

Joseph B. Lambert

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

No. 102
MARCH, 1967

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Deadline Dates: No. 103 - 15 April 1967
No. 104 - 15 May 1967

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

ILLINOIS INSTITUTE OF TECHNOLOGY
CHICAGO, 60616

DEPARTMENT OF CHEMISTRY

Concerning the Future of the [IIT]NMR Newsletter - the Second Exciting Chapter

In issue No. 100 of this Newsletter, I discussed the desire to disassociate myself, before too long, from this non-journal; the reasons cited included both the costs of production and the non-trivial demands on my time. During the last two months I have received many expressions of appreciation for the Newsletter (for which both IIT and I are duly grateful), several screams of pain at the idea of its demise (execution?), and a significant number of constructive ideas aimed at its continuance.

Among the positive ideas were several which involved my continuing to run the Newsletter, and I must admit my desire to do so if an appropriate mechanism can be found. I have grown rather fond of the Newsletter over the years - it is an albatross, but it's my albatross. Besides, how else could one get (for free) so many pretty foreign stamps - even from Tahiti!?

Some of the save-the-Newsletter ideas received were, unfortunately, impractical - these included the charging of money as well as contributions, page charges, advertising, etc. When considered in detail, all such things could raise only a small amount of money while increasing markedly the clerical and logistic problems. The one suggestion which has been made which I like is the following: perhaps the (now several) NMR hardware manufacturers and others with a substantial investment in NMR would find it possible and desirable to help subsidize the direct production costs of the Newsletter; the IIT administration has indicated its continuing enthusiastic support of the idea of the Newsletter and its willingness to help as much as possible, both with the costs and with the necessary secretarial and clerical help. Thus we are faced with the necessity of raising as much of the ca. \$5,000 per year in direct production costs as possible, and I would like to respectfully request that instrument manufacturers, other large NMR-using industries, research institutes and government labs consider how they might participate with IIT in sponsoring the NMR Newsletter. (I assume that all schools would find it impossible or prohibitively difficult to get involved, but I'll be happy to learn otherwise). We have already had one large international company volunteer to participate. Anyone else should also contact me directly.

As extra inducements to participation, I am sure we can offer at least (i) the high probability that such contributions could be made in a tax-deductible form, (ii) extra copies of the Newsletter, (iii) the inclusion of the company or organization name in a list of sponsors printed in the Newsletter.

A small, but possibly vital point: a potential sponsor would be understandably reluctant to help finance an endeavor in which his products are subjected to irresponsible and/or unwarranted criticism. Even though criticism and comparison are both valuable and inevitable, I am sure that we can by now rely on the good common sense of our subscriber-participants. In those few cases where it might be necessary, I will be willing to act as a filter, and I am confident that this can be done so as to preserve both the free interchange of ideas and information, and also everyone's good will and feelings.

I look forward to continuing to hear from anyone who has constructive ideas or offers whereby the NMR Newsletter can be maintained and improved. It will, however, be necessary to come to an equilibrium before very long.

BLS

B. L. Shapiro
 15 March 1967

102-1
Campo Grande, 15-II-1967.

Dr. R. A. Bothner - By

Dr. B. A. Shapiro

Mellon Institute

Pittsburg

Pensylvania U.S.A.

Will anyone who can help
Dr. Rivera please write
directly to him? BLS

Messieurs,

Etant intéressé à la Résonance Magnétique Nucléaire, je viens par la présente vous demander si la valeur $\frac{v}{h}$ de la R.M.N. du radionucléide B_{10}^7 (spin $\frac{3}{2}$, parité -, période : 53,6 jours) a déjà été déterminée ? Comme cela a été fait pour le radionucléide S_{16}^{35} ($\frac{3}{2}$ + période : 87,1 jours) dont la valeur $\frac{v}{h} = 5,08 \frac{Mc}{10^4 \text{ gauss}}$. Je n'ai pas à ma disposition : The monthly letters on NMR (MELLONMR).

J'espère ne pas abuser de votre bonté, et dans l'espoir d'une réponse de votre part, avec mes remerciements anticipés, je vous prie de croire, Messieurs, à l'expression de mes sentiments bien respectueux.

Jean-Marie Rivera

Labor. de Química Orgânica
Universidade Rural do Brasil
Estrada Rio-São Paulo - Km 47,
via Campo Grande. Z.G. 26.
(Estado da Guanabara) Brazil.

* Valeur tirée du livre : High-resolution Nuclear Magnetic Resonance de J.A. Pople, W.G. Schneider and H.J. Bernstein.

COMMONWEALTH



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CANBERRA, A.C.T.

WP:IVS.

Refer.:

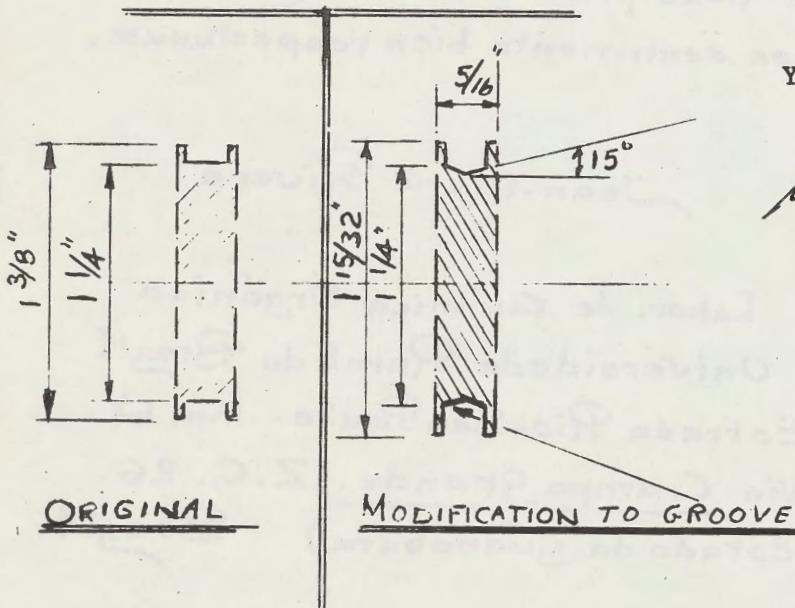
16th February, 1967.

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

Dear Professor Shapiro,

Re - Cording

Incessant trouble with the recorder stringing on our A-60 caused me to look for a solution. (The cord used to fall off the servo-motor clutch-pulley when the pen carriage hit the bottom). A modified clutch-pulley made in our workshop as per sketch appears to have completely overcome our difficulties. The increased flange and V shaped pulley makes the cord run true instead of piling up on the side or lapping over itself.



Yours sincerely,

(W.P.A. Pascoe)

PRESS RELEASE

4th Conference on Spectroscopy

The Hydrocarbon Research Group of the Institute of Petroleum will hold its fourth Conference on Spectroscopy at the Hotel Metropole, Brighton from 17th - 19th April, 1968. The Conference will be similar to those held previously and will include an instrument exhibition.

Progress will be reviewed in the following fields:-

Far Infra-red Spectroscopy, Laser/Raman Spectroscopy Attenuated Total Reflectance, Adsorbed Molecules, Photo-electron Spectroscopy, Nuclear Magnetic Resonance, Electron Spin Resonance, Transient Species and Data Retrieval.

The full programme and registration forms will be distributed later but applications for attendance or further details of the Conference or Exhibition should be sent to:-

The General Secretary,
Institute of Petroleum,
61, New Cavendish Street,
London, W.1.

UNIVERSITY OF CALIFORNIA

LAWRENCE RADIATION LABORATORY
P. O. BOX 808
LIVERMORE, CALIFORNIA

February 2, 1967

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

Dear Professor Shapiro:

Using Surplus V-2101 Voltage Regulators

The Varian V-2101 Voltage Regulator, which became surplus when the V-3506 Flux Stabilizer was introduced for stabilizing magnetic fields, can be used to reduce the current load on the 304TL's in the V-2100 Magnet Power Supplies and markedly increase the tube lifetime. The V-2101 is modified as shown in the accompanying diagram so that the magnet current is carried by twelve 304TL's (8 in the V-2100 and 4 in the V-2101) instead of eight tubes. Therefore, the current per tube is down by two-thirds and the power dissipated per tube is lowered by a factor of two. As a bonus there is an apparent increase in field stability at higher magnet currents as evidenced by slower drift rates when the flux stabilizer is not used.

To accomplish this modification connectors having at least 5 pins are added to the rear panels of the V-2100 and V-2101 along with a five conductor inter-connecting cable to carry the necessary voltages, currents, and interlocks. This is easier than trying to rewire the existing interchassis connectors. Connectors and wiring must be capable of handling 1500 volts and 2 amps.

To render the remainder of the V-2101 inoperative and remove possibly dangerous voltages on the unused rear connectors, T902 (V-2101) should be disconnected at TB903, the drive coil of K902 disconnected from the 110 volts and the leads between the plate, grid, and filament busses to TB902 are disconnected at the busses and taped off.

The coil K901 leads to J902 are removed and the coil reconnected to get its power when the V-2100 filaments are switched on. Inspection of the diagram reveals that after the modification the 4-304TL's in the V-2101 are merely in parallel with those in the V-2100 but have a separate interlocked filament supply. Two 5 ohm 20 watt resistors must be added across the filament of the V-2101 304TL's to give a balanced return to the corresponding point in the V-2100.

Sincerely yours,

Bert Holder

Bert Holder

Raymond Ward

James Happe

Richard Ryon

Chemistry Department

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Sincerely yours,

Bert Holder

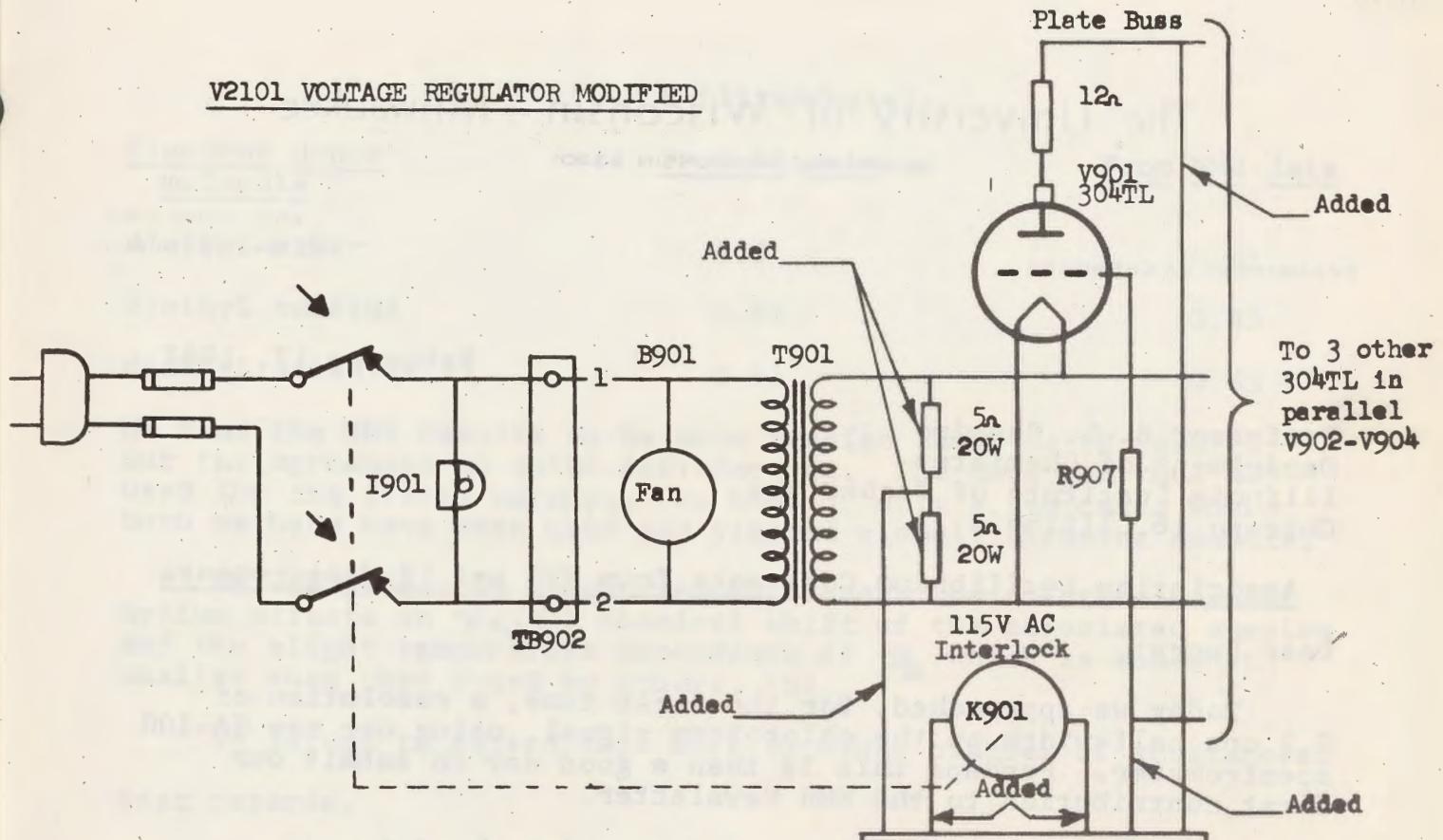
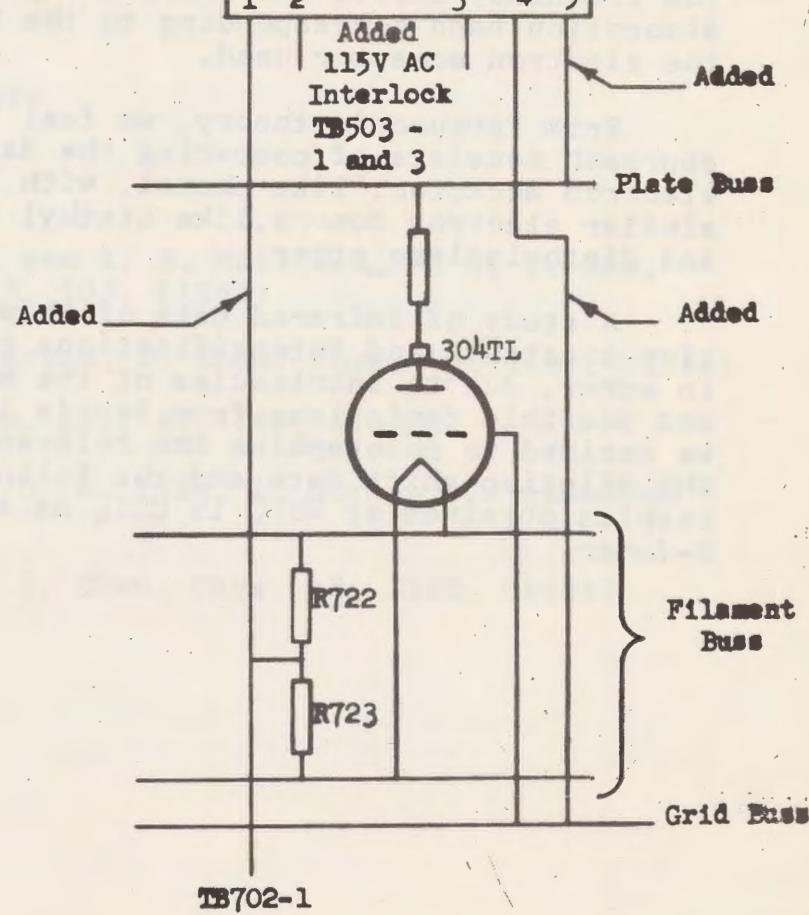
Bert Holder

