

*Joseph B. Lambert*

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

No. 108  
 SEPTEMBER, 1967

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Deadline Dates: No. 109: 7 October 1967  
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Reminder: For the period August 10, 1967 to August 15, 1968 inclusive, all Newsletter contributions, enquiries, etc., should be addressed as follows:

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

- continued on outside back cover

## University of East Anglia

From: Dr. R. K. Harris.

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

9th August, 1967.

COMPUTING IN NMR - SENIOR RESEARCH ASSOCIATE WANTED

Dear Barry,

I would like to use the columns of IITNMR to advertise that I am seeking a suitable person to fill the above position. The job involves co-ordinating and developing Computer Applications to NMR and is held in association with the Science Research Council Atlas computer at Chilton, Berks. The appointment is for two years.

I am looking for someone with a lot of initiative, since part of the task is to establish a library of efficient programs, covering the whole range of NMR, as part of the Atlas system. This means contacting scientists with working programs, as well as writing new routines. A good mathematical background is desirable, but prior knowledge of computing is not essential. The position would be ideal for a chemist wishing to gain experience in computing (where the highly-paid jobs are these days!). Someone of post-doctoral status (or of similar experience) would be preferred. There are also plenty of individual NMR research problems that are waiting to be tackled using computation. The salary scale is £1105 - £1340 per annum, with F.S.S.U. benefits. Commencement of the job would be by mutual arrangement, preferably this Autumn. Anyone is welcome to write for further details.

Best wishes,

*Robin*

R. K. Harris.

P.S. As I am not suggesting this letter should be counted as my IITNMR "contribution" (see the letter by Lauterbur, and sundry imprecations by Shapiro in number 106) I hope it can be "accepted for publication" without demur!!

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



THE UNIVERSITY OF OKLAHOMA  
NORMAN, OKLAHOMA, 73069

August 7, 1967

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

Dear Dr. Shapiro,

SOLVENT EFFECTS IN THE N.M.R. SPECTRUM OF 1,2,7,7-TETRACHLORONORBORNANE

N.m.r. spectra of the title compound have been obtained for 10% w/w solutions in benzene, pyridine, and chloroform; these spectra are shown in the accompanying diagram.

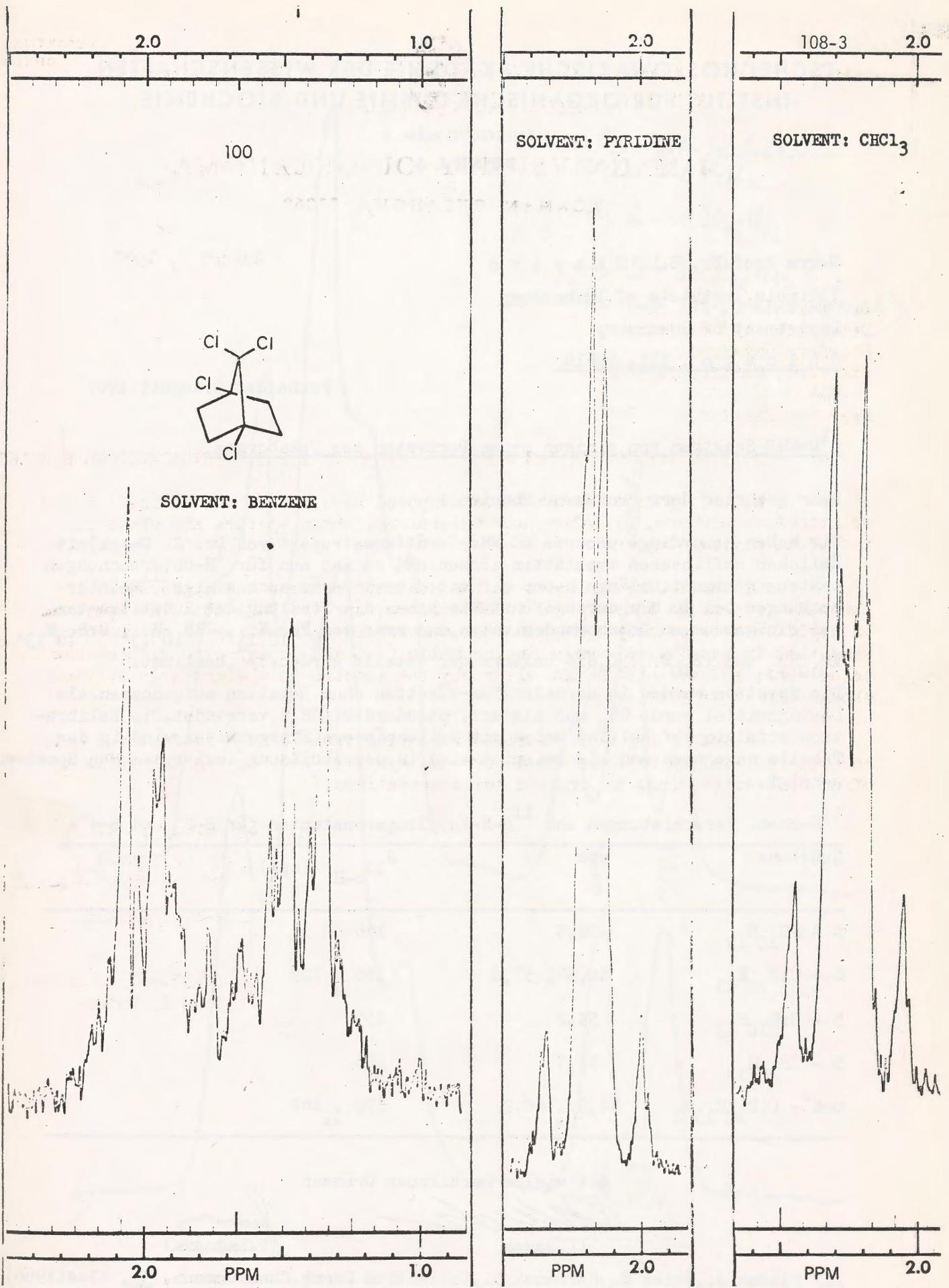
Of particular interest is the contrast between the appearance of the  $A_2B_2$  pattern in benzene and in chloroform, and the fact that the spectrum in pyridine more closely resembles the corresponding spectrum in chloroform than that in benzene. A change in the ratio ( $\Delta\delta/J$ ) is expected with change in solvent; however, it is not clear why the spectra in the aromatic solvents should differ so radically.

We would very much appreciate hearing from IITNMRN readers who have encountered similar behavior in AB or  $A_2B_2$  systems, and who might be able to offer some suggestions to explain our observations.

Sincerely yours,

Alan P. Marchand  
Alan P. Marchand

William R. Weimar Jr.  
William R. Weimar



TSCHECHOSLOWAKISCHE AKADEMIE DER WISSENSCHAFTEN  
INSTITUT FÜR ORGANISCHE CHEMIE UND BIOCHEMIE

FLEMINGOVO NÁM. 2

PRAHA 6

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

Praha, den 9. August 1967

<sup>11</sup>B-NMR-Spektren von einigen neuen Derivaten des Dekaborans

Sehr geehrter Herr Professor Shapiro !

Wir haben neuerdings unseres 80 MHz-Gerät (konstruiert von Dr. J. Dadok) mit üblichen Raffinessen ausstatten können und es ist nun für <sup>1</sup>H-Untersuchungen bestens geeignet. Unlängs haben wir mit diesem Gerät auch einige <sup>11</sup>B-Untersuchungen bei 20 MHz durchgeführt. Wir haben die Stellung der Substituenten bei einigen neuen Dekaboranderivaten, und zwar bei  $\text{FB}_{10}^{\text{H}}_{13}$ ,  $\text{ClB}_{10}^{\text{H}}_{13}$ ,  $\text{BrB}_{10}^{\text{H}}_{13}$ ,  $\text{IB}_{10}^{\text{H}}_{13}$  und  $(\text{B}_{10}^{\text{H}}_{13})_2\text{O}$ , die unlängs darstellt wurden/<sup>1</sup>, bestimmt.

Die Spektren wurden in normalen 5mm-Küvetten ohne Rotation aufgenommen. Als Lösungsmittel wurde  $\text{CS}_2$  und als ext. Standard  $\text{B}(\text{CCH}_3)_2$  verwendet. Die Kalibration erfolgte auf übliche Weise mit Seitenbändern. Einige Daten sind in der Tabelle angegeben und als Beispiel sind in der Abbildung zwei typischen Spektren gezeigt.

<sup>11</sup>B-chem. Verschiebungen und <sup>11</sup>B-H-Kopplungskonstanten für B-2 und B-4 :

Substanz	ppm	$J_{^{11}\text{B}-\text{H}}$ (cps)
5 - $\text{FB}_{10}^{\text{H}}_{13}$	56,5	166
6 - $\text{ClB}_{10}^{\text{H}}_{13}$	50,5 ; 57,6	155 ; 129
5 - $\text{BrB}_{10}^{\text{H}}_{13}$	54,2	155
5 - $\text{IB}_{10}^{\text{H}}_{13}$	51,7	165
6-6' - $(\text{B}_{10}^{\text{H}}_{13})_2\text{O}$	51,9 ; 60,2	170 ; 162

Mit vielen herzlichen Grüßen

(Z. Samek)

(P. Sedmera)

