R. JACQUIER

J. ELGUERO

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- (4) J.W. ApSIMON, W.G. CRAIG, P.V. DEMARCO, D.W. MATHIESON, L. SAUNDERS et W.B. WHALLEY, tetrahedron, 1967, 23, 2339.
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- (6) J.-R. DIDRY et F. CABARET, C.R. Acad. Sci., 1963, 257, 1466.
- (7) G.S. REDDY et J.H. GOLDSTEIN, J. chem. Phys., 1963, 38, 2736.
- (8) J.I. MUSHER, J. chem. Phys., 1961, 35, 1159 ; Mol. Phys., 1962, 6, 93.

Greenford · Middlesex

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

21st August, 1967.

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

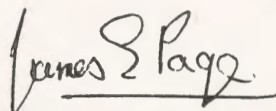
Dear Dr. Shapiro,

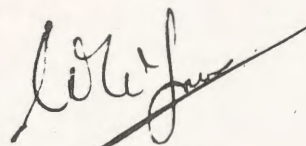
Varian A60 Spectrometer and Storage of N.M.R. Solvents

Our Varian A60 spectrometer after over five years of heavy use continues to be generally efficient, but we have had increasing trouble from bad contacts between valve pins and sockets, particularly in the control loop. The bad contacts gave intermittent trouble with the field lock and made the instrument sensitive to mains transients. Replacement (by a Varian engineer) of the valve holders in the transmitter, control receiver, and field modulator has given better stability during the last nine months. The integrator D.C. amplifier now needs attention and it has become necessary to run as a linearity test each morning an unbalanced integral trace (no sample in probe); this trace should, except for regular, small-amplitude peaks caused by pulses in the electrical mains supply, be a straight line. We were, therefore, interested by R.H. Elskén and R.E. Lundin's report (IITNMR Newsletter, 1966, (99), p. 50) on the replacement of the USA-3 valve amplifier in a Varian V-3521 N.M.R. integrator by a Fairchild solid state amplifier and would like to hear of an A60 spectrometer that has been modified in this way.

Readers may be interested in the routine methods that we use for storing n.m.r. solvents. We store all solvents except acids over molecular sieves. This not only dries, but also appears to stabilise (presumably by removing traces of acid) deuteriochloroform and carbon tetrachloride. The absence of acid from these solvents is demonstrated by the good splitting obtained when the solvents are used for solution-spectra of simple alcohols (e.g. methanol and ethanol), in which spin-spin coupling can occur between hydroxyl and α -carbon protons; a trace of acid in the solvent would catalyse exchange of hydroxyl protons and prevent the appearance of splitting patterns. Dimethyl sulphoxide and its hexadeuterated analogue, which are exceptionally hygroscopic and need special care, are stored in small quantities over fresh molecular sieves (1/16" pellets, Union Carbide type 4A) in special 25 ml. double-stoppered bottles (with the outer stopper greased).

Yours sincerely


Dr. J.E. Page


G.F.H. Green

UNIVERSITY OF COLORADO

BOULDER, COLORADO 80304

DEPARTMENT OF CHEMISTRY

October 3, 1967

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

Dear Dr. Shapiro:

We just obtained a HA-100 spectrometer a few months ago and much of our effort has been devoted to its operation rather than "unpublishable" results. We have looked at the C^{13} satellite spectrum of hexafluoroethane at -90° (just below the boiling point) and observed several very nice quartets which are obviously due to the vicinal fluorine-fluorine coupling. The vicinal coupling is sufficiently small compared to the "chemical shifts" that the spectrum can be analyzed as a A_3MX_3 type instead of the A_3XB_3 case which Johannesen, Farrar, Brinckman, and Coyle (J. Chem. Phys., 44, 962 (1966)) observed for the similar compounds $F_3^{28}Si^{29}SiF_3$. J_{FF} in $F_3^{12}C^{13}CF_3$ is 3.5 Hz. We claim, according to the equation for coupling constants vs. electronegativity (Ng and Sederholm, J. Chem. Phys., 40, 2090 (1964) or Abraham and Cavelli, Mol. Phys., 9, 67 (1965)) that this is even an example of a positive vicinal fluorine-fluorine coupling. (All other known vicinal FF couplings are negative). We hope to have spin decoupling equipment available in the near future to prove this.

We are still in the process of obtaining data on the internal rotation of halogenated ethanes (Newmark and Sederholm, J. Chem. Phys., 43, 602 (1965)). Our first project in this direction at Colorado is to compare the barriers in $CHClBr-Cl_2Br$ and $CFClBr-CCl_2Br$ in several solvents. We hope to achieve a reasonable control over the temperature by using internal capillaries of methanol (for the proton experiments) or a pair of saturated and unsaturated compounds for the fluorine experiments (e.g., the fluorine chemical shift between perfluorobenzene or $CF_2=CCl_2$ and $CFCl_3$ or CF_2Cl_2 changes about 0.8 Hz/degree at 94 MHz).

Sincerely yours,

Richard

Richard A. Newmark

RAN:rm

The University of Newcastle upon Tyne

School of Chemistry

The University
Newcastle upon Tyne 1

Telephone 28511

Department of Inorganic Chemistry

JWA/BMD.

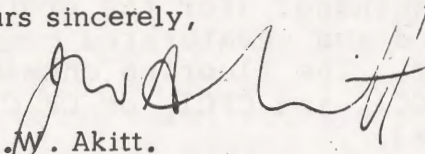
28th September 1967.

Dear Professor Shapiro, Al^{27} NMR of $\text{Al}_2(\text{SO}_4)_3$ Solutions.

The Al^{27} NMR signal of the hexa-aquo aluminium ion in different salt solutions has been reported by several workers as having a single line with a linewidth of 40 to 50 cps^{1,2,3}. We have found that by using a high resolution magnet and working in the absorption mode it is possible to obtain a linewidth of about 10 cps in dilute (i.e. the least viscous) aluminium sulphate solutions. The addition of acid produces progressive line narrowing, the linewidth varying exponentially with acid concentration until in the presence of excess acid the linewidth is reduced to about two fifths the width obtained from an aluminium sulphate solution of similar viscosity. Thus Al^{27} lines as narrow as 4 cps have been observed. The narrowing due to acid seems to be due to acid catalysed proton exchange involving the hydration water hydrogens, since water exchange is known to be too slow to affect quadrupole relaxation seriously.

Under the conditions of resolution attained in this work a second weak resonance can be seen 3.3 ppm to high field of the hexa aquo ion resonance. This indicates the presence of a second long lived ionic species. Its linewidth is also reduced on the addition of acid and its relative intensity can be increased by the addition of sulphuric acid so that at 60% v/v acid it accounts for 67% of the total aluminium present. The concentration of the new species is found to depend upon the bisulphate ion concentration in these solutions and it appears that it probably has the formula $\text{Al}(\text{H}_2\text{O})_{6-n}(\text{HSO}_4)_n^{(3-n)+}$ where $n = 1$ or 2 . The linewidth of the species is about 30% greater than that of the hexa-aquo aluminium ion and this is in accord with relaxation being induced by acid catalysed proton exchange since in the new species there are between 17% and 33% fewer protons available for exchange.

Yours sincerely,



J.W. Akitt.

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3. W.G. Movius, and N.A. Matwiyoff, Inorg. Chem., 1967, 6, 847.

Professor B.L. Shapiro,
Department of Chemistry,
Stanford University, Stanford,
California 94305, U.S.A.



Department of Chemistry

 5151 State College Drive, Los Angeles, California 90032
 (San Bernardino and Long Beach Freeways Interchange)

September 28, 1968

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

Proton Magnetic Resonance Coordination Number Measurements

Dear Barry:

As we recently demonstrated (J. Chem. Phys., 47, 1554 (1967); Tet.Let., (in press), at low temperatures, in aqueous solutions of Al (III) salts, separate proton resonance signals can be observed for bulk water and water molecules in the cation solvation shell. The same phenomenon had already been reported for a variety of non-aqueous solvents. Thus, by direct integration, coordination numbers can be measured. We have used this technique to study solvation in aqueous solvent mixtures. The results for aqueous mixtures of acetone (A) and dimethylsulfoxide (DMSO) are listed below.

Table I. Al (III) Coordination Numbers in Aqueous Solvent Mixtures

Solvent Mole Ratios	AlCl ₃ Concentration (moles/liter)	Al (III) Coordination Numbers H ₂ O	Organic Component
<hr/>			
H ₂ O/DMSO			
10:1	2.00	5.01 0.10	0.92 0.01
10:1	1.00	5.63 0.26	0.53 0.04
5:1	1.80	4.12 0.17	1.69 0.02
5:1	1.00	4.97 0.13	1.28 0.06
3.5:1	1.50	3.56 0.10	2.02 0.03
3.5:1	1.00	4.06 0.12	1.92 0.01
2:1	1.00	3.08 0.04	2.61 0.01

H₂O/A

10:1	2.00	6.3 0.3
10:1	1.75	5.7 0.1
10:1	1.50	5.8 0.1
10:1	1.00	6.1 0.2
7.5:1	1.50	5.8 0.1
7.5:1	1.00	6.0 0.1
5:1	0.70	5.8 0.1
5:1	0.40	5.6 0.2
3.5:1	0.50	5.7 0.1
2.5:1	0.30	6.0 0.1

As the data illustrate, DMSO is able to replace water in the Al (III) solvation shell while acetone is not.

