Joseph B. Lambert

Illinois Institute of Technology N-M-R Newsletter

No. 101
FEBRUARY, 1967

OVER

Deadline Dates: No. 102 - 15 March 1967
No. 103 - 15 April 1967

UNIVERSITÉ D'OTTAWA
FACULTÉ DES SCIENCES PURES ET APPLIQUÉES

UNIVERSITY OF OTTAWA
FACULTY OF PURE AND APPLIED SCIENCE

OTTAWA 2, CANADA

DÉPARTEMENT DE CHIMIE
365, RUE NICHOLAS

DEPARTMENT OF CHEMISTRY
365 NICHOLAS ST.

January 13, 1967

Title: A. Interpretation of Chemical Shifts in Aryl Tetrazoles.
B. Post-Doc Wanted

Dear Barry:

During work on the chemistry of tetrazoles we had the opportunity to assemble the chemical shift data given in Table I. This table contains the τ values for the phenyl protons in 5-phenyl tetrazole, and its two N-methyl derivatives (I, II, III) and also τ values for the three corresponding para nitro derivatives. Several workers[1,2,3] have previously observed significant deshielding of the ortho protons in various phenyl substituted aromatic heterocycles, specifically pyrazoles, pyridazines and triazoles. In each instance the deshielding was attributed to a neighbour-anisotropy effect. Our data indicates this to be an oversimplification.

It is seen that in III the 1-methyl substituent results in a coalescence of all phenyl absorption into one peak. From the lower λ max and ϵ of this compound relative to II it is obvious that the methyl group destroys the coplanarity of the two rings. (The U.V. of VI resembles p-nitro toluene). The destruction of coplanarity will of course decrease the size of the neighbour anisotropy effect.

.....2

It will also prevent resonance interactions, and this too will affect the chemical shifts.

To see the effect of rotation from coplanarity we can examine the τ values for II and III. Destruction of coplanarity causes are upfield shift of .49 ppm. for the ortho proton. However, the same geometrical change in V and VI causes only a .16 ppm. diamagnetic shift. Diehl[4] has already pointed out that additivity of substituent effects will occur only in systems in which resonance interactions are negligible. The above lack of additivity is thus an indication of a substantial resonance effect. There are other indications as well. For example, the para proton shifts appear at lower field than benzene in I and II and at still lower field in III. These facts are interpretable if the tetrazole ring withdraws electrons inductively but donates electrons via a resonance interaction. For this reason the effect of introduction of a nitro group into II to give V (where resonance interaction is present) is abnormal, whereas the effect in the change III → VI is very similar to the observed nitro group effect. in nitrobenzene (δ_o = -.97, δ_m = -0.3 ppm from benzene).

I think I should emphasize the fact that the above rationalization is somewhat speculative. The only important conclusion is that anisotropy alone cannot explain the chemical shift data.

Finally, I wish to make it known that I have an opening for a post-doctorate fellow. I am particularly interested in an individual with experience in organic chemistry.

.....3

(1) Tensmeyer and Ainsworth, J. Org. Chem., 31, 1881 (1966).

(2) J. Crossland, Acta Chem. Scand., 20, 258 (1966).

(3) Lynch and Hung, Can. J. Chem., 42, 1605 (1964).

(4) Diehl, Helv. Chem. Acta, 44, 829 (1961).

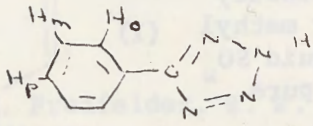
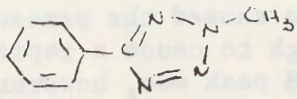
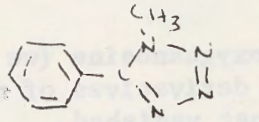
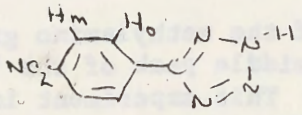
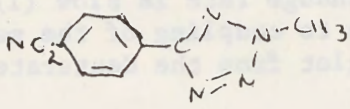
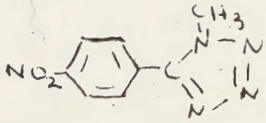
Yours sincerely,

Bob Fraser

Robert R. Fraser

RRF:ae

TABLE I -- τ VALUES

		$-NCH_3$	H_o	H_m	H_p
I		-	1.9	2.49	2.49
II		5.75	1.87	2.59	2.59
III		5.84	2.36	2.36	2.36
IV		-	1.6	1.6	-
V		5.58	1.79	1.79	-
VI		5.75	1.95	1.44	-

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Bob Fraser

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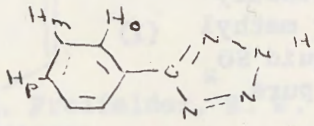
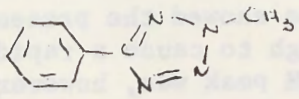
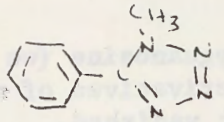
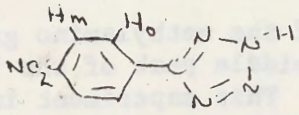
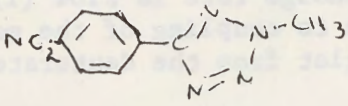
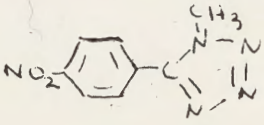
I		-	1.9	2.49	2.49
II		5.75	1.87	2.59	2.59
III		5.84	2.36	2.36	2.36
IV		-	1.6	1.6	-
V		5.58	1.79	1.79	-
VI		5.75	1.95	1.44	-

DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
PUBLIC HEALTH SERVICE

NATIONAL INSTITUTES OF HEALTH
BETHESDA, MD. 20014
AREA CODE 301 TEL: 656-4000

January 10, 1967

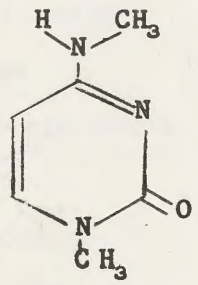
Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry:

D_2O as a Troublemaker in DMSO-d_6; Isotope Effect on Chemical Shift

We have recently encountered a problem with deuterated dimethylsulfoxide that may interest other NMR fans.

In the course of an investigation of the spectra of methyl derivatives of cytosine we were quite surprised to observe a triplet, instead of the expected doublet, for the methyl signal of several derivatives containing a $NHCH_3$ group such as I: Expansion of the triplet revealed that the middle peak was slightly displaced from the center of the other two. The integral showed the intensity of the triplet to be equal to that of other methyl groups, and spectra of the compounds in liquid SO_2 showed the expected characteristics of the pure materials.



(I)

A low intensity peak in the DMSO-d_6 sample showed the presence of a small amount of water, evidently not enough to cause a rapid exchange of the NH proton. The intensity of the NH peak was, however, lower than it should be.

Upon admixture of an equimolar amount of deoxyguanosine (we are presently studying base-base interaction on derivatives of nucleosides) the center peak of the "triplet" almost vanished.

We prepared a sample of I in which the NH of the methylamino group was replaced with ND and observed that the middle peak of the "triplet" was much more intense than the outer lines. This experiment indicates that the culprit in our DMSO-d_6 was D_2O or HDO, which gave partial exchange of the NH proton. Since the exchange rate is slow (1), we observe simultaneously the doublet due to coupling of the methyl group with the NH ($J \approx 5$ Hz) and the singlet from the deuterated species.

Professor B. L. Shapiro
January 10, 1967
Page 2

Our observation of a decrease in intensity of the middle peak upon admixture of deoxyguanosine fits the conclusion that we have a mixture of $NHCH_3$ and $NDCH_3$, since a relatively large amount of exchangeable protons is added this way.

From the position of the center peak of the triplet we could determine an isotope effect of 0.24 ± 0.04 Hz (0.0040 ppm) on the chemical shift of the methyl group protons four bonds removed from the substituted proton. The deuterated species is at higher field.

We have informed Merck, Sharp & Dohme of Canada of our findings and requested that they dry their DMSO better. They "normally" (not in this batch) keep D_2O to < 0.1%, but even this amount can cause difficulties occasionally.

Best regards,

Sincerely,

Ted

Edwin D. Becker
Regitze R. Shoup
H. Todd Miles

(1) cf. M. Freifelder, R. W. Mattoon and R. Kriese, J. Phys. Chem. 69, 3645 (1965).

UNIVERSITY OF BRADFORD

Bradford 7 Telephone OBR4 29567
ENGLAND.

Vice-Chancellor and Principal
E G Edwards PhD FRIC

Please quote ref DWJ/pep

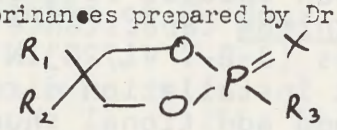
17th January, 1966.

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

Dear Dr. Shapiro,

Short title: Proton resonance of cyclic organophosphorus compounds

Recently, we have recorded the proton resonance spectra in $CDCl_3$ solution at 60 Mc/s of about thirty 5,5 - substituted 1,3,2 - dioxaphosphorinanes prepared by Dr. R.S. Edmundson. They have the formula



where X=O or S; R_1=R_2=Me or Et; R_1=Me, R_2=$ClCH_2$; R_3= alkyl, alkoxy, aryloxy, alkylamide, halogeno, or H

Despite the formal complexity of these spin systems, solutions have been obtained for many of the spectra by regarding the 4,6-methylene protons as AB parts of ABX systems; indeed the A and B frequencies often differ sufficiently for the AMX approximation to be justified. Couplings $J_{AA'}$ and $J_{BB'}$ across the rings and to the 5-methyl protons are all small. Both axial and equatorial 5-methyl peaks are broadened, presumably because of long-range coupling to each other and/or to the 4,6-protons; the broader 5-methyl resonance is taken to be axial.

The form of the R_3 part of the spectrum is of some interest in the cyclic phosphates, e.g. for X=O; R_1=R_2=Me; R_3=$OCHMe_2$. Methine-methyl coupling gives a 6.5 c/s doublet for the R_3 methyls; the corresponding 6.5 c/s septet for the methine protons is complicated by ^{31}P coupling to give an intensity sequence: 1.3: 4.6: 8.9: 8.3: 5.3: 2.6: 1.0. The B region of the approximate A_6BX system was in turn approximated by superposing two parts of A_6B systems[1] with the ratio J_{AB}/ν_{AB} equal to 0.025 in each part. Comparison of calculated spectra for J(P-O-CH) equal to J_{AB} and twice J_{AB} showed that ^{31}P-O-CH coupling in this compound was 6.5 c/s rather than 13 c/s.

For cases in which 4,6-methylene protons have been assigned, we find $J(P\text{-}O\text{-}CH)_{ax}$ in the range 7.8-10 c/s and $J(P\text{-}O\text{-}CH)_{eq}$ in the range 13.8-16.3 c/s. Further details of these spectra will shortly be published with Dr. R.S. Edmundson in Tetrahedron.

Thank you for your Newsletters.

Yours sincerely,

K.D. Bartle D.W. Jones J.E. Pearson

K.D. Bartle D.W. Jones J.E. Pearson

(1) J. Ranft, Ann. der Physik 10, 1 (1962).

Dartmouth College HANOVER · NEW HAMPSHIRE · 03755

Department of Chemistry · TEL. (603) 646-2501

January 16, 1967

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

Dear Barry:

With issue 101 of NMR Letters coming along, a heartfelt THANK YOU for your unique and invaluable contribution to NMR spectroscopy.

Item 1. A penny saved. Our 15MHz V4311 and probe can now be retuned to 19.25 MHz in ca 30 minutes. This trivial task at 56.4-60 MHz requires more heroic measures at lower frequencies, specifically major changes in capacitance in some of the tuned circuits to get optimum L/C ratios. Briefly, following the recommended values from the Varian special frequency schematic, we installed as necessary the minimum capacitance values for either frequency and use switches (J-B-T VL72031N subminiature lever action, modified to permit installation directly at or over the critical circuits) to add additional shunt capacitance (Vitramon ceramic) to obtain the maximum values needed at either frequency. Where more than 3-4 turns of inductor tuning were needed, better values of capacitance were chosen by trial and error. The unit now tunes well within specs on both frequencies. In the process we solved another small problem - low receiver gain. Some LOL in tennis shoes at Palo Alto had tuned our IF strip off center by 50 Kc. Retuning to center band and stagger tuning by ±50 Kc improved our gain and disposition nicely.

The V4331 probe posed a problem - there was no observable correlation between the probe RCVR and XMTR tuned circuits and the schematic. Trial and error (the last and only resort of ex-organic chemists faced with electronics problems) revealed the XMTR section worked equally well (poorly ?) at both frequencies and that the RCVR required a change in capacitance when changing frequencies.

We see no reason why the unused 3rd position on the switches couldn't be used to extend the change in frequency to 24 MHz although we haven't tried this yet.

Item 2. Spurious beats on internal lock operation.

a) First and most obvious is from superstabilizer loop oscillation, usually after bulb replacement and realignment. We cure this by settling for about 45/50 rather than 70/50 (change in fine current/change in meter) operation.

b) Second and less obvious was a beat which came in one day at about τ =3. The usual hocus pocus of detuning the reference phase section, etc., failed, but the beat frequency changed when we diddled the local oscillator. By correct adjustment of the local oscillator 1st stage L/C ratio (our old V4311 unit still has a variable C-333) the beat was completely eliminated and all other stages could be peaked.

Item 3. Work in progress. Besides the usual organic load, we're doing a fair amount of pulse operation, T_1 and T_2 measurements (Meiboom modification) on polymers, exchange rates for molecular complexes and metal ion-enzyme binding. Other work includes BF_4^- coupling constants, nmr asymmetry for systems with equal conformer populations and miscellaneous on ^{11}B, ^{23}Na, ^{79}Br, etc.

We are more than pleased with our DA-60-IL system performance especially since our down time is only about 1% and the original set of 304TL's is still going strong after 18 months of 24 hour operation.

Cheers,

Nick Karl

Paul R. Shafer
Karl F. Kuhlmann

מכון ויצמן למדע
THE WEIZMANN INSTITUTE OF SCIENCE

REHOVOTH · ISRAEL
P.O.B. 26 · PHONE 951721-7

רחובות · ישראל
ת.ד. 26 · טלפון: 951721-7

ISOTOPE DEPARTMENT

מחלקת האיזוטופים

January 19, 1967

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

Dear Professor Shapiro:

We are continuing our NMR studies of the hydration of paramagnetic rare earth ions. There has been some controversy in the recent literature regarding the nature of the acetate complexes of these ions: are they "innersphere" or "outersphere". We have measured the water proton shifts in solutions of the perchlorate salts of a series of rare earth ions in absence, δH_2O, and in presence, δH_2O, of ammonium acetate (AmAc), as well as the shift of the acetate methyl protons, δCH_3. The following results have been obtained, relative to the resonance of the uncomplexed pure ligand (in Hz at 56.4 MHz, water shifts are interpolated for 1.0 molal solution, CH_3COO^- shifts are for [Ln]/[AmAc] = 0.1).

Ln^{3+}	Pr	Nd	Gd	Tb	Dy	Ho	Er	Yb
δH_2O	15	18	170	-594	-392	-275	216	142
$\delta' H_2O$	83	37	-	37	257	35	107	43
δCH_3	-64	-24	-	-300	-497	-182	48	64

It seems that the proton shifts in these systems are dominated by pseudo-contact contributions (except Gd^{3+}). The results clearly indicate that the acetate is bound in the first coordination sphere of the rare earth ion. Assuming a coordination number of 3 with respect to acetate a lower limit of $10^4 sec^{-1}$ was calculated for the rate constant of ligand exchange between the acetate complex of Dy^{3+} and the ligand in bulk. The upper limit is set by the rate constant of water exchange, $4 \times 10^8 sec^{-1}$, which has been obtained from ^{17}O relaxation studies.

Yours sincerely,

Jack Reuben

Daniel Fiat

Jack Reuben

Daniel Fiat

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

1 Berlin 12, den January 17, 1967
Straße des 17. Juni Nr. 115 (Chemiegeb.)
Fernruf: 32 51 81, App. 252
Prof. B/Ma

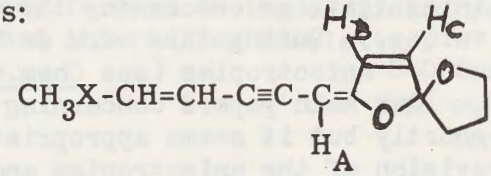
Professor B. L. Shapiro
Illinois Institute of Technology
Department of Chemistry

Chicago 60616 - Ill.
U S A

Long range effect on chemical shifts

Dear Professor Shapiro!

In connection with structural work on natural occurring polyynes we have measured a lot of sulfones. It turns out that the sulfone group has a surprising long range effect on the chemical shifts as you can see in the following examples:



X	H_A	H_B	H_C
S			
cis, cis	ddd 5.03	dd 3.22	dd 3.91
cis, trans	dd 5.35	d 3.75	dd 3.97
SO_2			
cis, cis	ddd 4.88 Δ =0.15	dd 2.89 Δ =0.33	dd 3.64 Δ =0.27
cis, trans	dd 5.20 Δ =0.15	s 3.62 Δ =0.13	Δ =0.35

$PhCH_2-C\equiv C-CH=CH-SCH_3$
d 6.30

$PhCH_2-C\equiv C-CH=CH-SO_2CH_3$
d 6.11 Δ = 0.19

$CH_3-C(SCH_3)=CH[C\equiv C]_2-C(H)=C(H)-CO_2CH_3$ H dd 3.22, H d 3.77

$CH_3-C(SO_2CH_3)=CH[C\equiv C]_2-C(H)=C(H)-CO_2CH_3$ H dd 3.13 Δ = 0.09, H d 3.58 Δ = 0.19

Yours sincerely,

F. Bohlmann

THE SCHOOL OF PHARMACY
UNIVERSITY OF LONDON

PHARMACEUTICAL CHEMISTRY
PROFESSOR W. B. WHALLEY
D.Sc., Ph.D., F.R.I.C.