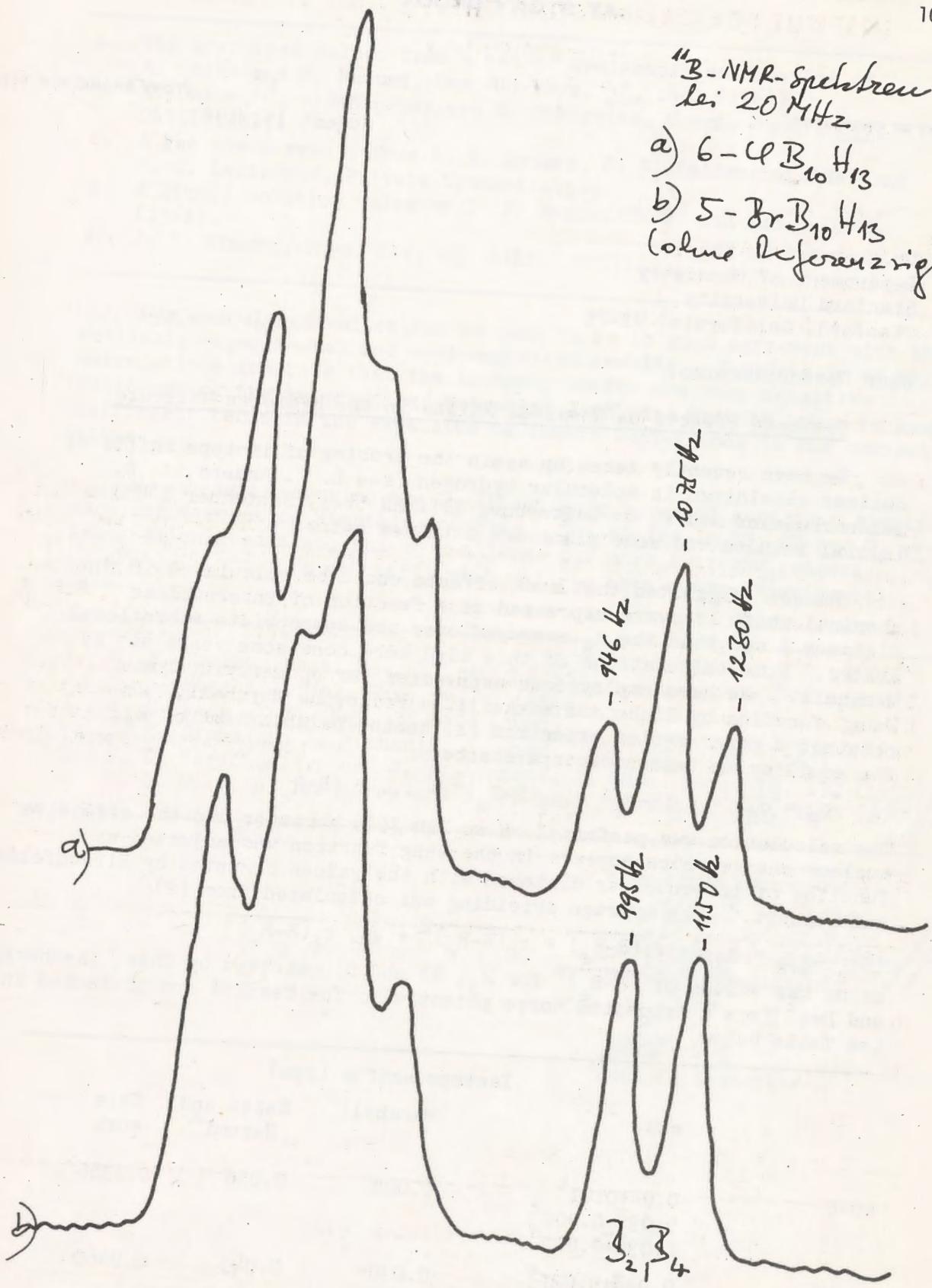
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<sup>11</sup>B-NMR-Spektrum  
bei 20 MHz

a) 6-CPB<sub>10</sub>H<sub>13</sub>

b) 5-BrB<sub>10</sub>H<sub>13</sub>

(oleine Referenzsignale)



STATE UNIVERSITY OF NEW YORK  
AT STONY BROOK

DEPARTMENT OF CHEMISTRY

STONY BROOK, N.Y. 11790

August 15, 1967

Dr. Bernard L. Shapiro  
Department of Chemistry  
Stanford University  
Stanford, California 93405

Dear Doctor Shapiro:

Isotopic Effects on Chemical Shifts in the Hydrogen Molecule

We have recently taken up again the problem of isotope shifts of nuclear shieldings in molecular hydrogen (see L. R. Anders, J. D. Baldeschwieler and P. C. Lauterbur, IITNMRN 84-1 (September 1965)). Initial results and some plans are outlined below.

Ramsey<sup>1</sup> suggested that such effects could be calculated if the chemical shift ( $\sigma$ ) were expressed as a function of internuclear distance  $R$  and then the  $\sigma_R$  averaged over the appropriate vibrational states. Some calculations of this kind were done some years ago by Marshall.<sup>2</sup> We have employed an expression for  $\sigma_R$  derived from a Wang<sup>3</sup> function by Sinha and Mukherji.<sup>4</sup> Following Marshall, we obtained a power series expansion (1) in the neighborhood of  $R_e$ , the equilibrium internuclear distance.

$$(1) \quad \sigma_R = \sigma_{Re} + c_1(R-R_e) + c_2(R-R_e)^2 + \dots + c_6(R-R_e)^6$$

The calculation was performed on an IBM 7044 computer and the effective nuclear charge which appears in the Wang function was adjusted as a function of internuclear distance with the values computed by Hirschfelder and Linnett.<sup>5</sup> The average shielding was calculated from (2)

$$(2) \quad \sigma_{ave} = \sigma_{Re} + c_1 \overline{(R-R_e)} + c_2 \overline{(R-R_e)^2} + \dots + c_6 \overline{(R-R_e)^6}$$

using the values of  $\overline{(R-R_e)^n}$  for  $H_2$ ,  $HD$  and  $D_2$  obtained by Chan, Ikenberry and Das<sup>6</sup> from a truncated Morse potential. The results are presented in the Table below.

	Isotope shifts (ppm)			
	exp.	Marshall <sup>2</sup>	Saika and Narumi <sup>a</sup>	This work
HD-D	0.04+0.01 <sup>b</sup> 0.038+0.008 <sup>c</sup> 0.036+0.002 <sup>d</sup>	0.025	0.036	0.0386
$D_2$ -HD	0.048+0.037 <sup>e</sup>	0.030	0.043	0.0460

To Dr. Bernard L. Shapiro  
August 15, 1967  
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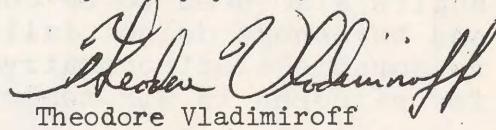
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- a. The preferred values from a set of semi-empirical results of A. Saika and H. Narumi, Can. J. Phys. 42, 1481 (1964).
  - b. E. Dayan, G. Widenlocher and M. Chaigneau, Compt. Rend., 257, 2455 (1963).
  - c. A gas phase result from L. R. Anders, J. D. Baldeschwieder and P. C. Lauterbur, Private Communication.
  - d. A liquid solution value by D. F. Evans, Chem. and Ind., 1960 (1961).
  - e. J. F. Wimett, Phys. Rev. 91, 476A (1953).
- 

Our calculated values can be seen to be in good agreement with the available experimental and semi-empirical results. The details of the calculations indicate that the isotopic shifts are very sensitive functions of the internuclear potential, and care must be taken to keep sufficient terms in the expansion to insure convergance to the correct value.

We are now measuring the  $D_2$ -HD shift in hopes of getting a significant improvement on Wimett's accuracy. The shifts in HT, DT and  $T_2$  are being calculated also, and we challenge our more intrepid readers (G. V. D. Tiers, are you still with us?) to obtain the experimental data.

- 
- 1. N. F. Ramsey, Phys. Rev. 87, 1075 (1952).
  - 2. T. W. Marshall, Mol. Phys. 4, 61 (1961).
  - 3. S. C. Wang, Phys. Rev. 31, 579 (1928).
  - 4. S. K. Sinha and A. Mukherji, J. Chem. Phys. 32, 1652 (1960).
  - 5. J. O. Herschfelder and J. W. Linnett, J. Chem. Phys. 18, 130 (1950).
  - 6. S. I. Chan, D. Ikenberry and T. P. Das, Phys. Rev. 135, A960 (1964).
- 

Very truly yours,

  
Theodore Vladimiroff

  
Paul C. Lauterbur

pb



## TEXAS CHRISTIAN UNIVERSITY

Fort Worth, Texas 76129

Department of Chemistry

August 15, 1967

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

Further J-Bond Order Examples

Dear Barry:

Several months ago we published work (J. Am. Chem. Soc., 89, 1438 (1967) extending the linear relation between bond order and vicinal coupling constants for a variety of olefins, diolefins, aromatics, and nonbenzenoid aromatics with both six and five membered rings. In searching for other systems where these relations might be of interest, I recently collected the m.o. bond orders and NMR data available for a series of six-membered heterocycles. These data are plotted in the accompanying figure; the line shown being that for the six-membered carbocyclics.

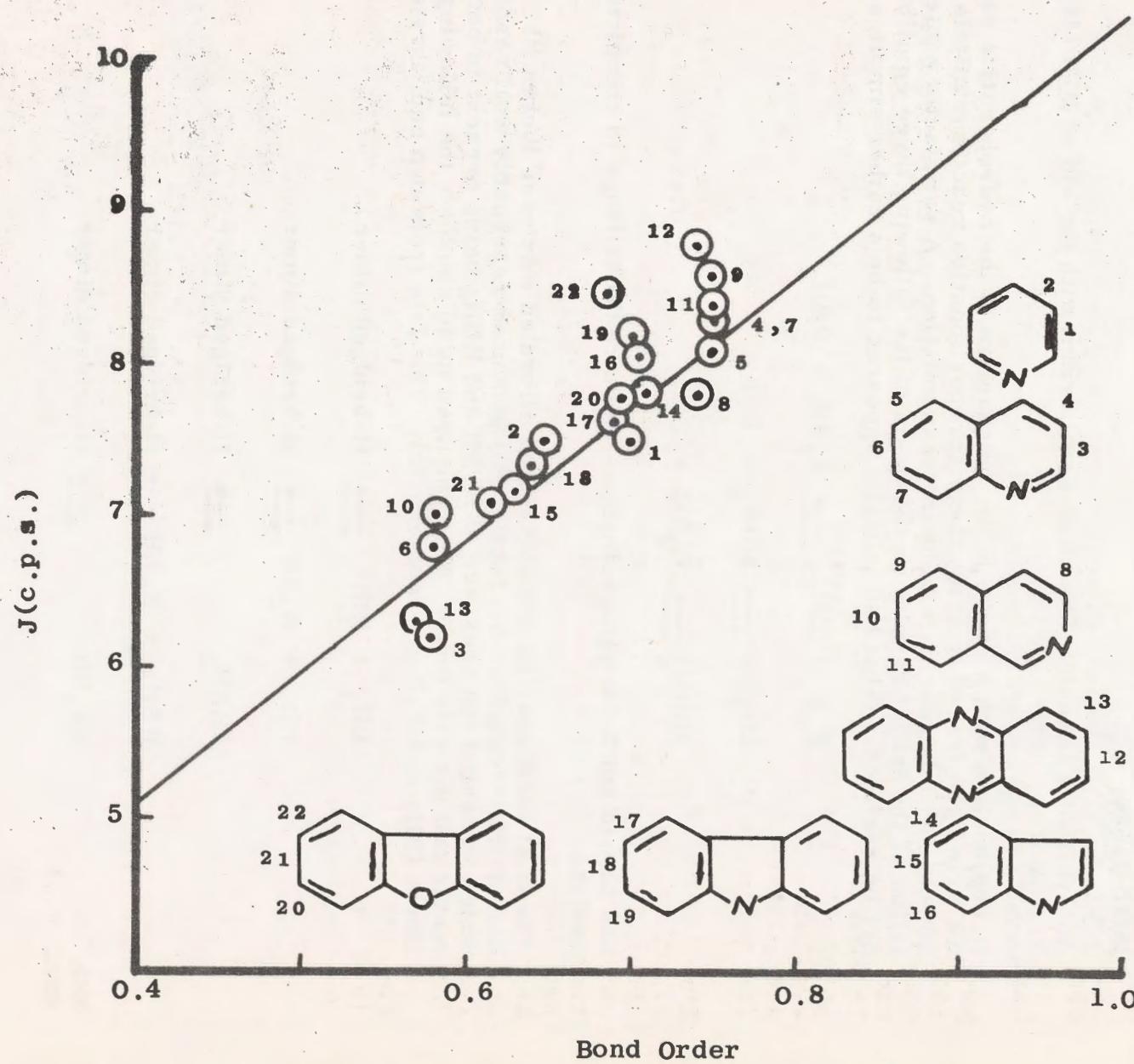
The vicinal coupling constants in these systems will be functions of other variables than the bond order. The electronegativity of the heteratom is of importance. From considerations of some vinyl amines the effect of nitrogen vs. carbon seems to require the addition of ca. 2 c.p.s. for bonds  $\alpha$ ,  $\beta$  to nitrogen. This correction has been made in the plot. Changes introduced by altered HCCH angles also need to be considered and may explain why furan, pyrrole, and thiophene do not fall well on the five-membered plot even with reasonable electronegativity corrections. The bond angle correction for nitrogen in six-membered rings does not appear to be significant.

