

Mailed:
31 July 1963

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Monthly
Ecumenical
Letters from
Laboratories
Of
N-M-R

No. 58

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DEADLINE FOR NEXT ISSUE
26 August 1963

Coleção mensal de cartas particulares de laboratórios de NMR. As informações aqui contidas são somente para entretenimento do leitor e para manter o editor ocupado. Não são permitidas citações, exceto por permissão direta do autor da carta. As citações devem ser referidas como "Comunicação Particular".

Policies and Practical Considerations Concerning MELLONMR

1. Policy: MELLONMR is envisaged as a means for the rapid exchange of information between active workers in the field of nuclear magnetic resonance. As such, it will serve its purpose best if the participants impart whatever they feel will be of interest to their colleagues, and inquire concerning whatever matters interest them. Since the participant is clearly the best judge of what he considers interesting, our first statement of policy is "We print anything.". (This is usually followed by the mental reservation "that won't land us in jail".) No editorial functions are performed. MELLONMR is not, and will not become, a journal. We merely reproduce and disseminate exactly what is sent in.

As suitable subjects for contributions, we have suggested the following:

1. Reproductions of spectra or parts of spectra.
2. Measurements and calculations of same.
3. Theoretical considerations and interpretations.
4. Novel experimental methods and techniques.
5. Complete or partial mysteries.
6. Comments on material appearing in earlier letters.
7. Requests for help in locating odd or rare compounds.
8. Announcements of forthcoming meetings, colloquia, symposia, books, etc. Also "Preprints Available".
9. Notices of "Positions Available" or "Situations Wanted".
10. Wild theories (this category added at the request of Dr. Ray Freeman).
11. Anything else which you feel your co-workers in the field might find interesting - even mild polemics.

Foreign participants should not feel obligated to render their contributions in English.

2. Bibliography: The titles and references appearing in the "Bibliography" section are merely those which the NMR people here at Mellon Institute come across in their perusal of the literature. No claims for completeness are made and we once again invite all participants to send in references and titles which we have missed for inclusion in a subsequent issue. This invitation is extended particularly to foreign, and most especially foreign-language, journals, since this is where our NMR searching is most apt to be late at best and also incomplete at worst.

3. Subscriptions: We will continue to send out MELLONMR without charge, although we would be grateful for the cooperation of all participants as outlined in the section below on "Practical Considerations" (see also page 1 of issue No. 51).

Participation is the prime requisite for receiving MELLONMR; in order to receive the Newsletter, you must make at least occasional contributions to its contents. We feel that we have to be ruthless in this connection and the following schedule is in effect: Eight months after your last contribution, you will receive a "reminder letter". If no contribution is then forthcoming ten months after your last contribution, you will receive the "ultimatum letter", and then the next issue will be your last. If you are dropped from the mailing list, you can be reinstated by sending in a contribution, and you will receive back issues (as available) and forthcoming issues at the rate of 9 per contribution. Frequent contributions are encouraged, but no "advance credit" can be obtained for these.

A subject of considerable interest and concern to several present and potential MELLONMR participants - as well as to ourselves - is whether MELLONMR ought to contain material which either appears essentially simultaneously in the

(cont'd.)

formal literature or is definitely scheduled to appear very shortly (*i.e.*, within a few weeks) after it would appear in the Newsletter. Our attitude is that a MELLONMR contribution should not duplicate, summarize or abstract material which has been published or which will appear in the formal literature within a small number of weeks of the MELLONMR account. On the other hand, let it be emphasized firmly that if the appearance in a journal is several months away - as is frequently the case - a brief account (as an abstract with or without a "Preprint Available" notice, a separate informal account, a selection of material from the manuscript, or what have you) sent in to MELLONMR fulfills one of the very functions which we feel this Newsletter can provide. We trust that a participant will in each case apply the criterion of whether or not his contribution will communicate some subject matter to the MELLONMR audience before they could read it elsewhere. The abundance of subjects, the imagination and interest of our participants and the natural limits of size and costs make it both desirable and advisable that MELLONMR not take on an NMR-abstract-service aspect.

4. Brevity: Brevity is good - see the section on Practical Considerations.

5. Public Quotation: Public quotation is not good, and reference to MELLONMR by name in the scientific literature is never permissible. We remind you that in order to quote results or use material from MELLONMR it is necessary, in each individual case, to obtain the prior permission of the author in question and then to refer to the material quoted as a "Private Communication".

6. Practical Considerations:

(a) All contributions to MELLONMR should be sent to the attention of B. L. Shapiro or A. A. Bothner-By, and will always be included in the next issue if received before about the 25th of the month. (The actual deadline date appears on the cover of the previous issue.)

(b) Effective immediately, we now prefer not to receive 200 copies of contributions. We feel we can keep our costs down best by doing all the reproductions, etc., ourselves. Thus, the physical requirements for the contributions are:

(i) Contributions should be on the minimum number of 8-1/2 x 11" (21 x 27.5 cm) pages, printed on one side only, with at least 3/4" (2 cm) margins on all sides.

Since reproductions of various kinds do not themselves reproduce too well, contributors are urged to submit their originals to us and we will be happy to return these if requested.

(ii) Please provide short titles of all the topics of your contributions, as this will ensure accuracy (and lessen our work) in preparing the title-page index.

(iii) Please try to avoid sending in manuscripts, theses, books, etc., and asking us to be your consciences in selecting what should and shouldn't go into MELLONMR.

(c) Brevity is getting more and more golden as our mailing list increases. Single spacing is almost as good as brevity, and in general, you will help us keep our costs down to a reasonable level by sending in as much material as you wish, but by planning it to fill the minimum integral number of pages. This will help cut the costs of both reproduction and mailing.

7. Suggestions: We welcome them.

A. A. Bothner-By

B. L. Shapiro

17 July 1963



CONTINENTAL OIL COMPANY

P. O. DRAWER 1267

PONCA CITY, OKLAHOMA

Research and Development Department

June 25, 1963

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

HR-60 Miscellanea

Moseley 2S Time Base - We have also found (cf. Landis, MELLONMR 55, 5) the time base of the Moseley 2S X-Y recorder to be inadequate for even routine applications. Checking the performance of the Moseley B-2 time base led us to abandon the idea of a motor-driven pot. Instead we have fitted a synchronous motor directly to the X-axis drive system through a magnetic clutch. All this is mounted inside the cover. Now a single button disconnects the servo system, connects the magnetic clutch and synchronous motor and drops the pen. At the end of the sweep a micro switch reverses all this. We now have a 3"/min. sweep of linearity better than we can measure. We continue to have troubles with the rest of the recorder. Has anyone found a dependable flat-bed recorder?

Air Conditioning - Last year we fitted our lab with a Westinghouse SU-603A air conditioner controlled by a Yellow Springs Model 71 thermistor controller. (The system suggested by Jim Shoolery.) Our SU-603A came equipped with a 40 psi low-pressure cutoff switch and a 40-45 psi normal operating back-pressure. Since nothing is adjustable on the unit and there is no automatic reset, we had quite a bit of trouble with the unit shutting down until we shorted out the low-pressure cutoff switch. The unit now has operated about a year on a 1-minute cycle with no mechanical problems (and the original contactor).

Spin Decoupler - For spin-decoupling with the V-3521 integrator (cf. Johnson, MELLONMR 43, 12), our electronics group inserted an in-line appliance switch in the cable from the V-3521 to the

adapter on the V-4352 linear sweep unit. The $0.01\mu\text{fd}$ capacitor was placed inside the switch across the switch contacts. The ON position shorts the capacitor, giving normal operation. Total cost of materials--less than \$2.

V-4340 Temperature Controller - The variable temperature apparatus for the HR-60 is supplied without a controller. Our electronics group built a simple thermistor unit (proportional control) that seems to work well. Long-term stability is quite good. We haven't thought of any way to measure short-term variations. The circuit diagram is shown below. The VECO 51A2 thermistor replaces the thermocouple in the probe insert. We have used the unit from 35 to 200°C . It may be necessary to change thermistors for low-temperature work. The temperature is set by the $30\text{K}\Omega$ adjustable pot.

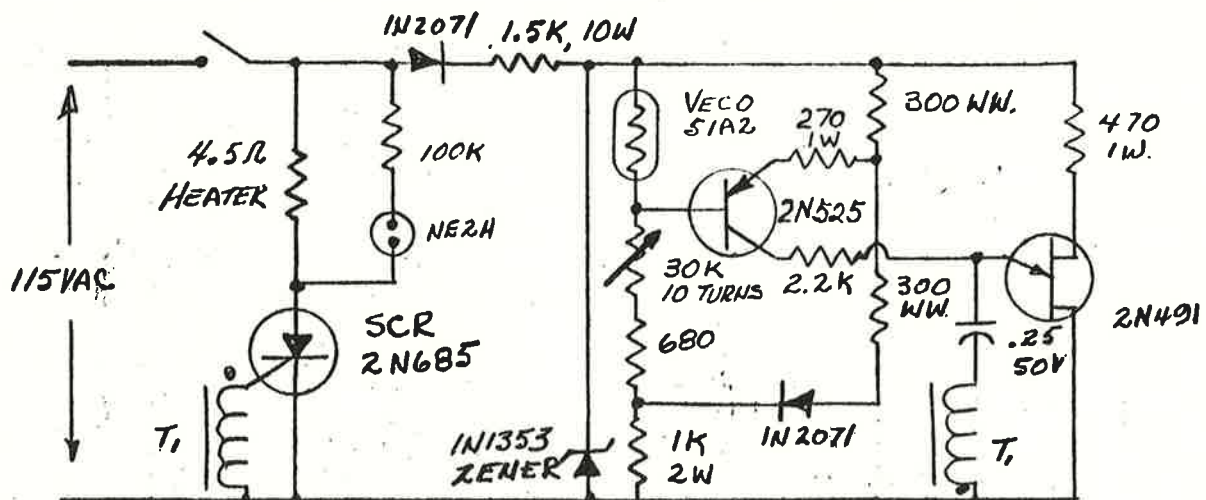
Pat W. Flanagan

Pat W. Flanagan

Charles E. Godsey

Charles E. Godsey

PWF-CC



$T_1 = \text{SPRAGUE 31Z382}$



EL PASO NATURAL GAS PRODUCTS COMPANY

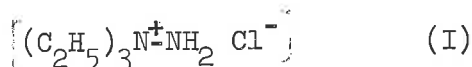
POST OFFICE BOX 1161, EL PASO 99, TEXAS

July 7, 1963

Dr. B. L. Shapiro
Mellon Institute,
4400 Fifth Avenue
Pittsburg 13, Pennsylvania

Dear Dr. Shapiro:

During the time I was with W. R. Grace Research Division, David Kramm and I carried out infrared and chemical studies on the structure of 1,1,1-triethylhydrazinium chloride.



We found that (I) has a strong intramolecular $-\text{NH}_2$ hydrogen bond ($-\text{NH}_2$ stretching vibrations the same in solid phase and in dilute CDCl_3) and the position of the $-\text{NH}_2$ stretching vibration is related to mass, charge, and geometry of the anion (M. Harvey and D. Kramm, TO BE PUBLISHED.)

Using our A-60, I have recently studied the NMR spectrum of (I) in CDCl_3 , CHCl_3 and acetonitrile. Figure 1 shows that the $-\text{NH}_2$ resonance of (I) in CDCl_3 moves upfield as concentration is increased. This led me to believe that (I) exists as a dimer in CHCl_3 (dielectric constant 4.8) and CDCl_3 . Figure 2 is a plot of molecular weight of (I) in CHCl_3 (Mechrolab Osmometer) and shows that (I) exists as a dimer in CHCl_3 .