Raymond Ward

James Happe

Richard Ryon

Chemistry Department

V2101 VOLTAGE REGULATOR MODIFIEDV2100 MAGNET POWER SUPPLY

The University of Wisconsin - Milwaukee

MILWAUKEE, WISCONSIN 53201

DEPARTMENT OF CHEMISTRY

AREA CODE - 414
TELEPHONE 228-4411

February 17, 1967

Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago 16, Illinois

Association Equilibrium Constants from NMR and IR Measurements

Dear Barry:

Today we approached, for the first time, a resolution of 0.2 cps half-width on the chloroform signal, using our new HA-100 spectrometer. Perhaps this is then a good day to submit our first contribution to the NMR Newsletter.

We have been very interested in testing the "electron-vibration" model suggested by Ferguson and Matsen and others (1) and elaborated on recently by Person (2) in so far as it applies to H-bonding. Basically, we are therefore interested in measuring the frequency shifts and increases in intensity of the infrared absorption band corresponding to the X-H stretching vibration of the electron acceptor used.

From Ferguson's theory, we feel that a most promising approach consists of comparing the data obtained for a single electron acceptor, like phenol, with a series of structurally similar electron donors, like diethyl ether, diethyl thioether, and diethylseleno ether.

A study of infrared data of this type shows that the association constants and intensifications to be determined might be in error, due to intricacies of the methods of evaluation (3) and possible deviations from Beer's law. (4) As a consequence, we decided to redetermine the relevant association constants from NMR dilution shift data and the following table shows representative results obtained at 40°C in CCl_4 as solvent, with phenol as H-donor.

K (liter/mole)

<u>Electron donor molecule</u>	<u>From IR data</u>	<u>From NMR data</u>
diethyl ether	4.3	4.61
diethyl sulfide	0.86	0.83
diethyl selenide	0.63	0.63

We find the NMR results to be more precise than the IR results, but the agreement is quite satisfactory. The data can thus be used for the stated purpose. We know of only a few cases where both methods have been used and yielded closely agreeing results.

We are presently attempting to discern the relatively small medium effects on δ_2 , the chemical shift of the associated species and the slight temperature dependence of δ_2 which is somewhat smaller than that found by others, (5).

We intend to extend this work to other classes of substances.

Best regards,

Werner W. Brandt

Werner W. Brandt
Associate Professor

Julian Chojnowsky
Postdoctoral Research Associate

Literature Cited:

- (1) For a convenient review, see R. S. Mulliken, W. B. Person, Ann. Revs. Phys. Chem. 13, 107, (1962)
- (2) H. B. Friedrich, W. B. Person, J. Chem. Phys. 44, 2161, (1966)
- (3) W. B. Person, J. Am. Chem. Soc. 87, 167, (1965)
- (4) P. H. Emslie, R. Foster, C. A. Fyfe, I. Hormann, Tetrahedron 21, 2843, (1965)
- (5) D. Muller, R. C. Reiter, J. Chem. Phys. 42, 3265, (1965)

WWB db

The Catholic University of America

Washington, D. C. 20017

DEPARTMENT OF CHEMISTRY

February 16, 1967

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

Subject: Solvent Effects Upon Geminal H-D Coupling.

Dear Dr. Shapiro:

In an earlier letter, IITNMRN 93, 26 (1966), we referred to our observation of a strong temperature dependence in H-D geminal spin-spin coupling. For several molecules containing the group -CHD-, the triplet proton resonance, $|J_{HD}| = 1.5$ Hz, was found to coalesce into a singlet peak in the region of about -20° . This decoupling is due to a slowing down of molecular motion, and concomitantly, a more effective quadrupolar relaxation of the deuteron. This can be understood in terms of the relationship between spin-lattice relaxation time T_1 and correlation time for molecular reorientation τ_c :

$$T_1^{-1} \propto (e^2 q Q)^2 \tau_q(t); \text{ where } \tau_q(t) = \tau_q^0 e^E / kT.$$

Slowed molecular motion due to a drop in temperature causes $1/\tau$ to become of the order of the spin coupling constant J_{HD} and coupling disappears.

One would predict that in addition to changes of τ_q due to thermal motion the same effect would result from dilution of the hydrogen bonded solute with a non-polar solvent. Molecular movement would be freer and the situation at higher temperatures would be approximated, i.e., $\frac{1}{\tau} \ll J_{HD}$.

We now report the observation of such spin-coupling by dilution of $(CH_3)_2CHCHDOH$ with solvents (Table 1).

At room temperature $(CH_3)_2CHCHDOH$ as a pure liquid shows a simple doublet for CH-CH-D-OH. Dilution with carbon tetrachloride, carbon disulfide, or chloroform caused a gradual transformation of the doublet into a doublet of triplets. The concentration at which this change occurred varied from solvent to solvent. For example, with pyridine and trifluoroacetic acid the change from doublet to doublet of triplets occurred at concentrations as low as 5% of solvent while with carbon tetrachloride, no change in the simple doublet was observed until 75% by volume of solvent was added. No change in the doublet CH-CHD-OH was observed upon dilution to the lowest observable concentrations with either cyclohexane or benzene.

Clearly the strongest force inhibiting rapid molecular motion for the pure alcohol is the presence of intermolecular hydrogen bonding. Solvents such as

-2-

CCl_4 , CS_2 and CHCl_3 act to break up this bonding by forming solvent separated solute molecules. The fact that much lower percent concentrations of $\text{C}_5\text{H}_5\text{N}$ and CF_3COOH is not readily explained. The effect is not simply one of acid or base catalysis, since other acids and bases at comparable concentrations do not cause the same change. We are continuing our study of these solvent effects.

Table 1

Effect of Dilution Upon Geminal Hydrogen-Deuterium

Geminal Coupling In $(\text{CH}_3)_2\text{CH CHDOH}$

% Volume of Solvent At Which Simple Doublet

Solvent	Changes to Doublet of Triplet at 25°
CCl_4	35%
CS_2	75%
CHCl_3	60%
$\text{C}_5\text{H}_5\text{N}$	5%
CF_3COOH	5%

Sincerely yours,

*Robert M. Moriarty*Robert M. Moriarty
Associate Professor of Chemistry*Stephen J. Druck*

UNIVERSITY OF Minnesota

INSTITUTE OF TECHNOLOGY

DEPARTMENT OF CHEMISTRY • MINNEAPOLIS, MINNESOTA 55455

February 24, 1967

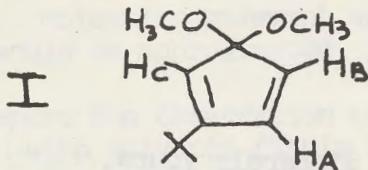
Frequency Sweep Double Resonance with a Varian A-60

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

Dear Barry,

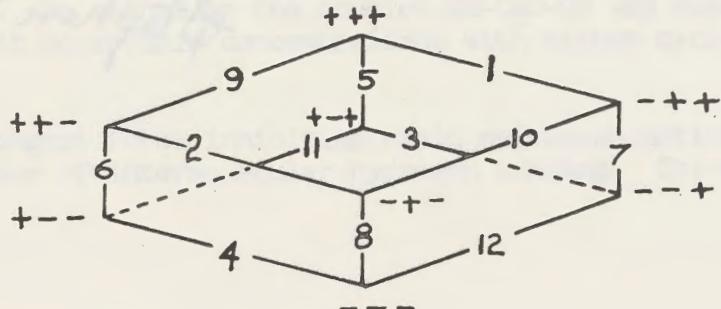
We recently modified our V6058A spin decoupler to extend frequency sweep double resonance capability to our Varian A-60 spectrometer. Possibly some of the IITNMR Newsletter readers would be interested in the details of the modification and in seeing the results of one of our experiments.

If the irradiating field is positioned using the A-60 recorder, we find that sweeping a Hewlett Packard model 3300A Function Generator and feeding the af output to a slightly modified V6058A leads to externally recorded field modulation frequency sweep double resonance spectra. For example, we were interested in determining the relative signs of the proton spin couplings in I. The A,B labelling in I was made by analogy to chemical shift assignments for ketals of α,β -unsaturated cyclic ketones and should be regarded as tentative. Analysis of the normal spectrum of I gave J_{Ac} and J_{Bc} to be of the same sign. This was confirmed from double quantum transition spacing and by analysis of normal spectra taken in different solvents where δ_{AB} was found to vary. The sign of J_{AB} relative to J_{Ac} and J_{Bc} , however, is not obtained as a result of the above analyses. The following frequency sweep double resonance experiment shows that J_{Ac} , J_{Bc} , and J_{AB} have the same sign.



$$\begin{array}{ll} J_{AB} = \pm 6.0 & T_A = 3.71 \\ J_{Ac} = \pm 1.9 & T_B = 3.82 \\ J_{Bc} = \pm 2.1 & T_c = 4.29 \end{array}$$

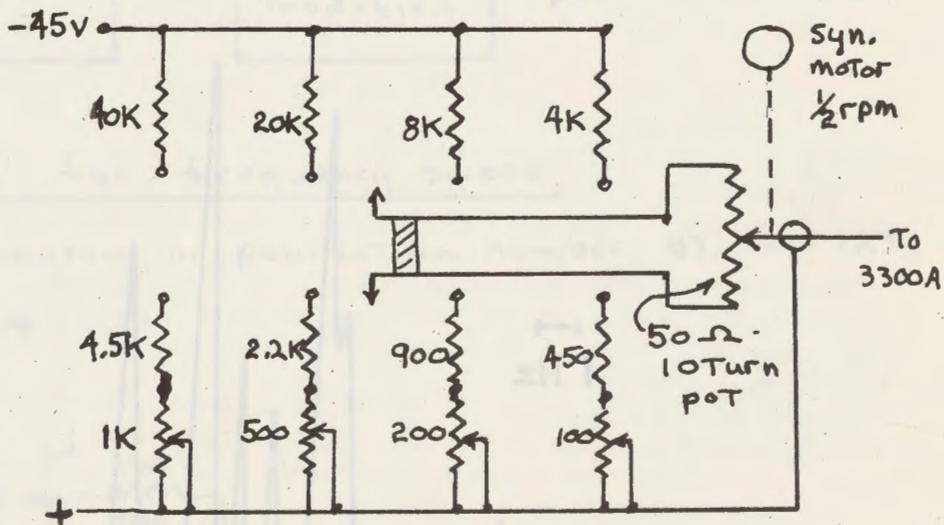
The top figure shows the normal spectrum of the ABC spin system of I (30% in methanol, v/v). Transition 12 was irradiated with $H_2 = 0.15$ mg (or 0.64 Hz) while observing the spectrum by linear af sweep. The resulting double resonance spectrum is shown in the bottom figure. Transitions 9, 11 and the beat frequency at 12 are not shown. If J_{AB} , J_{Ac} , and J_{Bc} have the same sign, the following "cube diagram" may be drawn. If the relative sign combinations are correct, irradiation



of transition 12 will lead to regressive transitions 4 and 8 splitting into well resolved doublets and progressive transitions 3 and 7 splitting into poorly resolved doublets in the double resonance spectrum (see: R. Freeman and W. A. Anderson, J. Chem. Phys., 37, 2053 (1962)). This is observed.