Considering the variety of sources for the NMR data and bond orders (all the way from simple HMO to fancy SCF) the fit seems remarkably good and potentially useful.

Best regards,

*Bill*  
 W. B. Smith  
 Chairman  
 Department of Chemistry

WBS/dc  
 Enclosure



University of Notre Dame  
College of Science  
Notre Dame, Indiana 46556

Department of Chemistry

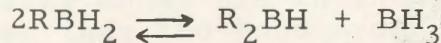
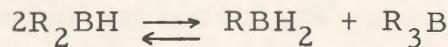
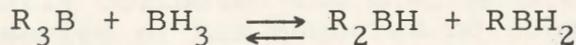
August 16, 1967

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

Dear Barry:

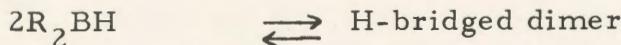
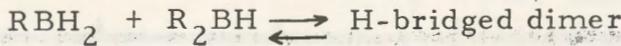
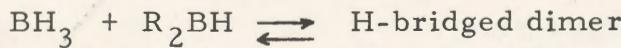
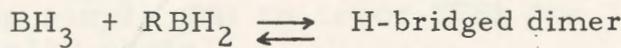
Analysis of a Complex Chemical Equilibrium with the aid of B-11 Magnetic Resonance.

In conjunction with a study on the mechanism of the hydroboration reaction, we have been forced to study the disproportionation reactions of trialkylboranes with borane in tetrahydrofuran solution. A satisfactory interpretation of the initial results on the basis of the following three equilibria could not be realized. What had initially appeared to be a rather simple system



has turned out to be quite complex - and a real challenge to completely analyze.

The B-11 resonance spectra of the equilibrated mixtures looked like ill-defined blobs! Heteronuclear decoupling (H) produced reasonably well resolved spectra which indicated the presence of  $R_2BH$  and  $RBH_2$  being present in both monomeric and dimeric form. This required us to consider the following five equilibria ( $BH_3$  and  $R_3B$  are monomeric). The data from two equilibrated mixtures

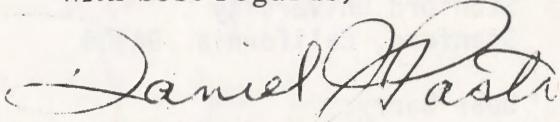


Professor B. L. Shapiro  
August 16, 1967  
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for each R system were analyzed by a complex computer routine giving what we feel are quite reasonable values for these five equilibrium constants. We are now attempting to calculate the rate constants involved in the three disproportionation equilibria.

It is obvious from this investigation that any approach to the study of the hydroboration reaction may be beset with many problems involving such equilibria.

With best regards,



Daniel J. Pasto  
Associate Professor of Chemistry

DJP:dw

UNIVERSITY OF HOUSTON  
CULLEN BOULEVARD  
HOUSTON, TEXAS 77004

CHEMISTRY DEPARTMENT

16 August 1967

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

Dear Barry:

THE AA'(BB')(BB')' SPIN SYSTEM: CYCLOBUTANONE

Bill Fairless and I have recently completed an analysis of the proton NMR spectrum of cyclobutanone. We undertook the project because we thought it would be a valuable test for our HA-100 spectrometer and would teach us a little about machine computation of spectra. Unfortunately we were unable to solve the problem completely using 100 MHz data as well as extensive spin-tickling. We had to obtain 60 and 40.5 MHz data to supplement the 100 MHz information. We did find out a good deal about symmetry in this rather complex spin system and have written the results for publication. A very limited number of pre-prints are available.

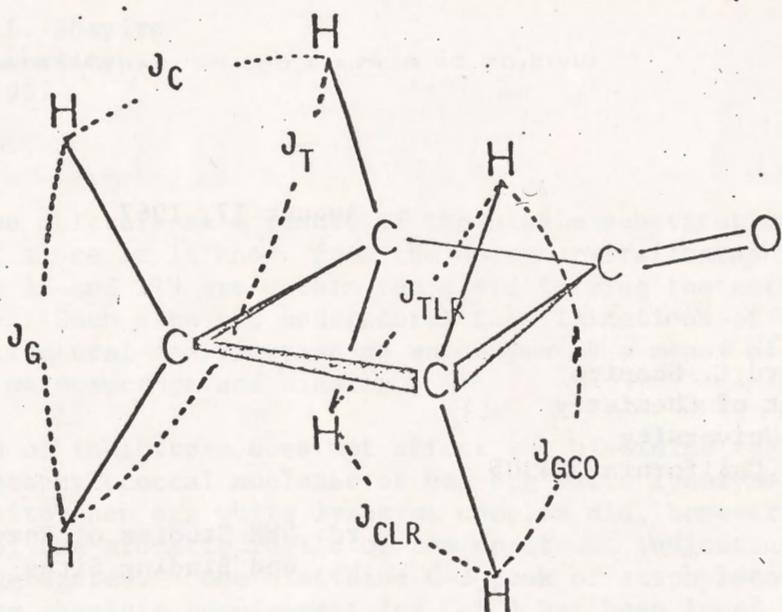
We have summarized the values that gave calculated spectra which fit at all three frequencies in the appended table. In addition, a diagram showing the nomenclature we have used for cyclobutanone is included.

Sincerely,

*BW*

M. R. Willcott  
Assistant Professor of Chemistry

MRW:la  
Enclosure



## CYCLOBUTANONE

NMR PARAMETERS DETERMINED FROM 100, 60, 40.5 MHz SPECTRA

$$\nu_B = 2.93573$$

$$J_{TL} = -2.283$$

$$\nu_A = 1.86036$$

$$J_C = 10.066$$

$$J_{GCO} = -17.576$$

$$J_T = 6.351$$

$$J_{CL} = 2.497$$

$$J_G = -11.750$$

## MERCK SHARP &amp; DOHME

## RESEARCH LABORATORIES

DIVISION OF MERCK &amp; CO., INC. RAHWAY, NEW JERSEY 07065

August 17, 1967

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

re: NMR Studies of Enzyme Structure  
and Binding Sites

Dear Barry:

Our present objective is to obtain information concerning the binding sites of a number of enzymes by direct observation of the resonances of the macromolecule. Even the higher frequency spectrometers with superconducting magnets are not expected to provide spectra subject to unambiguous interpretation in most cases due to the inevitable overlap or large numbers of broad peaks. Therefore, we are in the process of preparing a selectively protonated deutero-enzyme as a model system.<sup>1</sup> However, useful information has been obtained for some small proteins (MW~15,000) at 100 Mc, viz. bovine pancreatic ribonuclease A, staphylococcal (Foggi) nuclease, and hen egg white and human lysozymes. The imidazole C-2 proton resonances of the four histidine residues of ribonuclease and staphylococcal nuclease and the single histidine residues of the two lysozymes have been clearly resolved. Plots of the shifts of these peaks as a function of pH give an individual titration curve and pK for each histidine residue. In ribonuclease the pK values are 5.6, 6.0, 6.2, 6.6; in staphylococcal nuclease 5.6, 5.9, 6.1 and 6.6; in HEW lysozyme 5.8; and in human lysozyme 7.6, reflecting differences in protein structure.

Addition of nucleotide inhibitors to ribonuclease causes selective shifting and broadening of at most two of the four histidine C-2 peaks. These two correspond to the two titration curves which are also grossly shifted in the 3-carboxypropyl histidine-12 ribonuclease. This tends to confirm the assignments of these two peaks as His 12 and 119, and is consistent with previous data of their proximity and involvement in the binding site. Preliminary data with 1-carboxypropyl histidine-119 ribonuclease indicates that the same two peaks are affected, but to a lesser extent. It should be noted, however, that in both derivatives all the titration curves are affected to some extent, indicating a conformational

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Dr. Bernard L. Shapiro  
Stanford University  
August 17, 1967

change in the molecule as a result of the single substitution. This might be expected since it is known from the X-ray crystallographic structure that the His 12 and 119 are within the cleft forming the active site of the molecule. Such a result underscores the limitations of any technique requiring structural modification of an enzyme as a means of obtaining information on structure and binding.

The presence of inhibitors does not affect the histidine regions of the spectra of staphylococcal nuclease or hen egg white lysozyme. The formation of the inhibitor-hen egg white lysozyme complex did, however, result in sharpening of the aromatic region of the spectrum, indicating dissociation of enzyme aggregates.<sup>2</sup> One histidine C-2 peak of staphylococcal nuclease (which has an absolute requirement for  $\text{Ca}^{2+}$ ) has been found to shift down-field by 16 cycles/sec upon addition of  $\text{Ca}^{2+}$ . The positions of the other three peaks were only slightly affected by  $\text{Ca}^{2+}$ .

Titration curves determined in  $\text{H}_2\text{O}$  and  $\text{D}_2\text{O}$  have been found to be almost superposable, indicating that the  $\text{pK}_{\text{H}}$  (direct meter reading) is the same ( $\pm 0.2$  units) in both cases i.e.  $\text{pK}_{\text{H}}^{\text{H}_2\text{O}} = \text{pK}_{\text{H}}^{\text{D}_2\text{O}}$  and that the enzyme conformation in the histidine regions is not significantly different in the two solvents. The actual  $\text{pK}_{\text{H}}^{\text{D}_2\text{O}}$  should be obtained by the addition of 0.4 units<sup>3</sup> due to the constant deuterium isotope effect at the glass electrode.<sup>4</sup>

Regards,

(Mrs.) Donella H. Meadows      *Donella Meadows*

John L. Markley      *John Markley*

Jack S. Cohen      *Jack Cohen*

Oleg Jarretzky      *Oleg Jarretzky*

:ras

Refs.