In both systems, contact ion-pairing is negligible at these salt concentrations. It is interesting to note that although acetone and DMSO are both quite polar, only the later exhibits a solvating ability. DMSO is much more basic than acetone, however, and perhaps this property is more important.

We have also completed coordination number studies in aqueous mixtures of dimethylformamide, and tetramethylurea, and to a small extent, dioxane and tetrahydrofuran. The measurements are now being extended to other aqueous solvent mixtures. In addition we are studying the coordination behavior of other cations in water by this direct integration, p.m.r. technique. In progress also, are kinetic studies of the proton exchange processes occurring in these solutions. Reprints describing some of this work are available and will be sent to those requesting them.

Sincerely,

Tony

Anthony Fratiello

AF:tn

MCMMASTER UNIVERSITY

HAMILTON COLLEGE

HAMILTON, ONTARIO, CANADA

DEPARTMENT OF CHEMISTRY

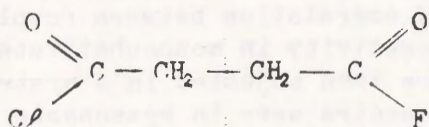
August 25, 1967

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

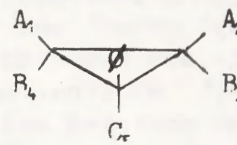
Dear Barry,

We recently received a Fortran deck of Bothner-By and Castellano's program LAOCN3 from the Mellon Institute. Before using this deck on our computer installation it was necessary to replace the Fortran II functions XMINOF, ABSF and SQRTF by the corresponding Fortran IV functions MINO, ABS and SORT - an interesting point, since the Fortran listing in the booklet accompanying the program has the Fortran IV functions! At this point we found LAOCN3 overflowed the storage capacity of our IBM 7040 by approximately 4000 locations. We therefore simply reduced the array sizes in the dimension statements of the program to those adequate for a six spin system or less. Reduction from seven to six spins saves approximately 10,000 locations, making the program executable on our computer.

We have thus far used the amended version of LAOCN3 twice, both times with iteration. In the first case, an iterative fit was obtained for the proton spectrum of succinyl chloride fluoride (I).



I



II

This spectrum has previously been analyzed in this laboratory by subspectral analysis¹ as the AA'BB' part of an AA'BB'X spectrum. Subspectral analysis yields a proton spectrum consisting of two overlapping aa'bb' subspectra giving² $\nu_{\text{O}} \delta_{\text{AB}} = 23.16$, $J_{\text{HF}} = 2.22$, $J_{\text{HF}}' = -0.30$, $J_{\text{HH}} = 5.36$, $J_{\text{HH}}' = 7.51$ and $J_{\text{gem}} = J_{\text{gem}}' = -20$ c/sec. These values were used as input parameters for a LAOCN3 iterative fit.

This gave $\nu_{\delta} = 23.04 \pm 0.03$, $J_{HF} = 2.13 \pm 0.03$, $J_{HF}' = -0.34 \pm 0.03$, $J_{HH} = 5.43 \pm 0.03$, $J_{HH}' = 7.50 \pm 0.02$ and $J_{gem} = J_{gem}' = -65 \pm 140$ c/sec., essentially the same as previously obtained. The large value obtained for J_{gem} is not particularly significant, since the spectrum is affected very little by this parameter.

We have also used LAOCN3 to analyze the 60 and 100 Mc/sec spectra of phenylcyclopropane (II) in the cyclopropyl region from scratch. The phenyl coupling to the cyclopropyl ring is small, hence an AA'BB'C spectrum for the cyclopropyl protons results. The spectra, shown in the accompanying figures, analyze to give the chemical shifts (from dioxane as internal reference) and coupling constants shown below

	<u>60 Mc/sec</u>	<u>100 Mc/sec</u>
ν_A	171.21 c/sec = 2.854 ppm	282.84 c/sec = 2.828 ppm
ν_B	160.23 = 2.671	264.49 = 2.645
ν_C	102.85 = 1.714	170.60 = 1.706
J_{12}	9.31 c/sec	9.45 c/sec
J_{13}	6.26	6.30
J_{14}	-4.50	-4.46
J_{34}	9.41	9.46
J_{15}	5.19	5.20
J_{35}	8.51	8.39

The 60 Mc/sec spectrum was analyzed by first calculating a trial spectrum using chemical shifts estimated from the experimental spectrum and coupling constants estimated by using electronegativity $E = 2.75$ for the phenyl group³ and the published correlation between coupling constants and substituent electronegativity in monosubstituted cyclopropanes.⁴ Spectral parameters were then adjusted in a systematic manner until the observed and calculated spectra were in reasonable agreement. Finally, iteration using 24 well resolved lines gave the "best values" given above. We found an iterative fit could be attempted with success only when very close to the final solution, as the interchange of line identification numbers occurs while approaching a solution. We feel this limits the usefulness of iteration in this case. The 100 Mc/sec spectrum was analyzed by using the chemical shifts and coupling constants

Dr. Bernard L. Shapiro

3

August 25, 1967

obtained from the 60 Mc/sec spectrum as initial parameters for an iterative fit. The two sets of parameters are in reasonable agreement.