Figure 3 shows that the $-\text{NH}_2$ resonance of (I) moves downfield with increasing concentration in acetonitrile (dielectric constant 35.7). This shows that (I) does not exist as a dimer in this polar solvent. Figure 4 shows the molecular weight of (I) as a function of concentration in acetonitrile and indicates that (I) is partially dissociated.

Sincerely yours,

MACK C. HARVEY,
Section Leader (Analytical)
Research Department

MCH:ps

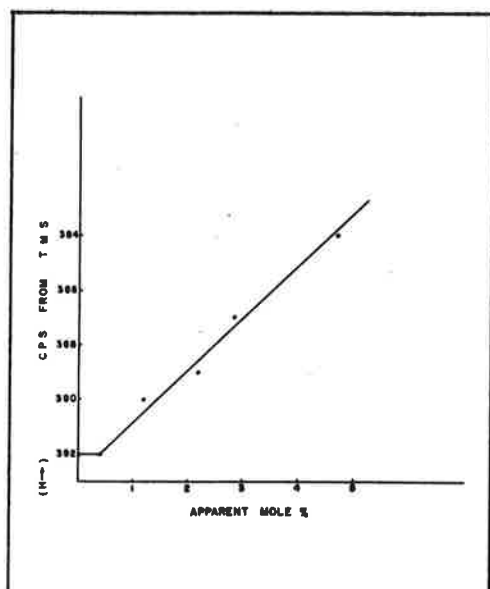


Figure 1, -NH_2 resonance of (I) as a function of concentration in CDCl_3 .

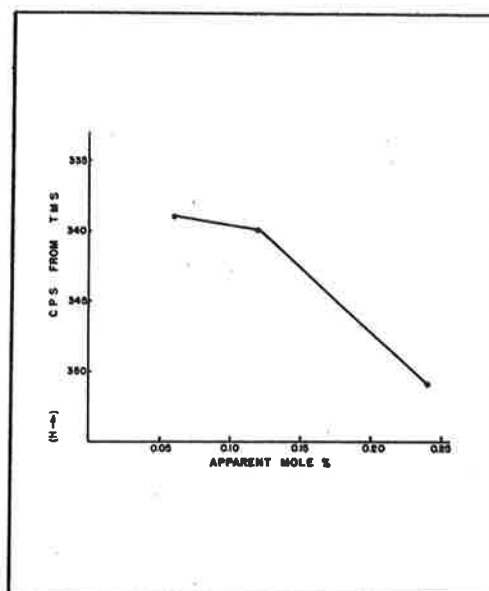


Figure 3, -NH_2 resonance of (I) as a function of concentration in acetonitrile.

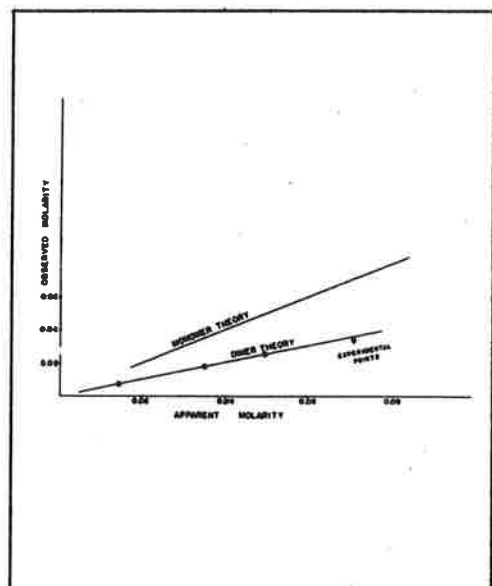


Figure 2, Molecular Weight of (I) as a function of concentration in CHCl_3 .

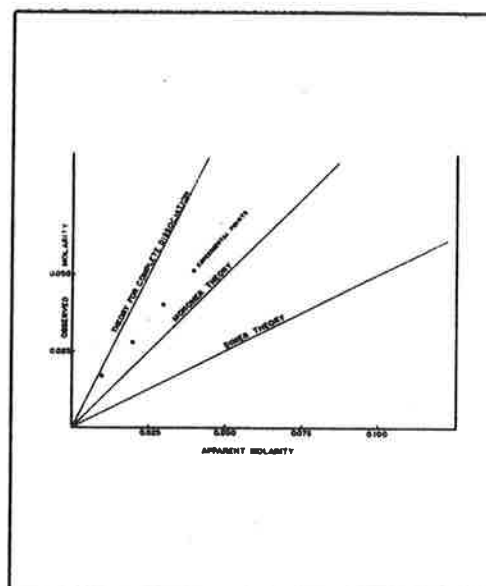
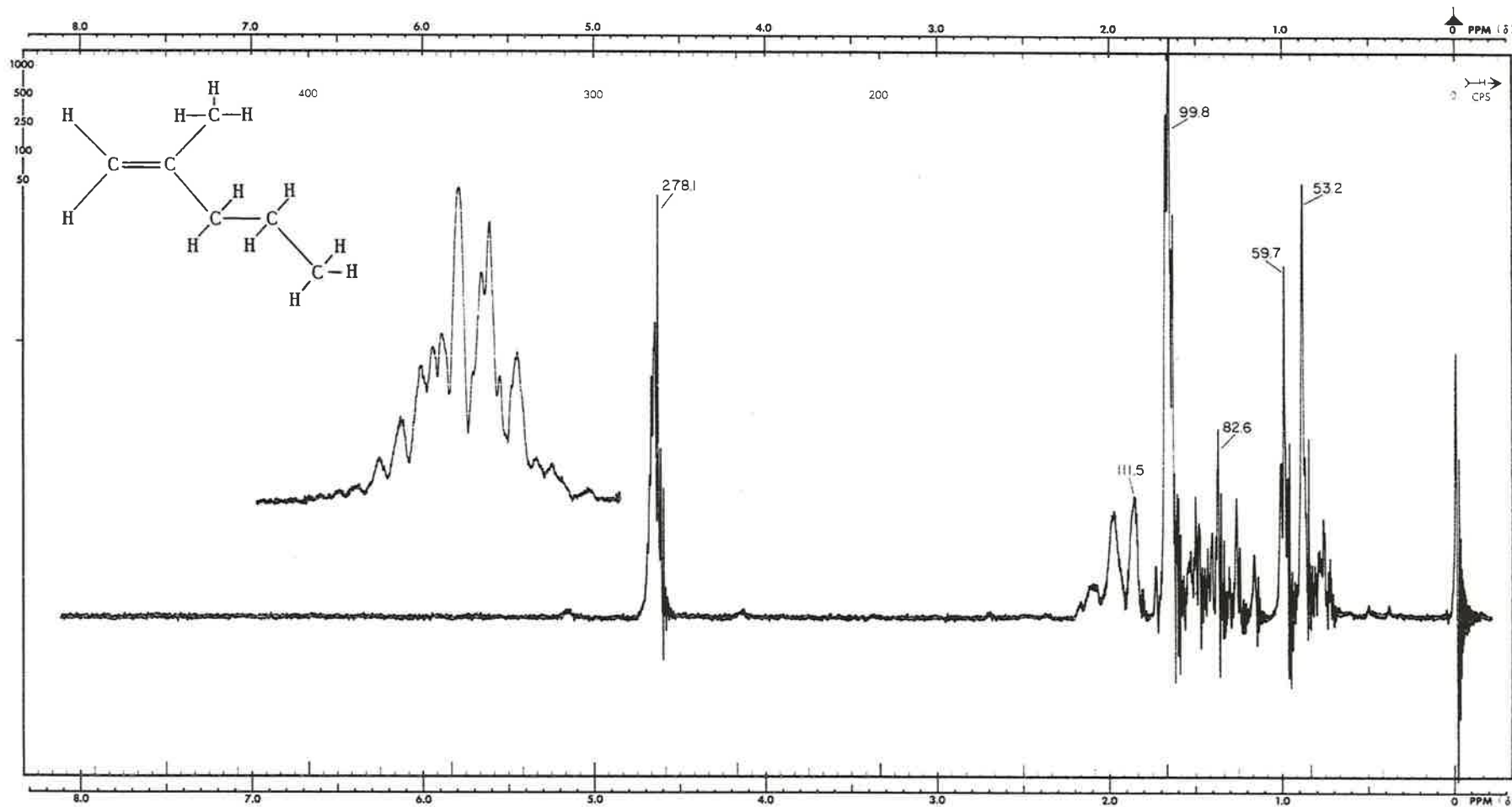
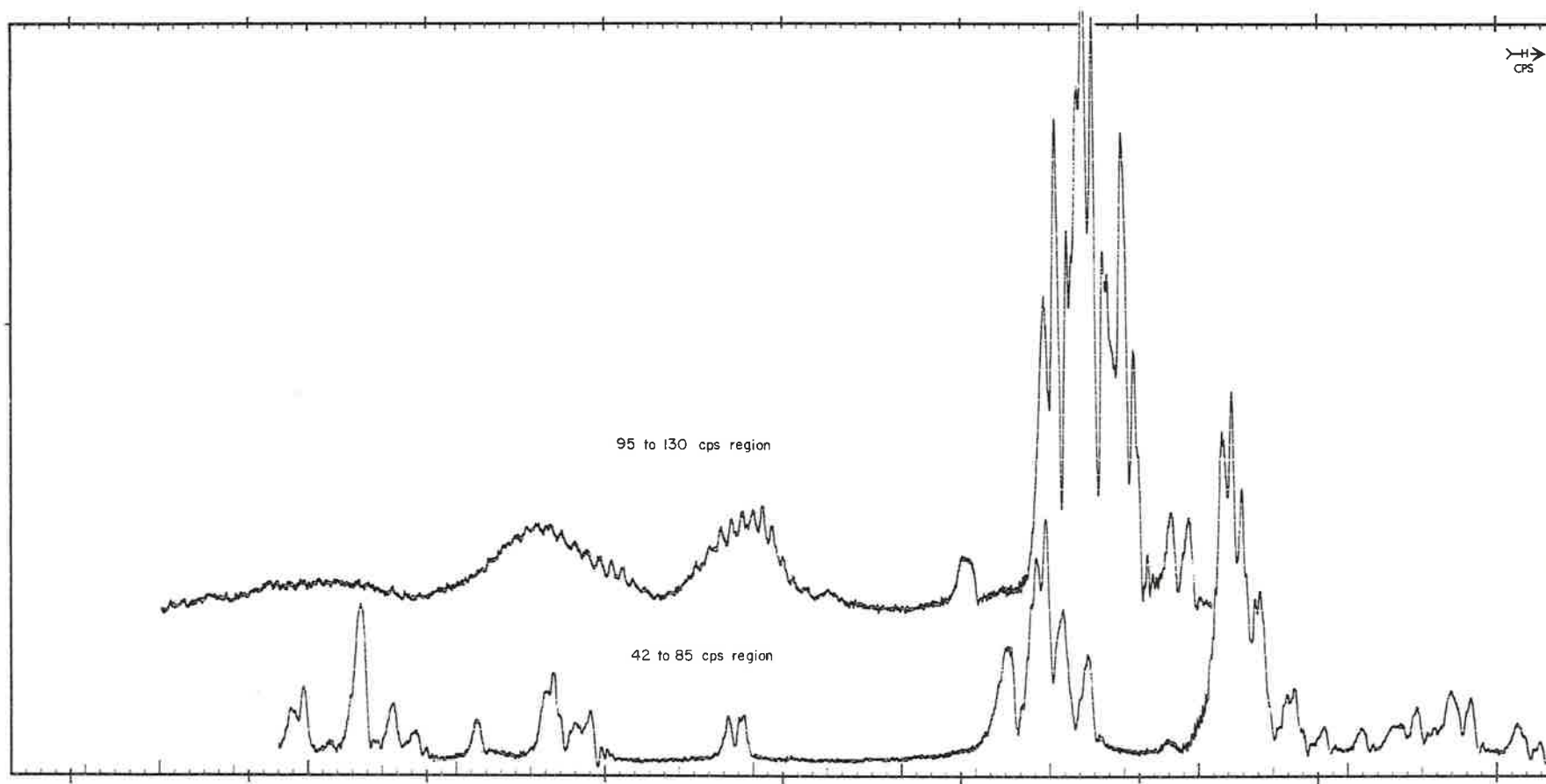


Figure 4, Molecular Weight of (I) as a function of concentration in acetonitrile.