1. O. Jarretzky, Int. Symp. on Magnetic Resonance, Tokyo, 1965, p.N-3-14.
2. M.R. Bruzzesi, E. Chiancone and E. Antonini, Biochemistry, 88, 1796, 1965.
3. N.C. Li, P. Tang and R. Mathur, J. Phys., Chem., 65, 1074, 1961.
4. P.K. Glasoe and F.A. Long, J. Phys., Chem., 64, 188, 1960.

## BELL TELEPHONE LABORATORIES

INCORPORATED

MURRAY HILL, NEW JERSEY 07971

TELEPHONE  
AREA CODE 201  
582-3000Air Mail

August 17, 1967

DR. BERNARD L. SHAPIRO  
Department of Chemistry  
Stanford University  
Stanford, California 94305

Dear Dr. Shapiro:

Mr. Perry Hood and myself have recently re-investigated the NMR of polypeptide solutions (cf. Bovey et al. J. Polymer Sci. 38, 73 (1959)) with particular attention to the  $\alpha$ -helix-random coil transition. We have studied both the right-handed helix of poly- $\gamma$ -benzyl-L-glutamate and the left-handed helix of poly- $\beta$ -benzyl-L-aspartate, with d-chloroform as the helix-supporting solvent and trifluoroacetic acid (TFA) as the helix-breaking solvent. Polymers of ca. 50 and ca. 1000 peptide units length have been used in each case. Both the glutamate and aspartate helices behave in essentially the same manner, except that the transition occurs at much lower TFA concentration in the latter, indicating its lower stability. The chemical shifts of the main-chain and side-chain protons as a function of TFA concentration are shown in Figs. 1 and 2. The main-chain protons (NH and  $\alpha$ -CH) show the largest changes in shielding as we pass through the transition (at ca. 20% TFA for glutamate and 3% TFA for aspartate helices), and they move in opposite directions. Similar results have been reported for poly-L-alanine (Stewart et al., Biochemistry 6, 143 (1967)). The position of the transition was independently confirmed by circular dichroism measurements on the same solutions. The dashed lines correspond to fractions of the polymer which are too short to sustain a helix or which undergo the transition at lower TFA levels than the rest. In these regions, the spectra show two  $\alpha$ -CH and NH peaks, but these must correspond to non-exchanging spin populations, as the lifetimes of the helix and coil in equilibrium are less than  $10^{-6}$  sec. (Lumry et al. Biopolymers 2, 489 (1964).)

In chloroform, the linewidths of all peaks are very broad and depend strongly upon molecular weight. The high molecular weight polymers appear to give virtually no spectrum, only the

Dr. Bernard L. Shapiro - 2

phenyl resonance being discernible at all (linewidth ca. 250 cps). This extreme broadening is due to the nematic liquid crystalline nature of these solutions. Addition of TFA well short of the helix-coil transition breaks up the nematic structure and causes great narrowing of the peaks, which still are markedly dependent upon molecular weight, as would be expected for rod-like helices. The transition itself causes a further but relatively much smaller line narrowing. In the random coil state, segmental motion dominates the nuclear relaxation, and linewidths are independent of molecular weight. Fine structure due to NH- $\alpha$ -CH vicinal coupling becomes evident. We believe, with Stewart et al. (above reference), that the function of TFA is not to protonate the peptide groups but to form hydrogen bonded complexes which reduce helical stability.

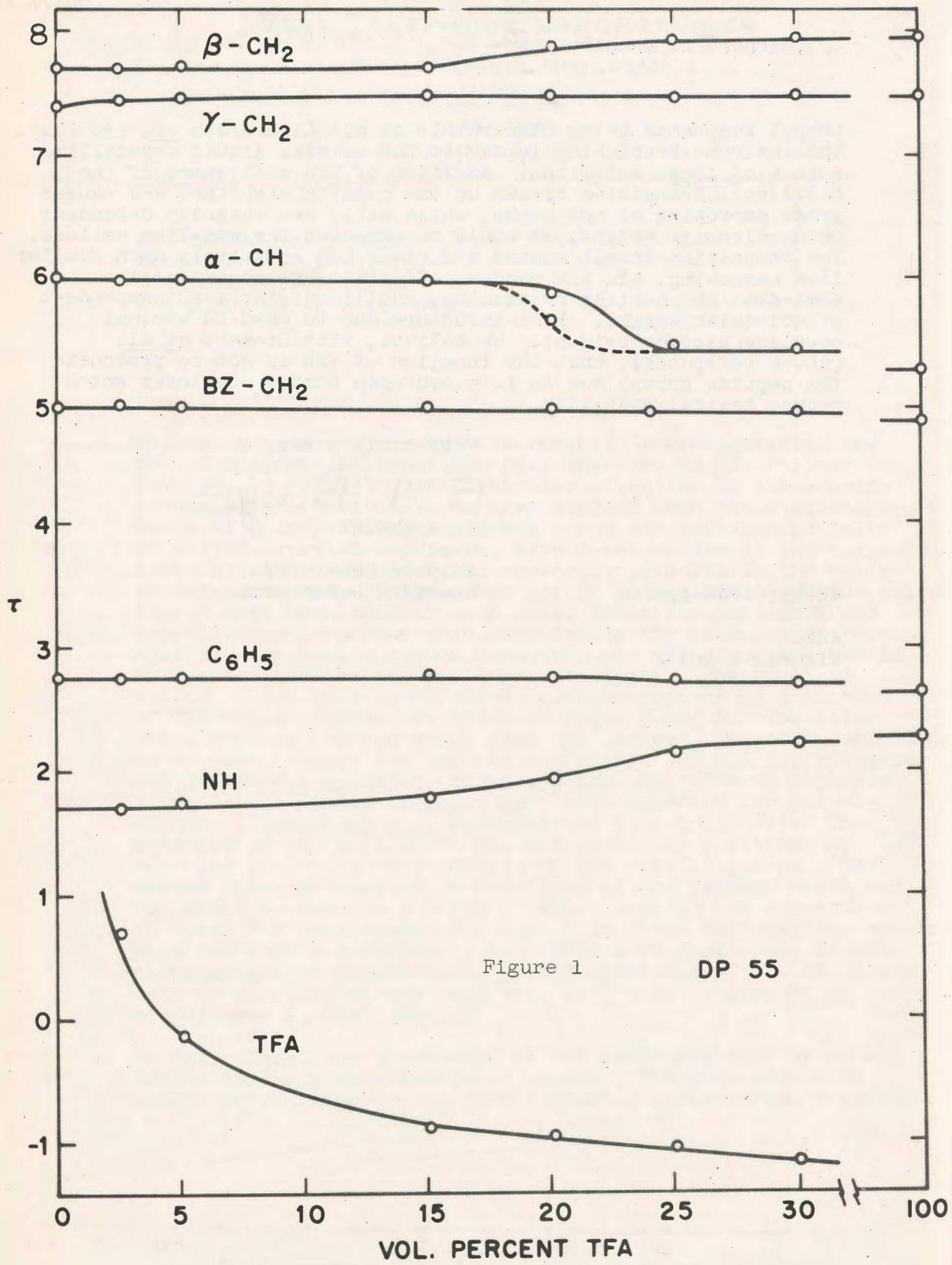
Very truly yours,

F. A. Bovey

F. A. BOVEY  
Head  
Polymer Chemistry  
Research Department

MH-1519-FAB-sjs

Att.  
Figures 1 and 2

POLY- $\gamma$ -BENZYL-L-GLUTAMATE, 50°

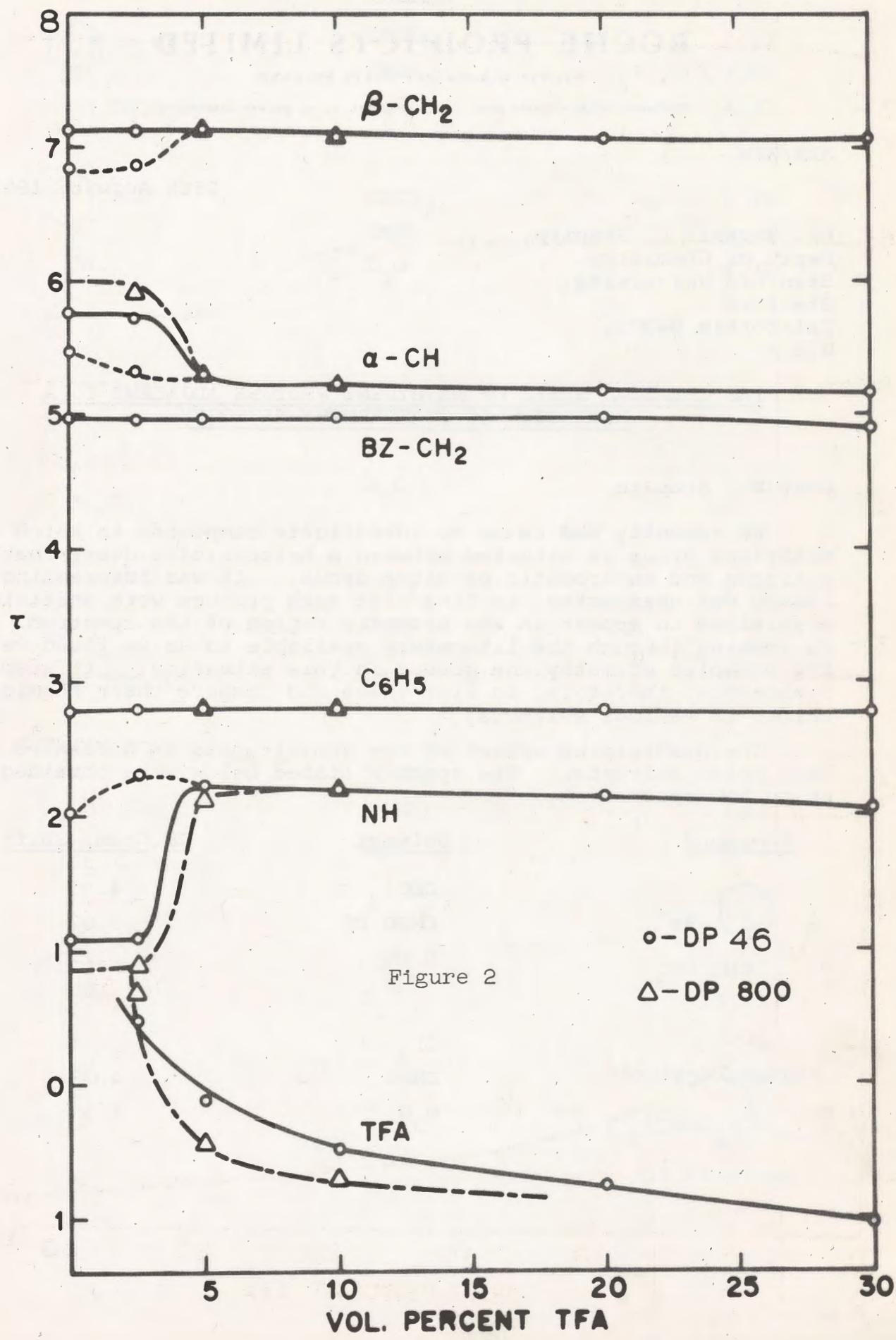
POLY- $\beta$ -BENZYL-L-ASPARTATE, 50°

Figure 2



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AAW/RKW

25th August, 1967

Dr. Bernard L. Shapiro,  
Dept. of Chemistry,  
Stanford University,  
Stanford  
California 84305,  
U.S.A.