Also shown are a block diagram of the frequency sweep decoupler and the schematic of the resistor card to replace the resistor card which came with the V6058A. After inserting the modified card and connecting the oscillator (set at 5kc) the VFO amplitude adjust was set so that full power from the oscillator gave 5v. peak to peak at pins J and L on J2118 (VFO amplifier). This voltage was measured with an oscilloscope and was that measured at the same pins in field sweep operation.

The linear sweep was obtained with the following arrangement.



The power was supplied from a Burgess Super B Battery # 21308.

With best regards,

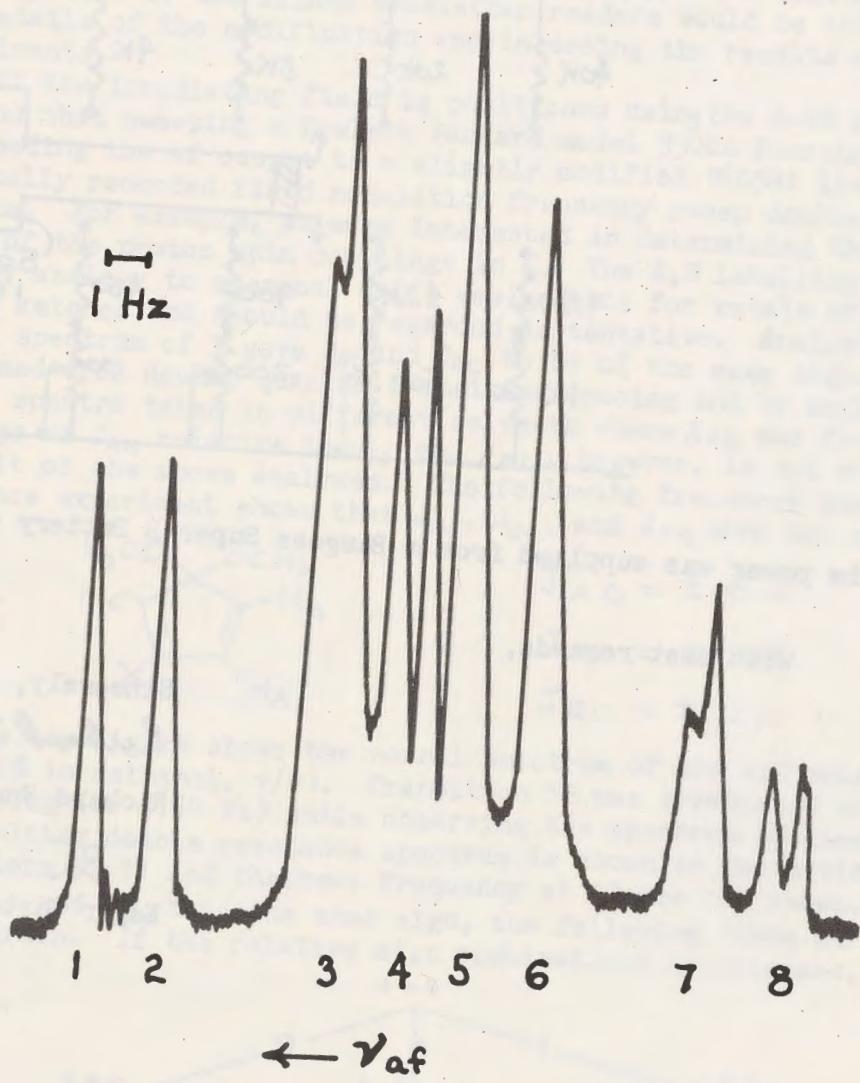
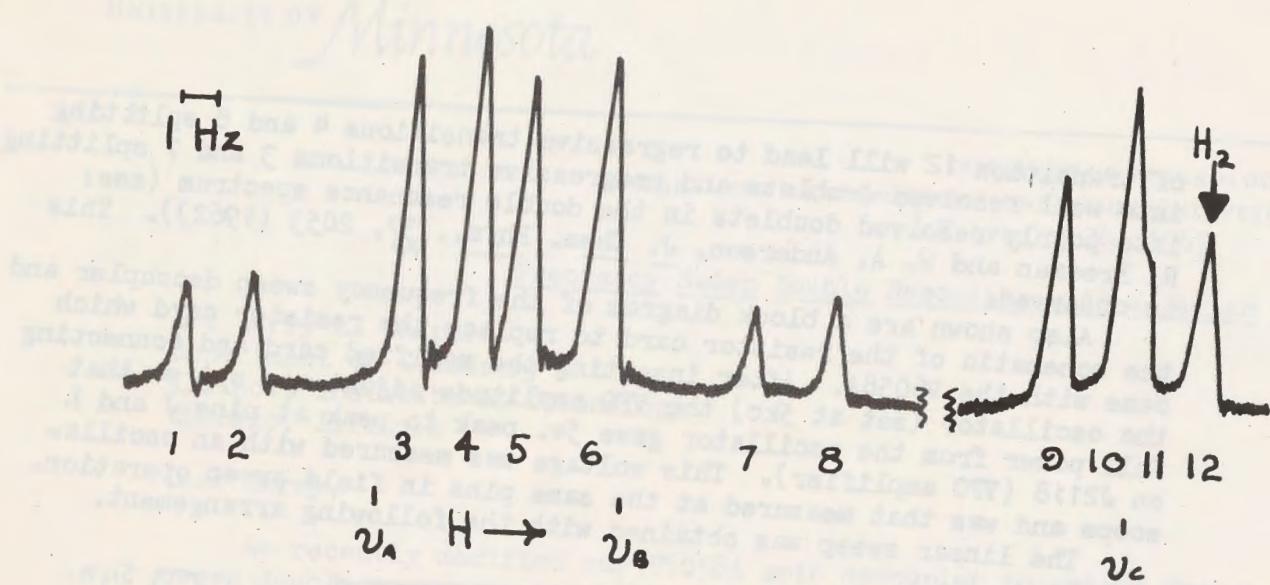
Sincerely,

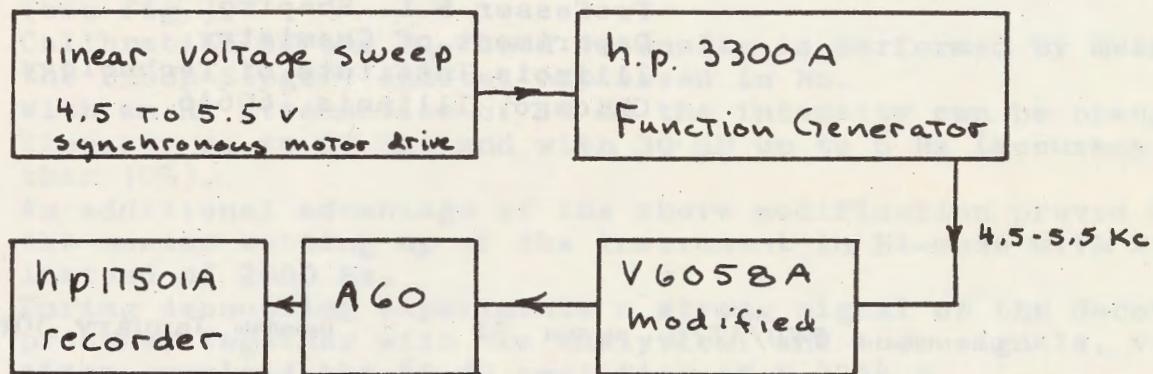
Richard F. Sprecher

Richard Sprecher

Ed

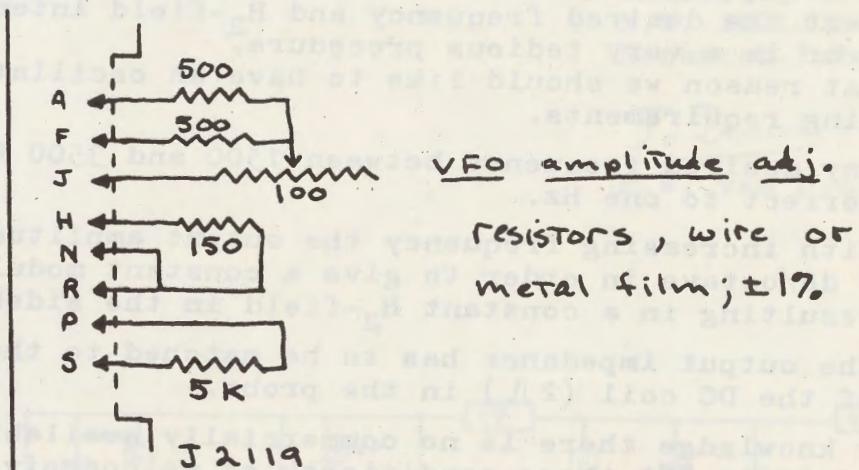
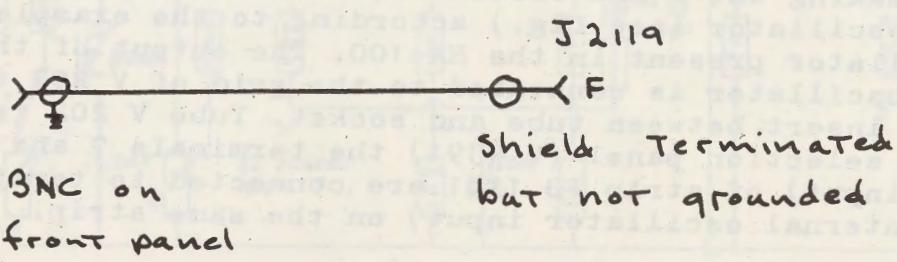
Edgar Garbisch



Block DiagramResistor Card for frequency sweep

to replace diagram in publication number 87-101-723

revision A 1164

V6058A modification

TELEPHONE: (02940) 2121

TELEGRAMS:

VITAMINE - WEESP

N.V. **PHILIPS-DUPHAR**

POSTBUS 2 - WEEESP - NEDERLAND

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

Uw ref./Your ref.

Onze ref./Our ref. FVD/AZ/6 Afd./Dept. 32

Datum/Date January 30th, 1967

Dear Dr. Shapiro,

Homonuclear spin decoupling on the Varian HA-100

As our first contribution to the IIT.NMR-Newsletter we want to report some modifications to the Varian HA-100 spectrometer which make it possible to perform decoupling experiments much more easily and quickly.

To select the desired frequency and H_2 -field intensity by trial and error is a very tedious procedure.

For that reason we should like to have an oscillator meeting the following requirements.

1. Any desired frequency between 1500 and 3500 Hz can be chosen correct to one Hz.
2. With increasing frequency the output amplitude must increase 6 dB/octave in order to give a constant modulation index, resulting in a constant H_2 -field in the sidebands.
3. The output impedance has to be matched to the low impedance of the DC coil (2Ω) in the probe.

To our knowledge there is no commercially available oscillator which fulfills all these conditions, so we conceived the idea of using the driver amplifier of the 2 KHz modulator, used in center-band mode, of the integrator-decoupler unit (V 3521 A), which meets the items 2 and 3 of the above requirements and is not in use in the HA-mode.

Instead of making the 2 KHz oscillator tunable, we built an additional oscillator (see fig.) according to the example of the manual oscillator present in the HA-100. The output of this additional oscillator is connected to the grid of V 203 tube by means of an insert between tube and socket. Tube V 202 is removed. On the mode selection panel (V 4391) the terminals 7 and 5 (2 KHz modulation input) of strip TB 1101 are connected to terminals 9 and 10 (internal oscillator input) on the same strip.

H.V. PHILIPS-DUPHAR

FVD/AZ/6

Dat. January 30th,
1967.

To have the oscillator conveniently at hand it is placed just below the V 4354 A ; the linear sweep unit, originally in that place, is moved to the empty place above the oscilloscope. For triple resonance experiments and additional input terminal for another oscillator is also connected to the grid of V 203 (see fig.).

Calibration of the H₂-field intensity is performed by means of the Bloch-Siegert shifts, expressed in Hz.

With an RF attenuation of 10 dB the intensity can be changed linearly up to 60 Hz, and with 30 dB up to 6 Hz (accuracy better than 10%).

An additional advantage of the above modification proved to be the easier setting up of the instrument in HA-mode with 2500 Hz instead of 2000 Hz.