COMPOUND		INSTRUMENT AND CONDITIONS	
Name: 2-Methyl-1-pentene	State: Liquid	Instrument: Varian Associates Spectrometer, Model A-60	RF Field Intensity (H ₁): 0.03 milligauss
C ₆ H ₁₂		Frequency: 60 Mcps	Scanning Rates: Standard (Spectrum A): 1.0 cps/second Other (Spectrum B):
Source: API Research Sample	Temperature: ~ 37°C	External Reference:	Internal Reference: Tetramethylsilane Concentration: ~ One percent by volume Resonance Position:
Purity: 99.92 ± 0.07 mole percent	Cell: Varian Precision Tube, 5 mm OD	Precision of Measurement: One cycle/second or better	
LABORATORY: Department of Chemistry, Agricultural and Mechanical College of Texas, College Station, Texas			

Figure 32



COMPOUND		INSTRUMENT AND CONDITIONS	
Name: 2-Methyl-1-pentene	State:	Instrument:	RF Field Intensity (H_1):
42 to 85 cps region		Frequency:	
		Scanning Rates: Standard (Spectrum A):	Internal Reference:
		Other (Spectrum B):	
Source:	Temperature:	External Reference:	Concentration:
Purity:	Cell:	Precision of Measurement:	Resonance Position:
LABORATORY: Department of Chemistry, Agricultural and Mechanical College of Texas, College Station, Texas			

Figure 33

AEROSPACE CORPORATION



Post Office Box 95085, Los Angeles 45, California, Osborne 9-4661

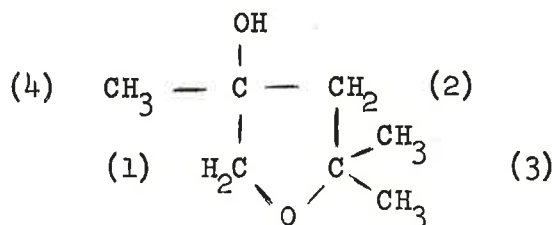
June 18, 1963

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

Dear Dr. Shapiro:

Our NMR studies at Aerospace Corporation have finally reached a point where it is producing some results. We are submitting the following data in the hope that it will be of interest to others doing structure studies. This is our first contribution, and we would like to be added to the mailing list to receive the MELION-M-R letters. We hope to be able to make many more contributions in the future.

Dr. D. J. Coyle of our laboratory obtained a photolysis product which we think has some interesting structure features. Its NMR spectrum taken at 56.4 m.c. is shown in Figure 1. A study of the compound by various methods indicates that it has the following structure:



The line at position E in the spectrum is due to the hydroxyl proton. The quartets at C and D are due to the CH_2 protons at locations (1) and (2) in the molecule respectively. The line at position B is due to the pair of essentially equivalent methyl groups at location (3) and the line at position A is due to the single methyl group at location (4). The line at position I is an impurity.

Although the ring in this molecule is not rigid, it holds the methylene groups in relatively fixed orientation. It holds the protons on one side of the ring in a somewhat different environment than those on the other side because of the presence of the OH group.

Hence, the methylene protons give rise to a four line AB type spectrum. The ring oxygen chemically shifts one group of lines down field relative to the other as indicated above. Figure 2 shows these four line groups at higher gain. The protons at position (1) gave $\delta = 7.2$ cy/sec. and $J = 8.3$ cy/sec; at position (2) $\delta = 5.8$ cy/sec. and $J = 5.5$ cy/sec.

Measurements of chemical shifts were made on the pure compound relative to tetramethylsilane. The values obtained are give below.

<u>Group</u>	<u>Chemical Shift</u> (pp m)
CH ₃ (4)	1.87
CH ₃ (3)	1.98
CH ₂ (2)	2.48
CH ₂ (1)	4.21
OH	4.88

(The values given for the CH₂ groups give the location of the center of the corresponding group of lines.)

Sincerely yours,

A. Greenville Whittaker

A. Greenville Whittaker
Staff Scientist
Department of Chemistry
Materials Sciences Laboratory

AGW/ea

Attachment: Figure 1



SHELL DEVELOPMENT COMPANY

A DIVISION OF SHELL OIL COMPANY
EMERYVILLE, CALIFORNIA

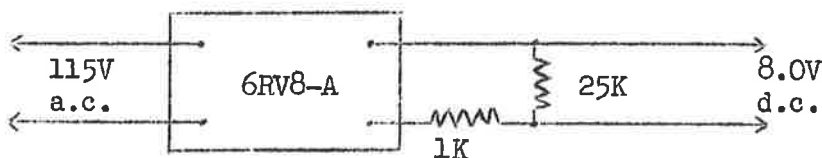
July 15, 1963

Dr. Barry Shapiro
4400 Fifth Avenue
Pittsburgh 13, Pennsylvania

Dear Barry,

Your reminder note arrived as anticipated. I hope the following item will be useful to NMR spectroscopists.

Ron Raymond of these laboratories has found that the mercury reference batteries in the Varian V-2100 magnet power supply can be replaced by an International Rectifier Zener voltage reference pack, Type 6RV8-A. This uses 115V power which is conveniently available from the pilot light socket. The output voltage is 8.4, which can be reduced by a simple potentiometer if desired. Mr. Raymond uses the following simple circuit:



The reference pack is of such a size that it and the voltage divider can be installed in the present battery box.

This source of reference voltage has operated successfully in the power supplies for our Broadline and our ESR spectrometers for several months now. We have not yet tried it in the power supply for our High Resolution spectrometer, but no trouble is anticipated.

Sincerely yours,

Charlie

C. A. Reilly

CAR: jel

THE SQUIBB INSTITUTE

FOR MEDICAL RESEARCH

NEW BRUNSWICK, N.J.

July 16, 1963

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

Dear Doctor Shapiro:

The last issue of MelloNMR (#56) arrived while I was trying to keep my last A-60 pen operating with water soluble red ink so that your solution to the pen problem appeared to be timely. However, after a brief discussion with the machine shop staff, I found it would take approximately four weeks to gather the materials and to do the machining. Stimulated by your success, it prompted me to seek an expedient solution to the A-60 pen problem.

The A-60 pen flows very well at the baseline (most of the time) but when asked to trace a full-scale deflection signal with a half bandwidth of 2.5 cps at a chart speed of 2 cps/sec., the pen balks and results in a curve unsuitable for reproduction. The remedy was simply to make the pen orifice larger which would allow drawing inks to flow. The procedure for modifying the pen is described below.

Fill the ink well of the pen with water. Place a 0.004" (38 gauge) wire¹ through the back of the pen so that the tip of the wire can protrude through the pen orifice. With the end of the wire even with pen orifice, carefully abrade the tip with very fine waterproof emery cloth² until the taper of the pen tip has been removed. To insure that intractable particles do not enter the orifice, the wire is pushed out and wiped a number of times during the sanding operation and the sanding is continued only while water flows from the pen. Finally, ream the opening with a glass scribe to the desired size.

A number of brands and types of inks were tested. Higgins non waterproof black drawing ink (No. 4425) was found to give very dark lines which were relatively skip-free. The ink could be washed out of the well with water and clogs in the tip only when the pen is not used for several hours. The pen could be started again by either moistening the tip or clearing with a wire.

I purchase specially cut 11" x 26" Ozalid 208S paper for the reproduction of the spectra.

Yours truly,

Allen I. Cohen

Allen I. Cohen

¹ "Gilbrator Music Wire", Masback Inc., Madison Street, N.Y., N. Y.

² Carborundum 600A silica carbide L Red-I-Cut type 3 was used.

MONASH UNIVERSITY

DEPARTMENT OF CHEMISTRY

Telephone 544 0481

Head of Department
Professor R. D. Brown

P.O. Box 92
Clayton, Victoria,

19th June, 1963.

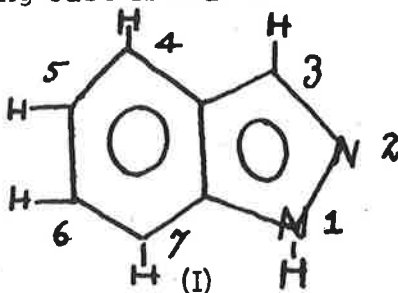
Dr. B. L. Shapiro,
Mellon Institute,
4400 Fifth Avenue,
PITTSBURGH, 13
P. A.

U. S. A.

Dear Dr. Shapiro,

Since our last communication we have had our Varian DP 60 spectrometer modified to become a HR-100 by fitting special tapered pole-caps. We have, of course, had to make other arrangements about our wide-line work. The unit is working quite well now at 23,490 gauss, although we did have some preliminary troubles with the 100 Mc/s probe. We have been looking at some proton spectra of heterocyclic systems and have been analysing the resulting four and five spin systems using eigenspectrum and Reilly-Swalen type (NMRIIT and NMREN2) iterative programmes written for our Ferranti Sirius computer. (4000 words).

One interesting case is that of indazole (I)



which has a fairly complicated spectrum at 56.4 Mc/s, but at 100 Mc/s it is almost a first order case. (Figures 1 and 2; both spectra refer to a 5 mole percent solution of indazole in acetone). The 3-proton showed evidence of cross-ring coupling which we assigned to the 7-proton in view of the well-known 4-8 coupling in quinoline and the recently published 3-7 coupling in benzofuran¹ (we have also independently observed this effect in benzofuran).

The individual multiplets in this rather favourable case can be assigned on the basis of the ortho coupling constants as 4- or 7- and 5- or 6-. To go further than this we have to resort to making methyl- or bromo-substituted indazoles or make use of proton-proton spin decoupling. Even this latter technique will not uncover the complete story and it is necessary to assign one multiplet, such as the 7-multiplet, on the basis of greater complexity due to cross-ring coupling or by means of the 7-methyl indazole spectrum. We used Roy Johnson's spin decoupling technique based on the Varian integrator to decouple the 3 and 7, the 4 and 5 and the 7 and 5 protons and found that it was fairly straightforward to carry out.

The first order parameters obtained from this assignment were then used to construct an energy level diagram for the four-spin (ABCD) and five-spin (ABCDE) cases. In the ABCD case the cross-ring coupling is treated as a small first order perturbation. The final values of the parameters for these two cases were obtained by an iterative scheme of the Reilly and Swalen² (NMRIT) type.

Spectral Parameters for Indazole at 100 Mc/s.