29/39, BRUNSWICK SQUARE
LONDON, W.C.1.
TELEPHONE TERMINUS 7651/8

WBW/JL

20th January, 1967.

Dear Dr. Shapiro,

The Anisotropies and Susceptibilities of the C-H, C-C, and C=C Bonds

I must apologise most sincerely for not having replied sooner to your reminder of 9th December, 1966, and plead the usual exigencies of pressure of work together with the diversions of the Christmas season! I trust the following will put us back on your circulation list.

During the last two years we have been carrying out an extensive programme of investigations concerning the calculation of chemical shift values. During this work we have derived the C-H, C-C, and C=C anisotropies (see Chem.Comm., 1966, 359, 361 and 754). Our main papers concerning this work will be published shortly but it seems appropriate at this time to report a revision of the anisotropies and susceptibilities previously recorded in our Communications.

One method for the derivation of these constants involved the use of the equation $\Delta\chi^{CC} - 2\Delta\chi^{CH} = -4.8 \times 10^{-6}$ which is derived from magnetic birefringence measurements upon ethane (Buckingham et al., Chem.Comm., 1965, 51). Professor A.D. Buckingham has informed us that the most recent and reliable value for the constant in this equation should now be -2.3×10^{-6}. Using this new constant we have rederived the anisotropies and susceptibilities (in $cm.^3$ $molecule^{-1} \times 10^{-30}$) for the C-H, C-C, and C=C bonds as follows:

$\Delta\chi^{C-C}$	= + 11.26	$\Delta\chi^{C-H}$	= + 7.54
χ_L^{C-C}	= + 1.37	χ_L^{C-H}	= - 1.37
χ_T^{C-C}	= - 9.87	χ_T^{C-H}	= - 8.90

Professor Bernard L. Shapiro 20th January, 1967.

$\Delta\chi_1^{C=C} = -12.00$ $\Delta\chi_2^{C=C} = -12.06$

$\chi_x^{C=C} = +2.69$
$\chi_y^{C=C} = -9.31$
$\chi_z^{C=C} = -9.37$

Recalculation of chemical shifts using these revised constants yields values which fall within the same limits of error and differ only marginally from those previously briefly reported and to be described in extenso in our complete publications. This work was performed in association with J.W. ApSimon, P.V. Demarco and D.W. Mathieson.

We look forward to the continued receipt of your most valuable and interesting Newsletter.

Best wishes,
Yours sincerely,

W.B. Whalley

W.B. Whalley

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

KEMISK INSTITUT
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DANMARK

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Telefon (06) 124633 HeL/EL

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

Long Range Coupling in Quaternary Heteroaromatic Compounds

Dear professor Shapiro,

During an investigation of the quaternization of pyridazines and related compounds with methyl iodide we have observed long range coupling between the N-methyl protons and protons in various positions. The following examples may illustrate this.

In 1,4,6-trimethyl-3-methoxypyridazinium iodide (I) Me(1) couples with Me(4) (J = 0.6 cps) and with Me(6) (J = 0.45 cps). Here the coupling Me(1)-Me(4) through seven bonds is slightly greater than the coupling Me(1)-Me(6) through five bonds. Me(4) also couples with H(5) (J = 1.1 cps).

Is Me(4) substituted with H, a weak coupling through six bonds between Me(1) and H(4) can be detected by spin decoupling. By irradiating Me(1) a sharpening of the signal from H(4) is obtained.

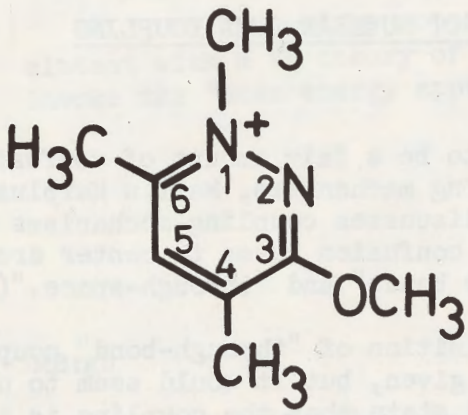
1,6-Dimethyl-3-methylmercaptopyridazinium iodide (IIa) and 1,6-dimethyl-3-chloropyridazinium iodide (IIb) both show a coupling between Me(1) and Me(6) with J = 0.6 cps. In the isomeric 2,6-dimethyl-3-substituted pyridazinium compounds no coupling between the methyl groups at N(2) and C(6) was observed.

In 1,4-dimethylcinnolinium iodide (III) the protons in the two methyl groups couple through seven bonds with J = 0.65 cps. Me(4) also couples with H(3) (J = 0.60 cps). In 2,4-dimethylcinnolinium iodide no coupling between the protons in the methyl groups has been detected. A coupling through six bonds between Me(1) and H(4) is found in 1-methyl-3-phenylcinnolinium iodide (J = 0.5 cps).

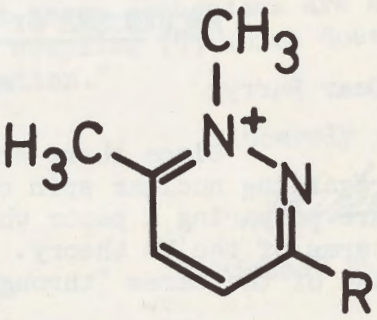
The above mentioned results seem to indicate that in aromatic heterocyclic compounds coupling between N- and C-methyl protons occurs only, when there are an uneven number of bonds between the protons. This may be of diagnostic value, as the quaternization of aromatic diazaheterocyclic compounds mostly gives a mixture of isomers.

Sincerely yours,

Henning Lund Per Lunde

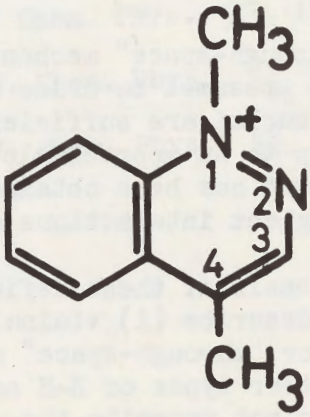


I



II a R = SCH_3

b R = Cl



III

THE UNIVERSITY OF ARIZONA

TUCSON, ARIZONA 85721

COLLEGE OF LIBERAL ARTS

DEPARTMENT OF CHEMISTRY

January 18, 1967

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

MECHANISMS OF CONTACT NUCLEAR SPIN COUPLING

Dear Barry:

Since there seems to be a fair amount of confusion regarding nuclear spin coupling mechanisms, Martin Karplus and I are preparing a paper which discusses coupling mechanisms in terms of the VB theory. The confusion seems to center around the use of the terms "through-the bonds" and "through-space."(1)

An unambiguous definition of "through-bond" coupling does not appear to have been given, but it would seem to us that a reasonable definition would state that the coupling is transmitted entirely through the bonds; that is, a path which includes the bonds containing the coupled nuclei and one bond between every intervening pair of atoms in the chain linking the coupled nuclei. This is the type of mechanism which was investigated by Koide and Duval (2), but these authors neglected to point out that such contributions are independent of dihedral orientations. Contributions of this type must be very small (3) for coupling over more than two bonds.

The "through-space" mechanism, as defined by Petrakis and Sederholm, is presumed to arise when the electrons associated with the coupled nuclei are sufficiently close that the electronic clouds can overlap to an appreciable extent. Satisfactory agreement with experiment has been obtained in calculations (4,5,6) which entirely neglect interactions of this kind.

On the basis of these definitions it seems clearly inappropriate to describe (1) vicinal H-H coupling in terms of a "through-bond" or "through-space" mechanism. Since this argument can be made for other types of H-H coupling, (5,6) it appears that these two terms do not describe the most important mechanisms.

Using VB perturbation theory, it can be shown that to a good approximation the contact coupling constant is proportional to terms of first and higher order in the Penney-Dirac bond orders for two-bond fragments of the molecule. The proposed nomenclature uses the terms "direct-pair" and "indirect-pair" to describe contributions from first and higher order terms, respectively. "Through-bond" and "through-space" mechanisms are special cases of these.

The strong angular dependence of the vicinal coupling (4) can be identified with contributions from the direct-pair mechanism, whereas the various long-range couplings (5,6) arose from indirect-pair mechanisms associated with individual π and/or σ bonds in the molecule.

It should be noted that these mechanisms are also consistent with a VB theory of spin coupling (7) which does not invoke the "mean energy approximation."

Sincerely yours,

Mike

Michael Barfield

MB:gb

1. L. Petrakis and C. H. Sederholm, J. Chem. Phys., 35, 1243 (1961).
2. S. Koide and E. Duval, J. Chem. Phys., 41, 315 (1964).
3. P. Chandra and P. T. Narasimhan, Mol. Phys., 11, 189 (1966).
4. M. Karplus, J. Chem. Phys., 30, 11 (1959).
5. M. Karplus, J. Chem. Phys., 33, 1842 (1960).
6. M. Barfield, J. Chem. Phys., 41, 3825 (1964).
7. M. Barfield, J. Chem. Phys., 46, to be published.

DEPARTMENT OF CHEMISTRY

UNIVERSITY OF ALBERTA
QUAECUMQUE VERA

EDMONTON, ALBERTA
CANADA

10 January, 1967

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

Dear Barry,

While we all appreciate the power and elegance of the Ferguson and Marquardt version of NMRIT and NMREN (J. Chem. Phys. 41, 2087 (1964)), which is available from the IBM SHARE Library (No. 3165), I imagine that many readers of this newsletter share our frustration with its curious limitations, intricate input formats, clumsy operation, tape mounting, card shuffling, etc.

Last summer, in the course of his work on vinylcyclopropane, Dr. George DeMaré noticed that certain sorting routines essential to parameter iteration do not work reliably. In addition, as you may recall, major surgery is required to fit the F-M version of NMRIT into a 32K core (IITNMR, 77, P. 9). All of this finally provoked Drs. DeMaré, Birss and myself to take drastic measures, i.e., figure out how it was supposed to work and write a program that does so.

The result of this is a completely rewritten chain incorporating the functions of both NMRIT and NMREN (thus brilliantly designated TWOSUM). The desired set of operations is controlled by the value of a single parameter, 'NIT'. The complete input writeup, as it appears on comment cards preceding the FORTRAN listing, follows this letter. I think that chemists, as opposed to computer specialists, will find this version a good deal easier to use. Most spectral parameters have the same significance as in the F-M program, unless specifically designated otherwise. All the original functions are retained.

Efficient matrix storage and operations, and a better Jacobi subroutine, plus use of system tapes for intermediate data storage, have removed the core overflow problems, and indeed enabled us to expand several critical dimensions of the program, as shown in the following table.

CRITICAL DIMENSIONS OF F-M NMRIT/NMREN versus TWOSUM.

	F-M	TWOSUM
Max. input transitions per submatrix, NMREN	600	600
Max. energy levels per submatrix, NMREN	75	100
Max. energy levels per submatrix, NMRIT	75	100
Max. energy levels per subsubmatrix, NMRIT	20	35
Max. energy levels per molecule, NMRIT	360	512
Max. number of transitions, NMRIT	1500	1500

The larger submatrix and subsubmatrix dimensions permit the treatment of a few new mildly exotic types, for example $A_2B_2C_2D_2$, $A_3B_3C_2D$, $A_6B_2C_3$, A_9BCD and A_6B_6C. The most important gain is the removal of any practical limitation on the number of transitions per molecule that NMRIT can handle. The older version permitted core storage of 1500 transitions of <u>any</u> intensity; TWOSUM stores 1500 transitions, but only transitions of <u>significant</u> intensity (By significant intensity, we mean intensity of at least 0.005, where the total molecular intensity is $n2^{n-1}$). In practise, any spectrum with more than 1500 significant transitions is hopelessly formidable. One commonly finds that 80 or 90 percent of transitions in a moderately complex system are edited out by the 0.005 criterion.

TWOSUM is written in FORTRAN IV for an IBM 7040-1401 using the IBSYS monitor. It fits into 24000 core locations, and requires four system tapes, in addition to the one carrying the chain. No manual data transfer between NMRIT in the parameter iteration mode and NMREN is required. We find that more time is wasted in this manual transfer of NMREN data to the NMRIT input than is ever likely to be wasted in useless NMRIT runs where NMREN failed to converge (we have also built in a convergence criterion for NMREN).

The program has been checked out in all modes on a number of four-, five- and six-spin systems. We have not checked out the missing-energy-level options, since we rarely need them. In practise, we find that 'missing' levels usually are simply isolated from other levels in a submatrix by effective factoring (symmetry or large chemical shifts) not explicitly accounted for in the Hamiltonian factoring program. We have had success in handling this by simply plugging into the NMREN input enough 'dummy' transitions derived from the first NMRIT run to connect the submatrix diagram completely. Since these 'dummy' transitions connect levels not otherwise related by observable transitions, their frequencies do not affect the derived parameters.

I would be glad to provide a FORTRAN listing and sample computation to anyone who requests them. If interest is sufficient, TWOSUM may find its way into SHARE. Before this happens, I would certainly want to hear about bugs that turn up in its operation (preferably with instructions for debugging!).