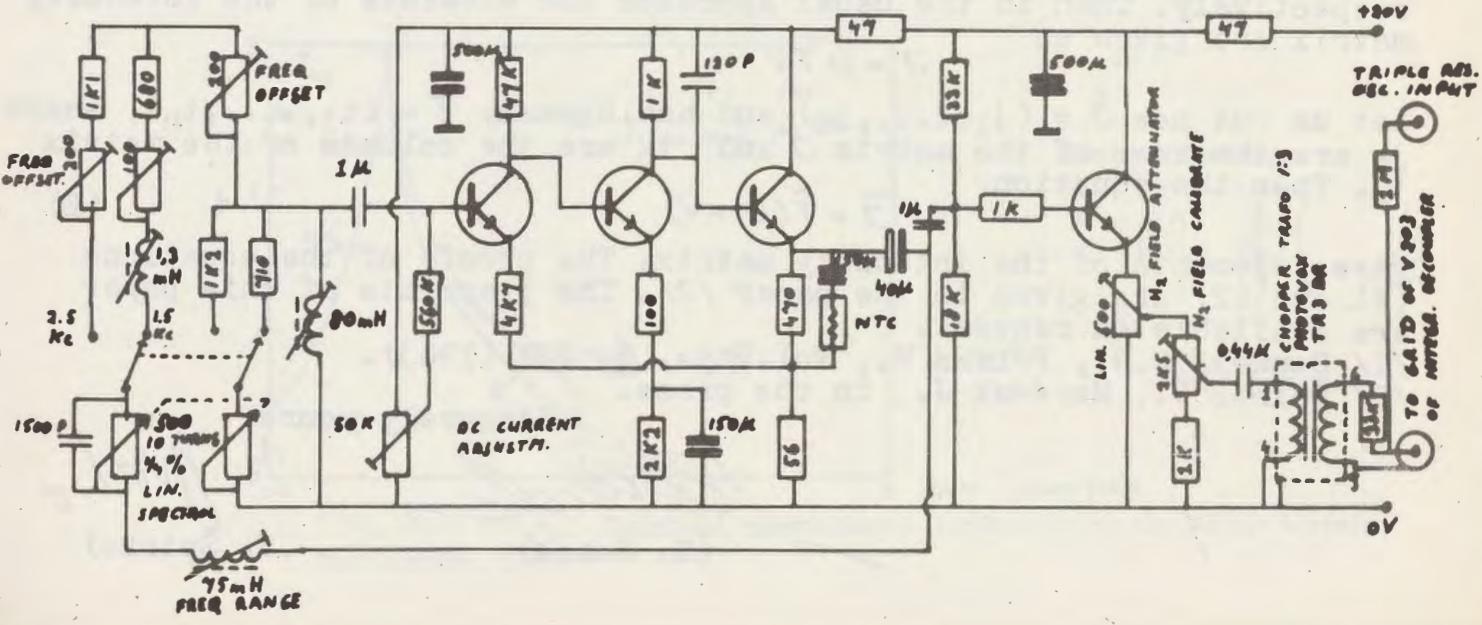
During decoupling experiments a strong signal of the decoupled protons, together with the analytical and lock signals, very often overload the 60-dB amplifier of V 4354 A.

This leads to loss of the lock, or at least to a weaker spectrum. The overloading sometimes occurs also during normal resonance experiments, and to avoid this a suitable attenuator (200 Ω potentiometer 10 turns) is placed between the output of the RF-unit and the RCVR input of V 4354 A. This potentiometer is used instead of the "spectrum amplitude" potentiometer, which is set to maximum. Due to the low impedance of the 60-dB amplifier this attenuator is, unfortunately, as little linear as the "spectrum amplitude".

Sincerely Yours,
N.V. PHILIPS-DUPHAR
Research Laboratories,
dept. 32

F. v. Denue

F.W. van Deursen



CZECHOSLOVAK ACADEMY OF SCIENCE
INSTITUTE OF ORGANIC CHEMISTRY AND BIOCHEMISTRY,

Br

FLEMINGOVO NÁM. 2

PRAHA 6

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

February 20th, 1967

On the "direct" method for calculating of NMR spectra

Dear Professor Shapiro:

Banwell and Primas /1/ proposed the so-called "direct"-method for calculating NMR spectra, which gave the resonance frequencies and intensities directly as solutions of an eigenvalue problem involving the derivation superoperator of the spin Hamiltonian. They have applied their method solely to the AB and An spin systems; any extension of the less trivial cases seems to be rather difficult. My colleague Dr. Spirko has proposed a new method for constructing the characteristic equation for a general spin system using the composition of matrices (usual matrix representation of the spin Hamiltonian in the basis of the product functions and selection rules are used) /2/.

Calculation of the frequencies: It is clear that the problem of finding the matrix for eigenvalue problem whose eigenvalues are the resonance frequencies consists in constructing a matrix the spectrum of which is given as differences between eigenvalues of submatrices χ_i (in usual representation) that form the transitions allowed by selection rules. Let us consider the ordered pairs of submatrices χ_i and χ_j , and let I_i and I_j be the unit matrices of the dimension of χ_i and χ_j , respectively. Then the eigenvalues of the symmetry matrix χ_{ij} , which is defined by

$$\chi_{ij} = \chi_i \cdot x I_j - I_i \cdot x \chi_j \quad (1)$$

(where the symbol $\cdot x$ denotes the direct product of matrices) are all possible differences $E_r^i - E_s^j$, where E_r^i and E_s^j are all eigenvalues of χ_i and χ_j , respectively.

Calculation of the intensities: Let U and V be eigenvectors matrices of χ_i and χ_j , respectively, and T the matrix proportional to the dipole moment matrix. Dimensions of U , V and T are (m, m) , (n, n) , and (m, n) , respectively. Then in the usual approach the elements of the intensity matrix are given as

$$J = U^T V$$

Let us put now $\hat{J} = (j_1, \dots, j_m)$ and analogously $\hat{T} = (t_1, \dots, t_n)$, where j_m are the rows of the matrix J and t_n are the columns of the matrix T . Then the equation

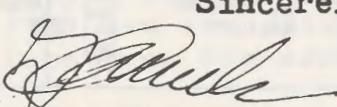
$$\hat{J} = \hat{T}(U \cdot x V) \quad (2)$$

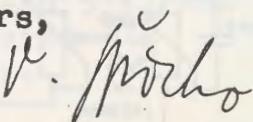
gives elements of the intensity matrix. The proofs of the equations (1) and (2) are given in the paper /2/. The preprints of this paper are available on request.

/1/ Banwell C.N., Primas H., Mol.Phys. 6, 225 (1963).

/2/ Spirko V., Morávek J., in the press.

Sincerely yours,


(Z. Samek)


(V. Spirko)

The University of Newcastle upon Tyne

School of Chemistry

The University
Newcastle upon Tyne 1
Telephone 28511

Department of Inorganic Chemistry

JWA/BMD.

8th March 1967.

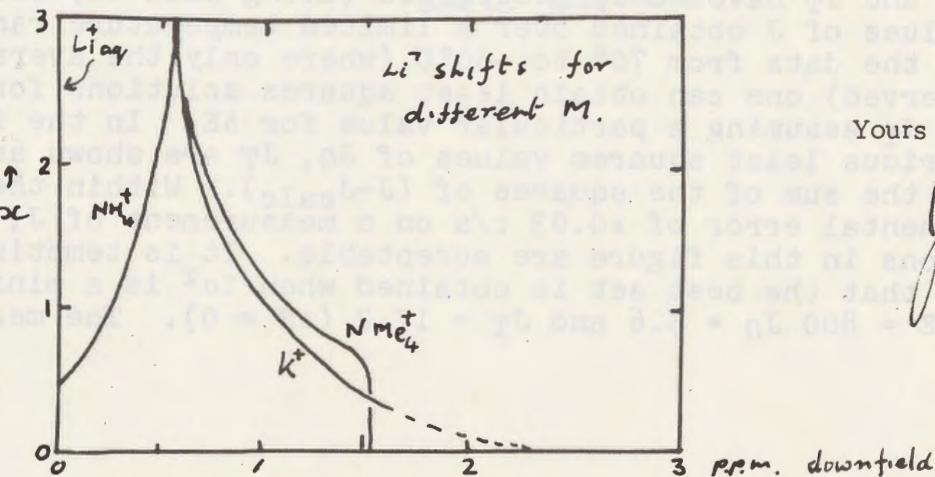
Professor B.L. Shapiro,
Department of Chemistry,
Illinois Institute of Technology,
Technology Center,
Chicago, Illinois 60616,
U.S.A.

Dear Professor Shapiro,

Li^7 shifts of complexed lithium ions.

Thank you for your reminder and my apologies for not writing sooner.

We have been looking at lithium solution chemistry using the Li^7 resonance. Some of the work has already been reported in detail¹ but I wish to present some recent conclusions about one aspect here. We have measured the chemical shifts of the Li^+ ion in aqueous solutions containing nitrilotriacetate (NTA) ions and various cations, i.e. solutions of mixed salts of the type $\text{Li}^+ \text{M}^{+x} \text{NTA}^{3-x}$. The Li^7 shift, which is downfield from aqueous Li^+ , is sensitive both to the value of x and to the nature of M since we have in solution an equilibrium between lithium - NTA complexes and aquated lithium. If M complexes strongly with NTA then the Li^7 shift moves upfield as x is decreased from 3 and less NTA becomes available to the lithium. If M complexes only weakly with NTA then the shift goes downfield as x is decreased since the proportion of lithium in the aquated form is decreased. From the form of the curves produced (see sketch) we have deduced that Li^+ forms two complexes with NTA, Li^+NTA and $\text{Li}(\text{NTA})_2$, the former having the larger downfield chemical shift. We hope also to be able to obtain formation constants for various M - NTA complexes using the known values for Li^+NTA and Na^+NTA .



Yours sincerely,

J.W. Akitt

1. J.W. Akitt and A.J. Downs. International Conference on the Alkali Metals, Nottingham, 1966.

DIVISION OF PURE CHEMISTRY
DIVISION DE CHIMIE PURECABLE ADDRESS
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RESEARCH

PLEASE QUOTE FILE NO.
NO DE DOSSIER À RAPPELERNATIONAL RESEARCH COUNCIL
CONSEIL NATIONAL DE RECHERCHES
CANADA

OTTAWA 2,

27 February, 1967

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

Dear Barry:

Dr. G. Govil has been investigating the temperature dependence of proton and fluorine resonances of CBr_2F CBr_2H in CFCl_3 over the temperature range of 70° to -100°C . This compound was chosen with the hope that at low temperatures the NMR spectra of the gauche and trans forms could be observed. This indeed turned out to be the case and as a result it was possible to measure J_G and J_T directly and find $\Delta E = E_G - E_T$, and ΔS , consistent with the equilibrium constant

$$K = \frac{J - J_G}{J_T - J} = \frac{1}{2} \cdot \ell \frac{\Delta S}{R} - \frac{\Delta E}{RT}$$

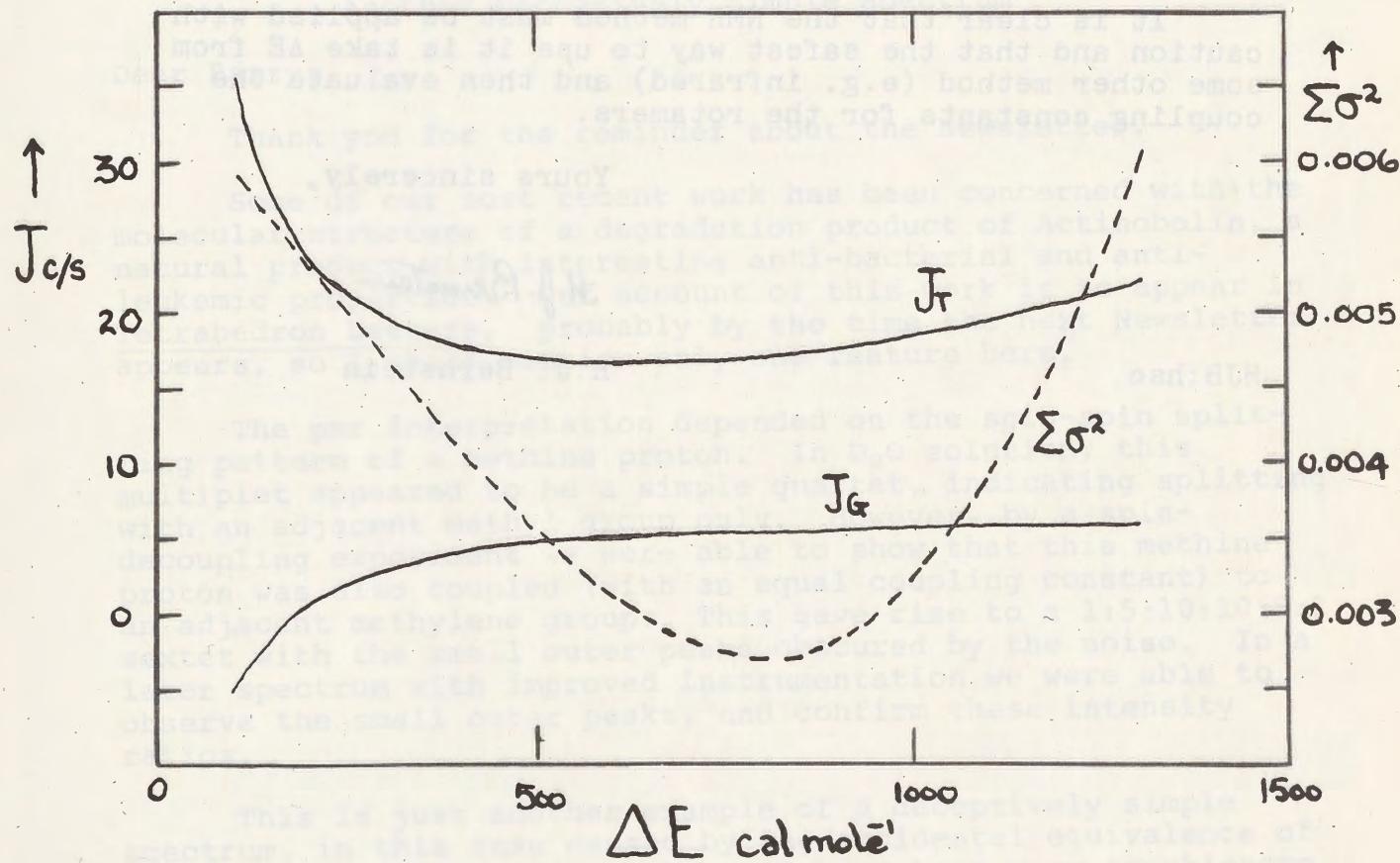
over the whole temperature range.