ABCD case	ABCDE case
$\tau_3 = 1.93_6$	1.93 ₆
$\tau_4 = 2.23_1$	2.23 ₁
$\tau_7 = 2.88_4$	2.88 ₄
$\tau_6 = 2.66_1$	2.66 ₁
$\tau_5 = 2.42_7$	2.42 ₇
$J_{45} = 8.15 \text{ cps}$	8.15 cps
$J_{56} = 6.89 \text{ "}$	6.89 "
$J_{67} = 8.39 \text{ "}$	8.36 "
$J_{46} = 0.95 \text{ "}$	0.95 "
$J_{57} = 0.82 \text{ "}$	0.82 "
$J_{47} = 1.03 \text{ "}$	1.04 "
$J_{37} = -$	0.99 "

We found that both sets of calculations gave virtually the same sets of spectral parameters. However, the final values of the coupling constants obtained by iterative solution were significantly different from

those deduced from the "nearly first order" spectrum.

The analysis for indazole was found to be relatively straightforward but we are currently investigating benzofuran, indole, quinoline and the nitroquinolines where the assignments are not so readily obtained. These results will be submitted to the Australian Journal of Chemistry.

Michael L. Heffernan

Michael L. Heffernan

Peter J. Black

Peter J. Black

1. Elvidge and Foster; J.C.S., 590; 1963
2. Swalen and Reilly; J. Chem. Phys.; 37; 21; 1962.

Fig. 1.

Experimental and calculated spectrum for 5 mole% indazole in acetone at 56.4 Mc/s

Fig. 2

Experimental and calculated spectrum for 5 mole% indazole in acetone at 100 Mc/s.

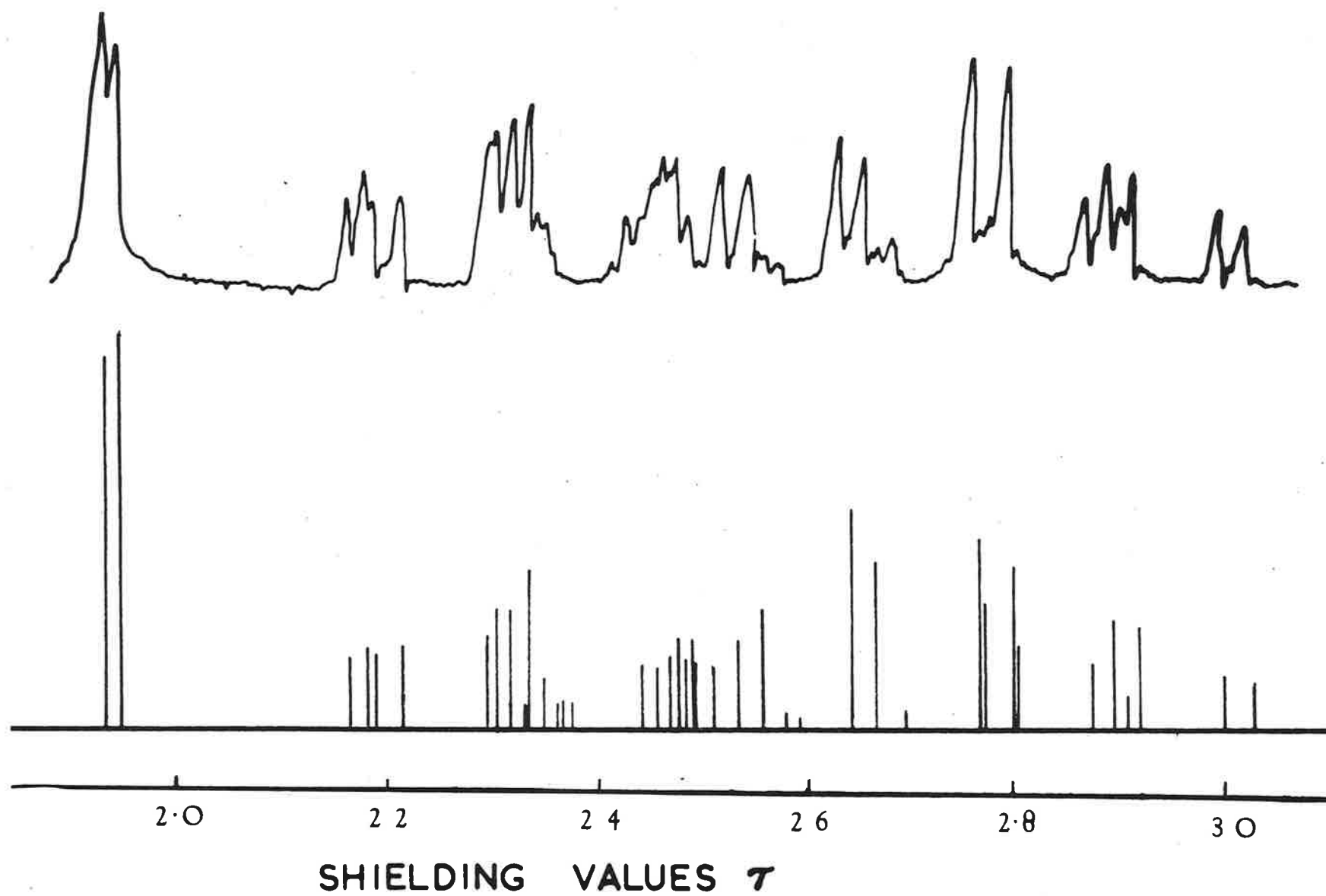
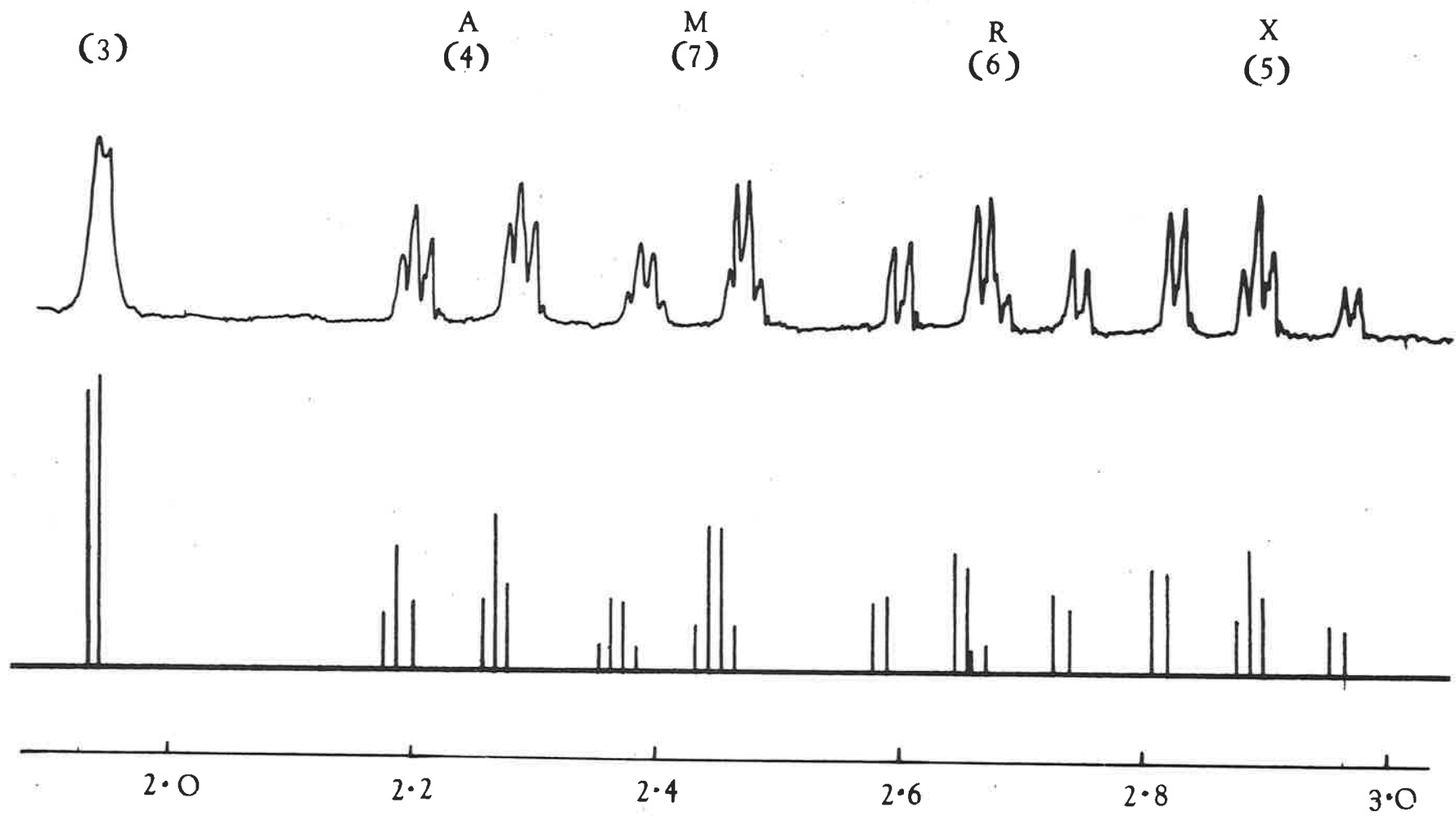


Figure 1



SHIELDING VALUES τ

Figure 2

THE UNIVERSITY OF TEXAS
DEPARTMENT OF CHEMISTRY
AUSTIN 12

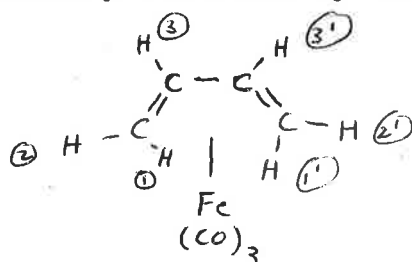
June 25, 1963

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

Dear Dr. Shapiro,

We have recently been doing some work with Dr. John Mahler and Dr. Rollie Pettit using spin decoupling which may be of interest to MELLONMR readers.

One series of compounds of interest have been iron carbonyl complexes of olefins. A number of peaks have been definitely established by synthetic and spin decoupling procedures. We have also used your Frequent IV program rewritten for our CDC 1604 computer. One interesting example involves a long-range coupling which was neatly verified by single and double irradiation.



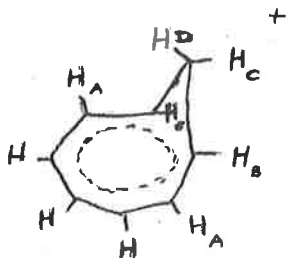
$$J_{12} = 2.4 \text{ cps}$$

$$J_{13} = 8.0$$

$$J_{23} = 5.4$$

$$J_{23'} = 2.2$$

Another interesting species was uncovered on looking at the proton spectrum of cyclooctatetraene in conc. H_2SO_4 . Peaks were found at 1.4, 3.4, 4.8, and 10.6 τ . The areas of the peaks indicated the assignments shown in the figure. The suspected coupling constants were verified by spin decoupling and deuterium substitution of the terminal protons the two techniques agreeing nicely.



	δ
A	1.4 τ
B	3.4
C	4.8
D	10.6

$$J_{BC} = 7.6 \text{ cps}$$

$$J_{BD} = 9.9$$

$$J_{CD} = 6.7$$

Sincerely,

Jeff C. Davis, Jr.
Jeff C. Davis, Jr.

EMORY UNIVERSITY

ATLANTA 22, GEORGIA

June 27, 1963

DEPARTMENT OF CHEMISTRY

Dr. Barry Shapiro
Mellon Institute
Pittsburgh, Pennsylvania

Dear Barry:

We have recently been investigating the effects of solvent and concentration on the NMR spectrum of the AB-system alpha-chloroacrylonitrile, $\text{CH}_2 = \text{C}(\text{Cl})\text{CN}$, which were first observed here by G. S. Reddy. The pattern consists, as expected, of two doublets, but, surprisingly enough, the two outer spacings turn out to be dependent on both the above-mentioned factors. This implies that the H-H coupling is not a constant for this molecule.