Sincerely,

John Martin

John S. Martin

```
C
C     ******************************************************************
C
C     NMR SPECTRAL ANALYSIS PROGRAM TWOSUM
C     BY GEORGE R. DEMARE, JOHN S. MARTIN AND FRASER W. BIRSS,
C     DEPARTMENT OF CHEMISTRY, UNIVERSITY OF ALBERTA, EDMONTON, ALBERTA
C
C     THIS PROGRAM CONTAINS MODIFICATIONS OF NMRIT AND NMREN,
C     BY FERGUSON AND MARQUARDT (J. CHEM. PHYS. 41, 2087, (1964)).
C     ALL OPERATIONS OF THESE PROGRAMS ARE INCORPORATED INTO A CHAIN.
C
C     THE MAINLINE, TWOSUM, CALLS LINKS AS NEEDED. THE OPTIONS USED ARE
C     CONTROLLED BY THE PARAMETER 'NIT' IN DATA ITEM 1.
C     ALL NECESSARY DATA TRANSFERS ARE MADE INTERNALLY VIA COMMON CORE
C     AND SYSTEM TAPES. FOUR TAPES ARE NEEDED, REFERRED TO AS FILES 1-4
C     IN THE PROGRAM, IN ADDITION TO TAPE 0, WHICH STORES THE CHAIN.
C
C     THE PROGRAM IS WRITTEN IN FORTRAN IV, FOR AN IBM 7040-1401 COMPUTER
C     USING THE IBSYS MONITOR, AND REQUIRES 24000 36-BIT CORE STORAGE
C     LOCATIONS NOT INCLUDING MONITOR.
C
C     THREE OPTIONAL MODES OF OPERATION ARE PROVIDED.
C
C     OPTION 'A'. NIT LESS THAN 0. ONLY LINK 1, NMREN, IS CALLED.
C     AN ENERGY-LEVEL DIAGRAM IS DEDUCED WHICH GIVES THE BEST POSSIBLE FIT
C     TO THE OBSERVED TRANSITION FREQUENCIES.
C
C     OPTION 'B'. NIT = 0. LINKS 2,3. NMRIT+NMROUT. A SPECTRUM IS
C     COMPUTED CORRESPONDING TO PARAMETERS SUPPLIED AS DATA. THERE IS
C     PROVISION FOR INCREMENTING CHEMICAL SHIFTS AND SUPPLYING SEVERAL
C     DIFFERENT SETS OF COUPLING CONSTANTS.
C
C     OPTION 'C', NIT GREATER THAN 0. ALL LINKS ARE USED. NMREN DEDUCES
C     THE 'OBSERVED' ENERGIES, AND NMRIT ITERATES THE INPUT SHIFTS AND
C     COUPLINGS NIT TIMES TO FIT THE ENERGIES FROM NMREN.
C
C     ******************************************************************
C
C     INPUT ON CARDS AS FOLLOWS.
C
C     ITEM 1. THIS ITEM, A CONTROL CARD, IS USED IN ALL MODES.
C       COLS. 1-4, FORMAT I4. NTYPE, THE NUMBER OF DIFFERENT GROUPS
C       OF EQUIVALENT NUCLEI IN THE MOLECULE.
C       COLS. 5-28, 6I4. THE NUMBER OF NUCLEI IN EACH OF THE GROUPS.
C       THE FIRST NTYPE GROUPS ONLY MAY BE USED.
C       COLS. 41-44, I4. NIT. THIS NUMBER DETERMINES THE MODE AND IS
C       THE NUMBER OF TIMES THE PARAMETERS WILL BE ITERATED IN MODE C.
C       COLS. 45-48, I4. (MODE B ONLY) NASET, THE NUMBER OF SETS OF
C       COUPLINGS TO BE SUPPLIED FOR THIS MOLECULE. IF BLANK, NASET=1.
C       COLS. 49-52, I4. (MODES B,C ONLY) NPLOT, THE NUMBER OF FREQUENCY
C       RANGES IN WHICH HISTOGRAM PLOTS OF THE NMRIT OUTPUT IS DESIRED.
C       THESE RANGES WILL BE PLOTTED FOR EACH SET OF PARAMETERS IN MODE B.
C       PLEASE NOTE THAT NPLOT CARDS MUST THEN BE SUPPLIED IN ITEM 3.
C       COLS. 61-70, F10.0. (MODES B,C) THE SPECTROMETER FREQUENCY, IN MHZ
C
C     ITEM 2. ONE CARD, USED IN ALL MODES.
C       COLS. 1-80, THE TITLE.
C
C     ITEM 3. THIS IS USED IN MODES B AND C UNLESS NPLOT = 0.
C       THIS ITEM CONSISTS OF NPLOT CARDS, ONE PER PLOTTING RANGE.
```

```
C       FOUR F10.0 FORMAT NUMBERS IN COLS. 1-40 ARE, RESPECTIVELY, THE
C       LOWER AND UPPER FREQUENCY LIMITS FOR THE PLOT, THE FREQUENCY
C       INCREMENT, AND THE SCALE FACTOR (SYMBOLS PER UNIT INTENSITY).
C       IF THE SCALE FACTOR IS LEFT BLANK, THE PROGRAM WILL AUTOMATICALLY
C       ADJUST THE STRONGEST LINE TO FULL SCALE.
C
C     ITEMS 4 AND 5 FOLLOW IN MODES A AND C ONLY. THERE WILL BE ONE (4,5)
C     SET FOR EACH SUBMATRIX IN THE MOLECULAR FACTORING SCHEME.
C
C     ITEM 4. A CONTROL CARD FOR NMREN.
C       COLS 1-4, I4. NFLAG. ANY NUMBER GREATER THAN ZERO INDICATES THAT
C       THE CURRENT SUBMATRIX IS THE LAST ONE.
C       COLS. 5-8, I4. MATD. THE DIMENSION OF THE COMPLETE MATRIX.
C       COLS. 9-12, I4. NSUBM, THE DIMENSION OF THE CURRENT SUBMATRIX.
C       THE NEXT TWO NUMBERS ARE USED ONLY IF SOME ENERGY LEVELS ARE TO
C       BE OMITTED FROM THE CALCULATION.
C       COLS. 13-16, I4. NOMIT. THE NUMBER OF LEVELS TO BE OMITTED.
C       COLS. 17-20, I4. NZ. THE INDEX OF THE LEVEL TO BE SET EQUAL TO
C       ZERO. NORMALLY (IF NOMIT IS LEFT BLANK) THE TRACE OF THE SUBMATRIX
C       IS SET EQUAL TO ZERO.
C       COLS. 21-24, I4.IEDIT. ANY NUMBER GREATER THAN ZERO CAUSES THE
C       VARIANCE-COVARIANCE MATRIX OF THE ENERGY LEVELS TO BE PRINTED.
C       COLS. 25-36, F12.0. VAR. THE VARIANCE (A FITTING CRITERION).
C       IF LEFT BLANK, THE PROGRAM SUPPLIES THE VARIANCE.
C
C     IF THERE ARE MISSING ENERGY LEVELS, THEIR INDICES MUST FOLLOW ITEM
C     4, TWENTY TO A CARD IN I4 FORMAT.
C
C     ITEM 5. TRANSITION CARDS, ONE PER OBSERVED TRANSITION ON WHICH THE
C     NMREN COMPUTATION IS TO BE BASED. EACH CARD CONTAINS THE FOLLOWING.
C       COLS. 1-8, 2I4. THE LOWER AND UPPER ENERGY LEVEL INDICES.
C       COLS. 9-18, F10.0. THE OBSERVED TRANSITION FREQUENCY, IN HZ.
C       A BLANK CARD SIGNALS THE END OF THIS ITEM.
C
C     ITEM 6, USED IN MODES B AND C ONLY. CHEMICAL SHIFTS.
C     THIS ITEM CONTAINS NTYPE CARDS, ONE PER GROUP. THEY MUST BE IN THE
C     ORDER SPECIFIED IN ITEM 1.
C       COLS. 1-12, F12.0. THE CHEMICAL SHIFT, IN HZ.
C       IN MODE B ONLY, THE CHEMICAL SHIFTS MAY BE INCREMENTED AS FOLLOWS.
C       COLS. 13-24, F12.0.  THE SHIFT INCREMENT DESIRED, IN HZ.
C       COLS. 25-30, I6. THE NUMBER OF INCREMENTS OF THIS SHIFT.
C       EACH INCREMENT LEADS TO A COMPLETE NEW CALCULATION.
C
C     ITEM 7, USED IN MODES B AND C ONLY.  COUPLING CONSTANT CARDS, ONE
C     PER NON-ZERO COUPLING. EACH CARD CONTAINS THE FOLLOWING.
C       COLS 1,2, 2I1. THE GROUP INDICES. THESE MUST BE CONSISTENT WITH
C       THE LABELING ESTABLISHED IN ITEMS 1 AND 6.
C       COLS. 3-12, F10.0. THE COUPLING CONSTANT, IN HZ.
C       A BLANK CARD SIGNALS THE END OF THIS ITEM.
C
C     IN MODE B ONLY, ITEM 7 MUST BE REPEATED NASET TIMES. THE SECOND AND
C       SUBSEQUENT SETS OF COUPLINGS NEED ONLY CONTAIN THOSE COUPLINGS
C       WHOSE VALUES ARE TO BE CHANGED. EACH SET OF COUPLINGS INITIATES
C       A COMPLETE NEW SET OF SPECTRAL COMPUTATIONS, INCLUDING THE
C       ENTIRE RANGE OF SHIFT INCREMENTS.
C
C     ***********************************************************************
C
C     ANY NUMBER OF PROBLEMS MAY FOLLOW ONE ANOTHER IN ANY ORDER.
C     THE COMPUTER RUN IS TERMINATED BY A BLANK CARD IN PLACE OF ITEM 1.
```

COMUNITA EUROPEA
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La Commissione
CENTRO COMUNE DI RICERCA

COMMUNAUTE EUROPEENNE
DE L'ENERGIE ATOMIQUE
La Commission
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EUROPESE GEMEENSCHAP
VOOR ATOOMENERGIE
De Commissie
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EURATOM

C C R

ISPRA

(ITALIA)

Magnetic Resonance

Ispra, January 20, 1967
HS/mg

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

Dear Barry:

Orientation of Methylcyanide-Methylisocyanide and "low boiling, small molecule contest".

During a study of the interesting five-spin system $H_3{}^{13}C^{14}NC$ I also looked at the ordinary isonitrile to compare it with its isomer acetonitrile.

Apart from the interesting fact that as in the normal spectrum of the liquid one observes a splitting due to the direct dipolar coupling of the nitrogen with the methyl protons in the isonitrile - the scalar coupling of 2.7 cps has not much of an observable effect - it seemed quite interesting to compare the intermolecular forces of the two isomers in different nematic solvents. The results are given in the following table:

Phase	concentr.	$J^{HH}_{CH_3CN}$ dip.	$J^{HH}_{CH_3NC}$ dip.	$J^{H^{14}N}_{CH_3NC}$ dip.
R_1-N=N-R_2	27.6 Mol%	3077 cps	3037	120
R_1-N=N-R_2	27.5 "	3149 cps	3066	123
R_1'-N=N-R_2 (O)	27.6 "	3216 cps	3189	131

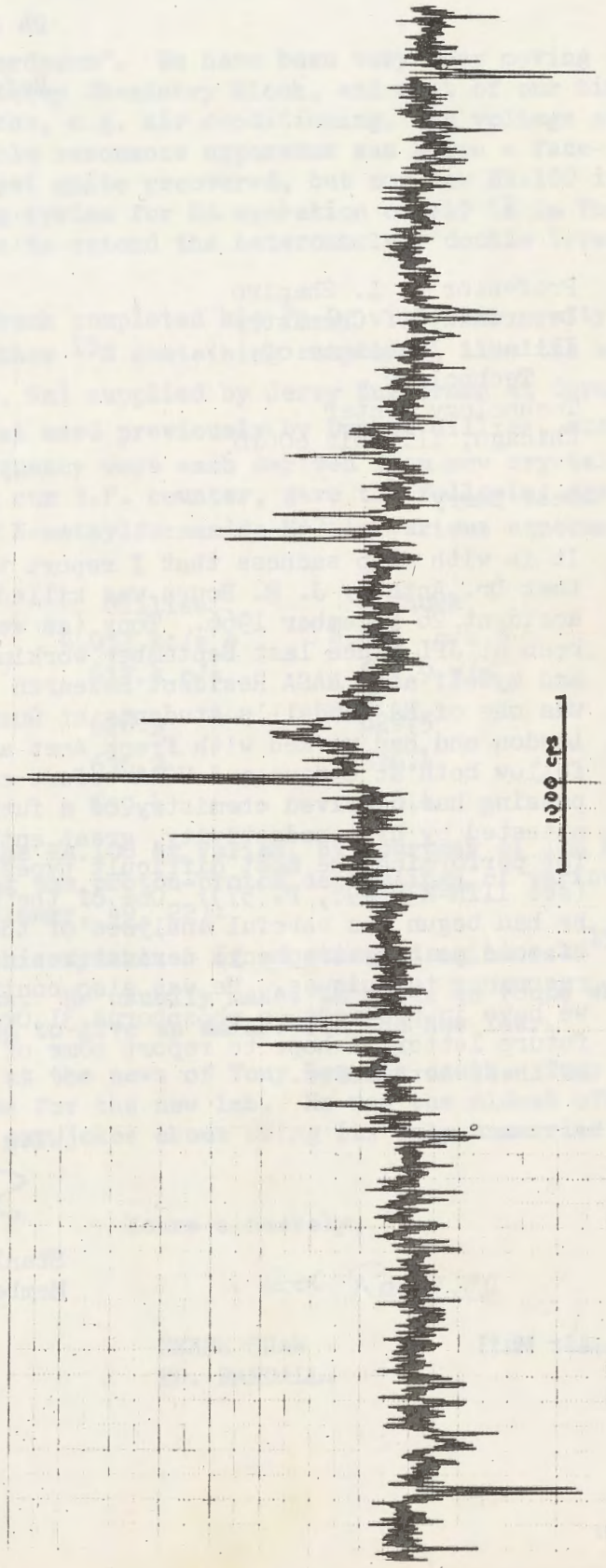
All spectra were taken as 1:1 molar mixtures of the isomers at 34°C. As to be expected the HH and HN couplings go parallel to one another. In all three cases acetonitrile is oriented slightly better than methylisonitrile. This cannot be due to the molecular dimensions since the isonitrile is longer and one would assume a higher degree of order everything else being equal. Unfortunately I have not found a value for the dipol moment of CH_3NC to speculate on other types of possible intermolecular interactions.

Recently I got quite hot on different four-spin systems. As an example I would like to show the spectrum of ethylene in a nematic solvent. As can be seen the degree of order is much less than in acetonitrile which leads me to believe that dispersion

forces only cannot be responsible for the orientation of acetonitrile.

Best regards

Dr. H. Spiesecke



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

24 January 1967

Refer to: 328-SLM:jm

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

Dear Barry:

It is with deep sadness that I report to the NMR community that Dr. Anthony J. R. Bourn was killed in a tragic auto accident 26 December 1966. Tony (as we all knew him) had been at JPL since last September working with Dan Elleman and myself as a NASA Resident Research Associate. Tony was one of Ed Randall's students at Queen Mary College in London and had worked with Frank Anet as a postdoctoral fellow both at Ottawa and UCLA before coming to JPL. His passing has deprived chemistry of a future bright star as attested by his productivity, great enthusiasm and ability for performing the most difficult types of experiments (see IITN-M-R #98, P. 57). One of the pieces of work which he had begun was careful analyses of the fluorine-19 spectra of some pentafluorophenyl derivatives using various multiple resonance techniques. He was also contributing to some work we have in progress on phosphorus-31 Overhauser effects. In future letters I hope to report some of the results from both of these activities.

With best regards,

Stan

Stanley L. Manatt
Member of Technical Staff

Air Mail

Telephone 354-4321 *Twx 213-449-2451*

QUEEN MARY COLLEGE
(UNIVERSITY OF LONDON)
MILE END ROAD E.1
TELEPHONE ADVANCE 4811

Even more N^{15} work

23rd January, 1967.

Professor B.L. Shapiro,

Dear Barry,

Our apologies for being 'overdrawn'. We have been very busy moving into our excellent quarters in the nine-storey Chemistry block, and most of our time seems to be spent in testing the services, e.g. air conditioning, and voltage stablised supplies. Our heteronuclear double resonance apparatus was given a face-lift during the move, from which it has not yet quite recovered, but our new HA.100 is in commission, complete with an extended locking system for HA operation on F19 (à la Phillips, Jenkins, and Turner), and we hope to extend the heteronuclear double irradiation facility to it.

Just before the move, Rod Chuck completed his Ph.D. with some pretty $H(N^{15})$ "tickling" work on $^{15}NH_4$ + and other ^{15}N containing compounds, like the samples of $Me_3MN^{15}H\ C_6H_5$ (where M = Si, Ge, Sn) supplied by Jerry Zuckerman at Cornell. The apparatus was essentially that used previously by Duncan Gillies, except the basic proton frequency and the ^{15}N frequency were each derived from new crystals. A check on the new instrumentation using our R.F. counter, gave the following agreement for the ^{15}N resonance frequencies of N-methylformamide-N^{15} in various aqueous solutions (referred to a common field).

% Concentration	Gillies	Chuck
	4,053 kc/s +	4,053 c/s +
100	617.4 c/s	617.9 c/s
75	628.9	629.5
50	636.2	636.4
25	639.4	640.4

Rod also gained his "wings" on the HA.100 at Varian, by courtesy of Jim Feeney, with whom he collaborated in reworking the proton-proton decouplings of various Santonins previously done at 60 M c/s (Mellonmr, 49, 22).

Malcolm Bramwell continues his syntheses of substituted anilines-N^{15} for N^{15} shift and $J(N^{15} - H)$ measurements. He usually makes an amide en route which is most disturbing, since we had resolved to give up amides for the New Year.

We have all been depressed at the news of Tony Bourn's death. Tony had helped in formulating our plans and designs for the new lab. He was the oldest offspring of our group here, and the butt of our jokes about being its only unmarried member. Sad, sad, sad.

Yours sincerely,

Ed Randall

Derek Shaw

DEREK SHAW
ED. RANDALL.

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

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

Styrene Derivative Complexes of Platinum

Dear Professor Shapiro:

We (with Prof. Kinugasa) have observed proton magnetic resonance spectra of styrene derivative complexes of platinum in an attempt to elucidate the nature of bonding in the complexes. It has been found (Table I) by the analysis of the spectra that for the ethylenic protons the nuclear magnetic shielding increases while the spin-spin coupling decreares on complexing with platinum. And it is worthy of notice that the values of Jpt-H are comparable in magnitude to those in alkyl-platinum complexes, where the carbon is σ-bonded to the platinum through an sp^3 orbital of the carbon. If only the π orbitals of the ethylenes were involved in the bonding, the coupling constants would not be as large as observed, the protons lying in the nodal plane of the π orbitals. This leads us to infer that the ethylenic carbon orbitals have some sp^3 character and the protons are displaced slightly away from the nodal plane. The slight saturation of the double bond by complexing with platinum may by partly responsible also for the increased shielding and decreased coupling of the ethylenic protons on complexing. Furthermore, polar effects of the substituents on the shifts of the ethylenic protons are generally lessened by complexing. Most of the experimental evidence appears to suggest the decreased double-bond character in the complexes.

Yours sincerely,

A. Saika

A. Saika

Table I. Chemical Shifts and Coupling Constants

	Complex									Free Ligand					
	Chemical shifts[a]			Coupling constants[b]						Chemical shifts[a]			Coupling constants[b]		
Substituent	δ_A	δ_B	δ_C	J_{AB}	J_{AC}	J_{BC}	J_{A-Pt}	J_{B-Pt}	J_{C-Pt}	δ_A	δ_B	δ_C	J_{AB}	J_{AC}	J_{BC}
p-CH_3O	370.1	284.0	245.4	13.6	8.5	0.7	73	61	70	400.8	338.1	304.3	17.5	11.1	1.1
p-CH_3	371.9	288.7	250.1	13.9	8.5	0.7	74	61	69	402.6	343.7	309.1	17.6	10.8	1.1
H	372.6	290.9	252.4	13.8	8.5	0.7	74	61	69	404.9	347.8	313.2	17.7	11.0	1.1
p-Cl	369.4	289.6	254.2	13.7	8.5	0.6	74	60	68	406.7	349.0	315.8	17.9	11.0	0.9
m-NO_2	372.8	297.4	261.3	13.0	8.1	0.3	75	60	67	414.3	362.1	327.5	17.7	10.9	0.5
p-NO_2	373.3	294.9	260.5	13.2	8.3	0.3	75	60	67	414.2	364.1	331.3	17.6	11.1	0.6

[a] In cps downfield from TMS as internal standard at 60 Mc. [b] In cps.