## THE CHEMICAL SHIFT OF METHYLENE PROTONS ADJACENT TO A HETEROCYCLIC QUATERNARY NITROGEN

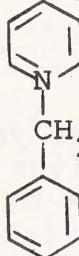
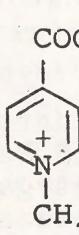
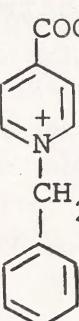
Dear Dr. Shapiro,

We recently had cause to investigate compounds in which a methylene group is situated between a heterocyclic quaternary nitrogen and an aromatic or ester group. It was interesting, though not unexpected, to find that such protons were sufficiently deshielded to appear in the aromatic region of the spectrum. On looking through the literature available to us we found very few examples of methylene groups in this situation. It seemed reasonable, therefore, to list these and compare their chemical shifts in various solvents.

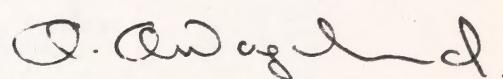
The deshielding effect of the substituents is decreased by more polar solvents. The spectra listed below were obtained on an A60 spectrometer.

<u>Compound</u>	<u>Solvent</u>	<u>CH<sub>2</sub> Chem. Shift</u>
	CDCl <sub>3</sub>	4.85
	DMSO D6	5.07
	D <sub>2</sub> O	5.17
	Cl <sub>3</sub>	-
	DMSO	4.03
	D <sub>2</sub> O	4.30

/contd.

<u>Compound</u>	<u>Solvent</u>	<u>CH<sub>2</sub> Chem. Shift</u> <u>T</u>
 Cl'	CDCl <sub>3</sub>	3.63
	DMSO	3.85
CH <sub>2</sub> COOC <sub>2</sub> H <sub>5</sub>	D <sub>2</sub> O	4.33
 Cl'	CDCl <sub>3</sub>	3.59
	DMSO	3.70
	D <sub>2</sub> O	4.02
 Br'	CDCl <sub>3</sub>	4.80
	DMSO	-
	D <sub>2</sub> O	5.00
 COOC <sub>2</sub> H <sub>5</sub>	CDCl <sub>3</sub>	3.55
	DMSO	-
	D <sub>2</sub> O	-
 Cl'	CDCl <sub>3</sub>	3.45
	DMSO	3.85
	D <sub>2</sub> O	4.27
CH <sub>2</sub> COOC <sub>2</sub> H <sub>5</sub>		
 Cl'	CDCl <sub>3</sub>	3.43
	D <sub>2</sub> O	3.93

Yours sincerely



A.A. Wagland



THE UNIVERSITY OF SUSSEX  
THE CHEMICAL LABORATORY FALMER BRIGHTON SUSSEX

Telephone Brighton 66755

25th August, 1967

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

Dear Dr. Shapiro,

We have spent quite a lot of time over the past two years in trying to understand why the Pople-Santry method gives good values for the meta and para proton coupling constants in benzene. What are the important delocalization terms?

Starting with a basis of localized C-H and C-C MO's constructed from the usual hybrids, one can introduce delocalization terms between these, which we will generalise as  $\beta'$ . One can now set up a perturbation expansion for the atom-atom polarizability having the form

$$\pi_{\mu\nu} = \frac{A \beta'^2}{\beta^3} + \frac{B \beta'^3}{\beta^4} + \dots$$

The leading term was obtained a couple of years ago (Murrell and Gil, Theoret. Chim. Acta. 4, 114 (1966); Pople and Santry, Mol. Phys., 9, 311 (1965)) and for the ortho coupling it gave good agreement to the full MO calculation. For the meta and para coupling, however, it gave a negligible contribution.

We have now sorted out the second terms and their details are in a paper just submitted for publication. We find these now give good agreement for the meta protons but are still a small part of the para coupling. The conclusion seems to be that long range coupling in the Pople-Santry theory is due to the combined effect of moderately large delocalization terms through several intervening bonds rather than the effect of just one or two small long range delocalization terms between the distant C-H bonds.

Yours sincerely,

J.N. Murrell

DEPARTMENT OF CHEMISTRY

## STANFORD UNIVERSITY

STANFORD, CALIFORNIA

September 1, 1967

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

Magnetic Fish: Prevention and Cure

Dear Dr. Shapiro:

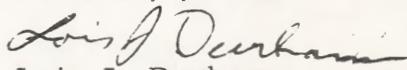
A problem which we frequently encounter as a result of running samples prepared by others (never in our own!) is the mysterious appearance of some small (and some not-so-small) particles of ferromagnetic material--possibly through some little understood mechanism of spontaneous generation. These magnetic fish usually play havoc with a well-tuned spectrometer--at least in proportion to their size and numbers, though some would argue for a much more complex relationship.

The idea of removing these particles from samples by fishing them out with a magnet is not a new one; however, it may be of interest to some that there is at least one supplier\* of very thin Alnico V magnets ( $1/16'' \times 4''$ ). These magnets are thin enough so that they may be sealed into a long, glass tube to protect both the magnet and the sample. By attaching a handle of  $1/8''$  glass cane of suitable length, the fishing rod may be inserted to the bottom of a standard NMR tube containing 0.3 to 0.5 ml. of sample without spilling sample, dissolving the magnet, or breaking the tube.

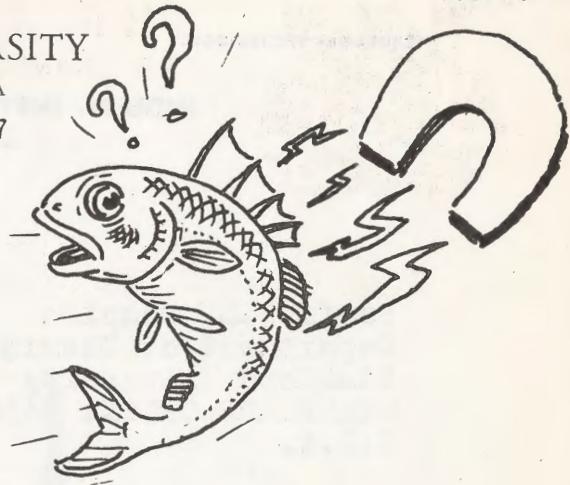
In order to convince our friends that we do not introduce these magnetic fish from our solvents (horrors, never!), we also keep small, glass-enclosed magnetic beetles in each of our solvent containers. These are made up from  $1/8'' \times 1/4''$  magnets which we obtained from the same supplier.

For those difficult-to-reach places, namely the business end of the spherical-type microcells at the end of a long, thin capillary, we grit our teeth and insert a piece of steel piano wire (usually  $0.007'' \times$  ca.  $7''$  or  $8''$ ) so it reaches to the bottom of the cell. Then the cell containing sample, fish, and wire are placed in the magnetic field to magnetize the wire. Removal of the wire usually takes out most, if not all, of the magnetic material on the first try. Of course, this technique is limited to samples and solvents which are unreactive towards steel wires. (Added bonus--the same size wire doubles nicely for cleaning recorder pen tips.)

Sincerely yours,


 Lois J. Durham

\* "centerless ground" Alnico V magnets,  $1/16'' \times 4''$  were obtained from the Dowling Magnet Co., 2472 Teagarden, San Leandro, Calif.; price in lots of 1 to 5 is \$2.75 each. The  $1/8'' \times 1/4''$  pieces run \$.30 each. No, we do not have stock in the company.



INDIAN INSTITUTE OF TECHNOLOGY KANPUR  
DEPARTMENT OF PHYSICS

I. I. T. Post Office

KANPUR August 25, 1967

Prof. B.L. Shapiro  
 Department of Chemistry  
 Stanford University  
 STANFORD Calif. 94305  
 U.S.A.

Short Title: Relaxation Effects in DR spectra  
 at sub-tickling strengths.

Dear Prof. Shapiro,

We are sorry for the delay in sending our contribution and thank you for the reminder. We have now in operation a frequency-sweep-homonuclear-double-resonance set-up on our Varian HR-100 spectrometer. The features of this otherwise standard setup are: (1) The field-frequency locking is done using the 2kc. Audio oscillator and lock-in-detector available in the Integrator. (2) A GR-1304-B Audio Oscillator is used for modulation in the observing channel. This is a good stable oscillator and furthermore, has a linear frequency increment dial (upto 100 cps) which can be conveniently driven to sweep through the spectrum. An EMC [Electronics Missiles and Communications Inc., 262 East Third St. Mount Vernon New York] Model RJB lock-in amplifier is used in this channel. This lock-in-amplifier comes made for specific frequencies, but can be easily modified for broad - band operation needed for this channel (essentially by eliminating the Twin-Tee cans in the circuit).

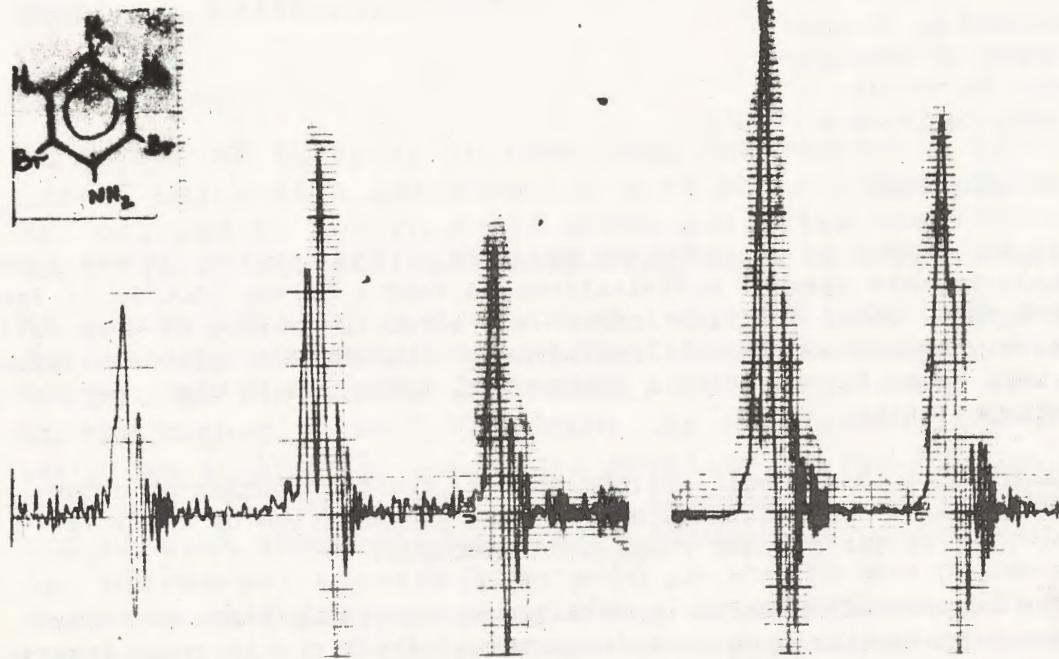
Double resonance experiments were performed on an AB<sub>2</sub> system (2,6 - dibromoaniline). The single and double resonance spectra are shown in the enclosed photograph. (We apologize for the poor photograph. We had to try making it ourselves as we faced some passive resistance from our photographing section here). The δ and J are 88.8 and 7.7 cps respectively. The DR spectrum which shows a marked change in relative intensities is obtained with a  $\sqrt{H_2}/2\pi$  of about 0.08 cps which one might call a : sub-tickling strength:. We are at present solving the density-matrix equations in the weak-irradiation limit and hope to report the findings pretty soon.