The spectrum has been observed in over a dozen non-aromatic solvents, and J^0 has been found to vary from 1.96 cps in TMS to 3.24 cps in dimethylsulfoxide. (J^0 is the coupling extrapolated to infinite dilution.) In general J^0 increases with the dielectric constant, ϵ , of the solvent, but appears to level off above a value of about 20 for ϵ .

The effect of concentration has been studied over a wide range in five solvents. In all these cases J apparently converges to the value of the coupling for the pure solute, 2.80 cps, as the concentration of solute approaches 100%. The total variation over the entire concentration range is, thus, 0.80 cps for TMS and 0.44 cps for DMSO.

We have submitted a preliminary report of these findings for publication and are now investigating the effect of aromatic solvents, mixed solvents, and other factors that may be related to the unusual behavior of this molecule.

Sincerely,



J. H. Goldstein
Professor of Chemistry

JHG:lt

MELLON INSTITUTE

4400 FIFTH AVENUE

PITTSBURGH 13, PA.

26 July 1963

Proton Magnetic Resonance Studies of Formaldoxime and Its Methyl Ether

We wish to make a preliminary report on three unusual features of the proton magnetic resonance spectra of formaldoxime and its methyl ether. Table I contains the relevant data, and the features in question are:

1. The unusually large magnitudes of $J_{H-H(gem)}$ for both compounds, the observed ranges being 7.63 to 9.95 cps for formaldoxime and 6.96 to 9.22 cps for the methyl ether. Usually sp^2 -type CH_2 groups show gem H-H couplings of $0 \pm$ ca. 3 cps (1) although values of between 7 and 8 cps have been reported for vinyl lithium (2) (3) and vinyl Grignard reagents (3) (4). The $J_{H-H(gem)}$'s reported here for the two $CH_2=N-OR$ compounds ($R = H$ or D , CH_3) form part of a consistent pattern of similar couplings observed in a variety of $CH_2=N-$ compounds (5). The sign of these couplings has not as yet been determined.

2. The most unusual medium dependence of J . In the vast majority of cases, H-H couplings are virtually independent of solvent, concentration, or temperature, unless these changes in the medium cause changes in (a) the abundances of two or more rapidly equilibrating isomers whose time-averaged J is being observed (6)(7), or (b) the bond order of a bond connecting the coupled nuclei (8). In the present cases, the geminal nature of the protons would seem to preclude sufficiently substantial medium-induced changes of geometry to account for the large changes observed in $J_{H-H(gem)}$. In fact, the only other cases of which we are aware are that reported by Goldstein (9) in the letter immediately preceding this one and a somewhat smaller variation observed in t-octylazomethine (5).

The source of this solvent-dependence of J is not as yet clear, but an examination of the data in Table I reveals an apparent correlation of J with the dielectric constant of the solvent in the case of formaldoxime methyl ether. A similar correlation does not obtain for the oxime itself, and it would appear that other influences, perhaps including ionization, self-association, and/or hydrogen-bonding, are being felt. Although a fuller discussion of this variation in J must be deferred for the present, it is our present feeling that the small variations in chemical shifts of both the $CH_2=N-$ and the $-OCH_3$ resonances argue against an explanation based upon solvent-dependent population changes arising from rotation about the N-O bond.

3. The unusually large line-widths of the resonances of both the CH_2 protons, particularly that high-field pair of lines in the AB pattern which arise from H_t , the proton trans to the methoxy group. $H^1(N^{14})$ double resonance studies* have shown that the line broadening has its origin in incomplete quadrupole washing out of J_{H_C-C-N} and J_{H_t-C-N} (The latter coupling is evidently considerably larger than the former.). As might be anticipated, these line-widths (in both the cases of the oxime and its ether) show no simple correlation with either the solvent dielectric constant (ϵ) or viscosity (η). Both of these will affect the observed line-widths, since J is a function of ϵ and changes in η will alter the N^{14} quadrupole relaxation time (cf. (8)).

* It is a pleasure to acknowledge our indebtedness for the performance of this important experiment to Mr. Robert C. Hopkins of the Department of Chemistry, Harvard University.

MELLON INSTITUTE

-2-

All the spectral data of Table I were obtained at 60 mc. on a Varian Associates Model A-60 NMR Spectrometer (sample temperature 37°C). The instability of both compounds precludes studies of the neat materials or concentrated solutions, and dilute (5% or less) solutions were used. In a few cases where moderately concentrated solutions could be obtained, no substantial dilution changes were noticed in chemical shifts, coupling constants or line-widths.

Work in this area is continuing; a more complete report will be published when the scope and nature of the abovementioned phenomena have been investigated more fully.

We acknowledge with thanks some helpful discussions with Drs. J. D. Baldeschwieler, A. A. Bothner-By, P. C. Lauterbur, and R. E. Richards.

B. L. Shapiro
S. J. Ebersole
R. M. Kopchik

References and Footnotes

(1) See for example:

- (a) C. N. Banwell, A. D. Cohen, N. Sheppard, and J. J. Turner, *Proc. Chem. Soc.*, 266 (1959).
- (b) C. N. Banwell and N. Sheppard, *Mol. Phys.*, 3, 351 (1960).
- (c) C. N. Banwell, N. Sheppard and J. J. Turner, *Spectrochim. Acta*, 16, 794 (1960).
- (d) E. B. Whipple, J. H. Goldstein and L. Mandell, *J. Am. Chem. Soc.*, 82, 3010 (1960).
- (e) A. A. Bothner-By and C. Naar-Colin, *J. Am. Chem. Soc.*, 83, 231 (1961).
- (f) G. S. Reddy, J. H. Goldstein and L. Mandell, *J. Am. Chem. Soc.*, 83, 1300 (1961).
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- (i) R. T. Hobgood, Jr., G. S. Reddy and J. H. Goldstein, *J. Phys. Chem.*, 67, 110 (1963).
- (2) C. S. Johnson, Jr., M. A. Weiner, J. S. Waugh and D. Seyferth, *J. Am. Chem. Soc.*, 83, 1306 (1961).
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- (4) R. T. Hobgood, Jr., and J. H. Goldstein, *Spectrochim. Acta*, 18, 1280 (1962).
- (5) B. L. Shapiro, G. J. Karabatsos, and S. L. Manatt, To be published.
- (6) e.g. (a) E. B. Whipple, *J. Chem. Phys.*, 35, 1039 (1961).
- (b) A. A. Bothner-By and C. Naar-Colin, *J. Am. Chem. Soc.*, 84, 743 (1962).
- (7) e.g. (a) R. J. Abraham and J. A. Pople, *Mol. Phys.*, 3, 609 (1960).
- (b) R. J. Abraham and H. J. Bernstein, *Can. J. Chem.*, 39, 39 (1961).
- (c) R. W. Fessenden and J. S. Waugh, *J. Chem. Phys.*, 37, 1466 (1962).
- (8) e.g. B. Sunners, L. H. Piette, and W. G. Schneider, *Can. J. Chem.*, 38, 681 (1960).
- (9) We thank Professor Goldstein for making a copy of his manuscript available to us before its publication.
- (10) G. V. D. Tiers, *J. Phys. Chem.*, 62, 1151 (1958).

(Submitted for Publication in *J. Mol. Spec.*)

TABLE I

			<div>$\begin{array}{c} \text{H}_t \\ \diagup \\ \text{C}=\text{N}^a \\ \diagdown \\ \text{H}_c \end{array} \text{O}-\text{H}(\text{D})$</div>					<div>$\begin{array}{c} \text{H}_t \\ \diagup \\ \text{C}=\text{N}^b \\ \diagdown \\ \text{H}_c \end{array} \text{O}-\text{CH}_3$</div>				
Solvent			$ J_{\text{H}_c \text{H}_t} ^d$	H_c		H_t		$ J_{\text{H}_c \text{H}_t} $	H_c		H_t	
	ϵ (37°)	η^c (37°)		τ^e	Width ^f	τ	Width		τ	Width	τ	Width
D ₂ O ^g	73.6	0.705	7.63	2.89	0.43	3.36	0.95	7.12	2.94	0.44	3.43	0.97
H ₂ O ^h	72.3	.695	7.67 8.18	2.88 2.89	0.45 0.44	3.36 3.37	0.96 0.70	7.12 6.96	2.91 2.90	0.50 0.57	3.39 3.35	1.18 1.57
(CH ₃) ₂ SO	46.0	1.77	9.15	3.05	0.38	3.57	1.38	-- ⁱ	--	--	--	--
CH ₃ CN	34.8	0.312	8.72	3.02	0.32	3.59	1.97	-- ⁱ	--	---	--	--
CH ₃ NO ₂	34.8	0.462	8.32	2.99	0.42	3.55	1.86	7.80	3.01	0.46	3.56	2.08
C ₂ H ₅ OH	22.6	0.885	9.30	3.04	0.34	3.65	1.00	-- ⁱ	--	--	--	--
<u>n</u> -C ₄ H ₉ OH	15.7	1.926	9.16	3.02	0.36	3.64	0.67	8.11	3.02	0.48	3.58	1.56
(<u>n</u> -C ₃ H ₇) ₂ CO	11.6	0.638	9.52	3.06	0.38	3.70	1.32	8.46	3.05	0.52	3.63	3.18
CH ₂ Cl ₂	8.46	0.383	8.35	2.94	0.38	3.52	1.31	8.28	3.04	0.47	3.64	2.35
CDCl ₃	4.61 ^j	0.478 ^j	8.58	2.92	0.45	3.50	2.04	8.32	3.00	0.50	3.60	2.04
(C ₂ H ₅) ₂ O	4.00	0.208	9.82	3.10	0.42	3.77	2.16	8.58	3.11	0.48	3.71	3.57
(<u>n</u> -C ₄ H ₉) ₂ O	2.25	0.533	9.95	3.12	0.48	3.80	1.59	8.85	3.14	0.62	3.77	3.70
CCl ₄	2.20	0.770	-- ^k	--	--	--	--	8.80	3.10	0.48	3.71	2.94
C ₆ H ₁₂ ^l	1.96	0.683	-- ^k	--	--	--	--	9.22	3.16	0.60	3.84	3.82

^aPrepared from the commercially available formaldoxime hydrochloride. ^bPrepared from the commercially available methoxyamine hydrochloride and formaldehyde. The assignments of the spectral lines to H_c and H_t are based on spectra of known isomeric aldoximes and will be discussed at length elsewhere. ^cViscosity in centipoises. ^dP.E. \pm 0.05 or less. ^eChemical shift on the τ -scale ($\tau_{\text{Me}_4\text{Si}} = 10.0$)(10). Estimated probable error of τ -values is \pm 0.01 or better. τ -values for the aqueous solutions were obtained by internal referencing with t-butanol, assigning $\tau_{\text{Me}_3\text{COH}} = 8.80$. ^fThe full line-width at half height; values are the average of the widths of the appropriate two lines of the AB pattern, and are accurate to \pm 0.1 cps or better. ^gSpectral data obtained on neutral solutions. ^hThe first value given in a particular column refers to data obtained on a neutral or slightly acidic solution, the second value to the data from alkaline solution. ⁱMaterial insufficiently stable in these solvents. ^jThe values are actually those for CHCl₃. ^kFormaldoxime not soluble in this solvent. ^lCyclohexane.