James Bacon

J. Bacon

R. J. Gillespie

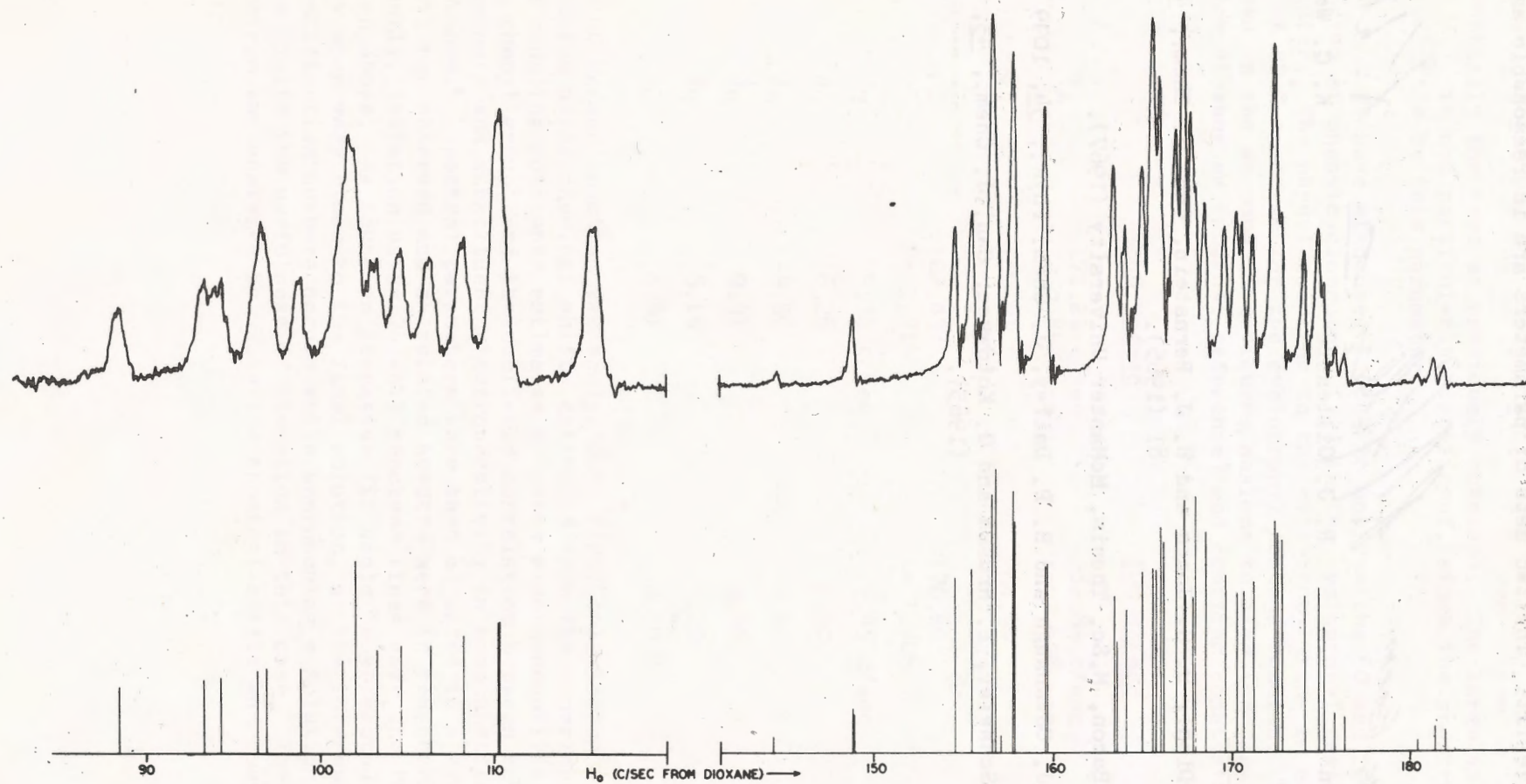
R. J. Gillespie

K. C. Westaway

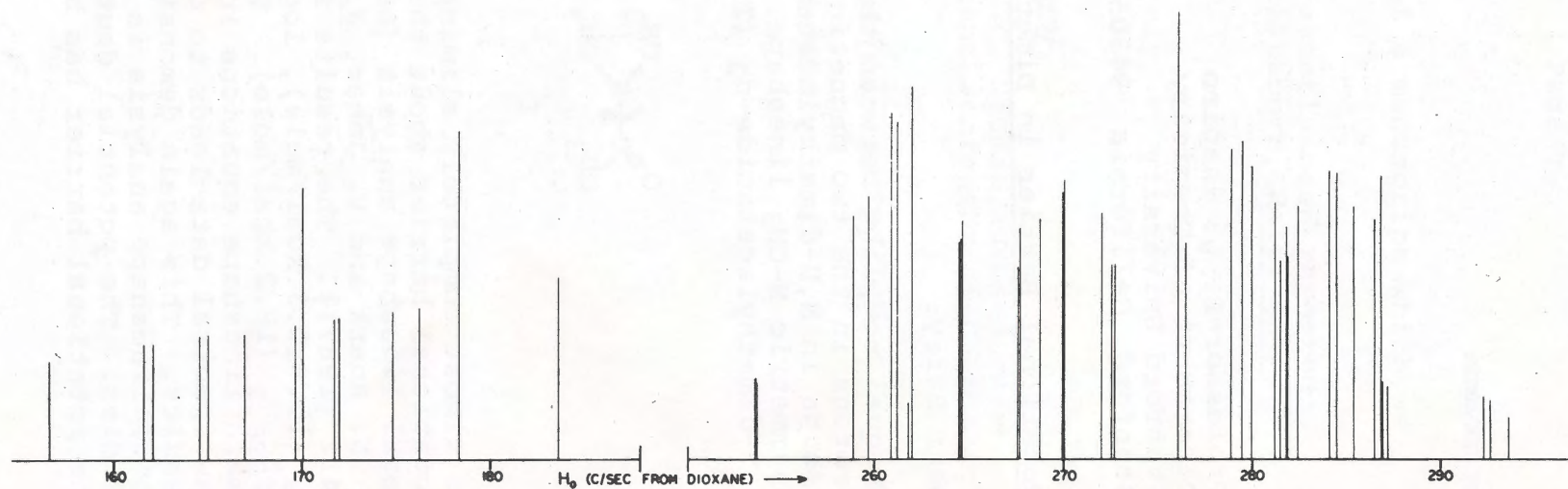
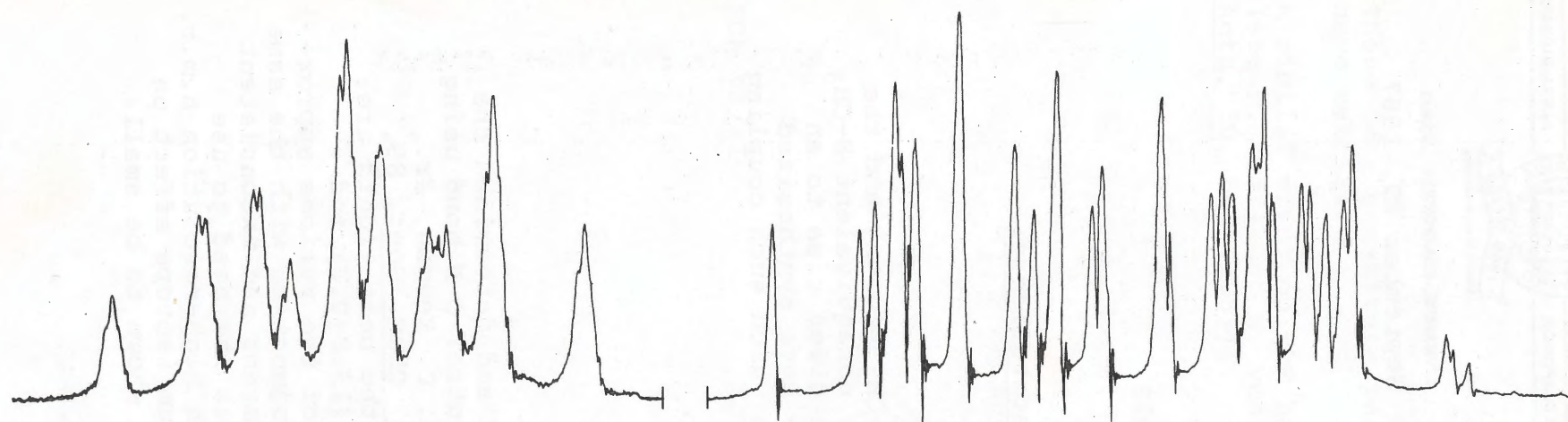
K. C. Westaway

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4. T. Schaefer, F. Hruska and G. Kotowycz, Can. J. Chem., 43, 75 (1965).



OBSERVED AND CALCULATED 60 MC CYCLOPROPANE SPECTRA



OBSERVED AND CALCULATED 100 MC CYCLOPROPANE SPECTRA

UNIVERSITY OF CALIFORNIA, RIVERSIDE

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DEPARTMENT OF CHEMISTRY

RIVERSIDE, CALIFORNIA 92502

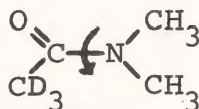
September 29, 1967

Professor B. L. Shapiro
 Department of Chemistry
 Stanford University
 Stanford, California 94305

Rotational Barrier in Dimethylacetamide-d₃

Dear Barry:

Unequal coupling between the C-CH₃ protons and the protons in the two magnetically nonequivalent N-CH₃ groups in N,N-dimethylacetamide gives rise to an asymmetric N-CH₃ lineshape. We have synthesized N,N-dimethylacetamide-d₃ (I) in which such coupling



I

is almost completely eliminated and determined the rotational barrier about the central C-N bond using total lineshape analysis (see R. C. Neuman, Jr., D. N. Roark and V. Jonas, J. Am. Chem. Soc., 89, 3412 (1967)). The results for the neat liquid are: E_a (19.6±0.3 kcal/mole), log A (13.8±0.2) and $\Delta F_{298.2}^*$ (18.2 kcal/mole). Use of the various approximate lineshape equations in conjunction with the same raw spectral data leads to different and inconsistent results. This again demonstrates the need to use total lineshape analysis in such high-resolution n.m.r. studies. The potential deuterium isotope effect on the rotational barrier has been shown to be small.

Professor B. L. Shapiro
September 29, 1967
Page Two

These data constitute part of a manuscript which we have submitted.

A similar study on DMF has recently been reported (see F. Conti and W. von Philipsborn, Helv. Chim. Acta, 50, 603 (1967)).

Sincerely,

Bob

Robert C. Neuman, Jr.
Assistant Professor of Chemistry

Violet Jonas

Violet Jonas

RCN/VJ/nl



University of Strathclyde

Department of Pure and Applied Chemistry

Thomas Graham Building Cathedral Street, Glasgow, C.1
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27th September, 1967.

Associate, Professor B. L. Shapiro,
Department of Chemistry
University of Stanford,
Stanford,
California 94305.

Dear Barry,

In collaboration with Professor D. W. A. Sharp we have been looking at the ^{19}F spectra of some monosubstituted pentafluorotellurium compounds [i.e. TeF_5R and $(\text{TeF}_5)_2\text{R}$]. The compounds themselves were made by Dr. A. Engelbrecht of the University of Innsbruck. The arrangement of groups round the tellurium atom is octahedral (as in the analogous SF_5R compounds) and preliminary analysis of the spectra can be made on the basis of the usual AB_4 or AB_4X algebraic calculations (of the corresponding SF_5R compounds¹). In all cases the resonances of fluorine attached to tellurium are in the region $\delta = +36$ to $+52$ p.p.m.; the apex fluorine being to higher field except in one case (TeF_5OH). The results are shown in the table.

$\text{TeF}_5\text{O} \cdot \text{SO}_2\text{F}$ is an interesting case in that the SO_2F fluorine is essentially coupled to the four equatorial fluorines only. Despite the small ratio ($J_{\text{BX}}/\delta_{\text{BX}} = 0.00087$) the X-multiplet is sufficiently asymmetrical to allow the sign of J_{BX} relative to J_{AB} to be determined, cf. Ref. 2 (the line at highest field in the quintuplet is split into three smaller lines). When we did a complete six spin calculation using LACON 3 the asymmetry of this multiplet was only correctly reproduced if J_{BX} was given a sign opposite to that of J_{AB} . (Like signs resulted in the mirror image of the multiplet). Computations on the other spectra are currently under way.

Compound	ϕ_A (apex F)	ϕ_B (equatorial F)	ϕ_X	J_{AB}	J_{AX}	J_{BX}
$\text{TeF}_5\text{O} \cdot \text{SO}_2\text{Cl}$	+48.4	+36.7	-	± 177.3	-	-
$\text{TeF}_5\text{O} \cdot \text{SO}_2\text{F}$	+52.33	+40.33	-49.38	± 178.9	± 0.19	± 2.95
$(\text{TeF}_5\text{O})_2\text{SO}_2$	+51.62	+40.00	-	± 177.7	$J_{BB'}$ ~ 1.0	$J_{AA'}$ ~ 0.0
TeF_5OH 50% in CCl_4	+44.5	+47.0	γ_H 4.42	± 182.2	-	-
$(\text{TeF}_5)_2\text{O}$	+48.1	+37.5	-	± 176.3	-	-

All results (except one) refer to neat liquids. Chemical shifts in p.p.m. w.r.t. internal CCl_3F . Coupling constants in Hz . All results from algebraic calculations except for $\text{TeF}_5\text{O} \cdot \text{SO}_2\text{F}$ which are computer refined.

We have had to modify LAOCOON 3 slightly to get it to work on our University's ICT 1905. The restricted store (32K) means that we can only do 6 spins but otherwise it works well. In addition we have written a plotting program NMRPLOT1900 which uses as input the optional tape or card output of LAOCOON 3. Details of NMRPLOT1900 and listings will be sent to anyone interested.