CARLETON UNIVERSITY

OTTAWA 1, CANADA

DEPARTMENT OF CHEMISTRY

January 17, 1967.

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

Dear Dr. Shapiro:

"More ballistic missiles. Possibility of position available for P.D.F."

We are continuing with the effects of anisotropic groups in the spectra of conformationally rigid molecules[1]. Here at Carleton we are interested in the effect of the epoxide group with possible extensions to aziridines, cyclopropyls and episulfides.

In the laboratory we are making suitable model compounds and haven't run a single spectrum yet! However, we have been thinking about the definition of the location of these groups using a model similar to that we have described before[2] and we ask for any suggestions or desire on the part of your readers for similar measuring devices for any functional groups. Our particular worry is to get some idea of the lone pair effects and perhaps the physical chemists out there can help this lowly organicer in the frozen North.

There is a possibility that I may have an opening for a Post-Doctoral to work in this field next year, and applications together with two references are solicited.

Yours sincerely,

J. W. ApSimon

JWA:jf

John W. ApSimon.

References

1. Chem. Comm. 359, 361, 754 (1966).

2. IIT NMR 92 - 46.
Chem. and Ind. 1792 (1966).

P.S. ALTERNATE TITLE – How to keep up your subscription without anything to report!

DUQUESNE UNIVERSITY
PITTSBURGH, PENNSYLVANIA
15219

Jan. 20,1967

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

Dear Barry:

Postdoctoral position available

Thank you for your reminder to send in our subscription to IIT NMR Newsletter. I should like to use this opportunity to bring to the attention of readers that a postdoctoral position will be available in our Department in September. We have a Varian A-60 instrument, and a time-averaging computer is due to arrive next month.

We are concerned with NMR study of hydrogen bonding and metal-ion complexation with purine and pyrimidine derivatives, peptides and amides, in aqueous, nonaqueous and mixed solvents. Since the only sure way of correcting for solvent effects is not to use any solvent, and since this is not possible with our systems, we are investigating systematically the nature of solvent interaction with the various species. Another portion of our NMR research has to do with the measurement of fast proton-exchange rates and activation energy, together with the role of solvent in proton-exchange rate processes, for the model amides.

Example of a paper from our Laboratory, dealing with the above system: "Proton Magnetic Resonance Studies of Metal Complexes of Imidazole, Purine and Pyrimidine Derivatives", S.M.Wang and N.C.Li, J. Am. Chem. Soc., 88 , 4592 (1966).

Sincerely yours,

Norman

Norman C. Li

Professor of Chemistry

MELLON INSTITUTE
4400 FIFTH AVENUE
PITTSBURGH, PA. 15213

January 26, 1967

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

Dear Barry:

Re: POSTDOCTORAL POSITION AVAILABLE

I would like to use I.I.T.N.M.R.'s columns to advertise for a research fellow.

This is an S.R.C. Postdoctoral research fellowship tenable at the University of Liverpool. Salary, F.S.S.U. benefits in accord with S.R.C. regulations; commencing date preferably about autumn of this year, when I will be back in Liverpool.

The areas of interest include high resolution N.M.R. (various nuclei), I.R. (particularly with relation to rotational isomerism studies) and theoretical N.M.R. calculations.

Anyone interested can contact me at the above address.

With best wishes,

Yours sincerely,

Ray

R. J. Abraham

RJA:sa

UNIVERSITY OF HOUSTON
CULLEN BOULEVARD
HOUSTON, TEXAS 77004

CHEMISTRY DEPARTMENT

February 6, 1967

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

Dear Barry:

The interest of the organic chemist is always increased when they see a report of an anomalous NMR spectra.

Meinwald (J. Am. Chem. Soc., 87, 3532 (1965)) reports a photo-isomer of α-phellandrene possesses an intact isopropyl group that exhibits a singlet resonance in the methyl region at about 1ppm. This behavior seems to be in substantial disagreement with the behavior that most of us would have predicted. Therefore, it seems reasonable that Einar Goerland, a senior at the University of Houston, should prepare some of this photo-isomer for NMR examination at 100 megacycles. We have done so and summarize some of our observations here. The vinyl resonance is not exceptional and needs no description. An allylic methyl group appears at ca. 1.95 ppm and the methyl of the isopropyl group appears at ca. .95 ppm. The ratio of intensities of these two resonances is within an experimental error 3:7. It thus appears certain that the appearance of the isopropyl group as a singlet is due to an unfortunate coincidence of chemical shifts of the methyl and methine protons.

Nugent Chamberlain has informed us that isopropyl cyclopropane also exhibits a badly distorted doublet for the isopropyl group and explains this result in the same manner.

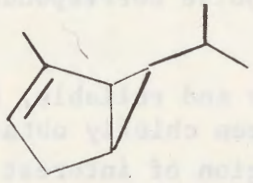
I would like to know if other readers of IITNMR have observed similar behavior of isopropyl groups.

Sincerely yours,

Bob

M. R. Willcott, III
Assistant Professor

MRW:la



Meinwald's isomer

MELLON INSTITUTE
4400 FIFTH AVENUE
PITTSBURGH, PA. 15213

20 January 1967

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

Dear Barry:

In the IITNMR Newsletter (Nov. 1965) we described an analog device for electronically sweeping a voltage variable frequency source. Since that time we have designed and built an all-solid state digital sweep instrument which has the added advantage of simultaneously controlling the frequency and advancing the address of Northern Scientific NS 544 (or similar) time averaging computer.

Operation of the system is as shown in Fig. 2. The sweep advances the 'cat', the recorder x-axis and the VCXO uniformly in time. Output of the VCXO feeds an A.C.amplifier, the output of which simultaneously passes to the coils and to the reference input of a phase sensitive detector. Detector signal output feeds a mode selector switch. A D.C. amplifier with a gain of 100, variable base line and filtered output precedes the NS544.

Referring to Fig. 3: The 'clock' output is ca. 100 HZ variable which acts as the master time base. The 'blanker' permits the sweep to be stopped at any point (frequency) and restarted from that same point. A spectrum can thus be calibrated during a run by stopping the sweep at several points and reading the frequency counter. In time averaging operation the end points can be accurately determined and monitored. 'Shaping' circuitry insures linearity and accurate advance. Eight sweep times (0.5, 1.0, 2.0, 4.0, 8.0, 16.0, 32.0, 64.0 minutes) are provided by the ten stage 'divider' which transfers the new time base to the 'ripple-carry counter'. Binary outputs are 'summed' in a selected resistor network. The resistance value for the fastest flip-flop being 2^{10} times the value for the slowest. A variable base line i.e. starting point, and an output adjustable from 0 to 7 V are provided in the 'final amplifier'. Thus the selection of frequency ranges is virtually limited by the frequency source. In our application the range is 0 to 1 MHZ using a G.R. 1164 A frequency synthesizer. 'Reset' immediately interrupts the sweep and returns it to the origin. Automatic reset is understood after 1024 steps. The 'gate advance' is the time base or 'stepper' for the NS544. A 0.625 usec triggering pulse corresponds to the leading edge of each step.

We have found the unit to be quite stable and reliable, reproducible spectra can be accumulated easily. Results have been chiefly obtained on the HR-60 in frequency sweep mode, scanning the region of interest while the

field is locked on a convenient sideband. The A-60 has been modified for digital field sweep and spectrum accumulation. The A-60 D. C. sweep mechanism is by-passed and replaced by the digital sweep through a resistor network. Variable baseline adjustment is employed to locate the starting point and to calibrate the sweep offset. In this operation, the reproducibility depends heavily upon spectrometer stability and some line broadening is apparent during extended accumulations. Although we have made no attempt as yet, we see no major obstacle in adapting the system to the HA-100.

Figure 1 shows the methylene proton resonance of 1% ethylbenzene in CCL_4. The "before" spectrum is straight out of the D. C. amplifier. Maximum enhancement was obtained after 110 scans of 1 minute each through the region 825.5 HZ to 859.7 HZ from TMS. It should be noted that the recorder sweep widths are not on the same scale.

Very truly yours,

Dennis Wisnosky

Dennis Wisnosky

Robert A. Bothner-By

DW:HB

P. S.: A complete set of schematics are available upon request.

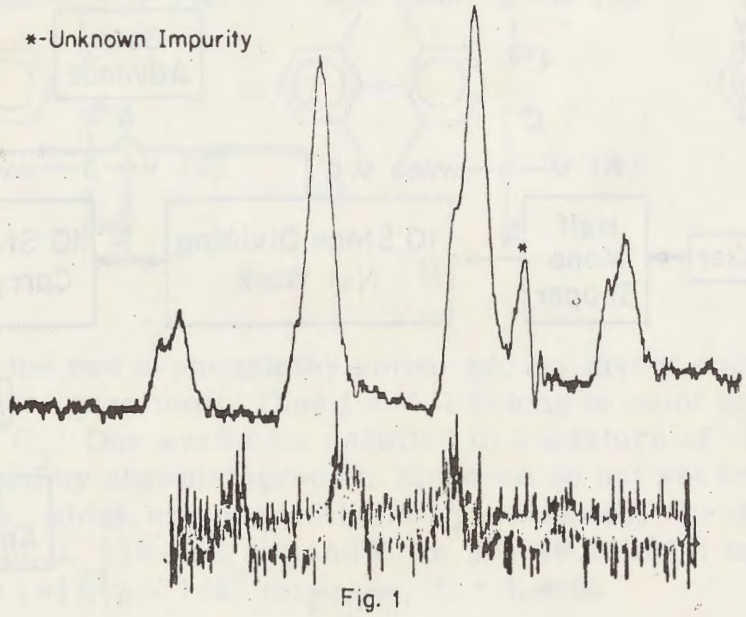
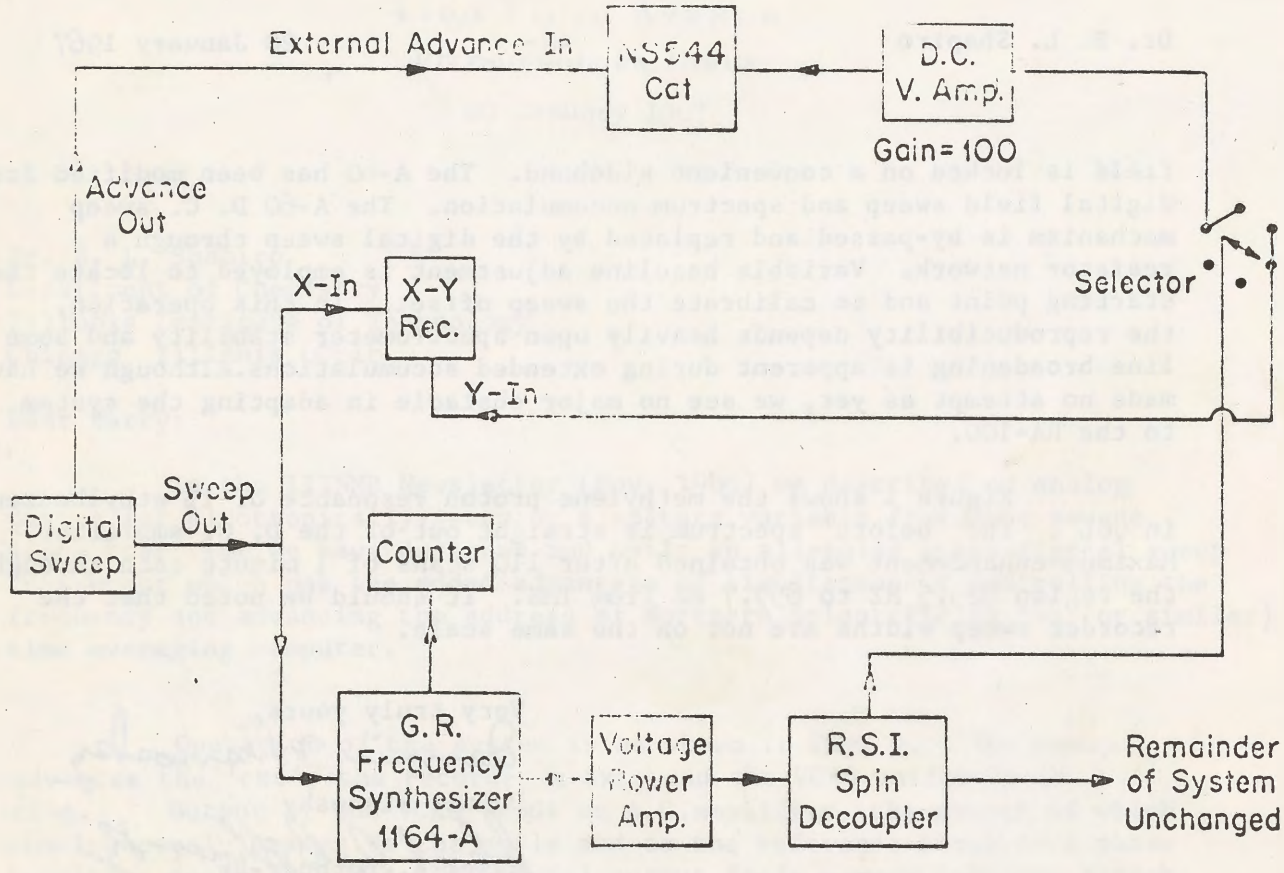


Fig. 1


External Advance In
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Cat
D.C.
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Gain=100
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X-In
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Y-In
Sweep
Out
Digital
Sweep
Counter
G.R.
Frequency
Synthesizer
1164-A
Voltage
Power
Amp.
R.S.I.
Spin
Decoupler
Remainder
of System
Unchanged

Fig. 2

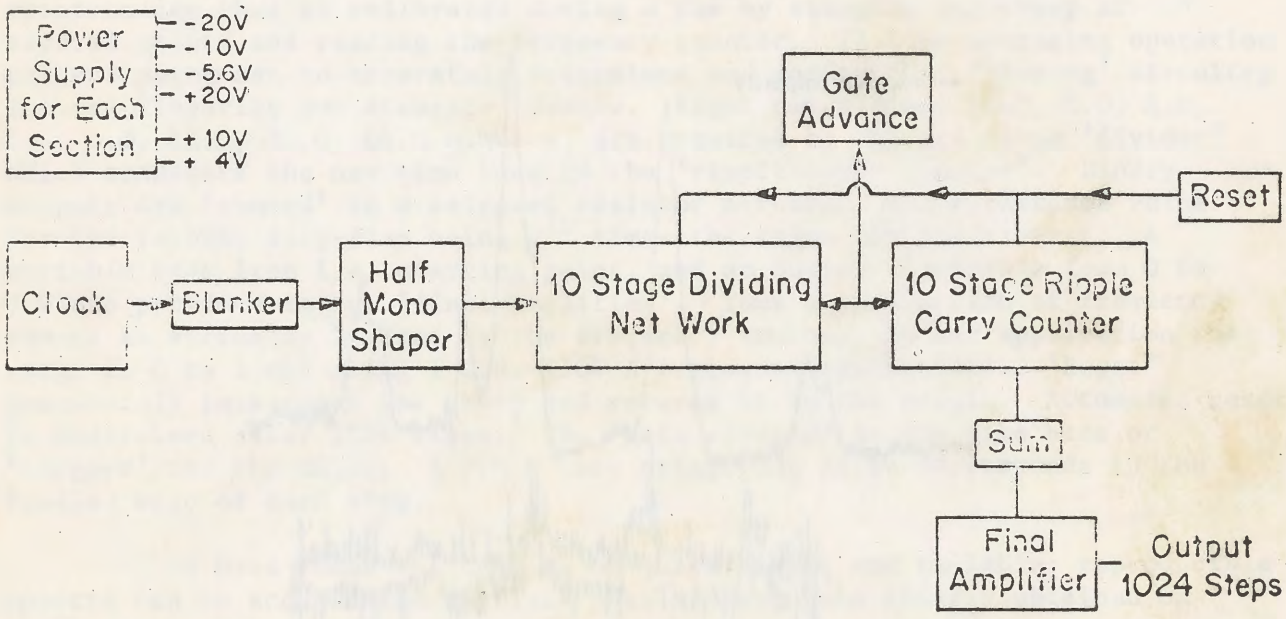

Power
Supply
for Each
Section
-20V
-10V
-5.6V
+20V
+10V
+4V
Gate
Advance
Reset
Clock
Blanker
Half
Mono
Shaper
10 Stage Dividing
Net Work
10 Stage Ripple
Carry Counter
Sum
Final
Amplifier
Output
1024 Steps

Fig. 3

Eidg. Technische Hochschule
Laboratorium für Organische Chemie
Zürich

8006 Zürich,
Universitätstr. 6/8
Tel. 051 / 32 73 30

January 24, 1967

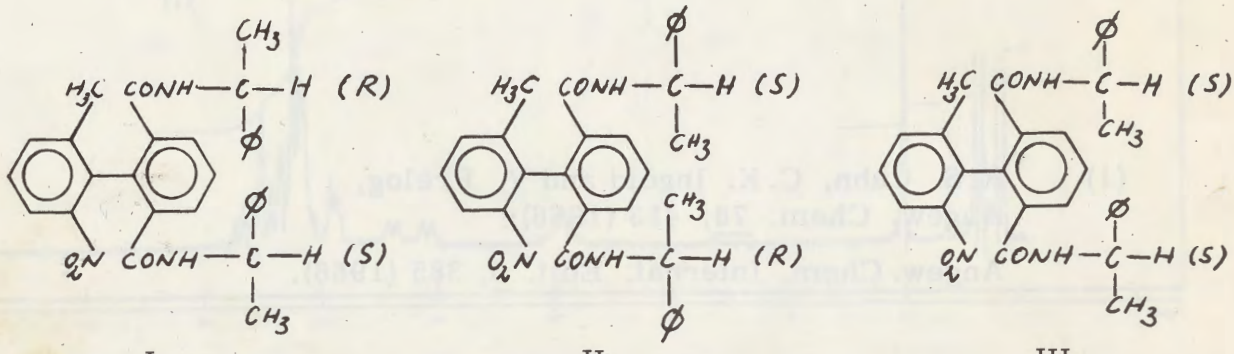
G. Helmchen

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

NMR-Spectra of Biphenyls with a Pseudo-asymmetric Axis.