THE UNIVERSITY OF MANCHESTER,

DEPARTMENT OF CHEMISTRY,

MANCHESTER, 13.

13th June, 1963

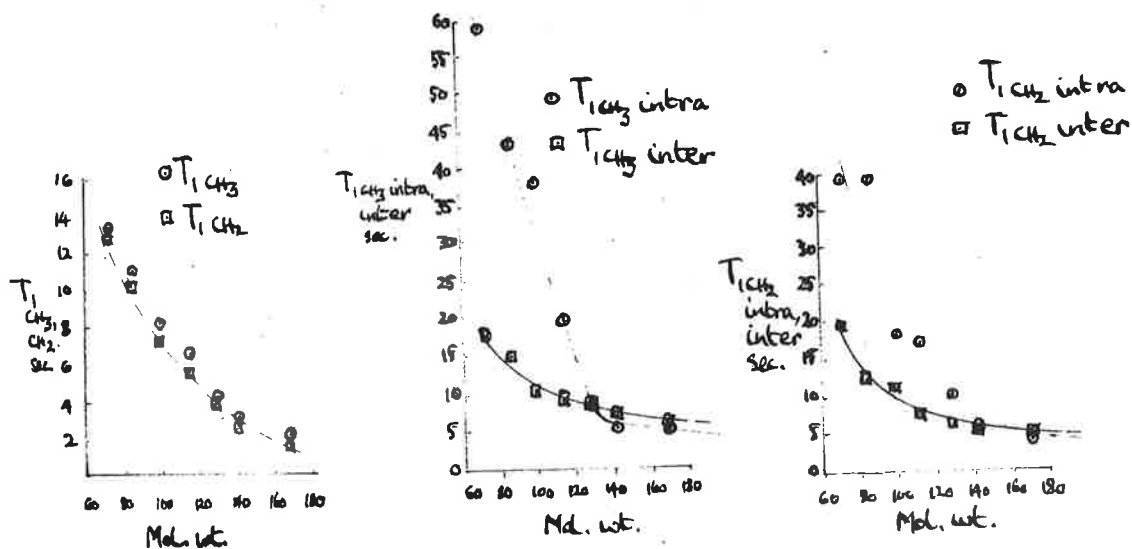
TELEPHONE: ARDWICK 3333

Dr. B. L. Shapiro,
MELLON INSTITUTE
4400, Fifth Avenue,
Pittsburgh.
PENNSYLVANIA. U.S.A.

Dear Dr. Shapiro,

As a contribution to MELLON M.R. from this laboratory, I should like to describe some spin-lattice relaxation measurements made on the series n-pentane, ..., n-dodecane on an A.E.I. R.S.2 spectrometer by the direct method. T_1 was measured for both the methyl and methylene protons, together with T_1 inter and T_1 intra for these groups. The latter measurements were obtained by dilution in CS_2 and extrapolation to infinite dilution to obtain T_1 intra.

T_1 inter was obtained from this value and that ascribed to T_1 itself by substitution in the equation: $\frac{1}{T_1} = \frac{1}{T_{1, intra}} + \frac{1}{T_{1, inter}}$. The results are summarised graphically below:-



T_1 CH_3, CH_2 are most influenced by molecular weight at the low molecular weight end of the series and reach a limiting value of ~ 2 secs. at n-dodecane. T_1 CH_3, CH_2 intra are much less molecular weight dependent, indicating that intra-methyl, methylene group relaxations are more important than extra-methyl, methylene group inter-actions within the same molecule. T_1 CH_3, CH_2 inter reach a limiting value at molecular weight ~ 140 . Results for liquid ethane due to Muller & Noble⁽²⁾ show that the inter- and intra-molecular components of T_1 show the same trend as above when interpreted using the conventional theory⁽³⁾ but on applying that due to Bloom and Sandhu⁽⁴⁾ they deduce that 90 per cent. of the interaction is due to intermolecular interaction. I suggest that relaxation mechanisms can best be described by theories similar to that in ref.3 and that both T_1 inter and T_1 intra make significant contributions to T_1 .

Yours sincerely,

D.J. Bleans.

1. R.W. Mitchell and M. Eisner. J.Chem.Phys. 33, 86.
2. B. H. Muller and J. D. Noble. J.Chem.Phys. 38, 777, (1963).
3. A. Abragam, The Principles of Nuclear Magnetism.
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המכון - מכון טכנולוגי לישראל

TECHNION - ISRAEL INSTITUTE OF TECHNOLOGY

Physical Chemistry

המחלקה לכימיה
DEPARTMENT OF CHEMISTRY

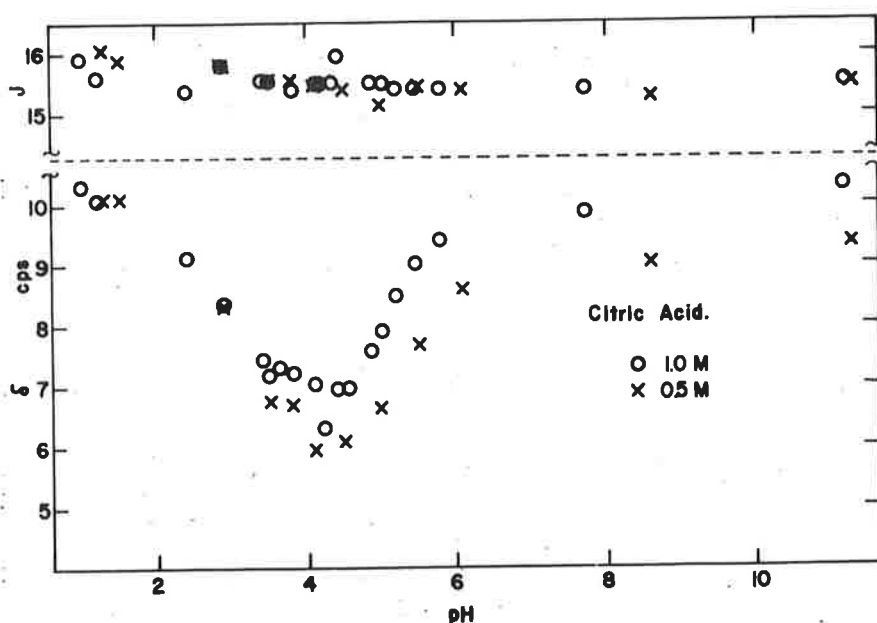
July 2nd, 1963

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

Dear Dr. Shapiro:

Sometime ago Dr. J. D. Roberts and I studied the ionization of citric acid by nmr. The results were published in the *J. Am. Chem. Soc.*, **82**, 2705 (1960). More recently Dr. R. Bruce Martin (*J. ~~Am. Chem. Soc.~~ Phys. Chem.*, **65**, 2053 (1961) reinvestigated the problem by titration of selected methyl esters of the acid. The results obtained by Martin contradicted those obtained by the nmr study. In private correspondence between ourselves and Dr. Martin, various reasons for the discrepancy were suggested.

As part of a program to study the pH and temperature dependence of the chemical shift (δ) and the spin-spin coupling (J), between the protons of the methylene group in molecules of the type $\text{CH}_2\text{X}-\text{CR}_1\text{R}_2\text{R}_3$, where the R's contain $-\text{COOH}$ or $-\text{OH}$, citric acid was restudied. Results are given in the attached figure.



.../2

Obviously our previous analysis, where the average chemical shift was used, is wrong. Temperature dependence studies show that δ and J change in the acidic but not in the basic solutions (10-80°C, pH 0 and pH 12). This result and the data shown in the figure indicate that in the ionized and to a lesser extent in the non or partially ionized species of citric acid, the rotations around the central **C-C** bonds are strongly hindered.

Through the use of simpler molecules an attempt is presently made to isolate the various contributions to δ and J .

Thank you for regularly mailing the Meliomr.

Sincerely yours,

A. Loewenstein

A. Loewenstein

H. Gilboa

H. Gilboa

Monthly
Ecumenical
Letters from
Laboratories
Of
N-M-R

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".

Glaxo Research Limited

Greenford · Middlesex

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

21st June, 1963.

Dear Dr. Shapiro

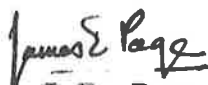
The British-made ancillary equipment (see MELLONMR, No. 49, p. 24) that we use with our A-60 n.m.r. spectrometer has, in general, worked satisfactorily, although regular maintenance is needed to ensure continuous operation. The gauze-filters of the Temkon air-conditioner require cleaning once a fortnight; the stator of the water-pump has been replaced twice during twelve months. The voltage stabiliser servo-motor failed one night and set "hard over", driving 270 volts through the spectrometer (set for 230 volts)! Fortunately no serious damage was done. We intend fitting an over-voltage trip to the "stabilised" voltage supply.

Streptomycin did not prevent the growth of dark green micro-organisms in our water-circulating system. The micro-organisms caused partial blockage of the magnet cooling coils; this was indicated by a reduced flow-rate and by the higher temperature of the exit water (rose from 35° to 45° C.). Acting on the advice of Mr. R. Wenk of Varian AG, we removed the dark green matter and restored the temperature of the exit water to 38° C. by blowing, alternately, compressed air (from the spinner supply, 30 p.s.i.) and water through the magnet coils. To admit air to the cooling coils, the magnet-solenoid valves must be opened with the instrument "Off". The valves are energised by disconnecting the wires from terminal L₁ on the magnet temperature controller (see Fig. 4-25 of the A-60 Maintenance Manual), connecting the wire from NO to L₂, and connecting L₂ and the loose leads from L₁ directly to the main electrical supply. The main two-pole circuit breaker must be opened to remove any risk of crossed polarity. We are now using dichlorophen ("Panacide", 2,2'-dihydroxy-5,5'-dichlorodiphenylmethane, sodium salt, supplied by British Drug Houses Ltd.) at a level of 100 p.p.m. to prevent the growth of micro-organisms.

We have found that the spectrum (420-470 c./sec. below T.M.S.) of pyridine (not especially pure, but reasonably dry) is useful for fine field trimming and for rapid checks on the field homogeneity of the A-60 spectrometer (see Fig.). Good Y-gradient trimming, which is all that we have found to be necessary on most days, may be obtained in this way.

We have submitted to the Journal of the Chemical Society a paper on the proton resonance spectra of griseofulvin and 44 analogues. As has been shown by Gerecke et al. (Helv. Chim. Acta, 1962, 45, 2241) and Arison et al. (J. Amer. Chem. Soc., 1963, 85, 627), n.m.r. is an excellent technique for studying such compounds and provides information, which complements that obtained from infrared studies (Page and Staniforth, J. Chem. Soc., 1962, 1292; 1963, 1814).