Yours sincerely,

P. Bladen

K.D. Crosbie

G.C. Forrest

P. Bladen.

K. D. Crosbie.

G. C. Forrest.

1. R. K. Harris and K. J. Packer J. Chem. Soc., 1961, 4736, 1962, 3077.
2. P. L. Corio, "Structure of High Resolution N.M.R. Spectra" Academic Press, New York. 1966 p.298.

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DEPARTMENT OF CHEMISTRY

September 26, 1967

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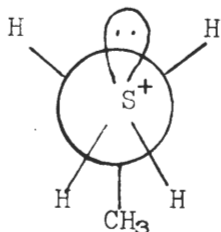
Dr. Bernard L. Shapiro
IIT NMR NEWSLETTER
Department of Chemistry
Stanford University
Stanford, California 94305

Dear Barry:

I was away for the summer and just now catching up with my back-log of mail. Thanks for being patient. I hope some recent findings will be of interest to your readers.

Some interesting features have emerged from our recent investigations of the nmr spectra of protonated sulfur compounds.

We have recently protonated thiols and sulfides in $\text{FSO}_3\text{H-SbF}_5\text{-SO}_2$ solution at low temperatures and find, in several cases, effects of molecular asymmetry leading to magnetic non-equivalence. Thus, in protonated diisopropyl sulfide the methyl groups are non-equivalent up to at least $+70^\circ$ in $\text{FSO}_3\text{H-SbF}_5$ (see G. A. Olah, D. H. O'Brien and C. U. Pittman, Jr., J. Am. Chem. Soc., 89, 2996 [1967]). Another example is protonated ethane thiol. We have recently decoupled the methyl protons from the methylene protons and find that the resulting spectrum constitutes an AA' XX' case. This we feel implies that the trans form is the preferred conformer and that the rate of inversion through positive sulfur is slow on the nmr time scale. This latter observation is not unexpected since optically active sulfonium ions are well known (for a recent discussion see R. Scartazzini and K. Mislow, Tet. Let. 28, 2719 [1967]).



CH_3 δ 1.48 (ppm from ext.
TMS)
 CH_2 3.37
 SH_2^+ 6.22

trans conformer of protonated
ethane thiol

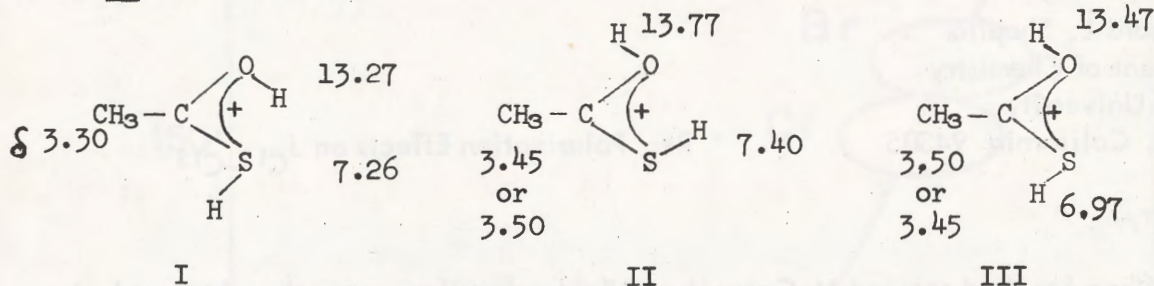
This result, and the spectra is reminiscent of the observations on primary Grignard reagents by Roberts and Whitesides (J. Am. Chem. Soc., 87, 4878 [1965]). We are at present analyzing this spectrum (which is unchanged from -80° up to at least 0°) with the assistance of LAOCN3 and a CalComp plotter and are investigating some further examples in this area.



2

September 26, 1967

In a related field we have found that protonated thiolacetic acid (at -80° in $\text{FSO}_3\text{H-SbF}_5\text{-SO}_2$) exists in three isomeric forms analogous to those found in protonated acetic and formic acids (see G. A. Olah and A. M. White, J. Am. Chem. Soc., 89, 3591 [1967]).



Isomer I is the predominate species observed, the approximate proportions being in 3:1:1 for I, II and III respectively. Of particular interest is isomer III in which there is a ⁴-bond coupling of 3.50 Hz between the OH and SH protons. The magnitude of this coupling evidently reflects the favorable planar zig-zag relationship of these protons to one another.

Yours sincerely,

George A. Olah
Anthony M. White

THE UNIVERSITY OF UTAH

SALT LAKE CITY 84112

DEPARTMENT OF CHEMISTRY
CHEMISTRY BUILDING

October 6, 1967

Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305Re: Polarization Effects on $J_{C^{13}-C^{13}}$


Dear Barry,

When Stanford secured McConnell and Baldeschweiler, many thought they had acquired their fair share of the world's resonators, and now I find they have captured the mentor of the group's official voice. If the trend persists, there is real danger that the center of gravity of magnetic resonators may cross from Palo Alto into Stanford, and how will we all adjust to the price increases resulting from such a catastrophe?

The accompanying figure from the work of Dr. William Litchman indicates that $J_{C^{13}-C^{13}}$ values are affected by polarizing groups in a manner similar to that noted for $J_{C^{13}-H}$ values. It is interesting to speculate, however, on the fact that the relative change ($25/125$) in $J_{C^{13}-H}$ is about twice as great as observed ($4/36$) for $J_{C^{13}-C^{13}}$. One is led to conclude that the electronic structure about both coupled nuclei is affected by the electronegative substituent and not merely the carbon atom which is bonded to the polar group. Otherwise, the two couplings might be expected to increase by the same relative amount. As inductive effects may be transferred on to additional atoms by an α -carbon atom, it is not unreasonable to expect that $J_{C^{13}-C^{13}}$ should be less sensitive to polar effects than observed for $J_{C^{13}-H}$. The reduced sensitivity of $J_{C^{13}-C^{13}}$ to charge polarization effects is fortunate as one can place even greater confidence in the use of $J_{C^{13}-C^{13}}$ values as a criteria for bond hybridization [Lynden-Bell and Sheppard (1962) and Frei and Bernstein 1963] than one is able to place in the very popular $J_{C^{13}-H}$ criteria of hybridization.

The data were secured with our AFS-60 spectrometer, using both time averaging and proton decoupling techniques. The Carbon-13 isotope appeared only in natural abundance, thereby obviating the need for isotope labeling procedures.

Sincerely yours,


David M. Grant,
Professor of Chemistry

DMG:vla

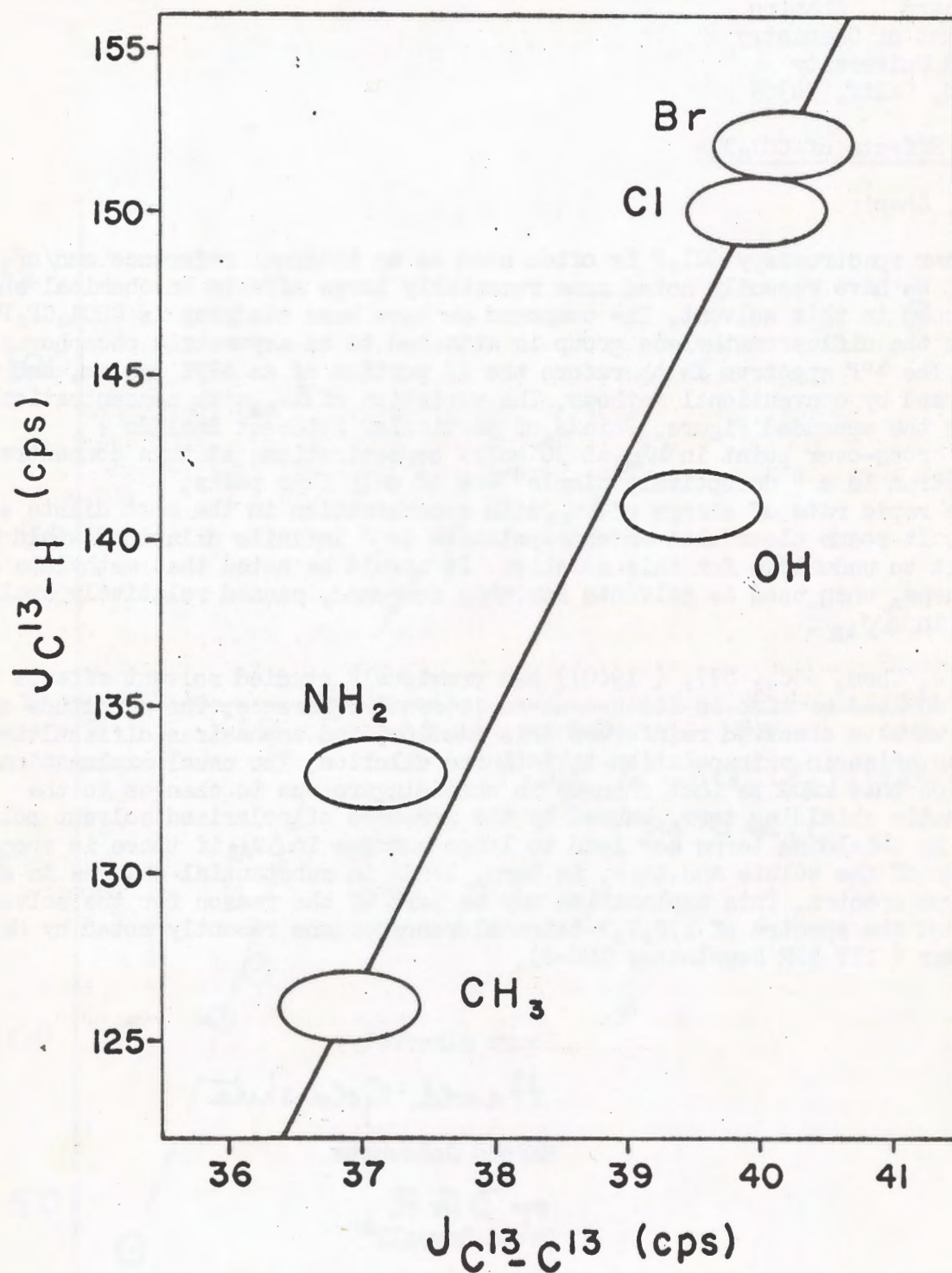


Fig. 1. The values of $J_{C^{13}-H}$ in $X-CH_3$ are plotted vs. $J_{C^{13}-C^{13}}$ in the similar $X-C(CH_3)_3$ compounds for X equal to CH_3 , NH_2 , OH , Cl , and Br .