Usually when one applies the NMR method to the problem of rotational isomerism, it is customary to assume $\Delta S = 0$, and J_G and J_T have to be determined (along with ΔE) from the values of J obtained over a limited temperature range. Taking the data from 70° to -50°C (where only the average J is observed) one can obtain least squares solutions for J_G and J_T assuming a particular value for ΔE . In the figure the various least squares values of J_G , J_T are shown as well as $\Sigma \sigma^2$ the sum of the squares of $(J - J_{\text{calc}})$. Within the experimental error of ± 0.03 c/s on a measurement of J , all solutions in this figure are acceptable. It is tempting to assume that the best set is obtained when $\Sigma \sigma^2$ is a minimum i.e. $\Delta E = 800$ $J_G = 5.6$ and $J_T = 17.2$ ($\Delta S = 0$). The measured

... /2

Southern Research Institute

8/25/55 Mrs E.E. (name) has 1/2 hrs pt for assav
 Prof. D.G. Fl. 8 E. 10th St. (Levittown)
 and .x. 10th. address not available on T
 village) equal pot size \$9.00 to movie Instnemiteqxs
 Professors were invited to discuss their work to R.D. Jacobs
 Department of Biochemistry & Nutrition at University of Illinois
 Chicago, Illinois 60612. -will send you a
 Chicago, Illinois 60612.



This is just a spectrum in the 1000-1500 cal/mole' range. The equivalence of the two coupling constants, which have been very doubtful without the spin-decoupler.

Very sincerely yours,
 John C. Duncanson
 Research Chemist

DIVISION OF PURE CHEMISTRY
DIVISION OF CHIMIOPURE

values for J_G and J_T are however, 1.2 and 22.2 c/s respectively and $\Delta E = 200$ cal/mole with $\Delta S = 0.14$ e.u. The discrepancy arises for two reasons. Firstly, the experimental error of ± 0.03 c/s is too large (usually about 5% of the total variation in J) for an accurate least squares fit involving 3 variables. Secondly the statistical treatment of the data includes only the mean values of J whereas values within ± 0.03 c/s can occur with a fairly high probability.

It is clear that the NMR method must be applied with caution and that the safest way to use it is take ΔE from some other method (e.g. infrared) and then evaluate the coupling constants for the rotamers.

Yours sincerely,

H.J. Bernstein

HJB:hsc

H.J. Bernstein

over the whole temperature range.

Southern Research Institute



2000 NINTH AVENUE SOUTH
BIRMINGHAM, ALABAMA 35205

TELEPHONE 205-323-6592

February 28, 1967

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

Another Deceptively Simple Spectrum

Dear Barry:

Thank you for the reminder about the Newsletter.

Some of our most recent work has been concerned with the molecular structure of a degradation product of Actinobolin, a natural product with interesting anti-bacterial and anti-leukemic properties. Our account of this work is to appear in Tetrahedron Letters, probably by the time the next Newsletter appears, so I shall mention only one feature here.

The pmr interpretation depended on the spin-spin splitting pattern of a methine proton. In D₂O solution, this multiplet appeared to be a simple quartet, indicating splitting with an adjacent methyl group only. However, by a spin-decoupling experiment we were able to show that this methine proton was also coupled (with an equal coupling constant) to an adjacent methylene group. This gave rise to a 1:5:10:10:5:1 sextet with the small outer peaks obscured by the noise. In a later spectrum with improved instrumentation we were able to observe the small outer peaks, and confirm these intensity ratios.

This is just another example of a deceptively simple spectrum, in this case caused by the accidental equivalence of two coupling constants, which would have been very troublesome without the spin-decoupler.

Sincerely yours,

Martha C. Thorpe
Research Chemist

mct pc



Technische Hogeschool Delft

Laboratorium voor Technische Natuurkunde

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

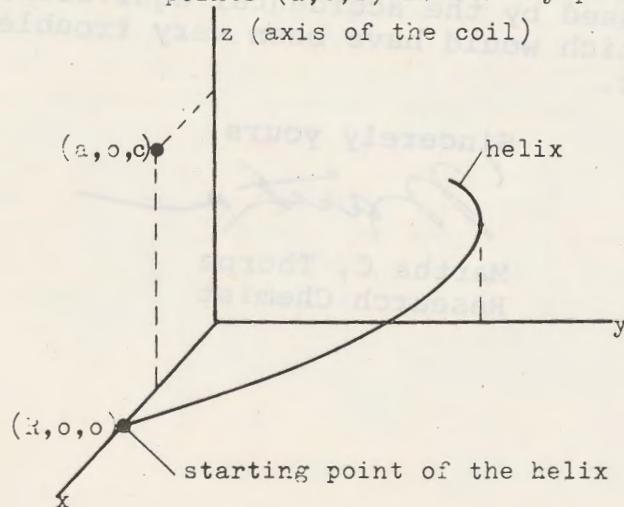
Uw kenmerk	Uw brief van	Ons kenmerk JS/WS	Delft, Nederland, Lorentzweg 1, tel. 01730-33222 1st February 1967 toestel: 5394
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Onderwerp

Dear Dr.Shapiro,

Title: For Do-it-yourselves.

1. Working with Overhauser dynamic polarisation of protons in liquids (high-resolution) we glued the sweepcoils (field H_s ; $H_{s \text{ max}} = 5 \cdot 10^{-6} H_0$) on the poles of our magnets without making them Helmholtzcoils. The results were disastrous: while in the centre of the sawtooth ($H_s = 0$) the H_0 -field was homogeneous, at the end of it (H_s maximum) no hr-nmr could be done due to the inhomogeneity of the H_s -field. Beware of this pitfall!
(Helmholtzcoils of course are the answer to the problem).
2. In our spin-echo work we use, amongst others, a single coil set-up. As we were interested in the homogeneity of the H_1 -field within the coil Mr.B.Sanders, a student in our department, made a calculation of the H_x and H_y components in points $(a,0,c)$ in the x-y plane (see sketch) with $0 \leq \frac{a}{R} \leq 0,5$



A.F.Mehlkopf.

Prof.Dr.J.Smidt.

Mehlkopf

J.Smidt

and $0 \leq \frac{c}{l} \leq 1$
(l is the length of the coil).
As communication of the re-
sults goes beyond the scope
of these letters, we suffice
with offering the Algol-pro-
gram for the calculation to
anybody who is interested in
it.

W. R. GRACE & CO.

RESEARCH DIVISION

GRACE

Washington Research Center, Clarksville, Maryland 21029

February 27, 1967

Professor B. L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago 16, Illinois 60616

Subject: Fine Tuning the H.P. 200 CD Audio Oscillator and a Slave Recorder for the A-60A.

Dear Barry,

We have made two instrumental modifications which may be helpful.

AUDIO OSCILLATOR FINE TUNING

When the Hewlett-Packard 200 CD Audio Oscillator is used to sweep a hetero nuclear decoupler (such as the NMR Specialties HD-60A) the dial is not sensitive enough to allow decent precision to be obtained quickly when searching for the exact decoupling frequency. Mr. Jim Light of our department, has modified the HP 200 CD in a simple way. Fig.1 shows the circuit modification and parts list. Fig.2 is a graph giving the characteristics of the new vernier dial.

SLAVE RECORDER FOR VARIAN A-60A

A slave recorder for the Varian A-60A allows smaller spectra to be recorded simultaneously. These are handier for notebook display, etc.

Mr. K. Ensing has constructed the slave recorder using a Varian X-Y Recorder, as follows:

A-60A Dual Sweep Potentiometer

In order for a slave recorder to be driven in synchronism with all the sweep times of the A-60A recorder, the single gang standard sweep potentiometer was replaced with a double gang sweep potentiometer. One gang for the A-60A DC sweep and the other gang for the X axis sweep on the slave recorder.

We used a 1.5 V long life telephone battery for the power source of the slave recorder sweep but the power could be tapped from the A-60A regulated 1.0 V DC power supply. Also the recorder up-down-auto pen switch and sweep switches can be wired in with the slave recorder pen such that the recorder pen controls on the A-60A will automatically control the slave recorder pen. This would entail no extra components except a few short lengths of wire.

Wiring details and structural modifications are shown in the enclosed sketches.

Sincerely,

A. J. Berlin

W.R. GRACE & CO.