Yours sincerely,


J.E. Page


G.F.H. Green

Pyridine

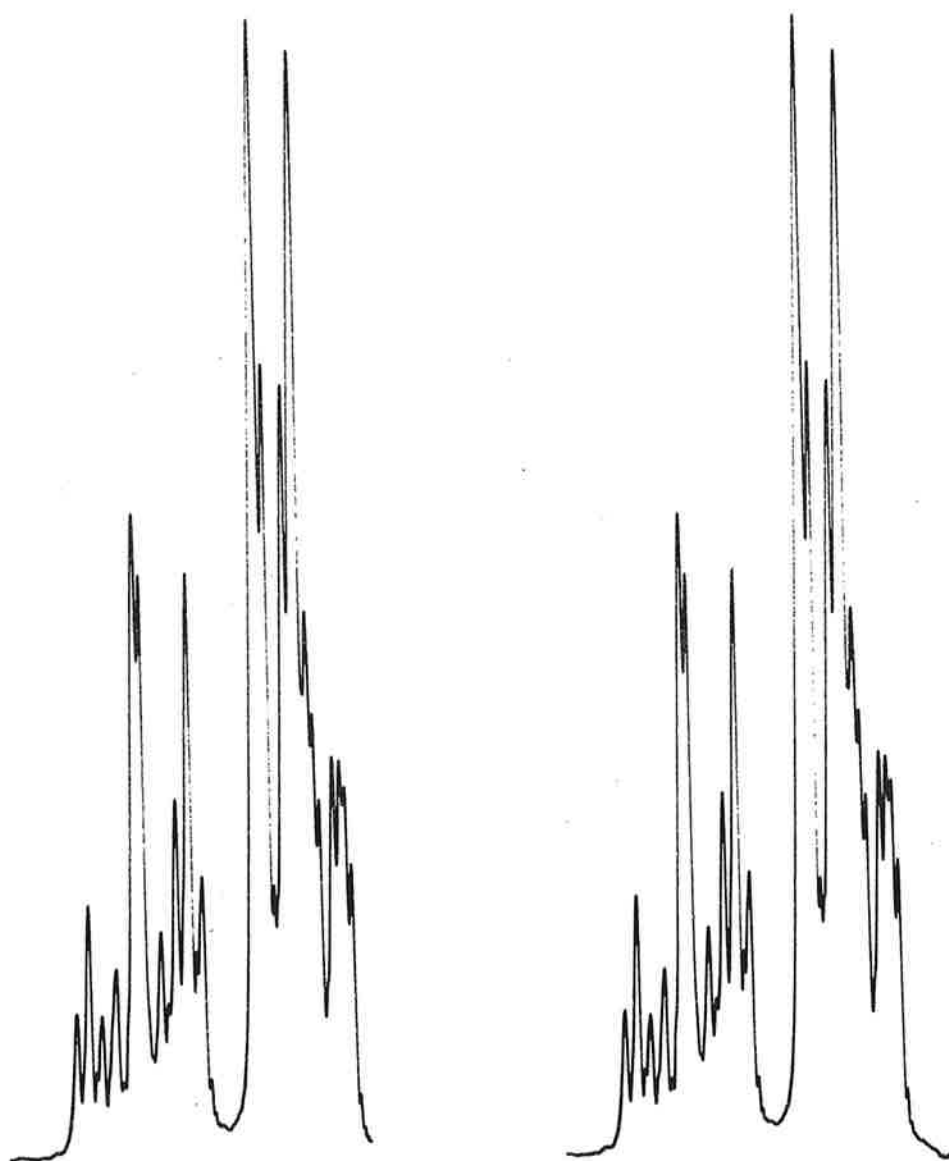
Filter Band width 0.4 c./sec.

R.F. Field 0.4 m.gauss

Sweep time 500 sec.

Sweep width 500 c./sec.

Amp. 4.0



**STERLING-WINTHROP RESEARCH INSTITUTE**

A DIVISION OF STERLING DRUG INC.
RENSSELAER, NEW YORK

July 5, 1963

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

Dear Dr. Shapiro:

We have often been chagrined by the tediousness of fishing decomposed cork stoppers out of necks of NMR tubes containing CF_3COOH as a solvent. Perhaps some of our colleagues have had similar annoying experiences and would like to share our means of making light-weight, resistant NMR tube closures.

Thin walled polyethylene tubing has been found to resist satisfactorily the action of CF_3COOH . The tubing is the type commonly used as "dip tubing" in certain types of pressurized aerosol dispensers. It is fabricated from low density (sp. gr. = 0.92 g/L) polyethylene, obtainable in a variety of diameters from suppliers to the aerosol packagers. (Two such suppliers are Anchor Plastics Co., 36-36 36th St., Long Island City 6, N.Y. and Colorite Plastics, 50 California Ave., Paterson 3, N. J.)

Tubing with an O.D. of 0.240", wall thickness 0.022", is cut into approximately 1 inch lengths. One end of the tube is collapsed by compression between metal clamps (laboratory tongs are convenient), with about 1/8" projecting beyond the edge of the clamp. The projecting end is carefully heated in a low gas flame until the open ends of the plastic fuses. Remove from the flame, keeping the clamp in place, and chill to room temperature by immersion in cold water. A little practice will indicate the correct degree of heating to fuse the plastic without over heating. The caps fit snugly over precision glass tubes and are quite impermeable to CF_3COOH fumes. As a matter of fact, they become more rigid in use.

With kind regards,

Sincerely yours,


S. Reznick
F. C. Nachod

FCN/rb



STATE UNIVERSITY OF NEW YORK

AT STONY BROOK

STONY BROOK, LONG ISLAND, NEW YORK

DEPARTMENT OF CHEMISTRY

July 8, 1963

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

Dear Barry:

Since our only computer here is an IBM 1620, it has been necessary to adapt FREQINT IV to its limited memory, its card input and output, and a few peculiarities of FORTRAN II. This has now been accomplished, with the help of our Computing Center personnel, and the result, called FREQINT IV A 1620, is running smoothly. At the moment, the program is limited to a maximum of four nuclei. Running times, including punch out of plot information, have been no more than two minutes for three nuclei and from three to nine minutes for four nuclei. For example, one typical A_2B_2 ran in three minutes, and the AB_2X test case in the FREQINT IV manual ran in nine minutes. The program as written requires a storage space of about 40,000 out of the 60,000 available in our machine.

We are considering the modifications required to enable the 1620 to handle five nuclei, but we will probably not put much effort into it unless a pressing need develops. The 1620 will be replaced by a 7040 within a year, which should permit us to do more nuclei and to run any of the iterative programs without much trouble.

I shall be happy to send FORTRAN decks, listings and operating instructions for FREQINT IV A 1620 to anyone who is interested. Does this note count as a subscription renewal for MELLONMR?

Yours truly,

Paul C. Lauterbur
Associate Professor

PCL:jv



DOW CHEMICAL OF CANADA, LIMITED

SARNIA, ONTARIO

June 7, 1963

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

Dear Dr. Shapiro:

Spectra of Aliphatic Fluorine Compounds

In this laboratory we have determined the spectra of a number of simple fluorine containing compounds. It is possible that the proton shifts and coupling constants may be of use to other readers.

The spectra were recorded on an A-60 spectrometer with approximately a 15 volume per cent solution in deuterated chloroform. Tetramethylsilane was used as an internal reference. Calibration of the instrument was checked with a solution of tetramethylsilane and chloroform in deuterated chloroform just prior to the recording of the spectra. The coupling constants were obtained from the 100 cycle sweep width.

Compound	Chemical Shift (c.p.s. from T.M.S.)	J_{HF} (c.p.s.)	J_{HF} (c.p.s. from other sources
CF_3CHCl_2	349 ^a	4.8	
$CF_3CH_2O-C(O)-CH_3$	128 ^b 268 ^a	8.7	
$CF_3CH_2-O-C(O)-CCl_3$	283 ^a	8.0	
$CF_2ClCHCl_2$	353 ^c	5.6	5.5 (1)
$CFCl_2CHCl_2$	360 ^d	4.5	4.5 (2)
$CCl_2FCHClF$	375 ^e	48.7 {gem} 4.0 {vic}	
CF_3CH_2Cl	228 ^a	8.6	8.49 (3)
CF_3CH_2Br	219 ^a	9.0	8.92 (3)
CF_3CH_2I	214 ^a	9.8	

a) Quartet
b) Singlet
c) Triplet

d) Doublet
e) Double doublet

- 2 -

I wish to express thanks to Dr. D.M. Young for supplying most of the above compounds.

References

1. R.W. Fessenden and J.S. Waugh, J.Chem.Phys. 37, 1466 (1962).
2. R.J. Abraham and H.J. Bernstein, Can.J.Chem. 39, 39 (1961).
3. Daniel D. Elleman, L. Carlton Brown and Dudley Williams, J. Mol. Spectroscopy, 7, 307 (1961).

Sincerely,



J. S. McIntyre
Exploratory Research Laboratory

Contribution No. 106
Exploratory Research Laboratory
Dow Chemical of Canada, Limited
Sarnia Ontario Canada

RF/DMT



Department of Scientific and Industrial Research
NATIONAL PHYSICAL LABORATORY
 TEDDINGTON, Middlesex

Telegrams: Physics, Teddington Telephone: TEDDINGTON Lock 3222, ext. 800

BASIC PHYSICS DIVISION

15th July, 1963

Please address any reply to
 THE DIRECTOR
 and quote: BP.5/6/01
 Your reference:

Dear Barry,

Double-Quantum Spectra

Although NMR spectra recorded at high settings of the radiofrequency field strength are usually rather messy, they are not always without interest. The eye of faith (or better still, some homework on the theory¹) may identify some new sharp lines, the double-quantum transitions. They may be thought of as arising through the coherent excitation of the "tails" of certain single-quantum lines - those pairs of transitions that span the interval between the initial and final levels of the double-quantum jump.

It turns out that the applications of double-quantum experiments show a very close parallel with the applications of double-resonance. In spectra where a first-order treatment is valid, both methods may be used to determine relative signs of spin coupling constants²; in strongly coupled spectra both techniques may be applied to the problem of assigning the transitions to an energy level diagram³ - the first step in the analysis.

Consider the example of an ABC spin system, the vinyl protons of methyltrivinylsilane. Fig. 1 shows a schematic energy level diagram, while the lowest trace of Fig. 2 shows the 60 Mc spectrum and the numbering of transitions. Line A may be taken as a typical double-quantum transition and it is a simple consequence of the frequency sum rules that its frequency is the mean of the frequencies of lines 3 and 5, and also the mean of 2 and 9, and of 1 and 10. In other words, each of the three pairs of transitions

Fig 1

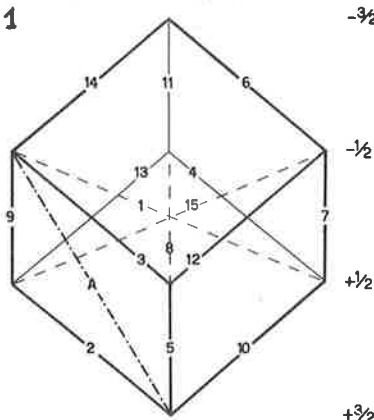
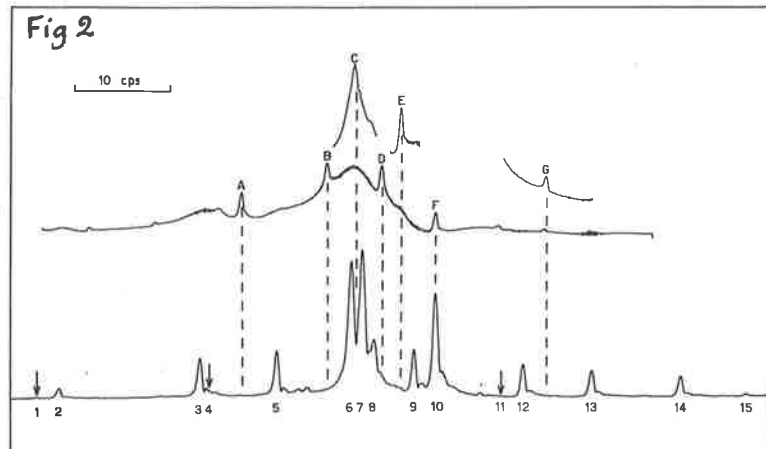


Fig 2



NATIONAL PHYSICAL LABORATORY

- 2 -

that connects the initial and final levels of the double-quantum transition may be identified simply by placing one leg of a pair of dividers at the position of the double-quantum line and by picking out transitions that have a partner symmetrically placed on the other side. Armed with this and similar information from the other double-quantum transitions, one has only a rather simple topological problem to solve in order to build up the complete energy level diagram. (Inversion of the entire diagram need not be considered here, since it corresponds only to the reversal of the signs of all the spin coupling constants).