Department of Chemistry

5151 State College Drive, Los Angeles, California 90032
(San Bernardino and Long Beach Freeways Interchange)

4 Oct. 1967

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

Solvent Effects of CCl_3F .

Dear Dr. Shapiro:

In ^{19}F nmr spectroscopy CCl_3F is often used as an internal reference and/or solvent. We have recently noted some remarkably large effects on chemical shift differences in this solvent. The compound we have been studying is $\text{CHCl}_2\text{CF}_2\text{P}(\text{Cl})(\text{N}(\text{CH}_3)_2)$ in which the difluoromethylene group is attached to an asymmetric phosphorus center. The ^{19}F spectrum is therefore the AB portion of an ABPX system, and can be analysed by conventional methods. The variation of $\Delta\nu_{\text{AB}}$ with concentration is shown in the appended Figure. Points of particular interest include:

- (i) the cross-over point in $\Delta\nu_{\text{AB}}$ at 30 mole% concentration; at this concentration the spectrum is a "deceptively simple" one of only four peaks;
- (ii) the rapid rate of change of $\Delta\nu_{\text{AB}}$ with concentration in the most dilute solutions studied. It seems clear that an extrapolation to "infinite dilution" would be difficult to undertake for this solution. It should be noted that methylene chloride and toluene, when used as solvents for this compound, caused relatively small changes in $\Delta\nu_{\text{AB}}$.

Evans (J. Chem. Soc., 877, (1960)) has previously studied solvent effects of CCl_3F and has advised caution in its use as an internal reference. The magnitude of the changes we have observed reinforces this caution, and emphasizes difficulties which can arise in extrapolation to infinite dilution. The usual explanation of effects of this kind is that changes in shielding are due to changes in the paramagnetic shielding term, caused by the presence of polarized solvent molecules. Changes in shielding terms may lead to large changes in $\Delta\nu_{\text{AB}}$ if there is specific solvation of the solute and this, in turn, leads to substantial changes in appearance of AB type spectra. This explanation may be part of the reason for the solvent effects on the spectra of 1,2,7,7-tetrachloronorborene recently noted by Marchand and Weimar (IIT NMR Newsletter 108-2).

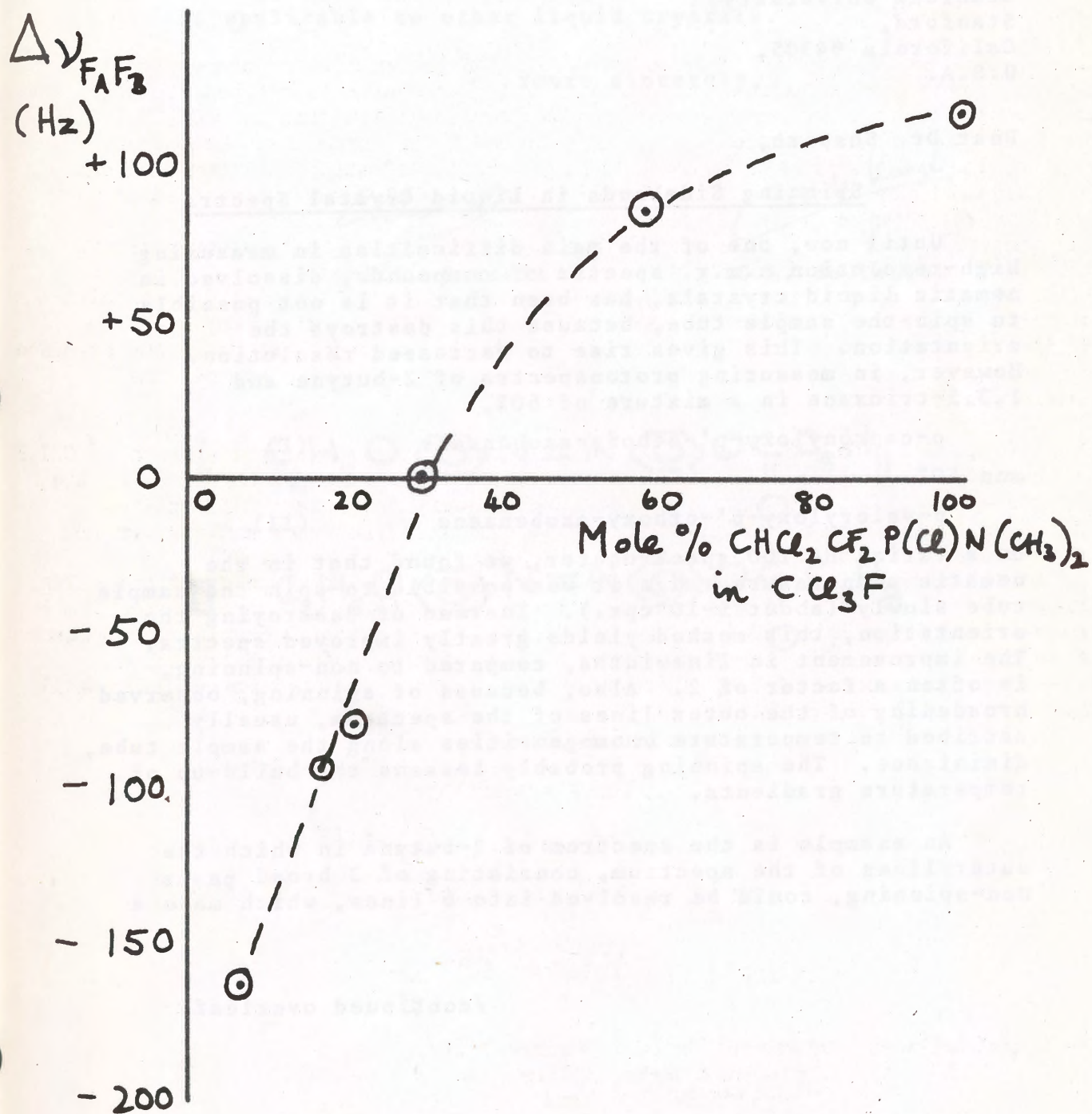
Yours sincerely,

Harold Goldwhite

Harold Goldwhite

PP DGR
David Rowsell*

* Present address: Wilkinson Sword (Research) Ltd.
Poyle Mill Works, Colnbrook
Slough, Bucks, England



University of Bristol

School of Chemistry

Telephone: Bristol 24161

Department of Theoretical Chemistry
Bristol 8
England

2nd October, 1967

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

Dear Dr. Shapiro,

Spinning Sidebands in Liquid Crystal Spectra

Until now, one of the main difficulties in measuring high-resolution n.m.r. spectra of compounds, dissolved in nematic liquid crystals, has been that it is not possible to spin the sample tube, because this destroys the orientation. This gives rise to decreased resolution. However, in measuring proton spectra of 2-butyne and 1,3,5-trioxane in a mixture of 60%,

p-capronyloxy-p'-ethoxy-azobenzene (I)
and 40%

p-valeryloxy-p'-ethoxy-azobenzene (II)
on a Varian HA 100 spectrometer, we found that in the nematic temperature range it was possible to spin the sample tube slowly (about 5-10 cps.). Instead of destroying the orientation, this method yields greatly improved spectra. The improvement in linewidths, compared to non-spinning, is often a factor of 2. Also, because of spinning, observed broadening of the outer lines of the spectrum, usually ascribed to temperature inhomogeneities along the sample tube, diminishes. The spinning probably lessens the build-up of temperature gradients.

An example is the spectrum of 2-butyne in which the outer lines of the spectrum, consisting of 3 broad peaks non-spinning, could be resolved into 6 lines, which made a

/continued overleaf:

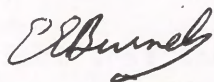
Dr. B.L. Shapiro2nd October, 1967

more accurate assignment possible.

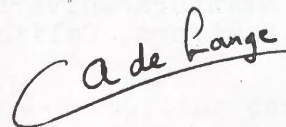
It is very important to keep the spinning speed as constant as possible, because the orientation parameter of the solute in the liquid crystal depends on this, which can give rise to considerable errors.

We have not tested yet whether or not this method is applicable to other liquid crystals.