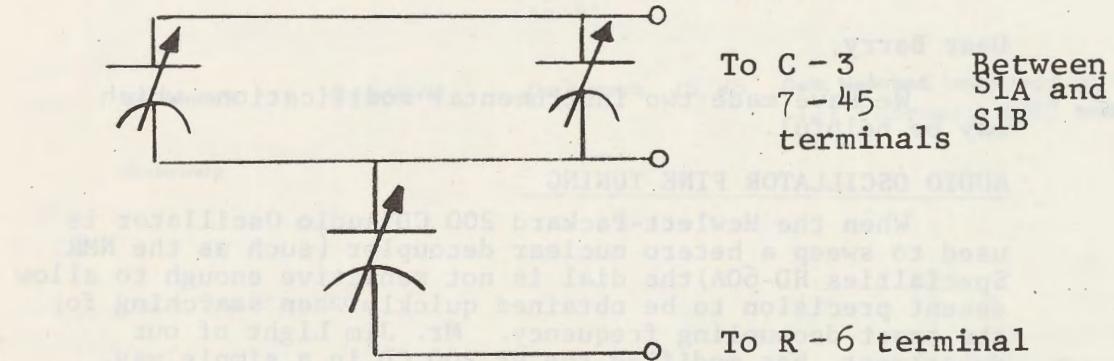
RESEARCH DIVISION

Washington Research Center, Clarksburg, Maryland

CONTINUATION

FIGURE I

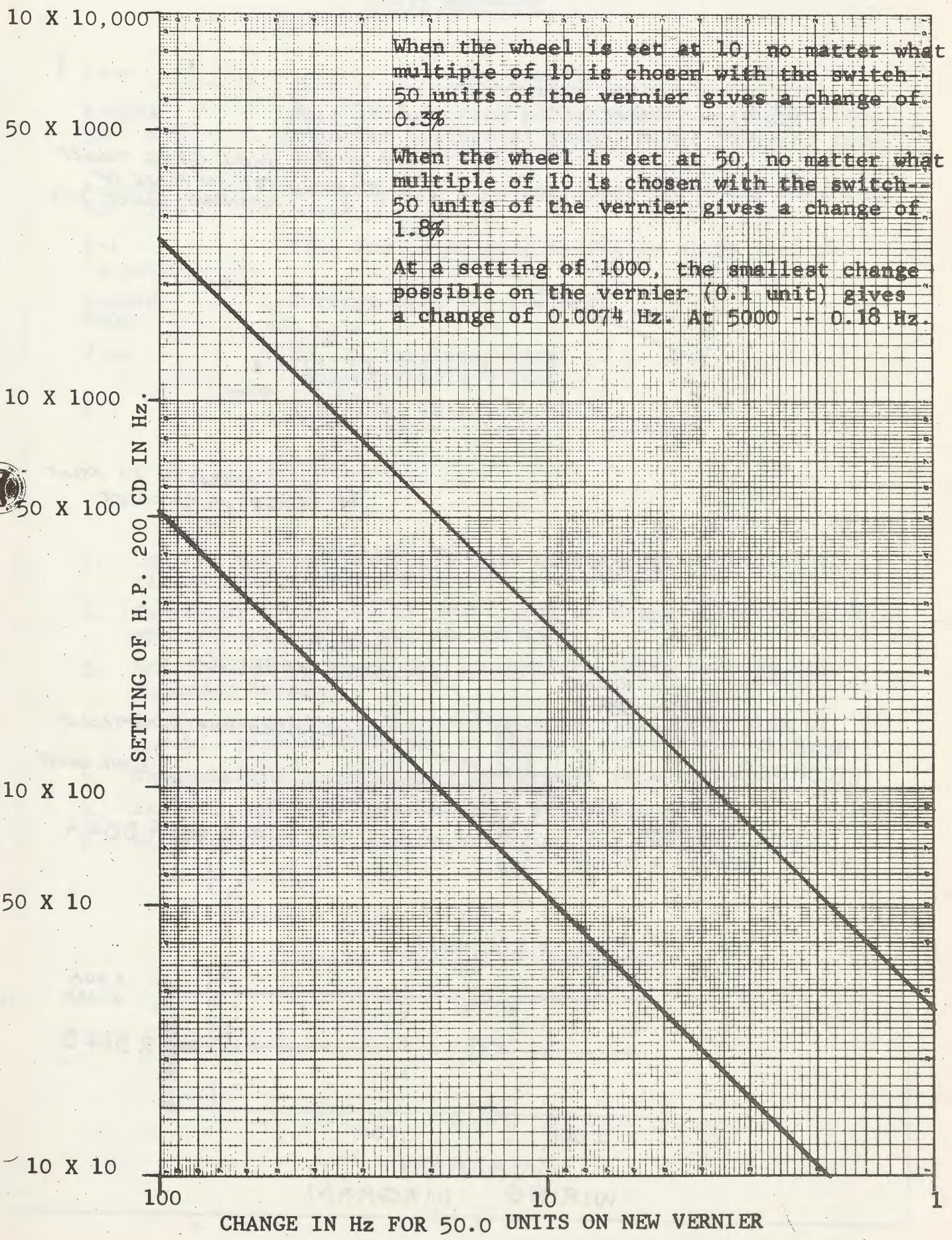
CIRCUIT MODIFICATION AND PARTS LIST



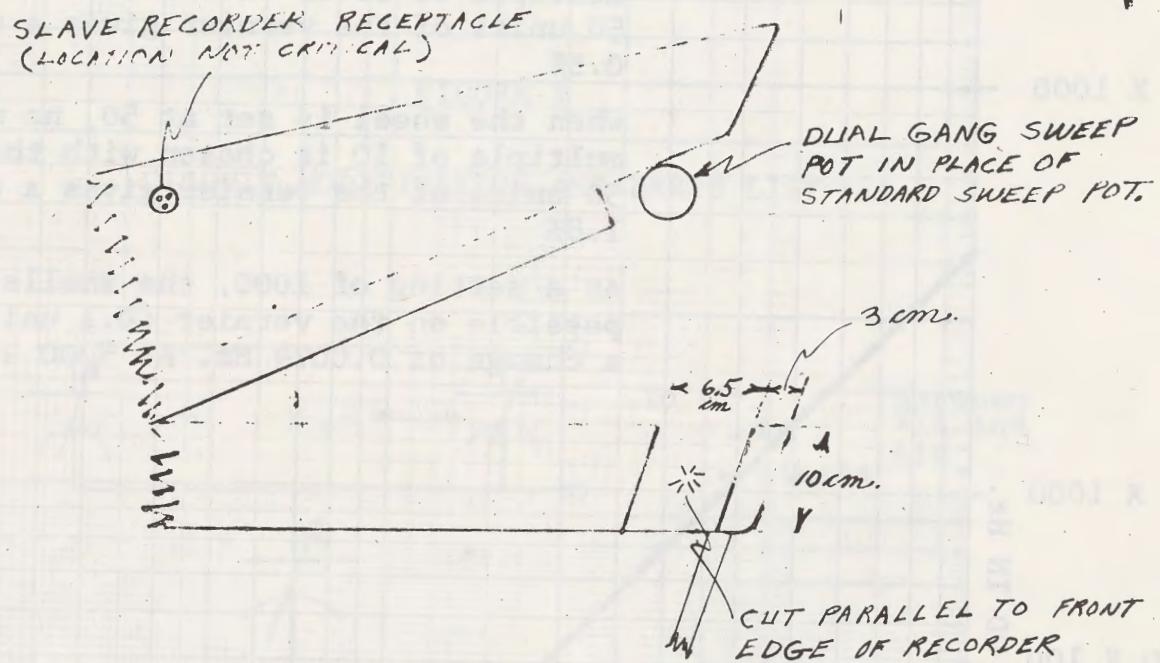
A 3-gang capacitor, 5-17 μfd per section is added. Remove all but 2 stator plates and 1 rotor plate from each section. Bud No. 1846, "Tiny Mite."

A Vernier Dial is mounted on the front of the H-P 200CD, connected to the rotor plates. Velvet Vernier Dial, Type N-2, National Company.

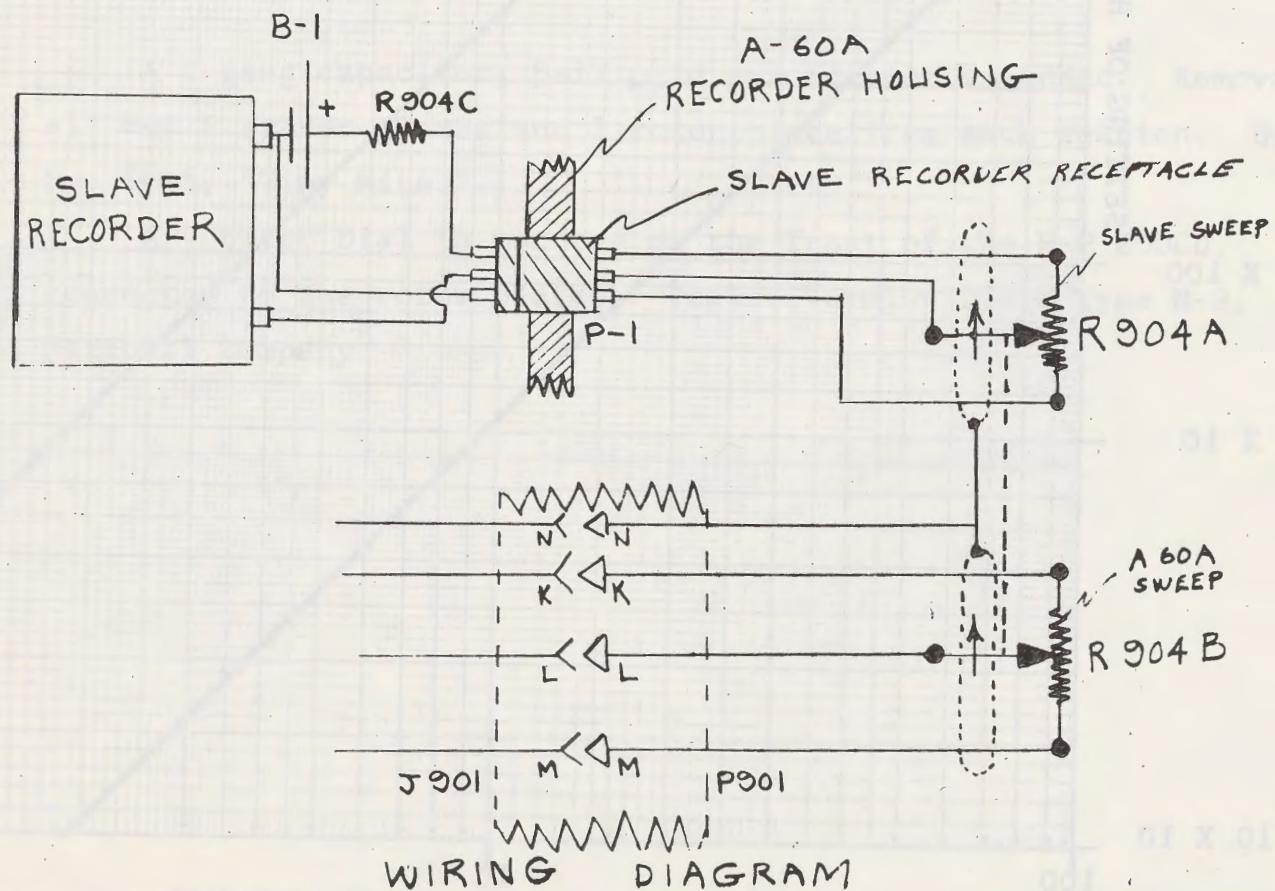
FIGURE 2



SKETCH # 1 - Side View of A-60A Console
And Wiring Diagram For Slave Recorder



LEFT SIDE VIEW



PARTS LIST AND MODIFICATION INSTRUCTIONS FOR
SLAVE RECORDER

Item	Description
R-904A 50 $\pm 3\%$	Half of dual gang potentiometer used for slave recorder horizontal sweep, Model 3600-723 from Duncan Electric Co.
R-904B 50 $\pm 3\%$	Half of dual gang pot. used for A-60A horizontal sweep
P-1 (3 term)	Plug for connecting R-904A to slave recorder
R-904C 4500	Current limiting resistor
Wire	No. 18 insulated wire (Approximately 5 ft.)
B-1	1.5V long life tel. cell

1. Cut out section from left side of recorder according to dimensions in sketch No.1.
2. Construct brace out of 1/8" steel or other suitable material with dimensions as per sketch No.2.
3. Drill holes in brace for appropriate bolts to bolt onto recorder housing.
4. Drill appropriate size hole in upper left hand side of movable part of recorder to receive slave recorder plug.
5. Remove R-904 (single gang sweep pot) from recorder.
6. Install dual gang pot in place of single gang.
7. Wire according to sketch No.3, use any suitable wire.

Institut für Elektrowerkstoffe

GEMEINNOETZIGES FORSCHUNGSIINSTITUT DER FRAUNHOFER-GESELLSCHAFT

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

INSTITUTSDIREKTOR:
 PROF. DR. R. MECKE

TELEFON 0761/45514

78 FREIBURG I. BR.
 ECKERSTRASSE 4

6. März 1967

Sehr geehrter Herr Professor Shapiro,

Bestimmung der Geschwindigkeitskonstanten der Aufspaltung
 eines Signals zum AB-Spektrum.

Hier unser Beitrag, um die drohende "Dead-Line" zu umgehen:

Zum Vergleich der Beweglichkeiten verschiedener flexibler Ringsysteme wird vielfach nur der Wert der freien Aktivierungsenthalpie ΔG^\ddagger herangezogen. Denn zur Bestimmung von ΔG^\ddagger wird nach der Eyring'schen Gleichung nur ein Wertepaar k und T , die Umklapphäufigkeit k für die Temperatur T , benötigt.

Ein solches Wertepaar (k_a für $T = T_a$) ist sehr einfach aus der Temperaturabhängigkeit der Protonenresonanz zu bestimmen, wenn die beiden Protonen (bzw. Protonengruppen), die ihre Plätze tauschen, nicht miteinander koppeln²⁾. Das PR-Spektrum einer derartigen Molekel ist ein Dublett-Spektrum, wenn die innermolekulare Beweglichkeit "eingefroren" ist. Der Wert k_a für die Aufspaltungstemperatur $T = T_a$ folgt näherungsweise aus der Beziehung von Gutowsky und Holm¹⁾:

$$k_a = \frac{\pi}{\sqrt{2}} \Delta v = 2,22 \Delta v$$

Δv = Chemische Verschiebung der beiden Signale.

Diese Gleichung ist gültig (vergl. unten die Kurve $J/\Delta v = 0$), wenn das Verhältnis $b_E/\Delta v$ klein ist (b_E = Eigenbreite der Signale, Näheres zur Nomenklatur s. Zit.2).

Um auch bei zwei koppelnden Protonen, deren PR-Signale beim "eingefrorenen" Molekül ein AB-Spektrum ergeben, eine rasche ΔG^\ddagger -Bestimmung (ohne Rechenmaschine) durchführen zu können, haben wir die k_a -Werte in dem uns interessierenden Wertebereich der Kopplungskonstanten J numerisch berechnet (s. Seite 2).