Yours Sincerely,

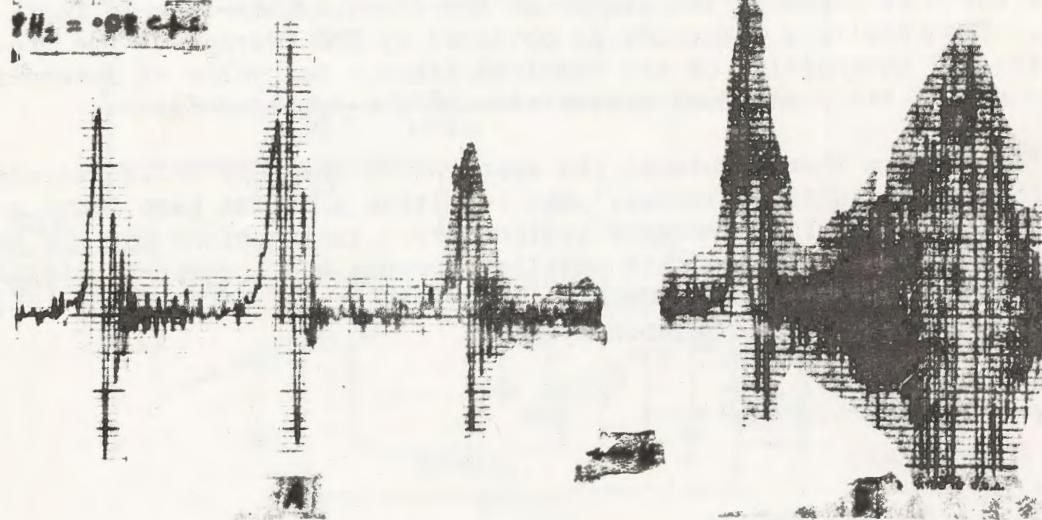
Anil Kumar  
(Anil Kumar)

B.D. Nageswara Rao.  
(B.D. Nageswara Rao)

P.S: BDNR is reminded of many happy memories about his :good old JDB gang: on seeing your new mailing address.



$\delta H_2 = 0.08 \text{ c.p.s.}$



**DEPARTMENT OF THE AIR FORCE**  
**AIR FORCE MATERIALS LABORATORY (AFSC)**  
**WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433**



REPLY TO  
ATTN OF: MAYH/Mr. R. E. Rondeau/255-2280

25 AUG 1967

SUBJECT: Modified LAOCOON Computer Program

TO: Dr. Bernard L. Shapiro  
Department of Chemistry  
Stamford University  
Stamford, California 94305

Dear Dr. Shapiro:

In the course of our computer analyses of NMR spectra, it was found desirable to make several modifications on Part I of the LAOCOON II Fortran Program. These modifications do not alter the method of computation, but rather present additional treatments of the computer spectrum output which were found to result in a substantial reduction in time required for interpretation.

Basically, these modifications are: 1) the compilation of a compressed table of ordered lines and, 2) the presentation of a printer display plot of the further compressed spectrum.

The compressed spectrum is obtained by combining lines which are degenerate in frequency or have frequencies within a 0.10 cycle interval. A number of lines so combined is printed just to the left of the line number which is taken as the number of the first summed line in that group. The resulting frequency is obtained by RMS average of the frequencies and intensities of the combined lines. The value of intensity is the sum of the individual intensities of the combined lines.

The program then tabulates the spectrum in one-half cycle increments and plots the resulting display. The resulting plot has been found most valuable in evaluating parameter assignments. Instructions for the preparation of data cards for this modified program and a complete listing are available in the form of an AFML Technical Report (AFML-TR-66-196). These reports are available upon request.

*Roger E. Rondeau*

ROGER E. RONDEAU  
HUGHEY A. RUSH\*  
Exploratory Studies Branch  
Materials Physics Division  
AF Materials Laboratory

\* New Address: Chemstrand Research Center  
Durham, North Carolina



## THE DOW CHEMICAL COMPANY

August 30, 1967

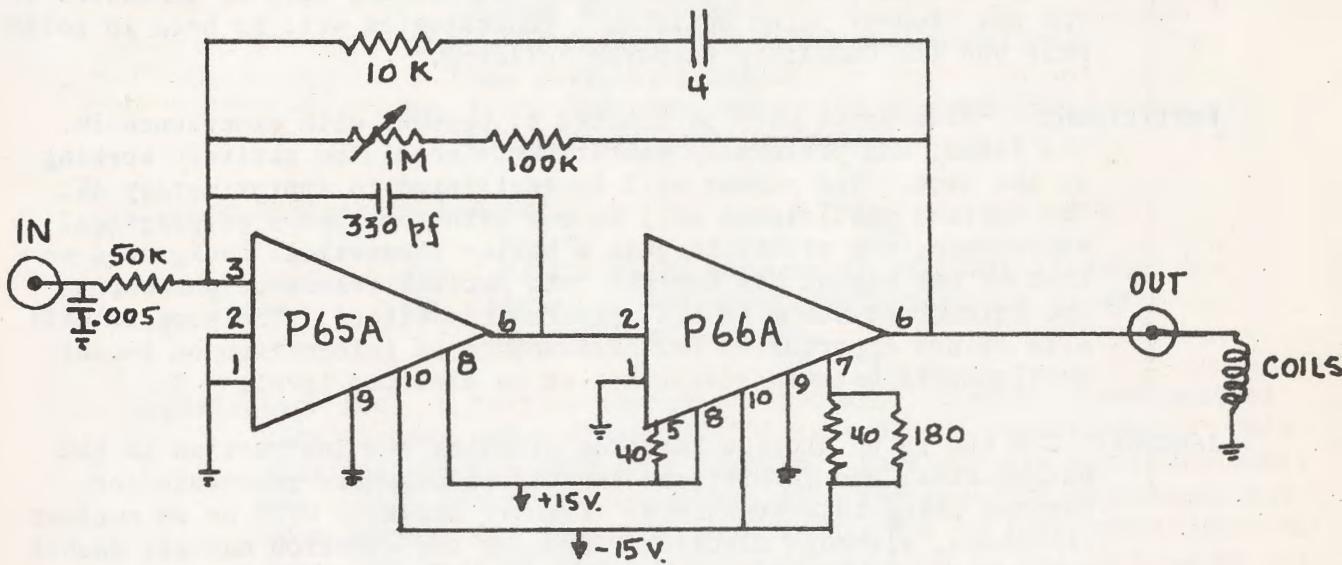
MIDLAND DIVISION  
MIDLAND, MICHIGAN 48640

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

Dear Barry:

It may be of interest to some that the venerable Superstabilizer, which is a galvanometer with photocell amplifier, can be replaced by modern solid state low drift operational amplifiers, at least in field-frequency lock applications.

We use the Philbrick Researches, Inc. P65A voltage amplifier followed by the P66A current amplifier (booster), which supplies a pair of coils (#24 wire, 2000 turns each) in series on the magnet poles. (No doubt the Superstabilizer coils would do.) The  $\pm 15$  volts are supplied by two Dynage, Inc. D-14.6-0.150 solid state power supplies. The feedback resistor network shown allows a 10:1 adjustment of dc gain. There is, of course, considerable gain in the rf and phase sensitive detector circuits preceding these dc amplifiers. The 4  $\mu$ f capacitor and 10K resistor are a loop stabilization network.



Sincerely yours,

*Ned*

Edward B. Baker  
 Physical Research Laboratory, 1712

Title: Replacement of a Superstabilizer with Low Drift,  
 Trouble Free, Solid State Operational Amplifiers

UNIVERSITY OF FLORIDA  
GAINESVILLE, 32601

DEPARTMENT OF CHEMISTRY

September 1, 1967

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

Dear Barry:

Some Newsletter readers may be interested in a workshop which we are planning for next January. The preliminary program is described in the following material, although the lineup of speakers is not necessarily final. Anyone who might wish to attend may secure further information and an application blank by dropping me a note.

Yours,  
*Wallace*  
Wallace S. Brey, Jr.

Advanced Workshop and Seminar in Nuclear Magnetic Resonance  
Conducted by:

Department of Chemistry of the University of Florida  
Division of Continuing Education of the University of Florida

With the assistance of Varian Associates.

Dates: Monday, January 22, through Friday, January 26, 1968.

Location: Gainesville, Florida. Lectures and classes will be conducted in the new Student Union Building. Laboratories will be held in Leigh Hall and the Chemistry Research Building.

Participants: Attendance will be limited to persons with experience in the field, and preferably participants should be actively working in the area. The number will be restricted to approximately 45. The typical participant will be one with some years of practical experience, who wishes to gain a better theoretical background so that he may expand his insight into nuclear resonance phenomena and extend the scope of his research activities. The program will also afford opportunity for interchange of information on recent developments between scientists at an advanced level.

Content: The aim is to provide advanced training and instruction in the mathematical and theoretical aspects of magnetic resonance for persons using this technique. Primary emphasis will be on nuclear resonance, although electron resonance and electron nuclear double resonance may be dealt with briefly.

The following topics will be included:

- (1) Mathematical methods of interpreting high-resolution spectra.
- (2) Methods of signal enhancement.
- (3) Study of rate processes.

- (4) Relaxation times and spin echo methods.
- (5) Spectra of oriented molecules.
- (6) Double resonance methods, homonuclear and heteronuclear; spin decoupling and spin tickling.
- (7) Use of computers in relation to the above areas.

Two to four lectures will be scheduled for each of the five days. A substantial amount of time will be spent in practice sessions with the participants engaged in self-study or in discussion under the direction of a discussion leader. Laboratory demonstrations and practice will be provided, with principal emphasis on various types of spin decoupling and spin tickling. There will be opportunity for participants to use the University of Florida IBM-360 computer.

Staff: Director, Wallace S. Brey, Jr., Professor of Chemistry, University of Florida.  
Assistant Director, Katherine N. Scott, Research Associate, University of Florida.  
Coordinator, W. T. Coram, Jr., Educational Director, Division of Continuing Education.