Dear Professor Shapiro,

The concept of pseudo-asymmetry as applied so far only to atoms as centers has been extended by R. S. Cahn, C. K. Ingold, and V. Prelog [1] to comprise also axes and planes. In the well-known case of the trihydroxyglutaric acids one can specify a pseudo-asymmetric carbon atom giving rise to two optically inactive diastereoisomers. In analogy, molecules having a pseudo-asymmetric axis can be conceived. To our knowledge, compounds of the latter kind have not been prepared yet. We have therefore synthesized examples with structures I and II, and for reasons of comparison also III.



I II III

In I and II the two α-phenylethylamino groups are of enantiomeric, in III of identical configurations. Thus I and II belong to point group C_s, whereas III belongs to C_1. Our synthesis resulted in a mixture of I and II, which could be separated by chromatography. Since we do not yet know the relative configurations, which are currently being elucidated, the diastereoisomers are called A (m. p. 152-152,5°) and B (m. p. 180,5-181°) in this context. III is an oil with $[\alpha]^{23}_{578}$ - 146° (benzene, C = 2,495).

In the NMR-spectra (100 MHz, $CDCl_3$) of either A or B the corresponding protons of the two α-phenylethylamino groups absorb at the same frequency, since they are enantioscopic (enantiotopic [2]). On the other hand, they are diastereoscopic (diastereotopic [2]) in III, which is reflected by a difference in their NMR-absorptions. The three NMR-spectra of A, B and III show differences revealing the diastereoisomerism of these compounds.

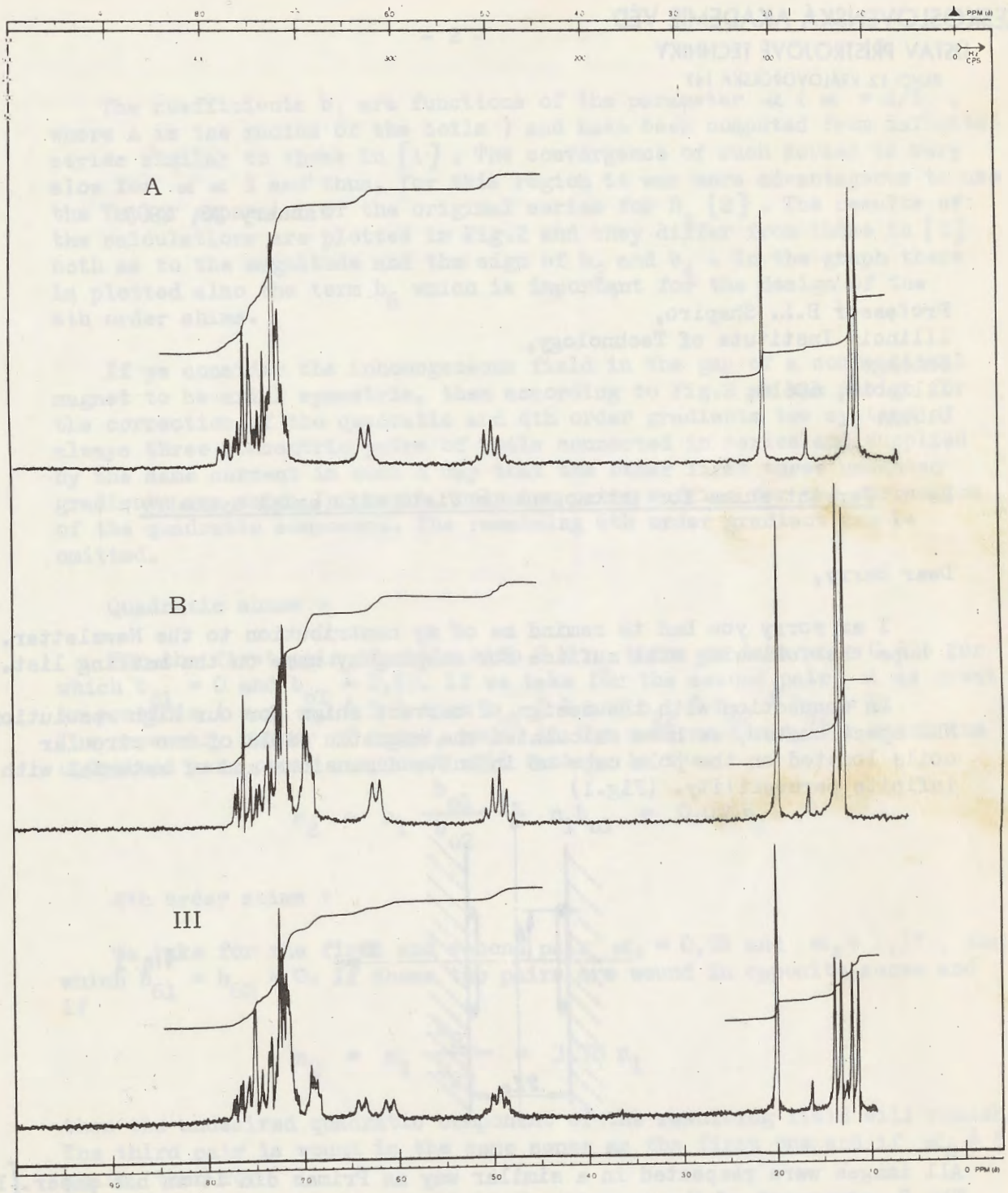
With kind regards from Professor V. Prelog

Sincerely yours,

Günter Helmchen

(1) R. S. Cahn, C. K. Ingold and V. Prelog, Angew. Chem. 78, 413 (1966); Angew. Chem. Internat. Edit. 5, 385 (1966).

(2) K. Mislow, M. Raban and P. Bickart IITNMRN 94-8 (1966)


A
B
III
PPM (δ)
Hz
CPS
0 PPM (δ)

ČESKOSLOVENSKÁ AKADEMIE VĚD
ÚSTAV PŘÍSTROJOVÉ TECHNIKY
BRNO 12, KRÁLOVOPOLSKÁ 147

January 26, 1967

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

Re : Current shims for inhomogeneous field with axial symmetry

Dear Barry,

I am sorry you had to remind me of my contribution to the Newsletter. I hope the following will suffice for keeping my name on the mailing list.

In connection with the design of current shims for our high resolution NMR spectrometer, we have calculated the magnetic field of two circular coils located on the pole caps of infinite dimensions and of material with infinite permeability. (Fig.1)

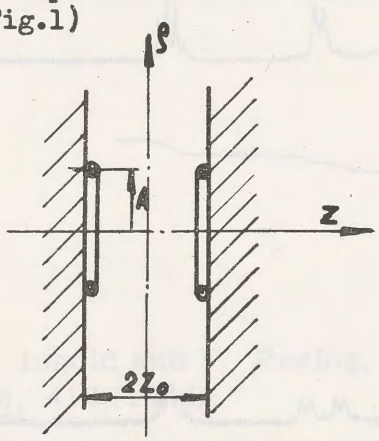


Fig. 1

All images were respected in a similar way as Primas did it in his paper.[1] The B_z-component of the magnetic field in the direction of the axis of the coils has been, however, expressed in a form

$$B_z(\rho, z=0) = \frac{nI\mu_0}{Z_0}\left[b_0 + b_2\left(\frac{\rho}{Z_0}\right)^2 + b_4\left(\frac{\rho}{Z_0}\right)^4 + b_6\left(\frac{\rho}{Z_0}\right)^6 \cdots\right]$$

which gives a better survey of properties of the system. Here ρ and z are the cylindrical coordinates with the origin at the centre of symmetry, n represents the number of turns, I the current flowing through the coil, μ_0 the permeability of free space and $2Z_0$ the width of the gap.

The coefficients b_j are functions of the parameter α ($\alpha = A/Z_o$, where A is the radius of the coils) and have been computed from infinite series similar to those in [1]. The convergence of such series is very slow for $\alpha < 1$ and thus, for this region it was more advantageous to use the Taylor expansion of the original series for B_z [2]. The results of the calculations are plotted in Fig.2 and they differ from those in [1] both as to the magnitude and the sign of b_2 and b_4. In the graph there is plotted also the term b_6 which is important for the design of the 4th order shims.

If we consider the inhomogeneous field in the gap of a conventional magnet to be axial symmetric, then according to Fig.2 we can design for the correction of the quadratic and 4th order gradients two systems of always three concentric pairs of coils connected in series and supplied by the same current in such a way that the other first three unwanted gradients are zero. In praxis, only two pairs suffice for the correction of the quadratic component. The remaining 6th order gradient can be omitted.

Quadratic shims :

For the first pair of coils with $2 \cdot n_1$ turns we take $\alpha = 0{,}835$ for which $b_{41} = 0$ and $b_{01} = 0{,}69$. If we take for the second pair α as great as possible (e.g. $\alpha_2 \doteq 5$) then $b_{02} \doteq 1$ and $b_{22} \doteq b_{42} \doteq b_{62} = 0$. The necessary number of turns, wound in the reverse sense, for which the undesired zero order component will vanish, is then

$$n_2 = n_1 \frac{b_{01}}{b_{02}} \doteq n_1 b_{01} = 0{,}69\, n_1$$

4th order shims :

We take for the first and second pair $\alpha_1 = 0{,}56$ and $\alpha_2 = 1{,}37$, for which $b_{61} = b_{62} = 0$. If these two pairs are wound in opposite sense and if

$$n_2 = n_1 \frac{b_{21}}{b_{22}} = 3{,}78\, n_1$$

then the undesired quadratic component of the resulting field will vanish. The third pair is wound in the same sense as the first one and if $\alpha_3 \doteq 5$ again, then for

$$n_3 = n_2 b_{02} - n_1 b_{01} = 3{,}07\, n_1$$

the zero order component of the whole system will vanish too.

In praxis, however, because of the finite dimensions of pole caps and because of their finite permeability, there will be a slight difference between the calculated and real value of the particular field gradients.

This is especially true for the zero order component at high values of α. It appears, therefore, advantageous to use a variable resistor connected in parallel with a part of the system and to adjust empirically this resistor for the vanishing of the zero order component. In the case of quadratic shims, the resistor is connected in parallel with the first pair of coils and in the case of the 4th order shims it is connected in parallel with the first and second pair of coils. Similar possibility exists for elimination of the remaining second order component in the 4th order system, but in praxis, this is rather difficult.

Sincerely yours

Josef Dadok

Institute of Scientific Instruments of
the Czechoslovak Academy of Sciences

Ref. : 1. H. Primas and H. Günthard : Helv. Phys. Acta 30, 331 (1957)
2. J. Dadok : To be published.

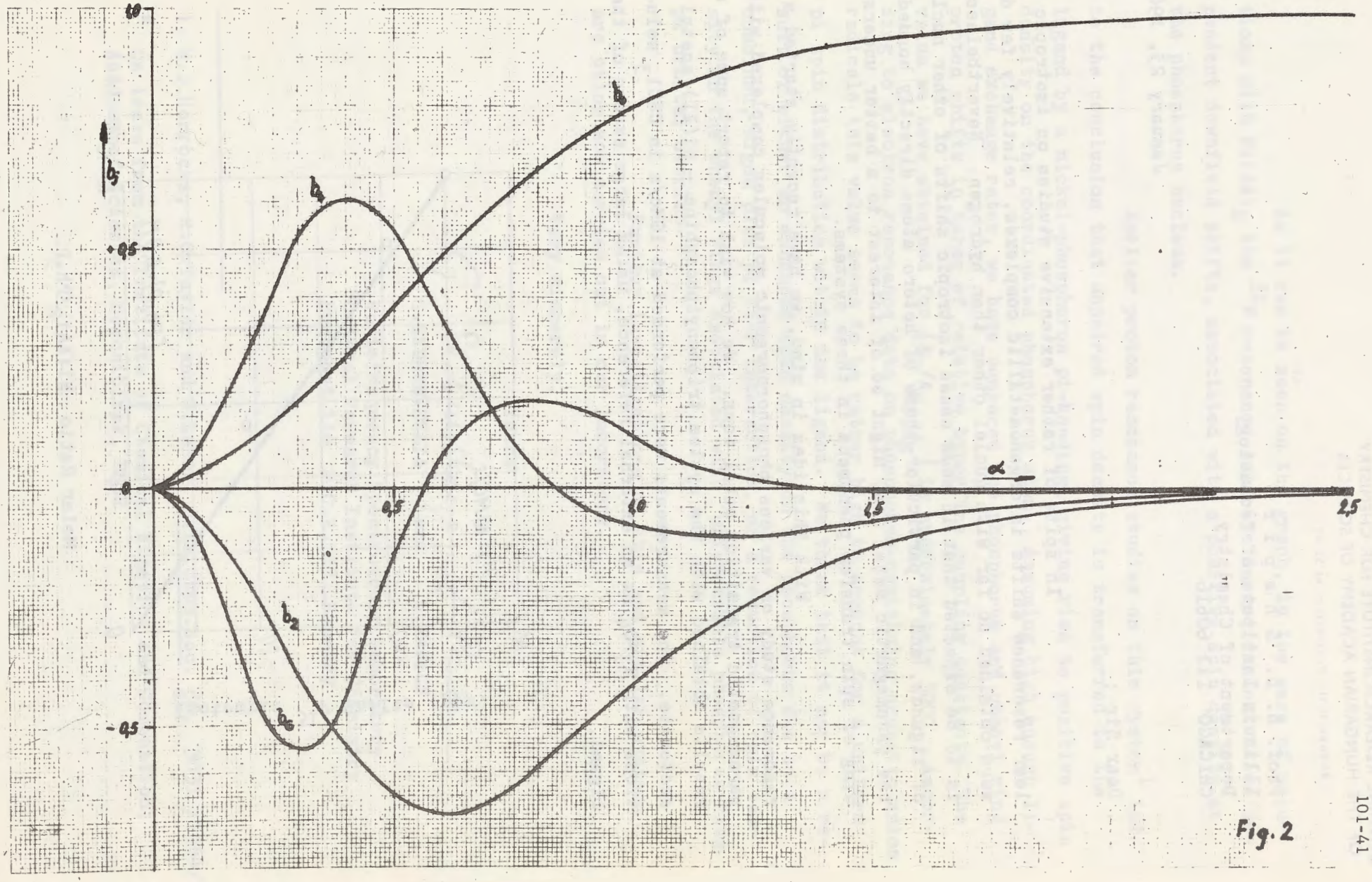


Fig. 2

CENTRAL RESEARCH INSTITUTE FOR CHEMISTRY
OF THE HUNGARIAN ACADEMY OF SCIENCES
Budapest, II., Pusztaszeri út 57-69

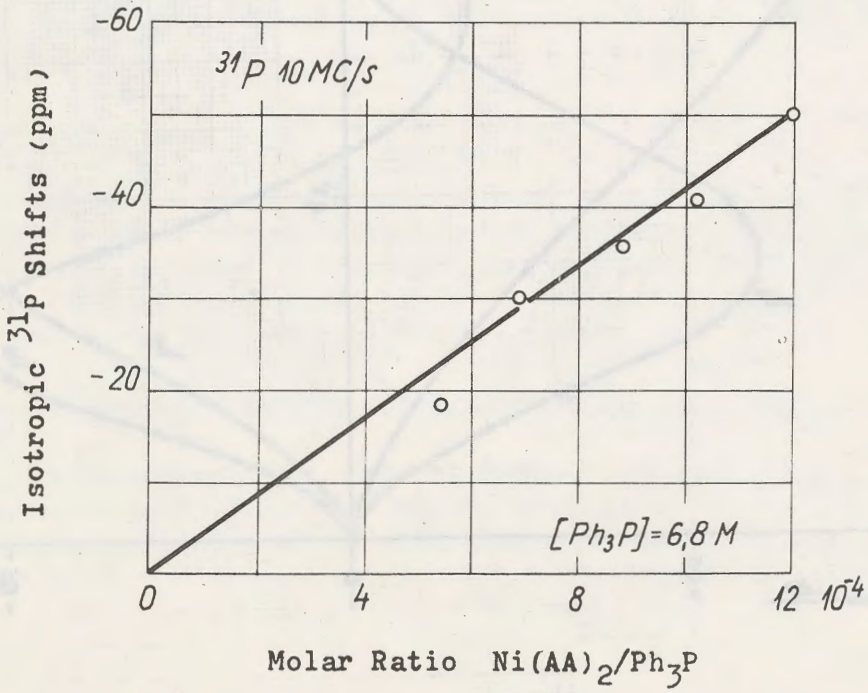
Prof. B. L. S h a p i r o
Illinois Institute of Technology
Department of Chemistry
CHICAGO Ill 60616

January 23, 1967

Dear Sir :

In spite of rather extensive studies on isotropic nuclear resonance shifts in organometallic complexes, relatively few data were obtained so far with nuclei other than hydrogen. Nevertheless, it is to be expected that in some cases isotropic shifts of other nuclei of ligands, and in particular those of hetero atoms directly bonded to the paramagnetic metal ion, might be of interest to a better understanding of spin transfer phenomena in these systems.