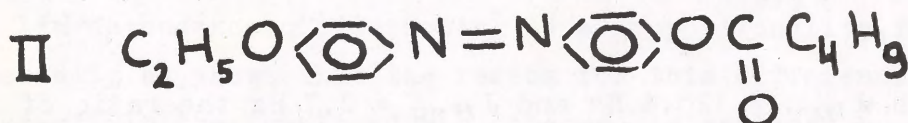
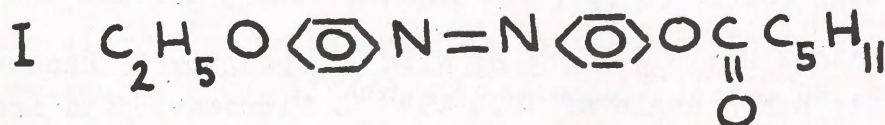
Yours sincerely,



E.E. Burnell



C.A. de Lange



COMUNITA EUROPEA
DELL'ENERGIA ATOMICA

La Commission
CENTRO COMUNE DI RICERCA



COMMUNAUTE EUROPEENNE
DE L'ENERGIE ATOMIQUE

La Commission
CENTRE COMMUN DE RECHERCHE

EURATOM

EUROPÄISCHE
ATOMGEMEINSCHAFT

Die Kommission
GEMEINSAMES FORSCHUNGSZENTRUM
Magnetic Resonance
Dr. H. Spiesscke

C C R

ISPRA
(ITALIA)

EUROPESE GEMEENSCHAP
VOOR ATOOMENERGIE

De Commissie
GEMEENSCHAPPELIJK CENTRUM
VOOR ONDERZOEK

September 29, 1967

VIA AEREA

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

Dear Barry:

In an attempt to determine the complete geometric structure of methylisocyanide I started with $^{13}\text{CH}_3\text{NC}$. Since the synthesis of $\text{CH}_3\text{N}^{13}\text{C}$ will be delayed here are some preliminary results obtained with the first species.

$P_{\text{HH}} = 1054 \text{ Hz}$; $B_{\text{CH}} = 784 \text{ Hz}$ with J_{CH} positive. These values lead to a HCH angle of $109^\circ 45' (1)$. Microwave data from Gordy as cited by Sutton's tables give $109^\circ 46'$ which compares very well.

With $\nu_{\text{NH}} = 128,4 \text{ Hz}$ and $J_{\text{NH}} = 2,7 \text{ Hz}$ the ratio of r_{CN} to r_{CH} is 1,290 assuming a negative sign for J_{NH} . The corresponding ratio from microwave data (r_0) is 1,304 whilst Costain's r_s values give 1,291!

Best wishes

Yours

Filmar

(1) A. Saupe, G. Englert, A. Povh, Advances in Chemistry Series, No. 63 "Ordered Fluids and Liquid Crystals" (1967)

October 2nd, 1967

Professor B.L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305
U.S.A.

Dear Professor Shapiro

M. O. CALCULATIONS OF SUBSTITUENT EFFECTS ON DIRECTLY BONDED
¹³C-H COUPLING CONSTANTS

The fact that the experimental ¹³C-H coupling constants in the simple hydrocarbons are almost rigorously proportional to the s-characters of the sp³, sp² and sp carbon hybrid orbitals explains that the localized m.o. description of the CH bonds should have been so widely accepted as valid. However, as the delocalized m.o. calculations by van Duijneveldt, Gil and Murrell (Theor. Chem. Acta, 4, 85 (1966)) have shown, such a proportionality must be regarded as fortuitous. In fact, for the symmetrical CH_n molecules (CH₂ linear, CH₃ trigonal planar, CH₄ tetrahedral), the delocalized m.o. values have an approximately n^{-3/2} dependence, against the 1/n dependence corresponding to a proportionality to s-character; it can easily be shown that the reason for this difference is that $\Delta E = 2\beta_{\text{H}i\text{C}_i} \approx \text{constant}$, in the localized m.o. theory, is replaced by $\Delta E = 2\beta_{\text{H}i\text{C}_i} + 2(n-1)\beta_{\text{H}i\text{C}_j} \approx \text{constant} \times n^{-1/2}$ in the delocalized m.o. treatment (see figure). This result shows the importance of σ -electron delocalization on CH coupling constants, for, even if $\beta_{\text{H}i\text{C}_j}$ is negligible for a given CH_n molecule, it will have a significant effect on J_{CH} of the others.

That a localized orbital treatment of a CH bond, in which the criterion defining the hybrid^{is} that they should point to the nuclei bonded to C, is not adequate (for the CH coupling) is clearly demonstrated by the nitid disagreement between J_{CH} - values and bond angles in substituted hydrocarbons.

Even if a maximum localization criterion for defining the carbon hybrids is used, the residual interactions leading to delocalization will, in general, be too large to be neglected; in any case, however, the

"best hybrids" do not have to follow the bond angles. Related to this is the common misunderstanding that changes in carbon hybridization due to substituents "arise" so as to minimize the total energy; obviously, no physical quantity depends upon the nature of the hybrids chosen for a given atom; these can only affect calculated values, and this if a localized bond approximation is made.

All the above considerations show that the localized bond approach to CH coupling constants should definitely give place to the more general delocalized m.o. treatment of nuclear spin coupling due to Pople and Santry. It has already been shown (Theor. Chim. Acta, 4, 85, (1966)) that, by means of a reasonable choice of parameters, the changes of J_{CH} from compound to compound can be closely reproduced.

For a better understanding of these changes we decided to study the variations of the polarizability π_{sh} when one of the hydrogen atoms in the symmetrical molecules CH_n is replaced by an atom X, this being assumed to differ from H by, either a) having a different coulomb integral, or b) having different overlap (and resonance) integrals with the carbon atom, or c) having an electron lone-pair.

Effect a) is calculated by making use of the relationship

$$\frac{\partial \pi_{sh_1}}{\partial \alpha_{h_2}} = \frac{\partial \pi_{h_1 h_2}}{\partial \alpha_s}$$

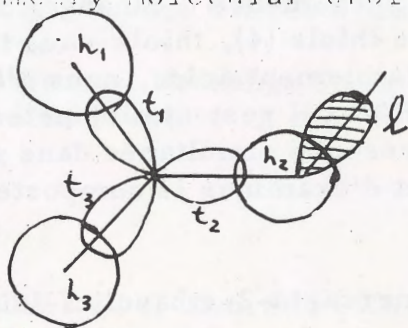
and then referring to the expression for $\pi_{h_1 h_2}$ obtained by Pople and Santry (Mol. Phys., 9, 311 (1965)) and by Murrell and Gil (Theor. Chim. Acta, 4, 114 (1966)). The expression for this effect turns out to be

$$\Delta \pi_{sh_1}^{(\alpha)} \approx - \frac{5\beta_{t_1 t_2} - \beta_{h_1 h_2}}{8n\beta_{h_1 t_1}^3} \Delta \alpha_{h_2}$$

For $n=3$, this gives an increase of about 8 c.p.s. in J_{CH} if $\Delta\alpha_{h_2} = -1$ e V. For CH_3X the effect is smaller; for CHX it will be larger.

Effect b) is being studied numerically. The preliminary results suggest that this is about as important as effect a) and, as the latter, increases with the electronegativity of X.

Effect c) was determined by finding the expression of π_{sh} when one allows for mixing of the lone-pair orbital with the CH symmetry molecular orbitals (Figure). The



first terms of the contribution due to the lone-pair are as follows:

$$\Delta\pi_{sh_1}^{(lp)} = \frac{3\beta_{sl}^2 - 8\beta_{h_2l}\beta_{t_1l}}{4n\beta_{h_1t_1}^3} + \frac{3\beta_{h_2l}^2}{4n^2\beta_{h_1t_1}^3}$$

where β_{sl} is the resonance integral between the carbon s-orbital and the lone-pair orbital. For the case illustrated in the figure,

$\Delta J_{CH_1}^{(lp)}$ turns out to be of the order of 20 c.p.s. whereas $\Delta J_{CH_2}^{(lp)}$ is negative and about the same order of magnitude. (Now that we have our new HA-100 spectrometer installed, I hope to investigate this effect experimentally).

Yours sincerely,

Victor F. Gil

Dr. Victor M.S. Gil

Paris, le 4 Octobre 1967

Professor B. L. SHAPIRO
 Department of Chemistry
 Stanford University
 Stanford
California 94305

PHENOMENES D'ECHANGES POUR LE MERCAPTO-2-ETHANOL

Cher Collègue,

Pour des mélanges de thiols et d'amines étudiés par RMN du proton nous avons décelé des phénomènes d'échanges rapides SH-NH (3). Pour des solutions binaires de thiols (4), thiols-eau, thiols-phénols, thiols-alcools, en milieu neutre ou faiblement acide, nous n'avons pas observé de tels phénomènes; les raies SH et OH restent distinctes.

En raison de la présence simultanée dans sa molécule de SH et OH, il nous a paru intéressant d'examiner le comportement du mercapto-2-éthanol ($\text{SH}-\text{CH}_2-\text{CH}_2-\text{OH}$).