Lecturers:

Dr. Ray Freeman, Varian Associates  
Dr. Charles S. Johnson, Jr., University of North Carolina,  
Chapel Hill.  
Dr. Lawrence C. Snyder, Jr., Bell Laboratories,  
Murray Hill, N. J.  
Dr. J. D. Swalen, IBM Research Laboratory,  
San Jose, California.  
Dr. D. E. Woessner, Mobil Oil Company Field Research  
Laboratory, Dallas, Texas.

Discussion Leaders:

Dr. Jeff C. Davis, University of South Florida.  
Dr. Charles G. Moreland, North Carolina State  
University at Raleigh.

Registration Fee: A tuition charge of \$100 will be made to non-academic participants and a charge of \$50 to academic participants. This will include the cost of a banquet. Participants must meet their own living and travel expenses, with the possible exception that some support for academic participants may be forthcoming from NSF. Rooms for most of the participants, as well as meals, will be available in the Union Building in which the classes will be held. In addition, there are a number of large motels within a mile of the campus.

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OUR REF.

YOUR REF.

DATE

Res/G.2050(B)/ACC/DJM/PMC.

31st August, 1967.

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

### Spin-rotation interaction in phosphorus

Dear Dr. Shapiro,

Studies of nuclear spin-lattice relaxation in several molecules containing both hydrogen and fluorine have shown that, at ambient temperatures, the longitudinal relaxation time,  $T_1$ , of the fluorine is considerably shorter than that for the hydrogen nuclei. The results have been interpreted in terms of relaxation mechanisms operating on fluorine alone; both anisotropy in the chemical shift and spin-rotation interaction have been proposed as likely contributors. However, the anisotropy contribution to the fluorine of  $\text{CFHCl}_2$  has been shown (1) to be very small, and for  $\text{C}_6\text{H}_5\text{CF}_3$  and  $\text{CHF}_3$ , the relaxation has been satisfactorily described (2) solely with reference to dipole-dipole and spin-rotation contributions.

In an attempt to discover the dominant relaxation mechanisms for  $^{31}\text{P}$ , a pure liquid sample of  $\text{P}_4\text{O}_6$  was examined at 25 and 15 Mc/s.  $T_1$  was found to be  $11.69 \pm 0.75$  sec. and  $11.27 \pm 1.5$  sec. respectively. Measurements on an oxygen-free sample at ambient temperatures, were made using the saturation recovery technique.

The separation of the intermolecular, intramolecular, anisotropy, and spin-rotation contributions is straightforward in this case. First, the anisotropy contribution can be ignored because of its dependence on the square of the applied field, in contrast to the field invariance of  $T_1$  found experimentally. Second, calculations of the dipolar interactions lead to the following very small values :

$$(1/T_1)_{\text{INTRA}} = 9.2 \times 10^{-5} \text{ sec.}^{-1}; \quad (1/T_1)_{\text{INTER}} = 8.1 \times 10^{-4} \text{ sec.}^{-1}.$$

The calculations were made using the well-known formulae (3).



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Dr. B.L. Shapiro.

$$(1/T_1)_{\text{INTRA}} = \frac{3}{2} \gamma^4 \frac{k}{\pi^2} \sum_j d_{ij}^{-6} \tau_c$$

$$(1/T_1)_{\text{INTER}} = \frac{3}{2} \gamma^4 \frac{k}{\pi^2} N \eta / kT$$

where the inter-phosphorus distance  $d_{ij} = 2.95 \text{ \AA}$ ,

the viscosity  $\eta_{21^\circ\text{C}} = 0.025 \text{ poise}$ ,

and the reorientational correlation time  $\tau_c = 4 \pi \eta a^3 / 3kT$ .

We are therefore, left with spin-rotation as the dominant mechanism for  $^{31}\text{P}$  relaxation in  $\text{P}_4\text{O}_6$ . According to Hubbard (4), for spherical molecules, the spin-rotation contribution to  $T_1$  can be expressed as

$$(1/T_1)_{\text{SR}} = 2 I \tau_{\text{SR}} \bar{C}^2 kT / \hbar$$

where the spin-rotation correlation time  $\tau_{\text{SR}}$  is given by (4)

$$\tau_{\text{SR}} = I / 6kT$$

Taking  $T_1 = 11.69 \text{ sec.}$ , and the moment of inertia of  $\text{P}_4\text{O}_6$  as  $7.824 \times 10^{-38} \text{ gm.cm}^2$ , the modulus of the spin-rotation coupling constant,  $\bar{C}$ , is  $19.5 \pm 1 \text{ k c/s.}$

Since there is no molecular beam data with which to compare this value, the following relationship between spin-rotational interaction and nuclear magnetic shielding (5) may be used to calculate the paramagnetic contribution to the chemical shift.

$$\sigma_{\text{PARA}} = e^2 / 3 mc^2 \left[ - \sum_i Z_i / r_i + \hbar^2 \bar{C} I / 4M \mu_N^2 g_P \right]$$

where  $Z_i$  and  $r_i$  are the nuclear charge and distance of the  $i$ 'th nucleus,  $g_P$  is the nuclear g-factor of  $^{31}\text{P}$  and the factor  $3 \bar{C} I$  replaces the summation of products of principal moments of inertia, and spin-rotation tensor components. Taking the magnetic moment of  $^{31}\text{P}$  as  $+1.1305$  Bohr magnetons

$$\sigma_{\text{PARA}} = (-346 \pm 7388) \times 10^{-6}$$

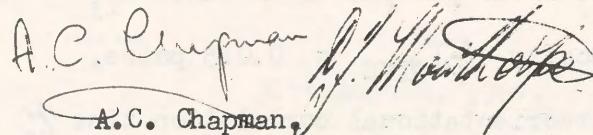
where the choice of sign for the second term depends on the sign of  $\bar{C}$ . The negative sign gives  $\sigma_{\text{PARA}} = -7734 \text{ ppm.}$  which is of the same order as the value of  $-11,828 \text{ ppm.}$  calculated by Letcher and Van Wazer (6) for the phosphorus paramagnetic shift in 85% phosphoric acid.

31.8.67.

Dr. B.L. Shapiro.

If these values are correct, the true magnetic moment of  $^{31}\text{P}$  is approximately 1% lower than the currently accepted value used above, which is derived from resonance measurements.

Yours sincerely,



A.C. Chapman  
D.J. Mowthorpe

A.C. Chapman,  
D.J. Mowthorpe.  
\_\_\_\_\_  
Research Department.

- (1) R.J.C. Brown, H.S. Gutowsky, and K. Shimomura, J.Chem.Phys. 38, 76, (1963).
- (2) R.H. Faulk, and M. Eisner, J.Chem.Phys. 44, 2926, (1966).  
J.H. Chaffin III, and P.S. Hubbard, J.Chem.Phys. 46, 1511, (1967).
- (3) N. Bloembergen, E.M. Purcell, and R.V. Pound, Phys.Rev. 73, 679, (1948).  
H.S. Gutowsky, and D.E. Woessner, Phys. Rev. 104, 843 (1956).
- (4) P.S. Hubbard, Phys. Rev. 121, 1155, (1963).
- (5) S.I. Chan, M.R. Baker, and N.F. Ramsey, Phys. Rev. 136, A1225, (1964).
- (6) J.H. Letcher, and J.R. Van Wazer, J.Chem.Phys. 44, 815, (1966).



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מחלקה איזוטופים

September 1, 1967.

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

Dear Prof. Shapiro,

We wish to make a comment on the dependence of pseudo-contact shifts on correlation times.

McConnel and Robertson [J. Chem. Phys., 29, 1361 (1958)], who have derived the expressions for the pseudocontact shifts, distinguish between two cases: solid and liquid. A system is defined as liquid only when the molecular tumbling is characterized by a correlation time.

$\tau_c \ll \frac{\hbar}{8H_0} |g_{\parallel} - g_{\perp}|^{-1}$ . Now, for  $H_0 = 1.4 \times 10^4$  gauss one has the condition  $\tau_c < 0.8 \times 10^{-11} |g_{\parallel} - g_{\perp}|^{-1}$  sec, and it is not immediately obvious that for a given complex even in solution, this requirement is fulfilled. Consideration of the correlation time for molecular tumbling should always be made in order to choose the correct expression for the pseudocontact contribution. We should like to point out, however, that in those cases where  $\tau \approx 10^{-11} |g_{\parallel} - g_{\perp}|^{-1}$  sec both the expressions, for solids and liquids, do not hold.

The Fermi-contact and the pseudocontact contributions to the shifts are linear with  $1/T$  and should extrapolate to zero at  $1/T = 0$ , whereas greater slopes and large intercepts are expected for systems with  $\tau_c \approx 10^{-11} |g_{\parallel} - g_{\perp}|^{-1}$  sec. In fact we have observed such phenomena in aqueous solutions of rare-earth ions.

Sincerely yours,

*Jack Reuben*  
Jack Reuben

*Daniel Fiat*  
Daniel Fiat

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RADIOLOGIQUES DE LA DÉFENSE**

OTTAWA 4, ONTARIO

AIR MAIL

5 September, 1967.

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

Subject Index for IITNMRN

Dear Dr. Shapiro,

With the number of issues of IITNMRN now over 100 we, and perhaps others, have found it increasingly difficult to retrieve information. Some time ago Miss P.M. Lutley of our laboratories started to make a subject index on which she has worked in her rare free moments. This index now covers the period July 1965 to June 1966 inclusive, and as you can imagine, it turned out to be a much more formidable task than we anticipated. Apart from a few entries and editorial changes, it is now ready for typing, and we should be able to send it to you in the next couple of months.

I would like to suggest that it may be a good idea to have a mandatory list of index words, including names of chemicals, with each contribution, as well as the now mandatory title. People can then make their own indexes much more quickly and even an "official" subject index becomes a possibility.

In the spirit of the recent exchange of letters, Epexegesis and Son of, I should perhaps entitle this one "Dangling a Carrot". Since, however, there is liable to be a misunderstanding in which I would probably get axed, I thought better of it.

Yours sincerely,

M.A. Weinberger.

STANFORD UNIVERSITY  
STANFORD, CALIFORNIA

DEPARTMENT OF CHEMISTRY

7 September 1967

Dr. M. A. Weinberger  
Defence Chemical, Biological and Radiation Laboratories  
Ottawa 4, Ontario  
Canada

Indexes for IITNMRN

Dear Dr. Weinberger:

Thanks for your September 5 letter about a Subject Index for the IIT NMR Newsletter. Your Miss Lutley has, I am sure, accomplished a most Herculean and useful task, although I shudder to think of the size of this one-year's index, and the cost of producing it; in any event, I look forward to receiving it.

Some time ago (would you believe several years?) we prepared an Author Index covering a few year's span. This proved very useful, although again rather large and costly. Perhaps it is time to do this again, and I herewith call for a volunteer to step forward, although I am compelled to warn that it is not a small task (Zero-order calculation: (Say) 30 issues x ca. 25 contributions per issue x ca. 2.74 authors per contribution = a large number of entries to be processed and typed.). I am, however, prepared to be most generous toward anyone submitting such an index (the details of which should probably be ironed out in advance with me for logistic reasons): the reward will be one subscription credit (i.e. + 9 months) to the Newsletter, plus the original copy of the Freeman etching or Lauterbur letter of your choice.