With this idea in mind, we have recently started a ^{31}P resonance study on various phosphoroorganic molecules complexed with paramagnetic transition metal ions. We now wish to report some of our results obtained with the system triphenylphosphine - Ni(II) acetyl - acetonate. The measurements were performed at 10Mc/s in $CHCl_3$ solutions with H_3PO_4 as an external reference, using large excess of the ligand.



As it can be seen on the graph, in the case of solutions with $Ni(AA)_2$ the ^{31}P resonance exhibits large, concentration dependent downfield shifts, associated with a positive spin density at the phosphorus nucleus.

Earlier proton resonance studies on this system[1] led to the conclusion that unpaired spin density is transferred to the ligand by a nickel-phosphorus pi-bonding, giving rise to positive spin density on the coordinated phosphorus atom. Assuming high enough ligand exchange rates, we have compared the phosphorus and phenyl ring proton shifts in terms of relative hyperfine coupling constants. The value we have obtained for $|A_P/A_{H_{ring}}|$ is approximately 3000. According to electron resonance data on phosphorus-containing aromatic anion radicals this value seems to be rather high to account for a uniform pi spin distribution within the ligand. We think that it may be a result of either an enhanced spin density on the phosphorus due to a combined, sigma plus pi Ni-P interaction, or a reduction of spin density on the phenyl rings associated with unfavourable phosphorus-ring pi orbital overlap.

Further investigations of this and similar systems are being now carried out in our laboratory.

Very sincerely,

Yours

G. Skubnevskaya L. Radics

G.SKUBNEVSKAYA* and L.RADICS

Radiofrequency Spectroscopy Laboratory
Central Research Institute for Chemistry
Budapest 114 P.O.B. 17 Hungary

---

1. W.D.Horrocks, R.C.Taylor and G.LaMar, J.Am.Chem.Soc. 86, 3031 /1964/

* On leave from the Institute of Chemical Kinetics and Combustion, Academy of Sciences, Novosibirsk, USSR

RESEARCH LABORATORIES
GENERAL MOTORS CORPORATION

25 January 1967

Professor Barry L. Shapiro
Editor, IIT NMR Newsletter
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry: Subject: Reprints and preprints available.

Lately my interests have been almost exclusively in the areas of NQR and wideline NMR of "plastic crystals". I have available some copies of reports on "Nuclear Spin-Spin Coupling, Hyperfine Coupling, and Effective Nuclear Change for Outer s-Electrons" (GMR-444), and "Some Computer Programs for Calculation of NMR Parameters" (GMR-540), for those interested.

Here are more recent items:

"Proton Magnetic Resonance of Solid Triethylenediamine - Molecular Structure and Motions," J. Chem. Phys. 43, 4325 (1966).

"Nuclear Quadrupole Resonance by Nuclear Induction: Theory and Experiment; With an Extension of the Theory to Absorption Methods," Phys. Rev. 149, 346 (1966).

"Molecular Rotation and Diffusion in Solid NF_3," J. Chem. Phys. 45, 3483 (1966).

"Plastic Crystals," General Motors Research Publication GMR-608, 11 November 1966. (A modified version of this report appeared in International Science and Technology as "Liquid-Like Solids," Int. Sci. & Tech., 1 January 1967, No. 61, p 72.)

Yours truly,

George

George W. Smith
Physics Department

ks

GENERAL MOTORS TECHNICAL CENTER
12 MILE AND MOUND ROADS WARREN, MICHIGAN 48090

Université Libre de Bruxelles

FACULTE DES SCIENCES
Avenue F.-D. Roosevelt, 50

Service : Organic chemistry

Dir.: Prof.R.H.MARTIN.

(A rappeler dans la réponse)

Bruxelles 5, le January 28, 1967.

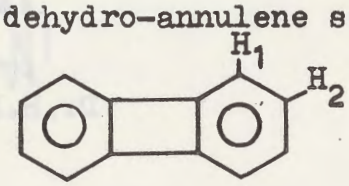
INDUCED PARAMAGNETIC RING-CURRENT IN BIPHENYLENE AND RELATED HYDROCARBONS.

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

Dear Professor Shapiro,

We have recently carried out LCAO calculations of the ring-current effects in biphenylene using the perturbation theory developped by McWEENY [1][2]. The required exchange integrals for the various bonds were calculated by a modified WOLFSBERG-HELMHOLTZ approximation [3] [4] from the values of the X-rays analysis of MAK and TROTTER [5].

The results show that the six-membered rings have comparatively small diamagnetic ring-currents, while the central four-membered ring has a large paramagnetic ring-current (+ 0,536 and -1,028 respectively; benzene = +1). This is an interesting new example of a "reversed" ring-current, other cases being known in the 4 Nπ-electron annulene and dehydro-annulene series [6][7][8].



The calculated chemical shifts are in good agreement with the experimental values of CAVA [9] and KATRITZKY [10], as shown in Table 1. The theory however suggests that proton 2 is more deshielded than proton 1, as opposed to KATRITZKY's attribution based on STREIT-

WIESER's presumptive evidence that tritiation (by extrapolation deuteration) goes faster in the 2 than the 1 position [11].

TABLE 1.*

"Shielded" hydrogen	"Deshielded" hydrogen	H resonance in benzene	ΔH_{1-2} (cps)	Ref.
6,598	6,702	7,342	6,24	9**
6,47	6,60	7,24	7,80	10$
6,662	6,756	7,276	5,64	Calc.

* Chemical shifts expressed in ppm from TMS, unless otherwise stated.

** Measurements : 5% in $CDCl_3$.

$ Measurements : 0,5 M. in cyclohexane.

In order to settle this problem experimentally, we plan to synthesize biphenylenes deuterated in known positions.

Calculation of the ring-currents of benzo[b]biphenylene also reveals an induced paramagnetic ring-current in the four-membered ring; here again, calculated chemical shifts are in fair agreement with the experimental values [12]. Full details will be published shortly.

Please credit this letter to Professor R.H.MARTIN, Head of the Department.

With kind regards,

Yours sincerely,

Dr.H.P.FIGEYS.

REFERENCES

1. R.McWEENY, Mol.Phys. 1, 311 (1958).
2. H.P.FIGEYS, Tetrahedron Letters, 4.625 (1966).
3. M.WOLFSBERG and L.HELMHOLTZ, J.Chem.Phys. 20, 837 (1952).

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11. A.STREITWIESER and I.SCHWAGER, J.Am.Chem.Soc.85, 2.855 (1963).

12. R.H.MARTIN, J.P.VAN TRAPPEN, N.DEFAY and J.F.W.Mc OMIE, Tetrahedron 20, 2.373 (1964).

Universidad de Buenos Aires
Facultad de Ciencias Exactas
y Naturales

Buenos Aires; January 30 1967

Professor Bernard L. Shapiro.
Illinois Institute of Technology.
Chicago.

Dear Dr. Shapiro:

Negative J_{26} ring coupling in 3-substituted pyridines

On applying the Ipdor technique (J.Mol.Spectr. 20, 203, 1966) to several 3-X pyridines (X: CN; OC-CH_3; OCH; Br; Cl; CH_3) it was possible to obtain the line positions of all the lines of protons 2 and 6 in spite of the quadrupolar broadening of their signals by the ^{14}N.

In this way, and after recording and measuring about 600 spectra, it was possible to detect negatives J_{26} couplings in all the 3-pyridines studied. The results (obtained in collaboration with a Physics student, E. Ferrá) do not show any regularity of j_{26} with substituent.

It is interesting to compare the almost superimposed C_3 C_4 D_1 D_2 lines in the normal 3 Cl pyridine spectrum (Kowalewski; J.Chem.Phys. 36, 266, 1962) with their clear line-shapes obtained by this technique.

Please, credit this note to our B.A. Laboratory, as unfortunately for our NMR work, the amount of Valdemar various non-research duties is still enormous.

Yours sincerely,

Dora G. de Kowalewski.

Duke University
DURHAM
NORTH CAROLINA

DEPARTMENT OF CHEMISTRY

POSTAL CODE 27706
TELEPHONE 919-684-2414

February 7, 1967

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

Dear Professor Shapiro, Re: P^{31} Chemical Shifts

We have recently measured the P^{31} chemical shifts of some isomeric pairs of heterocyclic organophosphorus compounds and think that the data given below may be of interest to others working with P^{31}-NMR.

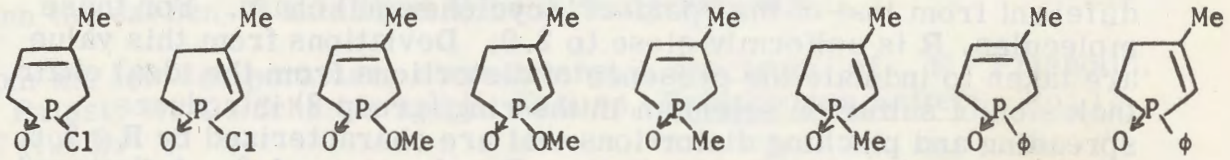
The chemical shifts were measured on a Varian 4300B spectrometer at 19.3 Mc. All values reported are with respect to external 85% H_3PO_4 with the + sign indicating an upfield shift, though in fact P_4O_6 was used experimentally for the phospholene oxides. The samples were provided by Dr. L. D. Quin and his research group. Spectra were taken without further purification.

The spectra of Compound I was a pair of quintets with J_{PCH} = 12 cps and J_{PCCH} = 42 cps. Other coupling constants determined by H'-NMR were consistent with the P^{31} spectra and will be furnished to any interested persons. Compounds XI and XII were run as a mixture of the two isomers as were Compounds XIV and XV.

We expect at a later date to contribute additional P^{31}-NMR data for some related compounds.

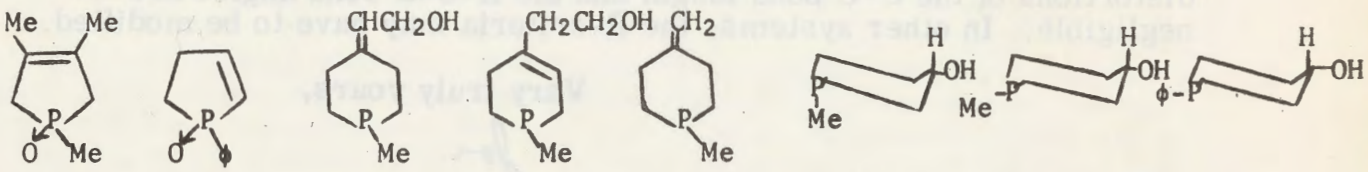
Sincerely,

Marcus E. Hobbs Stephen W. Dale



I II III IV V VI VII VIII

$\delta(P^{31})$=-83.2 -81.0 -73.8 -75.5 -61.6 -69.1 -58.4 -61.7 ppm



IX X XI XII XIII XIV XV XVI

-59.8 -56.2 +52.9 +70.0 +55.8 +61.0 +55.3 +32.3

NORTHWESTERN UNIVERSITY
EVANSTON, ILLINOIS 60201

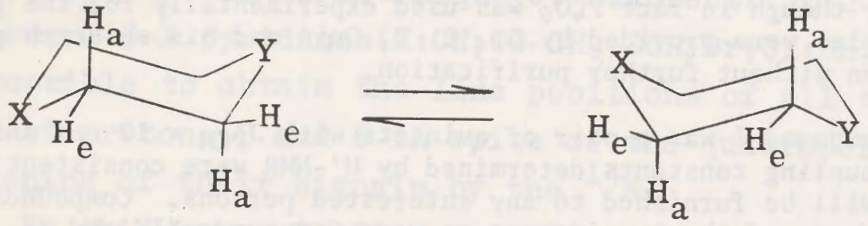
DEPARTMENT OF CHEMISTRY

February 9, 1967

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

Dear Barry:

We have found that the ratio (R) of the average $^3J_{trans}$ to the average $^3J_{cis}$ in six-membered rings of the type I is nearly independent

I 

$$J_{trans} = \tfrac{1}{2}(J_{aa} + J_{ee})$$

$$J_{cis} = \tfrac{1}{2}(J_{ae} + J_{ea}) = J_{ae}$$

$$R = J_{trans}/J_{cis}$$

of the electronegativity of X and Y. Hence, R becomes a direct measure of conformational effects. With reference to the Table, the group-1 compounds are known to exist in chair conformations not too different from that of the "perfect" (cyclohexane) chair. For these molecules, R is uniformly close to 2.0. Deviations from this value are taken to indicate the presence of distortions from the ideal chair. Inclusion of sulfur or selenium in the ring (group 2) introduces spreading and pinching distortions that are characterized by $R \geq 2.6$. Molecules in the flexible family have R-values near 1.3. A flattened chair such as cyclohexanone has an R-value of 1.7. We are examining the limitations of this method throughout cyclic chemistry. So long as we stay within the range of compounds of the type I, relative distortions of the C-C bond length and the H-C-C bond angles are negligible. In other systems, the R-criteria may have to be modified.

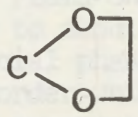
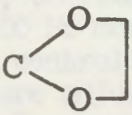
Very truly yours,

Joe

Joseph B. Lambert

JBL/kc

TABLE

	X	Y	J_{trans}, hertz	J_{cis}, hertz	$R=J_{trans}/J_{cis}$	Source
1.	O	O	6.11 ± 0.05	2.78 ± 0.05	2.20	a
	NH	O	6.66	3.04	2.19	b
	NCH_3	O	6.65	3.05	2.18	b
	NH	NH	6.54	3.04	2.15	a
	O	CD_2	7.41	3.87	1.91	a
	NH	CD_2	7.88	3.77	2.09	a
	$C(CH_3)_2$	$C(CH_3)_2$	8.27[c]	4.03[c]	2.05[c]	a
			8.34[d]	4.30[d]	1.94[d]	a
			8.82	4.59	1.92	a
2.	S	CD_2	8.51	3.26	2.61	a
	S	O	7.35	2.65	2.77	b
	S	S	8.11	2.40	3.38	a
	Se	Se	8.49	2.43	3.49	a
3.	C=O	C=O	8.05	6.23	1.29	a
	C=NOH	C=NOH	7.3	6.5	1.12	e
	$C=CH_2$	$C=CH_2$	7.52	5.31	1.42	a
4.	C=O	CD_2	8.61	5.01	1.72	a

[a] This work.

[b] Adapted from data of W. B. Smith and B. A. Shoulders, J. Phys. Chem., 69, 579 (1965).

[c] From the carbon-13 satellites at room temperature.

[d] From the low-temperature, slow-inversion spectrum, cf., H. Friebolin, W. Faisst, H. G. Schmid, and S. Kabuss, Tetrahedron Letters, No. 12, 1317 (1966).

[e] H. Saitô and K. Nukada, J. Mol. Spectry., 18, 355 (1965).

UNIVERSITY OF BRISTOL

SCHOOL OF CHEMISTRY,
Cantock's Close,
BRISTOL, 8,
E N G L A N D.

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

7th February, 1967.

Dear Dr. Shapiro,

Modification of the HA 100 Spectrometer for ^{19}F Spectra

We have recently modified our HA 100 along the lines kindly suggested by Jenkins, Phillips and Turner* of Imperial College so as to extend the range of the spectrometer. The results obtained for ^{19}F spectra are so encouraging that it seems worth communicating some details to the Newsletter.

The basic step is to substitute an external oscillator e.g. a Muirhead-Wigan Decade Oscillator D-890-A, for the Manual Oscillator of V 4354 by means of a two way switch inserted between R 1304 and switches S1303, S1305, and S1306. The correct amplitude may be obtained by equalising the Manual Oscillator signals on the oscilloscope for the internal and external oscillators. However, the phase of a sideband signal after amplification in V 4354 is frequency dependant, so that for increased Manual Oscillator frequencies it is no longer possible to obtain pure absorption signals on the recorder while the reference signal is in the correct dispersion phase to stabilise the field. This may be put right by altering the phase of the Sweep Oscillator reference input to the Audio Amplifier and Phase Detector (910-872), but the resistance-capacitance circuit used at Imperial College seemed to attenuate signals excessively, and we have used a simple capacitance method as is used to give the 90° shift in the Control Detector of the standard set up. An 8-way switch has been inserted in the lead to J1319 R in order to put 0.0001, 0.00047, 0.001, 0.0035, 0.01, 0.047, 0.1μ or a dead short in series with C 11 of 910-872. This switch allows nearly 90° phase shift but the smaller capacitances cause some attenuation and these positions are not used if possible. The signal may be inverted (180° shift) by a reversing switch in the signal input leads to J1319 A and B of this unit. With the phase tolerance (about 30°) of the Control Detector, these two phase adjustments are probably sufficient to cope with any phase relationship of the signals but we have also modified the phase of the reference input of the Control Detector. This is a similar 8-way switch arrangement to that for the Audio Detector except that since C 11 is 0.001μ, this has been shorted out and the eighth position of the switch is 2μ instead of shorted. The normal, i.e. proton spectra, positions of these switches are 'shorted' and 0.001μ respectively. If the phasing of the Control Detector is altered greatly, the Autoshim will not function correctly.