Les spectres du mercapto-2-éthanol ci-joints permettent d'observer :

a) une raie OH unique, une raie SH unique, une structure CH_2-CH_2 pour le produit commercial "pur" Fluka (I, T \neq 310°K)

b) l'apparition du couplage $J_{\text{CH}_2-\text{OH}}$

par refroidissement du produit commercial (II, T \neq 260°K)
 après distillation de ce même produit (III, T \neq 310°K)

c) l'apparition du couplage $J_{\text{CH}_2-\text{SH}}$ par refroidissement du produit commercial (IV, T \neq 220°K)

d) l'apparition nette des couplages $J_{\text{CH}_2-\text{SH}}$ et $J_{\text{CH}_2-\text{OH}}$, par dilution dans CCl_4 (V, T \neq 310°K)

e) la coalescence des raies OH et SH par addition d'une faible quantité de pipéridine ($\leq 5\%$) dans la solution de mercapto-2-éthanol très dilué dans CCl_4 (VI, T \neq 310°K).

...

En conséquence, dans le produit non redistillé, il apparaît des phénomènes d'échanges intermoléculaires SH-SH, OH-OH éventuellement liés à la présence d'une impureté dans le produit ; ces échanges sont ralentis par purification ou par abaissement de la température. La structure J_{CH_2-OH} apparaît avant celle de J_{CH_2-SH} . D'autre part le ra-

lentissement des vitesses d'échanges par dilution dans un solvant inerte est très efficace et à rapprocher de celui observé par plusieurs auteurs (2) qui ont relié ce phénomène à la "séparation" des molécules par le solvant. L'introduction d'un groupement NH (pipéridine $K_b \approx 1,6.10^{-3}$) fait apparaître l'échange SH-OH.

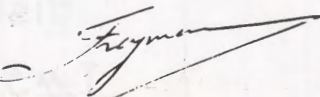
Nos observations soulignent l'importance d'une purification soigneuse des produits : en effet l'échange peut être influencé par une impureté qui participe au phénomène ou le catalyse par sa contribution au pH du milieu. On peut ainsi comparer le spectre χ avec celui obtenu par Kreevoy et coll. (1) pour la solution à 20% de mercapto-2-éthanol dans CCl_4 ; la raie OH reste unique, contrairement à ce que nous avons observé.

Quant à l'échange SH-OH, dans le cas que nous avons envisagé où l'amine présente un proton échangeable, celui-ci participe au phénomène.

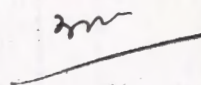
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2. - M. L. MARTIN - Thèse Paris 1961
3. - M. M. MARCIACQ-ROUSSELOT, N. BELLAVITA
(à paraître aux Comptes Rendus Ac. Sc.)
4. - M. M. ROUSSELOT - Comptes Rendus Ac. Sc. série C, 263, 649, 1966.

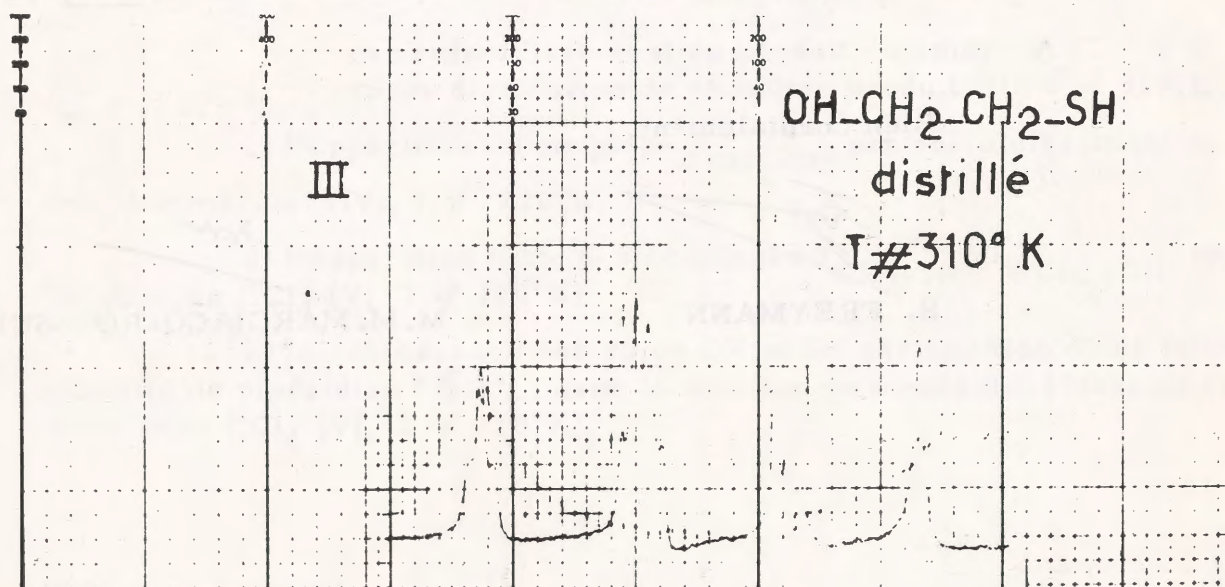
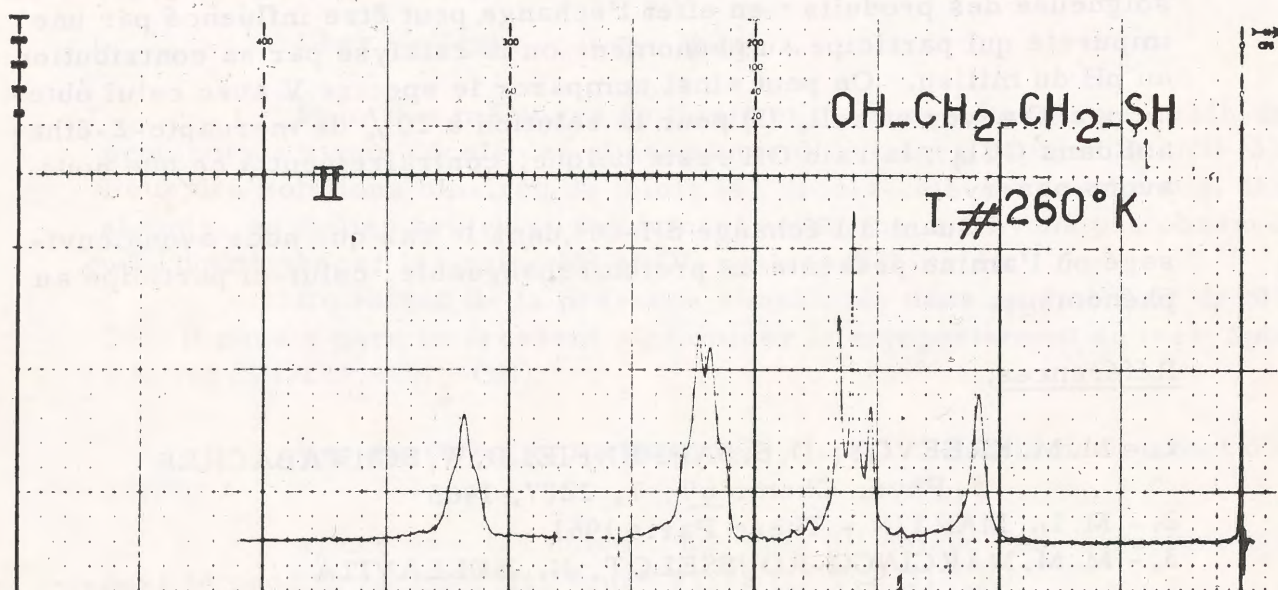
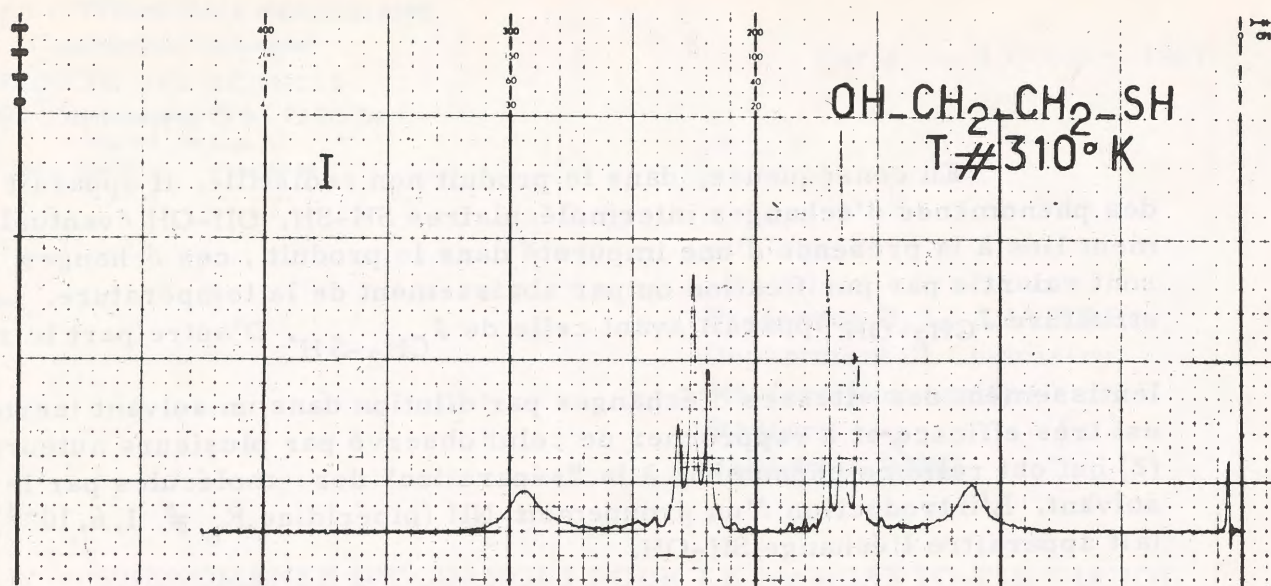
Bien cordialement.