Returning (!) to your letter, I must reject, tentatively at least, your idea of making it mandatory for each contributor to prepare a list of index words for his contribution. This would, in my opinion, raise significantly the energy barrier to contributing for many of our busy participants, and would also add to the space and other logistic problems, etc. Besides, such a step would make the Newsletter more formal and journal-like, which would be Bad. I feel it would be much better for you and Miss Lutley to take this task on as a continuing mission, if you can find your way clear to do so; perhaps you could keep a running index which could be included in the Newsletter every (say) 6 months or so. Suggestions from one and all re this, or other "off-line", major contributions to the production of the Newsletter, would be most welcome. We will be happy to attend to the production and distribution of such things, as long as the money holds out.

With all best regards,

*Barry Shapiro*

Bernard L. Shapiro  
Visiting Scholar (sic)

P.S. I do not understand your last paragraph, although it sounds vaguely improper. I will therefore consult with my learned neighbors Messrs. Ettinger and Freeman, who will, I am sure, be able to explain it to me.

BLS

## University of Notre Dame

College of Science  
Notre Dame, Indiana 46556

Department of Chemistry

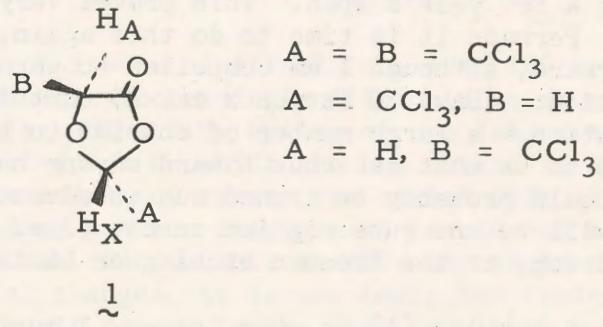
August 28, 1967

Professor Barry Shapiro  
Department of Chemistry  
Stanford University  
Stanford, California 94305

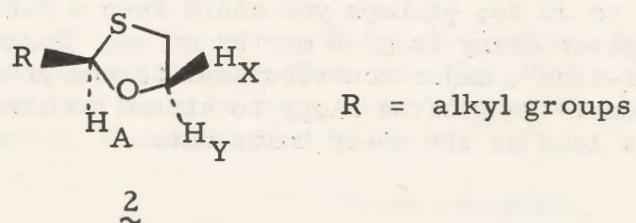
Dear Barry:

## Long Range Coupling in Heterocyclics

I am sort of reluctant to send in two contributions within two weeks, particularly after a request for our next contribution. However, I am stimulated to do so after reading the recent contribution by Dr. H. Fritz (IIT NMR Newsletter, 107-4) concerning the observed long range coupling between the indicated trans-hydrogens in 1.



In our recent contribution (IIT NMR Newsletter 100-38) we discussed the NMR spectra of 2-substituted-1,3-oxathiolanes 2. The spectra of these compounds were recorded on our HR-60, and due to field stability problems associated with very slow scans on the HR-60 we were restricted to employing fairly rapid scans. We observed that the signals representing  $H_A$  (pseudoaxial)



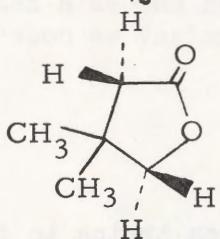
and  $H_X$  (pseudoequatorial) were slightly broadened. A few months ago we received our A-60-A. Slow scans (1000 sec sweep time, 100 Hz sweep width) of the spectra of these compounds revealed that  $H_A$  and  $H_Y$  were spin-coupled

Page 2

Professor Barry Shapiro  
August 28, 1967

$J = 0.55\text{Hz}$ . We have not determined the sign of the coupling constant. The half-height peak width for the resonance lines of  $\text{H}_Y$  and TMS are 0.45 Hz.

This observation is very similar to that of Dr. Fritz's. Dr. Fritz suggested coupling via five bonds by  $\sigma-\pi$  interaction. However, he did not observe this long range coupling with compound 3. This would seem to indicate that the



3

coupling might involve a four-bond interaction through the oxygen atom. It is interesting to note that there is no long range coupling observed through the sulfur atom.

With best regards,

*[Signature]*  
Daniel J. Pasto  
Associate Professor of Chemistry

DJP:dw

## NORTH CAROLINA STATE UNIVERSITY | AT RALEIGH

## SCHOOL OF PHYSICAL SCIENCES AND APPLIED MATHEMATICS

DEPARTMENT OF CHEMISTRY  
Box 5247 ZIP 27607

September 7, 1967

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

Dear Dr. Shapiro:

For the last year we have been trying to follow the fluorine exchange with pmr of compounds of the type  $R_3MF_2$ , where  $R = CH_3-$  or  $C_6H_5CH_2-$  and  $M = As$  or  $Sb$ . Until this past week we have not been able to get reproducible results for any of the compounds in the various solvents tried; the problem being that the compounds attack glass and are extremely sensitive to  $H_2O$ . We are now using teflon cells (NMR Specialties) and are getting interesting (and very reproducible) results. Some of our preliminary findings are listed in Table I below. At present we think that the first-order exchange for  $(C_6H_5CH_2)_3AsF_2$  in  $CCl_4$  and  $C_6H_6$  involves an ionization mechanism. We would be interested in hearing from anyone who has a general line-shape equation program which will run on the IBM 360/75.

Table I

Fluorine-Exchange Data<sup>a</sup> For 0.10M, 0.15M and 0.20M  $(C_6H_5CH_2)_3AsF_2$  in

		<u><math>CCl_4</math></u>		$\Delta E_a$ , kcal/mole
$t^\circ, C$	$\tau$ , sec	$1/\tau$ , sec <sup>-1</sup>		
40	0.0610	16.4		
60	0.0138	72.5		$15.8 \pm 2$
70	0.00613	163		
80	0.00428	233		
<u><math>C_6H_6</math></u>				
48	0.0118	85.0		
60	0.00613	163		$9.0 \pm 2$
70	0.00382	262		
80	0.00304	330		

- a. Tables of Exchange Broadened NMR Multiplets. The Weizmann Institute of Science, Rehovot, Israel, 1960.

-2-

One of us (CGM) spent June and July at the University of Florida working with Professor Wallace S. Brey on F<sub>19</sub> n.m.r. and computer analysis of second-order spectra. We were able to get LAO3 as revised by Roger Reaville of Procter and Gamble to run on the IBM 360 at Florida and NCSU.

We have recently purchased a second spectrometer, the DA-60EL, which we hope to have in operation as soon as Varian sends an engineer.

Sincerely yours,

*C. G. Moreland*

C. G. Moreland  
Assistant Professor

*J. Long*  
G. G. Long  
Associate Professor

L. J. Gerenser

CGM:GGL:ad

 **hooker research center**

NIAGARA FALLS, NEW YORK 14302, PHONE (716) 284-9965

September 5, 1967

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

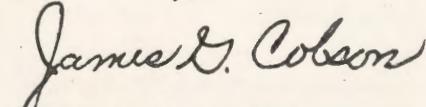
Dear Dr. Shapiro:

I would like to notify IIT Newsletter readers of an opening in our laboratory for an NMR spectroscopist with a good organic background to be in charge of our NMR group. Duties will at least entail: (1) interpretation of the more difficult spectra in  $^{31}\text{P}$ ,  $^1\text{H}$ ,  $^{19}\text{F}$  and  $^{13}\text{C}$ ; (2) setting up complex mixture analyses, (3) utilizing computer analysis to give more detailed structural data, (4) continuing a training program for other laboratory research chemists in NMR and (5) updating our instrument further to give better performance with the less sensitive nuclei.

We have an HA100 and routinely run hydrogen phosphorus and fluorine. Carbon is run on special request. Technicians are available to run the instrument, two electronics personnel repair the instrument and a chemist to do routine analysis.

If anyone is interested please contact Mr. A. V. Thorpe in our personnel department.

Yours truly,



James G. Colson

HOOKER CHEMICAL CORPORATION

CARNEGIE INSTITUTE OF TECHNOLOGY  
SCHENLEY PARK  
PITTSBURGH, PENNSYLVANIA 15213

September 5, 1967

DEPARTMENT OF CHEMISTRY

TELEPHONE: 621-2600  
AREA CODE 412

"CRACKING PARAMAGNETIC SHIFT PEANUTS WITH A FIELD-FREQUENCY CONTROL  
STEAM ROLLER ON THE HA-100"

Dear Barry:

Chien Ho, Don Davis and I have been interested for some time in paramagnetic Hemin derivatives. The proton spectra of these compounds show broad lines (of the order 100 Hz half-width) shifted ca. 30 to 70 ppm downfield from TMS. The 60 MHz spectra are, in several cases, complicated by overlapping lines; we therefore decided to use the HA-100 to spread things out and, while we were about it, to modify (temporarily) the HA-100 for field-frequency control operation with the required large sweep ranges and offsets.

A rough description of our set-up follows. The frequency sweep channel of the HA-100 "Lock-Box" is used in the "Field Sweep" mode with the following modifications: the sweep frequency is provided by a General Radio 1161-C audio frequency synthesizer, the output of which is connected to the "Sweep Frequency Out" connection at the rear of the Lock Box, after the "Oscillator Amplifier" and "Sweep Oscillator Network" cards are removed from the Lock Box. The GR frequency synthesizer is swept by a triangular voltage sweep ramp built by Dennis Wisnosky here at Mellon. The detection channel uses a Princeton Applied Research JB-5 audio phase sensitive detector in an "Internal Reference" mode; the reference frequency from the JB-5 is used, without amplification, to frequency modulate the V-4311 r.f. unit. A Mosely XY-recorder driven by the voltage sweep ramp, is used, rather than the HA-100 recorder.

With a two percent (V/V) DMSO line used as a lock signal in the field sweep mode one can sweep (and stay locked) over a range of 1 kHz to 9 kHz in a sweep time of 60 seconds, with no adjustments of the rf phase control required. Sufficient modulation is provided by the PAR for the large rf power needed to detect the lines in these paramagnetic substances, even at modulation frequencies in the 10 to 15 kHz range.

In the interests of space (and in order to meet the deadline for this month's contribution) I've omitted block diagrams of the experimental set-up and copies of spectra. With regard to the former, either Dennis Wisnosky (who was largely responsible for getting the modifications set-up) or I will be happy to supply more information. With regard to the latter, we're awaiting the results of variable temperature experiments before completing spectral assignments, but we'll be glad to correspond with those interested in our results so far.

Sincerely yours,

*Bob*

Robert J. Kurland  
Carnegie-Mellon University

108-42

DIVISION OF PURE CHEMISTRY  
DIVISION DE CHIMIE PURE



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G. Govil  
21.8.67.

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