Unlike the Imperial College arrangement, this system can be operated in Field Sweep as well as Frequency Sweep, although the latter gives greater sensitivity. Field Sweep has the advantage that to change the region scanned only requires the external oscillator frequency to be altered, whereas for Frequency Sweep the field must also be shifted and lock re-established. Field Sweep is also less prone to interference from unwanted sidebands.

The system is operated in the following manner. If the reference line is downfield of the region to be investigated, the usual sidebands for HA operation (e.g. upper sideband for Frequency Sweep) are used, but if the reference is upfield, the other first sideband is used. In the latter case, the recorder works in the reverse sense, the upfield end being on the left. For Field Sweep, the lock is set up in the usual way and the external oscillator set to about 3000 Hz more than the shift of the lines to be observed. The special phasing controls are used to get approximate absorption signals on the recorder which are adjusted accurately by the RF Phase control of V 4311. One approach to Frequency Sweep operation is to set up the lock on an external oscillator frequency of about 2000 Hz and then increase this in suitable steps, re-establishing lock and correcting the RF phase of V 4311 for maximum locking signal each time. The other method we have used involves centering, on the oscilloscope, the appropriate sideband of the resonance to be investigated whilst in HR mode. The external oscillator is then adjusted to about 3000 Hz more than the shift of the line (e.g. as estimated from a quick HR scan), and the field swept slowly by V 3507 until the locking signal is found. The correct RF Phase setting is found by experiment using a low Manual Oscillator Field. Using either method, the phase of the signals on the chart is then adjusted in the same way as for Field Sweep.

The shift of a signal from the reference is the difference between the external oscillator and Sweep Oscillator frequencies, and accurate values are obtained using a frequency counter. The Frequency Difference Phase Detector of V 4354 does not operate properly for increased external oscillator frequencies.

Yours sincerely,

R. J. Goodfellow

Dr. R. Goodfellow.

*P.N. Jenkins, L. Phillips and D.W. Turner, Private Communication.

Telephone: Coventry 24011

School of Molecular Sciences
University of Warwick, Coventry
England.

7-2-1967

Dear Professor Shapiro,

^{13}C resonance in a Coal-Tar Fraction

The usefulness of ^{13}C resonance in the study of coal tar (M.L.Retcofsky and R.A.Friedel, NTIS, 91-18) and petroleum fractions (C.Knight, 98-32) is now well recognized. As Dr. Knight points out, absorption-mode signals are particularly valuable in analytical work or studies of the molecular structure of the fraction. As an example, the enclosed figure gives the ^{13}C spectrum at 15.0896 Mc/s of an approximately 25% solution of a coal-tar fraction in CS_2; the label 6FU refers to the fraction un-precipitated by n-heptane in the solvent extraction of a coke-oven tar. This spectrum was kindly run for us by Perkin-Elmer of Beaconsfield, England, using their ^{13}C accessory, which incorporates an R3-544-RE digital memory oscilloscope; non-spinning, 8.5 mm. o.d. tubes were used and the spectrum represents 920 accumulated scans, with 20 sec. for each scan followed by a 36 second delay. This delay time could well be shorter for fractions of higher molecular weight, which contain more paramagnetic impurities.

As in Retcofsky and Friedel's spectrum, the aromatic band is a doublet. The larger peak to low field is thought to contain the low-field components of the aromatic CH doublets superimposed on the singlets due to the substituted and ring-joining C atoms. The smaller peak to high-field probably contains the high-field components of the CH doublets only. As a coal-tar sample contains C atoms in many different aromatic environments, the peaks are expected to be broad. If this tentative assignment is correct, and it is supported by the doublet splitting of 150 c/s (142-162 in many pure aromatic hydrocarbons) and its position (62 ppm upfield from $^{13}CS_2$), then the area of the weaker peak subtracted from that of the stronger should represent the signal due to substituted and ring-joining C atoms - which are of course inaccessible to proton resonance studies. In this sample, a value of 23% is obtained for the ratio of substituted and ring-joining C to total aromatic C; this is not in very good agreement with the value of 35% for the same parameter obtained from an analysis of the ^{1}H spectrum, but the technique is clearly going to be of great importance in tar studies.

With appreciation of the usefulness of your Newsletter,

Yours sincerely,

J. C. Hammel (p.p.) John A. S. Smith

J.C.Hammel J.A.S.Smith.

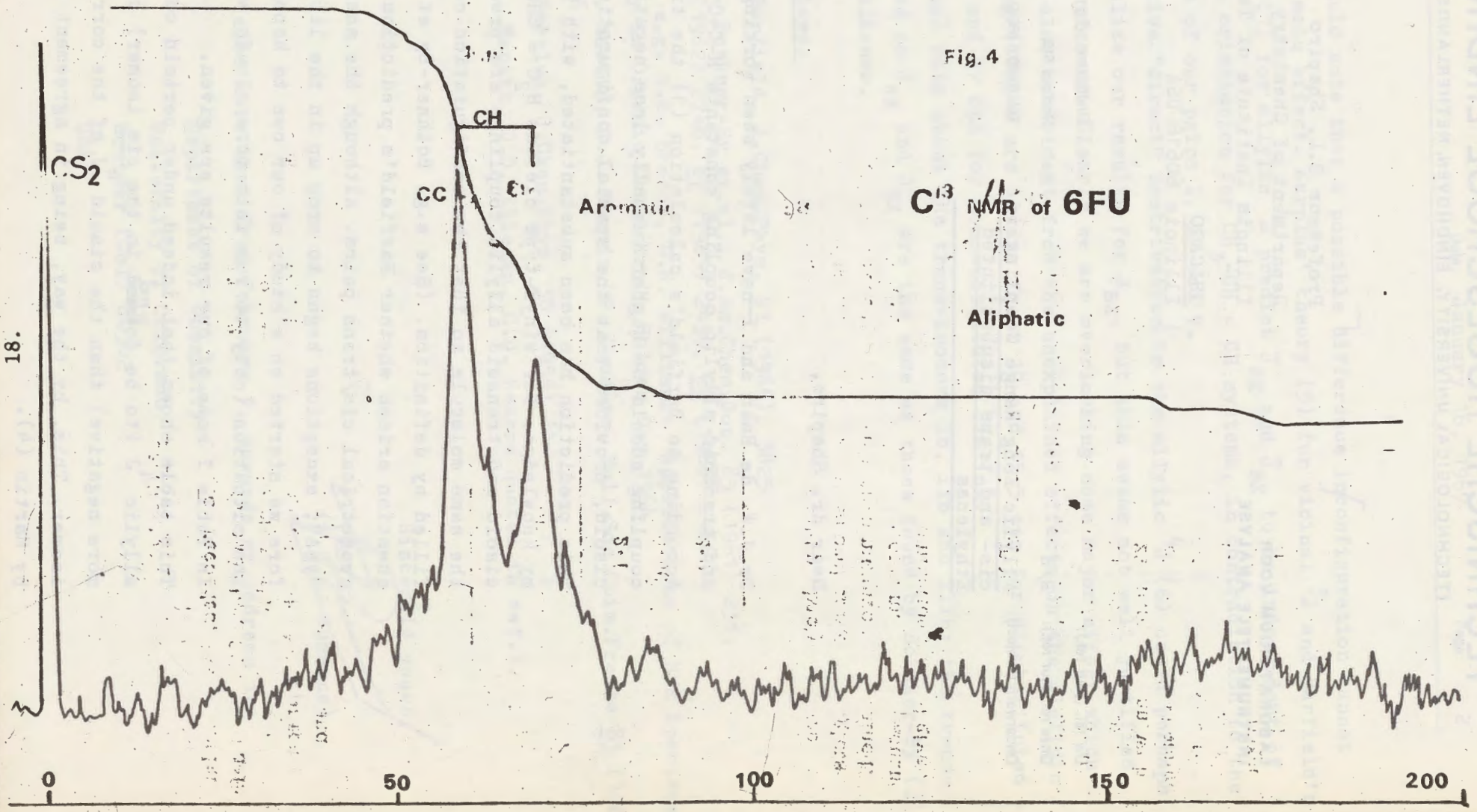


Fig. 4

C^{13} NMR of 6FU

TECHNISCHE HOGESCHOOL EINDHOVEN
(TECHNOLOGICAL UNIVERSITY, EINDHOVEN, NETHERLANDS)

LABORATORIUM VOOR
INSTRUMENTELE ANALYSE

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

Uw ref. (your ref.):
Onze ref. (our ref.): R/S
Onderwerp (subject): Allylic Long Range Coupling in cis- and trans disubstituted Ethylenes

Eindhoven, January 16, 1967
Postbus 513
Tel. (04900) 68000

Dear dr. Shapiro,

Dr.J.W. de Haan and I have lately been working on cisoid and transoid allylic coupling constants H-C-C = C-H. According to Barfield's calculation (1) the transoid allylic coupling should be higher (usually less negative) than the cisoid, provided that the spacial configuration is the same. This prediction has been substantiated, with no exception to my knowledge, for vinyl-type olefins H_2C = CHR, where both the cisoid and transoid allylic couplings are present in one and the same molecule so that the configuration condition is fulfilled by definition. (See e.g. Bothner-By et.al.(2)). The question arises whether Barfield's prediction still holds for geometrical cis/trans pairs. Although the answer seemed to be "yes", exceptions began to crop up in the literature (3). Therefore we started on a study of our own to hopefully clear up the situation (only to run into more clouds as it turned out). In Table I some of our results are given.
This table shows that indeed under certain conditions the transoid allylic ^4J (to be found in the cis isomer) can be lower (i.e. more negative) than the cisoid ^4J of the corresponding trans isomer. This, by the way, being in agreement with results found by Martin (4).

TECHNISCHE HOGESCHOOL EINDHOVEN (Technological University, Eindhoven)

One should note that a possible difference in configuration cannot be the main effect. Karplus' theory (5) for vicinal ^3J and Barfield's theory (1) for allylic ^4J predict $\bar{J}_{AX}$ and $\bar{J}_{BX}$ to be independent of spacial orientation for CH_3-CH = CH systems, in contradiction to the results of our pairs I, IV and V.

A negative "direct" contribution to the allylic ^4J (6) could perhaps rationalize our results for J_{BX}, but this seems not well justified at the present. Clearly we are overlooking some major effect which should also be evident from the unexplained effects on the olefinic couplings. These are always about 12 and 15.5 cps for disubstituted - and 10 and 17 cps for monosubstituted olefins.

One final note about the trans-isomers Ib, IIb and IIIb; the trends observed on J_{AX} and J_{BX} are the same as those found by Bothner-By (2) for 1-alkenes.

References:

1) M. Barfield, J.Chem.Phys. 41 (1964), 3825

2) A.A. Bothner-By et.al., J.Am.Chem.Soc. 83 (1961) 231, 84 (1962) 2748, 87 (1965) 2439, 88 (1966) 2466

3) See e.g. N.S. Bhacca, D.H. Williams: "Applications of NMR Spectroscopy in Organic Chemistry", Holden Day 1964

4) M.L. Martin, G.J. Martin, P. Caubère, Bull.Soc.Chim.France 83 (1964 3066

5) M. Karplus, J.Chem.Phys. 30 (1959) 11

6) S.L. Manatt, D.D. Elleman, C.D. Pearce quoted in ref.1.

Sincerely yours,

(F.H.A. Rummens)

P.S. Please note that from February 1, onwards my address is:

Department of Chemistry
University of Colorado
Boulder (Col) 80302
USA

Table I

Coupling constants of $R_1R_2H_XC-CH_A = CH_B-R_3$

No.	R_1	R_2	R_3	compound		J_{AB}	J_{AX}	J_{BX}
Ia	H	H	t-Bu	$CH_3-CH = CH-C(CH_3)_3$	cis	12.04	7.41	-1.89
Ib	"	"	"		trans	15.58	6.44	-1.69
IIa	H	CH_3	t-Bu	$CH_3-CH_2-CH=CH-C(CH_3)_3$	cis	12.03	7.40	-1.58
IIb	"	"	"		trans	15.57	6.37	-1.38
IIIa	CH_3	CH_3	t-Bu	$(CH_3)_2CH-CH=CH-C(CH_3)_3$	cis	synthesis in progress		
IIIb	"	"	"		trans	15.66	6.93	-1.27
IVa	H	H	Ø	$CH_3-CH=CH-Ø$	cis	11.56	7.15	-1.82
IVb	"	"	"		trans	15.69	6.62	-1.70
Va	H	H	COOH	$CH_3-CH=CH-COOH$	cis	11.46	7.32	-1.65
Vb	"	"	"		trans	15.53	6.94	-1.72

All compounds measured as neat liquids, except the crotonic acids (10 Mole % in $CDCl_3$).

THE UNIVERSITY OF WISCONSIN
MADISON 53706

DEPARTMENT OF CHEMISTRY

February 6, 1967

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

VIA DOGSLED

SHORT TITLE: Experimental Evidence of Anisotropic Rotational Diffusion

Dear Barry:

Anisotropic rotational diffusion has been discussed theoretically on a number of occasions[1,2,3,4] but there is so far very little unequivocal experimental evidence of such an effect. The existence of such an effect was recently confirmed by our measurement of the ^{35}Cl and ^{14}N relaxation times in $CCl_3 - C \equiv N$. The N T_2 was found to be $(1.16 \pm 0.03) \times 10^{-3}$ sec. at 27.5°C and the T_2 of Cl to be $(1.44 \pm 0.10) \times 10^{-5}$ sec. at 34.5°C. The Cl T_2 was corrected to 27.5° using an approximate ΔE_a (1400 cal as in CCl_4 [5]) and correlation times were calculated by the usual theoretical expression which assume spherical rotational diffusion one gets

$$\tau_c(Cl) = (2.67 \pm 0.15) \times 10^{-12} \text{ sec.}$$

$$\tau_c(N) = (3.55 \pm 0.10) \times 10^{-12} \text{ sec.}$$

indicating that anisotropic diffusion effects must be taken into account.

Having confirmed the existence of this effect, we are now proceeding to make more accurate measurements with the temperature controlled more precisely and hope to have more complete data in the near future.

Yours very truly,

Kenneth T. Gillen

Joseph H. Noggle

1. D. E. Woessner, J. Chem. Phys. 37 647 (1962).
2. D. E. Woessner, J. Chem. Phys. 40 2341 (1964).
3. H. Shimizu, J. Chem. Phys. 37 765 (1962).
4. H. Shimizu, J. Chem. Phys. 40 754 (1964).
5. D. E. O'Reilly, J. Chem. Phys. 39 1768 (1963).

101-60

STATE UNIVERSITY OF NEW YORK
AT STONY BROOK

DEPARTMENT OF CHEMISTRY

STONY BROOK, N.Y. 11790

February 6, 1967

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

Dear Barry:

It has been a long time since I wrote to IITNMRN and I am regretting its absence from my desk more every day. Since a number of projects are about to bear fruit but still lack a critical experiment or calculation, I shall just give a run-down on active programs here at this time.

Peter Wells left recently to return to the University of Queensland. While he was here for six months we straightened out (we think) some aspects of the proton NMR of CH_3HgX solutions, did some INDOR experiments on Pb^{207} with our A-60 and HD-60A (looking at various lead compounds and studying solvent effects on lead shieldings) and measured some new long range Hg-H and Pb-H couplings.

Seán Cawley also left a short time ago for Battelle Memorial Institute after a post-doctoral stint during which he helped whip our Computing Center into shape while getting Stevens' program for calculating integrals in diatomic molecules to run on our IBM 7040. Calculations are now almost complete for isotropic and anisotropic shieldings in a variety of ionic crystals. He also completed experimental measurements on a number of fluorides, and the results are being written up.

Dave Haddix is working on proton shift anistropics in single crystals. We now think that all significant experimental difficulties have been surmounted for at least one compound, and good data are emerging.

Skip Hutton is continuing the work on isotope effects on shifts in transition metal complexes, concentrating on $Co(CN)_6^{-3}$, for which some of the data and approximate calculations have been published.

José Ramirez is investigating solvent isotope effects on shieldings, especially D_2O effects on ions. Preliminary data on a number of electrolytes have been obtained, and some of the results are so remarkable that we are sitting on them until we have checked them several ways.

After an unfortunately abbreviated stay, Don Lemmon returned to Pittsburgh to take a position at Westinghouse. During his short stay, however, he turned up a rather peculiar long-range isotope effect on fluorine shieldings that we will try to follow up by long-range collaboration.

Next week, Ted Vladimiroff will be joining us as a post-doc to continue Seán's work on shieldings in solids (and on our Computing Center, which now has an IBM 7044 system).

As you can see from the list above, everyone is doing difficult experiments and spectrometer time is somewhat tight. If any of your readers have an old spectrometer system for sale cheap (or even just a magnet and power supply) we might be able to make a deal. We now have only an A-60 and an HR-100 (with lots of frequencies).

More details on the various things mentioned above should be available soon. In the meantime, how about a shipment of back issues?

Yours truly,

Paul

Paul C. Lauterbur
Associate Professor

PCL:pb

101-62

Argonne National Laboratory
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TELETYPE TWX ARGONNE, ILL. 312-257-2409
TELEGRAM WUX LB ARGONNE, ILL.