1) H.S.Gutowsky und C.H.Holm, J.Chem.Phys. 25, 1228 (1956)

2) H.G.Schmid, H.Friebolin, S.Kabuß und R.Mecke, Spectrochimica Acta, 22, 623 (66)

3) R.J.Kurland, M.B.Rubin und W.B.Wise, J.Chem.Phys. 40, 2426 (1964)

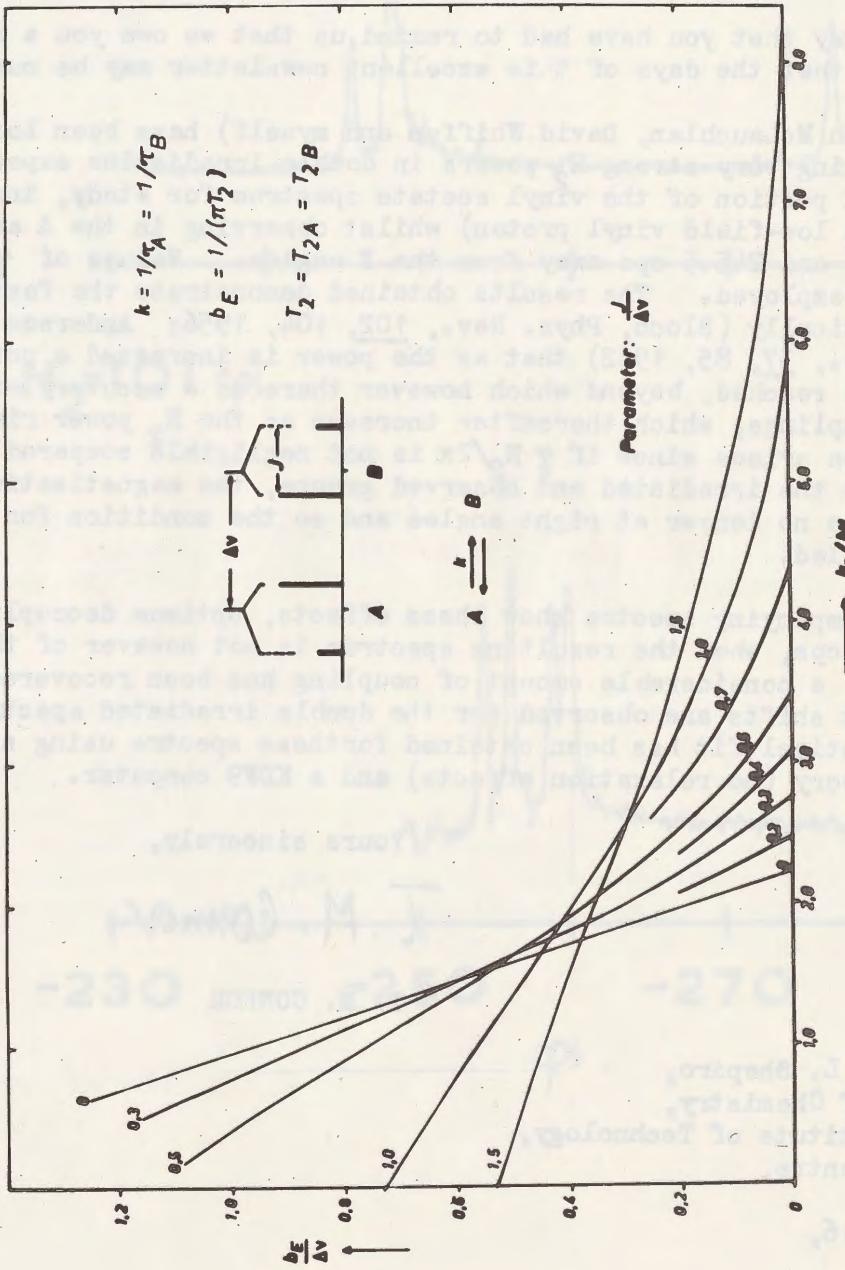
- 2 -

Dieses Nomogramm zeigt, dass der Wert $b_E/\Delta v$ einen großen Einfluß auf den Wert $k_a/\Delta v$ hat, wenn $J/\Delta v > 0,3$ ist. Für $b_E/\Delta v = 0$ gilt die von Kurland, Rubin und Wise³⁾ angegebene Gleichung:

$$k_a = \frac{\pi}{\sqrt{2}} \sqrt{\Delta v^2 + 6J^2}$$

Mit vorzüglicher Hochachtung

Helmut Feinend





Ministry of Technology

NATIONAL PHYSICAL LABORATORY

TEDDINGTON, Middlesex

Telex: 262344 Telegrams: Physics, Teddington, Telex

Telephone: Teddington Lock 3222, ext.

Please address any reply to

THE DIRECTOR

and quote:

Your reference:

DIVISION OF MOLECULAR SCIENCE

1st March 1967

Dear Barry,

Double Resonance using High Field Strengths

I am sorry that you have had to remind us that we owe you a contribution, and also to hear that the days of this excellent newsletter may be numbered.

We (Keith McLauchlan, David Whiffen and myself) have been looking at the effects of using very strong H_2 powers in double irradiation experiments. We chose the AMX portion of the vinyl acetate spectrum for study, irradiating in the X region (the low-field vinyl proton) whilst observing in the A and M regions which are 277.5 cps and 245.5 cps away from the X region. Values of $\gamma H_2/2\pi$ of up to 101 cps were employed. The results obtained demonstrate the fact which has been shown theoretically (Bloch, Phys. Rev., 102, 104, 1956; Anderson and Freeman, J. Chem. Phys., 37, 85, 1962) that as the power is increased a point of optimum decoupling is reached, beyond which however there is a recovery of part of the spin-spin couplings, which thereafter increase as the H_2 power rises. Qualitatively, this situation arises since if $\gamma H_2/2\pi$ is not negligible compared with the chemical shift between the irradiated and observed groups, the magnetisation vectors for these two groups are no longer at right angles and so the condition for optimum decoupling is not fulfilled.

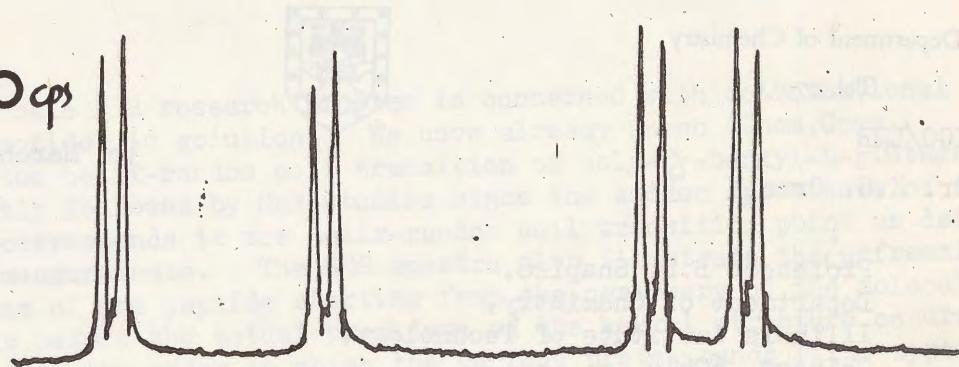
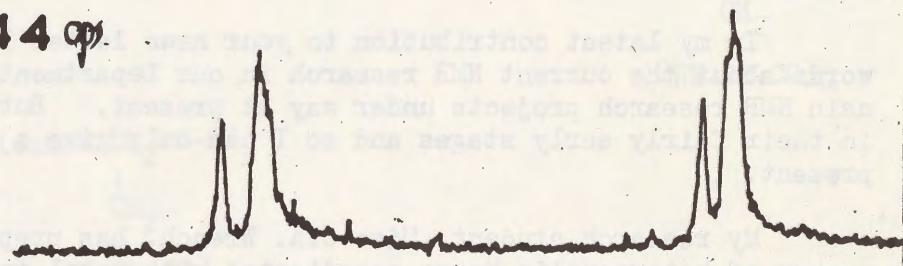
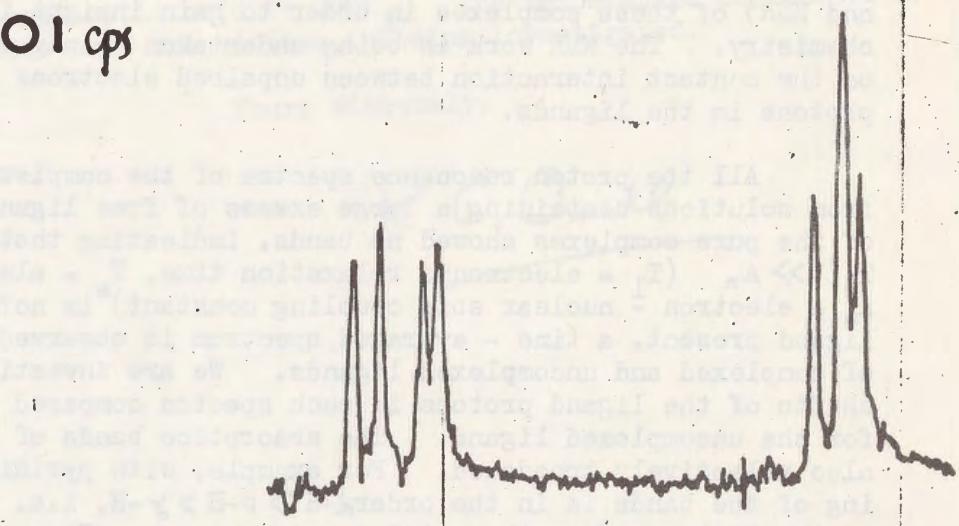
The accompanying spectra show these effects, optimum decoupling occurring when $\gamma H_2/2\pi = 44$ cps, when the resulting spectrum is not however of the AX type. At higher power, a considerable amount of coupling has been recovered. Also large Bloch-Siegert shifts are observed for the double irradiated spectra as expected. A good theoretical fit has been obtained for these spectra using simple double resonance theory (no relaxation effects) and a KDF9 computer.

Yours sincerely,

T. M. Connor

T. M. CONNOR

Professor B. L. Shapiro,
Department of Chemistry,
Illinois Institute of Technology,
Technology Centre,
Chicago 16,
Illinois 60616,
U.S.A.

$H_2=0 \text{ cps}$  $H_2=44 \text{ cps}$  $H_2=101 \text{ cps}$ 

-230 -250 -270 -290

cps.

UNIVERSITY OF EXETER

Department of Chemistry

Tel. 77911

KGO/LMB

Dr. K.G. Orrell

Stocker Road,
Exeter

3rd March, 1967

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

Dear Professor Shapiro,

In my latest contribution to your news letter I want to say a few words about the current NMR research in our Department. There are two main NMR research projects under way at present. Both projects are still in their fairly early stages and so I can only give a progress report at present.

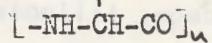
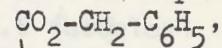
My research student, Miss C.A. Wrench, has prepared a considerable number of heterocyclic bases coordinated with metal trifluoroacetates. There appear to be two main series of complexes, $M(CF_3CO_2)_2L$ and $M(CF_3CO_2)_2L$, where $M = Co(II)$, $Ni(II)$ and $Cu(II)$, $L = pyridine$, mono and di-substituted pyridines, quinoline and isoquinoline. We are carrying out spectroscopic investigations (I.R., solution U.V., diffuse reflectance U.V. and NMR) of these complexes in order to gain insight into their stereochemistry. The NMR work is being undertaken mainly to obtain information on the contact interaction between unpaired electrons on the metal and the protons in the ligands.

All the proton resonance spectra of the complexes have been obtained from solutions containing a large excess of free ligand since the spectra of the pure complexes showed no bands, indicating that the condition $T_1 \gg A_n$ (T_1 = electronic relaxation time, T_e = electronic exchange time, A_n = electron - nuclear spin coupling constant) is not held. With excess ligand present, a time - averaged spectrum is observed due to rapid exchange of complexed and uncomplexed ligands. We are investigating the chemical shifts of the ligand protons in such spectra compared with the chemical shifts for the uncomplexed ligand. The absorption bands of the ligand protons are also selectively broadened. For example, with pyridine complexes the broadening of the bands is in the order $\alpha\text{-H} \gg \beta\text{-H} > \gamma\text{-H}$, i.e. the broadening decreases as the distance from the metal ion increases. These broadening effects have some very interesting but complicated temperature dependences which we hope to investigate in more detail very shortly.

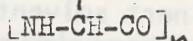
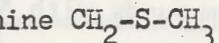
cont'd

The other main N.R research problem is concerned with conformational studies of polypeptides in solution. We have already shown (Chem.Comm. 1965, 518) that the helix-random coil transition of poly- γ -benzyl-L-glutamate can be conveniently followed by NMR studies since the sudden appearance of the -N-H absorption corresponds to the helix-random coil transition point as determined by O.R.D. measurements. The NMR spectra also illustrate the 'unfreezing' of the side-chains of the peptide starting from the periphery of the molecule and going inwards before the actual breakdown of the helical structure occurs. This is deduced from the order in which the various proton bands first appear and then progressively sharpen up.

This work has recently been extended by J.C. Haylock of this department to two other polypeptides, namely poly- β -benzyl-L-aspartate



and poly-L-methionine



In both cases the helix-random coil transition has been observed. For poly- β -benzyl-L-aspartate this occurred over a solution range of 5-8% dichloro-acetic acid in CDCl_3 , and for poly-L-methionine in the mixture range of 50-60% trifluoroacetic acid in CDCl_3 . The temperature dependence of these helix-random coil transitions is being investigated.

Yours sincerely,

K.G. O'Neill

The Standard Oil Company

(An Ohio Corporation)

Research Department

4440 Warrensville Center Road
Cleveland, Ohio 44128

E. C. HUGHES
VICE PRESIDENT

January 17, 1967

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

Dear Barry:

Interaction of Organic Nitrile
Compounds with Aromatic Solvents

We have obtained the NMR spectra of a number of organic nitrile compounds in aromatic solvents and noted an appreciable diamagnetic shift relative to the chemical shift in inert solvents. This phenomenon has been known for some time and has been documented by several investigators for a number of solute-aromatic solvent systems.¹⁻⁴

Tabulated below (Table I) are chemical shift data for several typical nitriles in inert and aromatic solvents. There are several interesting observations which may be made concerning this compilation. Klinck and Stothers suggest in their investigation of substituted benzaldehydes that the association site is governed by electron distribution in the solute. The same effect may be noted in Table II in which halo substitution on propionitrile results in a greater $\Delta\delta$ for both the α and β protons. The halo atom apparently enhances complex formation by increasing the net δ^+ on the nitrile carbon.