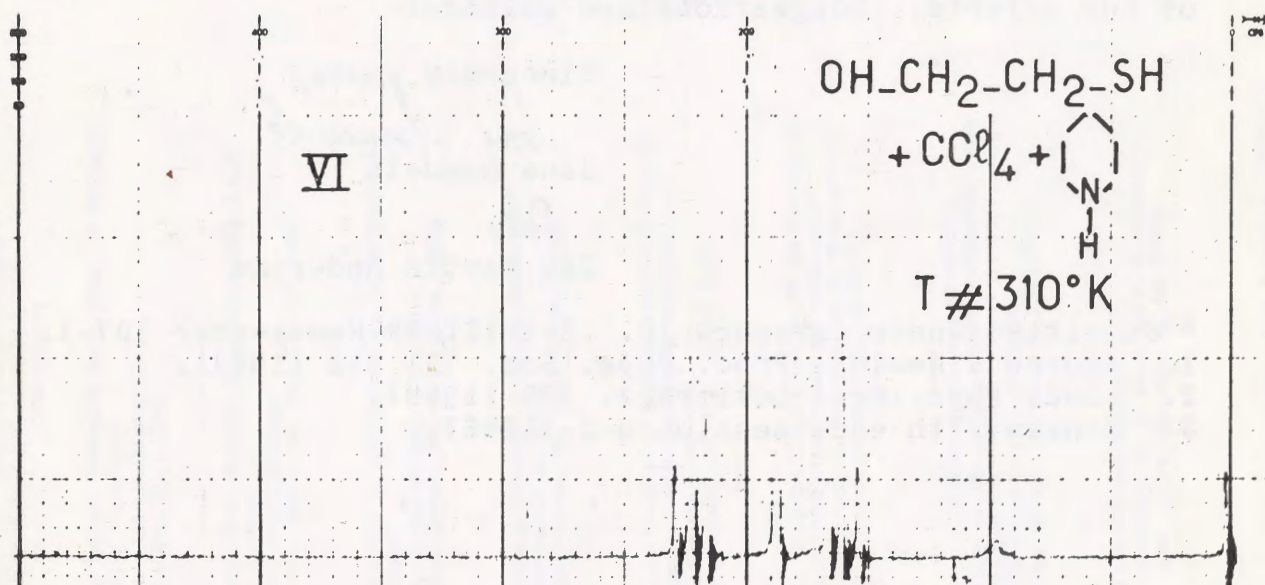
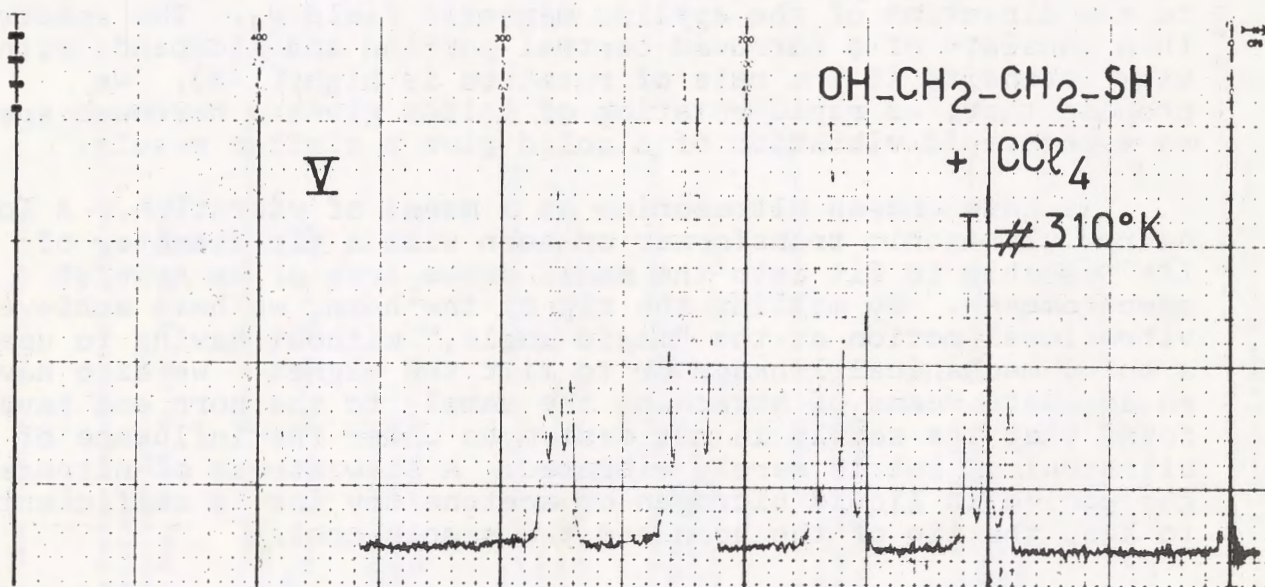
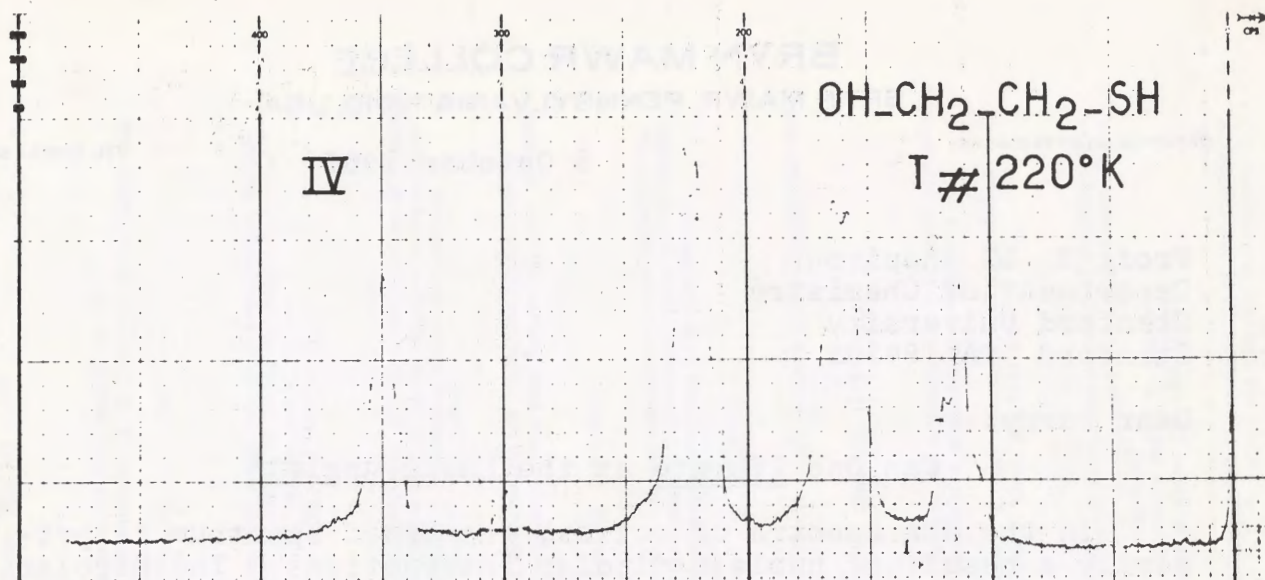


R. FREYMANN



M. M. MARCIACQ-ROUSSELOT





BRYN MAWR COLLEGE
BRYN MAWR, PENNSYLVANIA 19010, USA

DEPARTMENT OF CHEMISTRY

5 October 1967

TEL: (215) LA 5-1000

Prof. B. L. Shapiro
Department of Chemistry
Stanford University
Stanford CA 94305

Dear Barry:

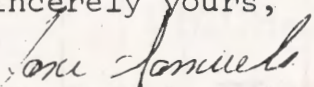

Can One Vibrate at the Magic Angle?*

In the NMR spectra of solids, the broad spectrum is primarily a result of nuclear dipolar interactions. The dipolar interaction may, however, be removed by rapid rotation of the sample about an axis inclined at an angle $54^{\circ}44' = \cos^{-1}(1/\sqrt{3})$ to the direction of the applied magnetic field H_0 . The spectrum then consists of a narrowed central portion and sidebands with weak intensity if the rate of rotation is high [1-3]. We propose that, as rapid rotation of solids gives a narrowed spectrum, so might rapid vibration of a solid give a similar result.

We have chosen ultrasonics as a means of vibration. A long narrow ultrasonic transformer or horn with a tip diameter of $1/4"$ is able to fit into the small probe area of an A-56/60 spectrometer. By milling the tip of the horn, we have achieved vibrational motion at the "magic angle," without having to use a bulky mechanical linkage or to tilt the magnet. We also have an adequate means of attaching the sample to the horn and have found that the sample is not destroyed under the influence of ultrasonics, but is merely vibrated. A slow stream of nitrogen gas cooled in liquid nitrogen or acetone/dry ice is sufficient to keep the tip of the horn and the sample cool.

We are now in the process of adapting an A-60 probe for the ultrasonic horn. We are also seeking theoretical justification of our efforts. Suggestions are welcome!

Sincerely yours,


Jane Samuels
Jay Martin Anderson

- * Submitted under category 10. See IITNMR Newsletter 107-1.
1. Andrew & Newing, Proc. Phys. Soc. 72, 959 (1958).
 2. Lowe, Phys. Rev. Letters 2, 285 (1959).
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CARNEGIE-MELLON UNIVERSITY

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