February 14, 1967

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

Oscillator Drive for Voltage Controlled Oscillator

Dear Barry:

In IIT NMR Newsletter No. 93 (June 1966, page 52) we described a voltage controlled oscillator (VCO) for time-averaging with the HA-100 spectrometer. We have now extended the use of the VCO to normal operation of the spectrometer, thereby making a substantial improvement in the linearity of the instrument. A stable voltage source, whose output is regulated by the sweep potentiometer in the recorder, is used to provide the driving voltage for the VCO.

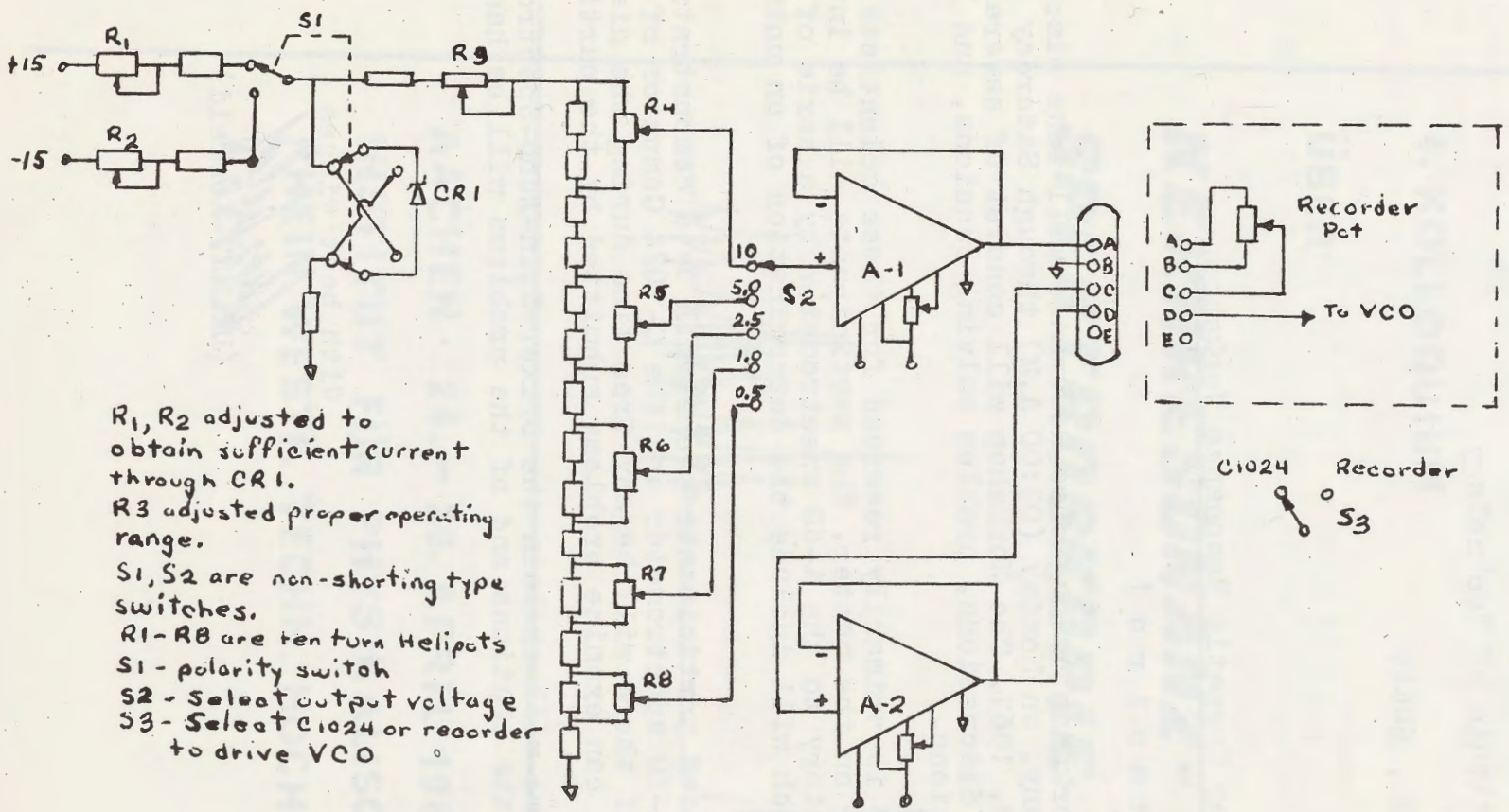
A well-regulated ± 15 volt power supply is the source for our oscillator drive, details of which are shown in the appended diagram. Outputs of 10, 5, 2.5, 1.0 and 0.5 volts are available, and these voltages correspond to sweeps of 1000, 500, 250, 100, and 50 cps. full scale in the recorder. The potentiometers R_1 and R_2 are used to provide adequate current for the Zener reference diode CR1. R_3 provides proper current through the divider string to provide 10 volts between ground and the slider of the recorder potentiometer. R_4, R_5, R_6, R_7 and R_8 are five range adjustments. Nexus SL-6 operational amplifiers are used to prevent loading. R_1 through R_8 are ten-turn Helipots. S_1 is a polarity reversal switch, and S_3 makes it possible to switch readily between the C-1024 and the spectrometer. The ranges are easily set, since each range is independent, and the long term stability of the voltage source is such as to provide oscillator frequencies stable to 0.1 cps. Linearity is very good, and it is practical to read chemical shifts from recorded spectra almost as precisely as can be done by the difference between the sweep and fixed frequency oscillators.

Preliminary work on this device was carried out by Dennis Matter, and the circuit was designed to our specifications by W. P. McDowell of the Electronics Division, Argonne National Laboratory.

Sincerely yours,

A. G. Kostka G. D. Norman J. J. Katz

A. G. Kostka G. D. Norman J. J. Katz



R1, R2 adjusted to obtain sufficient current through CR1.
R3 adjusted proper operating range.
S1, S2 are non-shorting type switches.
R1-R8 are ten turn Helipots
S1 - polarity switch
S2 - Select output voltage
S3 - Select C1024 or recorder to drive VCO

Oscillator Drive for Voltage Controlled Oscillator

101-64

Institut für physikalische Chemie
der Rhein.-Westf. Techn. Hochschule
Aachen, Direktor Prof. Dr. W. Ubranck

51 Aachen, den 1. 2 1967
Klosterbongard 12
Fernruf 422 2154

Hr.
Dr. Bernard L. S h a p i r o
Illinois Institute of Technology

C H I C A G O , 60616

U S A

Fourth Nuclear Magnetic Resonance Workshop

Dear Dr. S h a p i r o !

The fourth Workshop on Magnetic Resonance will take place at Aachen, Germany, on Monday (09:00 A.M) through Saturday (5:00 P.M) April 24 - 29, 1967. The Workshop will consist of several series of lectures, discussions, problem solving sessions, and laboratory demonstrations.

The first day is primarily reserved for those scientists without any knowledge on the matter. The participants will be introduced in the laboratory to the A-60 spectrometer by a serie of demonstrations which will include the determination of an unknown spectrum.

For the advanced participants there will be a demonstration of the Varian DA-60 spectrometer and the C-1024 Computer of Average Transients. If there will be any free time during the discussion sessions, one can examine problems submitted by the participants.

In addition, we will explain the Bruker-Spin-Echo-Spectrometer. Abstracts of the lections and of the problems will be handed to registrants.

With best wishes

(Dr. R. Kosfeld)

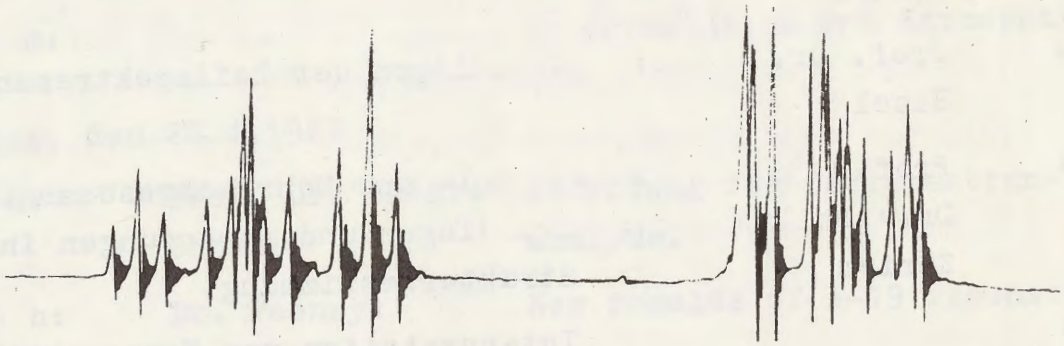
EINLADUNG

ZUM

4. KOLLOQUIUM

ÜBER

KERNRESONANZ-SPEKTROSKOPIE



AACHEN · 24. – 29. APRIL 1967

INSTITUT FÜR PHYSIKALISCHE CHEMIE

RHEIN. WESTF. TECHN. HOCHSCHULE

AACHEN

P R O G R A M M zum 4. Kolloquium über Kernresonanzspektroskopie

Montag, den 24.4.1967

9.oo h s.t.:	Dr. Joop: Leverkusen	Physikalische Grundlagen der Kernresonanzspektroskopie.
11.oo h:	Dr. Günther: Köln	Spin-Spin-Wechselwirkung zwischen Protonen (Grundlagen).
14.3o h:	Doz. Dr. Suhr: Tübingen	Lösungsmitteleinflüsse bei der Kernresonanzspektroskopie (Grundlagen).
16.30 h:	Dr. Scheidegger: Zürich	Einführung in die Interpretation von Kernresonanzspektren.
18.oo h:	Dr. Mannschreck: Heidelberg	Linienverbreiterung und chemische Kinetik.

Dienstag, den 25.4.1967

9.oo h s.t.:	Dr. Scheidegger: Zürich	Analyse von Spinsystemen.
11.oo h:	Prof. Dr. Ernst: Basel	Grundlagen der Teilspektrenanalyse.
14.3o h:	Prof. Dr.v.Philipsborn: Zürich	Doppel- und Mehrfachresonanz. Grundlagen und Anwendungen in der Strukturbestimmung.
16.3o h:		Interpretation von Kernspektren.

Mittwoch, 26.4.1967

9.oo h s.t.:	Dr. Melera: Zürich	Neuere instrumentelle Entwicklungen in der hochauflösenden Kernresonanz.
11.oo h:	Priv.Doz.Johnsen: Darmstadt	Untersuchung der Stereoregularität von Hochpolymeren.
14.3o h:	Prof. Dr. Simon: Zürich	Kombinierter Einsatz spektroskopischer Methoden in der Strukturauflösung organischer Verbindungen.

16.oo h:	Dr. Kosfeld Aachen	Anwendung der Breitlinienspektroskopie.
17.3o h:		Interpretation von Kernspektren.

Donnerstag, den 27.4.1967

9.oo h s.t.:	Dr. Günther: Köln	Spin-Spin-Wechselwirkung zwischen Protonen (Beziehungen zur chemischen Struktur).
10.3o h:	Doz. Dr. Brune: Karlsruhe	Spin-Spin-Kopplung zwischen Protonen und Kohlenstoff-13.
12.oo h:	Dr. Mannschreck: Heidelberg	Kinetik schneller intramolekularer Reaktionen.
14.3o h:	Doz. Dr. Suhr: Tübingen	Untersuchung zwischenmolekularer Wechselwirkungen.
16.3o h:	Prof. Dr.v.Philipsborn: Zürich	Doppel- und Mehrfachresonanz. Spezielle und neuere Methoden in der Spektrenanalyse.
18.oo h:		Interpretation von Kernspektren.

Freitag, den 28.4.1967

9.oo h:	Prof. Dr. Diehl: Basel	Anwendung der Teilspektrenanalyse.
10.3o h:	Dr. Feeney: London	New results of F-19 resonance.
14.3o h:	Dr. Luckhurst: Zürich	Nuclear magnetic resonance in liquid crystal solvents.
17.oo h:	Doz. Dr. Fluck: Heidelberg	Kernresonanzspektroskopie schwerer Kerne, insbesondere von ^{17}O, ^{13}C, ^{14}N, ^{15}N, ^{31}P.

Samstag, den 29.4.1967

Ab 9.oo h besteht die Möglichkeit, Spektren von mitgebrachten Proben aufzunehmen.

101-68

RAH-1

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
DEPARTMENT OF CHEMISTRY
CAMBRIDGE, MASSACHUSETTS 02139, U.S.A.

February 14 1967

On Chemical Exchange and High-resolution NMR Line Shapes

Dear Barry:

Some time ago, while reading through various papers on NMR methods for studying chemical exchange reactions, I became interested in the interrelations between the different approaches to the calculation of high-resolution NMR line shapes[1] and I reproduce here some of my notes which may possibly be of general interest.

A convenient point of departure is the McConnell formulation[1]: let $M_{+j} = (M_{xj} + iM_{yj})$ be the contribution of site j to the complex transverse magnetization in a frame rotating with the rf field, let P_j be the fractional probability for occupancy of site j and let p_{jk} be the probability that a nucleus from site j becomes transferred to site k; in the absence of rf saturation the McConnell equations may be written:

$$\frac{d}{dt} M_{+j} = -\alpha_j M_{+j} - M_{+j}/\tau_j + \sum_k{}' p_{kj} M_{+k}/\tau_k + i\gamma H_1 M_o P_j \qquad (1)$$

where $\alpha_j = i(w_{oj} - w) + 1/T_{2j}$ is the resonance parameter of site j, τ_j is the residence time at site j and M_o is the total equilibrium z-magnetization of the spin system. The prime on the summation sign signifies that the term with j=k is to be excluded. Normalization and detailed balance imposes the restrictions:

$$\sum_k{}' p_{jk} = 1 \qquad \sum_j P_j = 1 \qquad \text{and} \qquad p_{jk}/\tau_j = p_{kj}/\tau_k \quad .$$

When the spectrum is recorded under slow-passage conditions all the time derivatives vanish and (1) reduces to a system of linear equations from which the M_{+j} may be solved to give the absorption line shape I(w) :

$$I(w) = \mathrm{Im}\,(\sum_j M_{+j}). \qquad (2)$$

If we define a normalized magnetization at site j as $m_j = M_{+j}/P_j$ equation (2) may be rewritten:

$$I(w) = \mathrm{Im}\,(\sum_j P_j\, m_j) \qquad (3)$$

and equation (1) may be rewritten as:

$$\frac{dm_j}{dt} = -\alpha_j m_j + (\sum_k{}' p_{jk}\, m_k - m_j)/\tau_j + i\gamma H_1 M_o \;, \qquad (4)$$

where use has been made of the detailed balance equatons. Equations (3) and (4) represent a formulation akin to the density matrix and stochastic approaches[1] to the exchange problem in that (4) describes the time rate of change of the normalized magnetization at site j in terms of the single residence time τ_j . Let us now introduce a matrix $\underline{\underline{A}}$ with diagonal elements A_{jj} and <u>off</u> -diagonal elements A_{jk} defined according to:

$$A_{jj} = -(\alpha_j + 1/\tau_j) \quad \text{and} \quad A_{jk} = p_{jk}/\tau_j \quad (j \neq k)$$

and let us also define column vectors $\langle|$ and row vectors $|\rangle$ such as $\langle \underline{P}|$ with components P_j or $|\underline{m}\rangle$ with components m_j or $|\underline{1}\rangle$, a vector with all elements equal to unity. This enables us now to rewrite (4) as a matrix equation:

$$\frac{d}{dt}|\underline{m}\rangle = \underline{\underline{A}}\,|\underline{m}\rangle + i\gamma H_1 M_o\,|\underline{1}\rangle \qquad (5)$$

with the stationary solution

$$|\underline{m}\rangle = -i\gamma H_1 M_o\; \underline{\underline{A}}^{-1}\,|\underline{1}\rangle \;,$$

RAH-3

and the absorption spectrum will be given by

$$I(w) = -\gamma H_1 M_o \; \mathrm{Re}\left\{\langle \underline{P} \,|\, \underline{\underline{A}}^{-1} \,|\, \underline{1} \rangle\right\} \quad . \qquad (6)$$

Equation (6) is the well-known formula of Anderson and Sack[1]. The slow and fast exchange limits may be obtained from equation (6) by carrying out the appropriate perturbation expansions on the matrix $\underline{\underline{A}}^{-1}$. In this way one may obtain the slow and fast exchange formulas of Piette and Anderson[1] and also the generalized fast-exchange formula of Allerhand and Thiele[2]. The limits of validity of the perturbation expansions are indicated in a forth-coming letter to the Editor in the Journal of Chemical Physics.

Sincerely Yours,

Ragnar

Ragnar A. Hoffman

references:

1) a general reference on this whole subject is C. S. Johnson's article in"Advances in Magnetic Resonance" (J.S. Waugh, ed.), Volume 1, Academic Press 1965.

2) A. Allerhand and E. Thiele, J. Chem. Phys. 45 902 (1966) (section IV C of this paper contains a brief discussion of high-resolution line-widths)

P.S. I am writing this letter at MIT, where I am working with John S. Waugh during a sabbatical leave from Uppsala, but I hope that you will nonetheless acknowledge it as a valid contribution for my Uppsala subscription.

MELLON INSTITUTE

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