Table III illustrates the effect of increasing chain length in $\Delta\delta$ of the α methylene groups. Malononitrile experiences a very large $\Delta\delta$ for its methylene resonance. Dreiding models show that both nitrile groups in this molecule can associate to benzene, since the nitrile carbon in each case can align itself directly over the π cloud at either end of the ring. Increasing the number of methylene groups between nitriles results in a drastic reduction of $\Delta\delta$, obviously because on the time average, interaction is occurring between only one of the nitrile groups and the aromatic species.

The Standard Oil Company
(In The Corporation)

Professor B. L. Shapiro

-2-

January 17, 1967

Table I $\Delta\delta = (\delta_{\text{inert}} - \delta_{C_6D_6})$

Compound	Solvent	Resonance α to-CN (ppm)	$\Delta\delta$
Acetonitrile	CCl ₄	1.94	
"	C ₆ D ₆	1.18	0.76
Acrylonitrile	CCl ₄	~5.86	
"	C ₆ D ₆	~5.23	~0.63
1,2 Dicyanocyclobutene	CCl ₄	2.91	
"	C ₆ D ₆	2.06	0.85
trans-1,2 Dicyanocyclobutane	CDCl ₃	3.50	
"	C ₆ D ₆	2.83	0.67

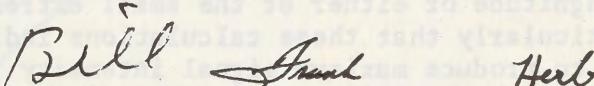
Table II Effect of Halides on $\Delta\delta$ of $\alpha\text{-CH}_2$ -

Compound	$\Delta\delta$ (ppm)
Propionitrile	0.55
3-Chloropropionitrile	0.83
3-Bromopropionitrile	0.93

Table III Effect of Chain Length on $\Delta\delta$ of $\alpha\text{-CH}_2$ in Dinitriles

Compound	Structure	$\Delta\delta$ (ppm)
Malononitrile	NC-CH ₂ -CN	1.92
Succinonitrile	NC-CH ₂ -CH ₂ -CN	0.91
Adiponitrile	NC-(CH ₂) ₄ -CN	0.62

Sincerely yours,



 W. M. Ritchey, F. J. Knoll, Herb Grossman

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WMR:FJK:HG:c1j

UNIVERSITY OF FLORIDA
GAINESVILLE, 32601

DEPARTMENT OF CHEMISTRY

AIR MAIL

March 9, 1967

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

Dear Barry:

The following is our contribution to the Newsletter, and I hope that it will reach you in time to maintain our subscription intact.
Subject: Line Shape Corrections for Modulation Amplitude.

A commonly encountered problem in wide line magnetic resonance is the effect of modulation amplitude on line shapes and intensities. Wahlquist [J. Chem. Phys., 35, 1708 (1961)] solved the problem in closed form for a Lorentzian absorption line. Smith [J. Appl. Phys., 35, 1217 (1964)] obtained a numerical solution for a Gaussian absorption line. Although the dispersion mode is encountered less often than the absorption mode, it is used in the method of Pake and Purcell [Phys. Rev., 74, 1184 (1948)] to provide a measure of the line shape. If one is to calculate accurately the value of T_2 from the line width, it is necessary to establish the Gaussian or Lorentzian character of the line.

Accordingly, we have been investigating the effect of modulation amplitude on the shape of dispersion mode lines. The Lorentzian solution has been obtained in closed form by a contour integration analogous to that used by Wahlquist. The results are shown in the accompanying figure. The modulation amplitude, h_m , is expressed as a fraction of H_1 , the true width at half height of the conjugate absorption line. The small diagram shows the significance of the line shape factor, R, which is the ratio of the magnitude of the large extreme of the derivative curve exactly on resonance to the magnitude of either of the small extremes above and below resonance. Note particularly that these calculations indicate the modulation amplitude required to produce maximum signal intensity in the absorption mode ($h_m/H_1 = 1$) reduces the value of R from that for a Lorentzian line, 8.0, to very nearly that for a Gaussian line, 3.5.

A paper is being prepared and preprints should soon be available for anyone interested in details.

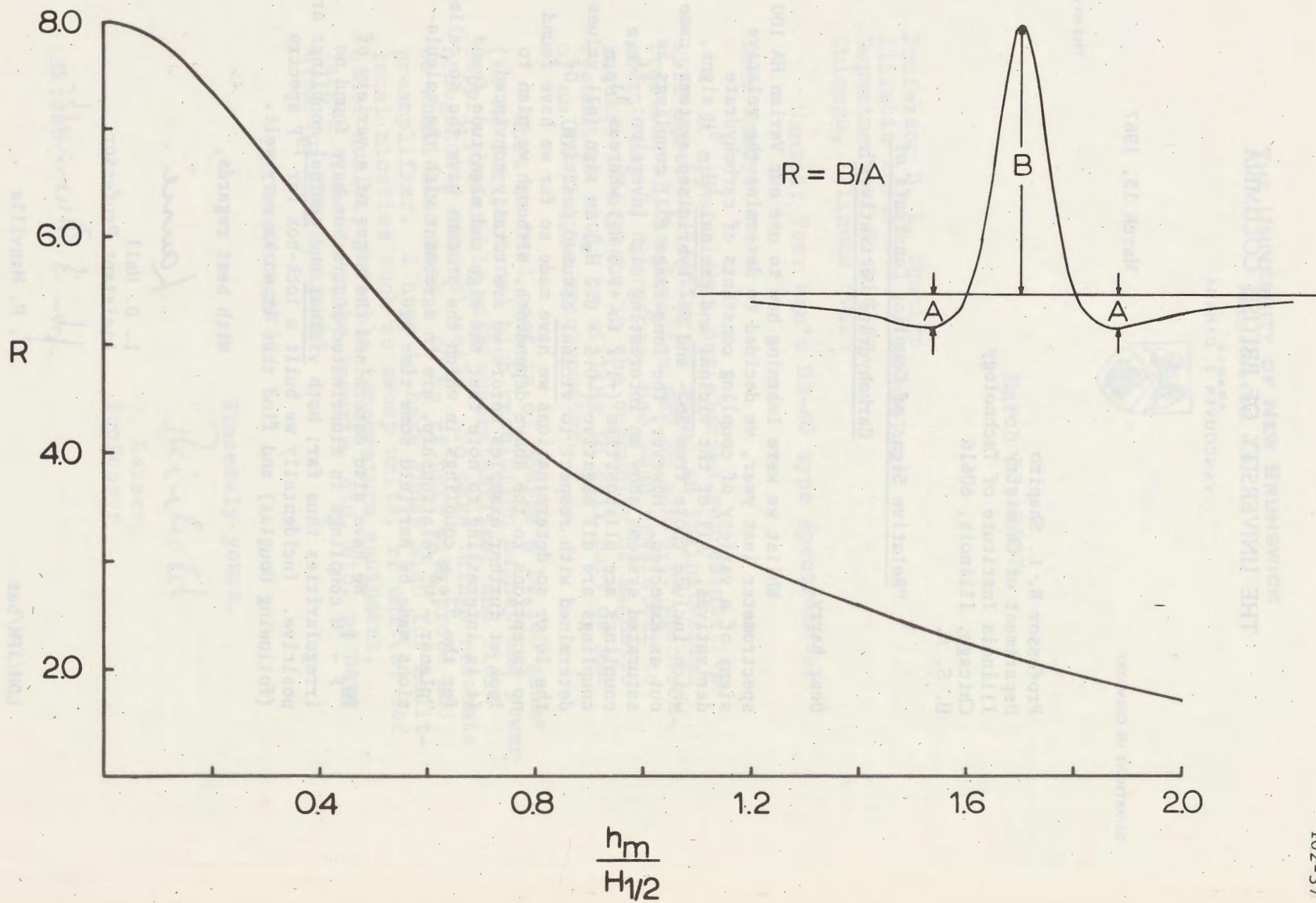
Cordially yours,

Wallace S. Brey Jr.
 Wallace S. Brey, Jr.

Thomas E. Evans
 Thomas E. Evans

Enclosure

WSB:EH



THE UNIVERSITY OF BRITISH COLUMBIA

VANCOUVER 8, CANADA

DEPARTMENT OF CHEMISTRY

March 13, 1967

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

"Relative Signs of Coupling Constants of
Carbohydrate Derivatives"

Dear Barry:

Whilst we were learning how to use our Varian HA 100 spectrometer last year, we decided to determine the relative signs of a variety of coupling constants of carbohydrate derivatives. All of the vicinal and geminal ^1H - ^1H signs, which include those from Sp^3 - and Sp^2 -hybridised systems, came out as expected. However, the long-range (^4J) couplings in saturated systems show an interesting sign inversion:- $^4\text{J}_{e,e}$ couplings are all positive (+1.2 to +1.6 Hz) whereas $^4\text{J}_{a,e}$ couplings are all negative (-0.5 ± 0.2 Hz) in sign (all values determined with respect to vicinal assumed positive). Of the 16 or so determinations we have made so far we have found no exceptions to the above dependence, although we plan to look at further examples before we are totally convinced. It is interesting to note that the sign and magnitude found for the $^4\text{J}_{e,e}$ couplings in which the protons have the so-called "planar - M" relationship, are in agreement with the calculations made by Barfield some time ago.

We have also determined the signs of a variety of ^{19}F - ^1H couplings in fluorinated sugars but have found no irregularities thus far; both vicinal and geminal couplings are positive. Incidentally we built a lock-box for ^{19}F spectra (following Douglas) and find that it works very well.

With best regards,

Laurie

L. D. Hall
 Assistant Professor

J. F. Manville

J. F. Manville

THE UNIVERSITY OF NEW BRUNSWICK
 FREDERICTON, N.B.
 CANADA



PHYSICS DEPARTMENT

March 13, 1967

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

Wanted: Poor Man's HR-60 type spectrometer

Dear Barry:

It appears that our elected representatives will agree to letting me spend an infinitesimal fraction of the Canadian GNP on a n.m.r. spectrometer. May I, therefore, use your newsletter to inquire if someone in the community of magnetic resonators would be willing to sell part or all of an HR-60 type spectrometer.

What we need most is of course a 14,100 gauss (60 Mc for protons) high resolution magnet and power supply, preferably with superstabilizer and field homogeneity control coils. To make things resonate we shall also need a 60 Mc rf unit with probe and preamplifier. I hope to have funds also for additional niceties such as sweep units, rf unit(s) for other nuclei, variable temperature equipment, larger diameter probe inserts, etc. etc. perhaps even for EPR equipment.

With best regards,

Sincerely yours,

Reinhold

R. Kaiser,
 Professor

RK:seb

PURDUE UNIVERSITY
 DEPARTMENT OF CHEMISTRY
 LAFAYETTE, INDIANA 47907

March 13, 1967

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

Dear Barry:

As I mentioned at Pittsburgh, it seems too bad that your reminders often hit one at a time when there seems nothing to do except to pick a rather green fruit. Since I'd hate to lose my IITNMR subscription, I'd like to contribute some comments on work by Dr. Harold Allen and myself, which we are preparing for publication.

Apparent non-constancy of bond-anisotropy values

We have used Krishnan's flip-angle method to measure the principal diamagnetic susceptibilities of γ - and δ -1,2,3,4,5,6-hexachlorocyclohexane, in the hope of determining, independently of n.m.r. spectra, the C-H, C-C and C-Cl bond anisotropies. If it is assumed that each bond has a unique anisotropy, independent of its environment, the data for each isomer can be used, alone, to evaluate the three anisotropies. The results do not agree well, as shown in the table, although the C-C and C-Cl anisotropies are at least in the same ball park as some values reported by J. Villepin in Compt. rend. 257, 2278 (1963).

Isomer	$\Delta\chi \times 10^6$		
	C-C bond	C-Cl bond	C-H bond
δ	3.93	-5.04	-0.85
γ	4.98	-7.04	5.16

Any conclusions drawn from these results must be taken cum grano salis, for the following reasons: (1) The overall crystal anisotropy, per molecule, is no larger than that of one C-Cl bond; i.e., there is a lot of cancellation when the individual bond anisotropies are added, which no doubt magnifies the errors in the calculation. (2) Under the circumstances, the errors in the available crystallographic structure determinations may be the main source of the observed inconsistencies. In particular, the X-ray work does not locate H atoms, so that some assumptions had to be used to get C-H bond direction cosines. However, it is possible that the inconsistencies reflect the breakdown of the assumption that the anisotropy of a given sort of bond has in fact a constant value. Until future work clarifies the situation, it would seem that this assumption should be used, if at all, only with the greatest caution.

With best regards.

Sincerely,

Muller
 Norbert Muller
 Associate Professor

MELLON INSTITUTE

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