Not all the double-quantum lines require the same optimum rf driving field, hence in practice it is necessary to run several traces at different strengths of H_1 , and some care is required to distinguish single, double and triple-quantum signals and spinning sidebands etc. All six double-quantum transitions of methyltrivinylsilane (A,B,C,E,F and G) and the one triple-quantum line (D) are shown in the upper traces of Fig. 2.

Now the presence of a strong rf field is known to perturb the energy level diagram to some extent, since the various transitions experience different effective fields in the rotating frame of reference - an effect closely allied to the Bloch-Siegert shift, and also to the shift of modulation sideband responses, and one of the corrections applicable in spin-decoupling experiments. This results in a very small displacement of the frequency of the double-quantum transition from its "expected" position, but fortunately it can be shown that for the optimum setting of H_1 this shift should be of the order of the natural line width and may therefore normally be neglected in the type of application described above.

Yours sincerely,

Ray Freeman
Ray Freeman

1. S. Yatsiv, Phys. Rev., 113, 1522 (1959).
2. Double-resonance: J. P. Maher and D. F. Evans, Proc.Chem.Soc., 108 (1961).
Double-quantum: K.A. McLauchlan and D.H. Whiffen, Proc.Chem.Soc., 144 (1962)
3. Double-resonance: R. Freeman and W.A. Anderson, J.Chem.Phys., 37, 2053, (1962).
Double-quantum: W.A. Anderson, R. Freeman and C.A. Reilly, J.Chem.Phys., to be published.

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

BASF



Badische Anilin- & Soda-Fabrik AG

LUDWIGSHAFEN AM RHEIN

Luftpost

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

IHRE ZEICHEN

IHRE NACHRICHT VOM

UNSERE NACHRICHT VOM

UNSERE ZEICHEN

TAG 15. 7. 1963

BETREFF

Dear Dr. Shapiro,

it is time for me to make a contribution again to Mello-NMR. However, I can send you in this moment only a minor technical hint which was of considerable help to us and may be useful for some other reader.

In our laboratory we are working with a VARIAN High Resolution Spectrometer and since a short time with a VARIAN A-60-Analytical Spectrometer too. The high resolution instrument, one of the first that came to Europe, was originally built for 40 Mc but later on changed to 60 Mc. We found out immediately that it is good to keep the instrument always switched "on". The reasons are lifetime of tubes, readiness to work and many others. For a long time, however, we had difficulties caused by a slowly variable thermal drift. In spite of many attempts we never succeeded to make it completely disappear. While taking spectra this factor is unimportant, but not if the instrument is not used for a longer period, e.g. at night. It then goes considerably off its working point and had to be adjusted again. Especially since the baseline stabilization and the integrator with its 2000 cps side bands had been built in this was tedious because of possible confusion of the side bands with the center band by untrained personnel. One of my coworkers found a simple way to avoid this difficulty: At the beginning of a longer period of rest the phase is altered from the usual absorption signal to the dispersion signal. In doing so we normally use a simple signal as given e.g. by water, benzene etc. The spectrometer is then adjusted to the center of the signal and the output is given to the superstabilizer as shown in the figure. As long as the instrument stays at the given working point the voltage at the output is zero. If it shifts there is a voltage at the output which takes the field back to the working point, if the sign of the voltage is in the right direction. This may be checked by using the slow sweep unit.

This measure has another advantage in producing the optimal form of the field by the electric shims or the field trimmer. For this purpose a small alternating voltage, e.g. of 100 cps, is given to the sweep coils of the probe (as for the sideband modulation). Thereby the lightspot on the oscilloscope is dissipated in the vertical direction to a line. Its length depends on the slope of the dispersion signal in the center. The

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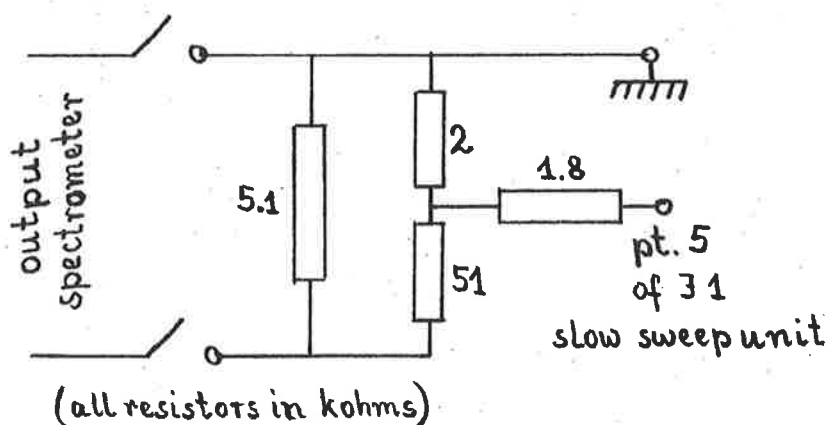
Betreff

slope is largest if the respective absorption signal is narrowest. By use of the electric shims the length of the line is made as large as possible. For untrained personnel the judgement of such a length is easier than that of the height of a periodically displayed signal or of the numbers of wiggles (the number of which is reduced with the high sensitivity insert in any way). -

Our laboratory mainly serves to aid many synthetically working chemists. Besides the considerable daily routine we are concentrating on **smaller** groups of spectroscopically interesting problems in order to get a general survey. In this respect we are directing our attention at the moment on the NMR spectra of naphthalenes and on the conformational analysis of cyclohexylamines, this latter in cooperation with Dr. FELTKAMP of the Pharmazeutisch-Chemisches Institut of Tübingen University. I hope that I can report on this subject in the near future.

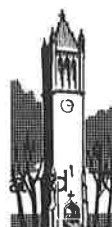
Yours sincerely

W. Brügel
W. Brügel



IOWA STATE UNIVERSITY

of Science and Technology



AMES, IOWA

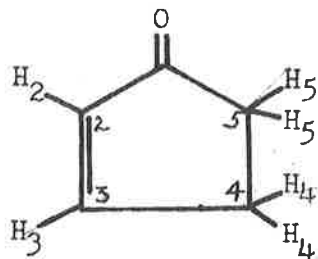
Department of Chemistry

Dr. Barry Shapiro
Mellon Institute
Pittsburgh 13, Pa.

Dear Dr. Shapiro:

During the past few years we have been doing a lot of synthetic work with substituted cyclopentenones, and because NMR was so valuable for identification and structure proof we did some very careful spectral work on a number of pure, standard compounds. This work is just being readied for publication and I thought that MELLONMR readers might be interested in a summarizing table of chemical shift and coupling constant data.

Fig. I Coupling Constants in Substituted Cyclopentenones.



$$J_{2,3} = 5.8 \pm 0.1 \text{ (6)}$$

$$J_{3,4} = 2.4 \pm 0.2 \text{ (9)}$$

$$J_{4,4} = J_{5,5} = 19.2 \pm 0.2 \text{ (5)}$$

$$J_{\text{trans}} = 6.4 \pm 0.1 \text{ (3)}$$

$$J_{\text{cis}} = 2.1 \pm 0.3 \text{ (3)}$$

$$J_{2,4} = 1.7 \pm 0.4 \text{ (5)}$$

The numbers given are the averages of the values from the number of compounds given in parentheses. The cross-ring coupling constant $J_{2,4}$ has rather deceptively large deviations. Actually in three compounds in which there is no substituent on carbon 4, $J_{2,4} = 2.1 \pm 0.1$ and in three compounds in which there is a substituent (Br, OAc, and alkyl) $J_{2,4} = 1.3 \pm 0.1$. In two compounds there was a methyl substituent at C₁. Then there was coupling between the methyl hydrogens and both H₃ and H₄, with $J_{\text{CH}_3, \text{H}_3} = 1.6 \pm 0.3$ and $J_{\text{CH}_3, \text{H}_4} = 2.4 \pm 0.2$.

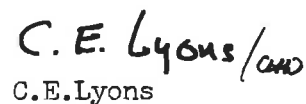
Fig. II Chemical Shifts in Units of Tau in CCl_4 Solution.

COMPOUND	H ₂	H ₃	H ₄	H ₅	OTHER
4-Bromocyclopentenone	3.79	2.39	4.87	7.03, 7.37	
4-Acetoxy-cyclopentenone	3.75	2.49	4.22	7.28, 7.79	7.95 (CH ₃).
5-Oxalylcyclopentenone	3.64	2.44	6.50		-2.7 (OH), 5.68, 8.62 (C ₂ H ₅)
5-Ethoxycyclopentenone	3.95	2.49			
3,4-Diethoxycyclopentenone	4.81		5.63	7.45, 7.79	5.94, 8.55 (C ₂ H ₅) 6.39, 8.81 (C ₂ H ₅)
Cyclopentene-3,5-dione	2.69	2.69	7.10		
1-Methylcyclopentene-4,5-dione		2.28	6.97		8.03 (CH ₃)
2-Methyl-5-diazocyclopentenone		3.29	6.43		8.20 (CH ₃)
Cyclopentenone	3.89	2.37	7.4	7.8	
Cyclopentenone Ethylene Ketal	4.04	4.42	7.7	8.0	6.17 (CH ₂)
2-Hydroxycyclopentenone		3.54	7.6	7.6	3.19 (OH)

Sincerely



C.H. DePuy



C.E. Lyons

HARVARD UNIVERSITY

DEPARTMENT OF CHEMISTRY

*12 Oxford Street
Cambridge 38, Massachusetts, U.S.A.*

July 19, 1963

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

Dear Barry:

After using the Varian Variable Temperature Probe a while, we have a few modifications and caveats which others may find helpful.

We have noted that on one occasion when cycling from the high temperature range to the low range (liquid nitrogen cooling) and back, the calibration suddenly shifted about 10°C. Although this may just be our system, it is advisable to check the calibration after such a cycle. A surge tank between the gas regulator and the temperature controller is helpful in smoothing out the regulator pulsations.

In the present low-temperature kit for the Variable Temperature Probe, the tygon tubing is joined to the stainless heat (cold is a better term) exchanger below the level of the liquid nitrogen. Trouble can (and does) arise from the fittings working loose or the tygon tubing splitting where it slips over the hose connector (which can be a small disaster). All this probably happens when the liquid nitrogen level in the dewar is allowed to fall too far and then the dewar is refilled. However, by supplying the Varian Cold Exchanger with long exit and entrance tubes, such that the tygon tubing joins the metal outside the nitrogen dewar, the unions are where they can be seen and minded.

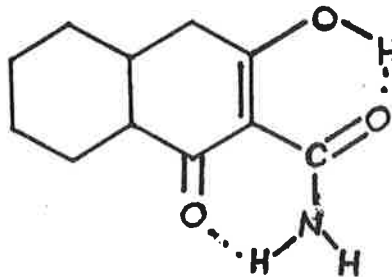
The spinners for the room temperature probe have been slightly modified. The original bottom containing the O-ring has been turned down to a taper and a left-handed thread machined into it to form the inside of a chuck. A crosscut into the bottom of the new spinner gives flexibility to the piece for the gripping action. A new bottom with a matching left-hand thread was made to fit. The pair screw together as a chuck and can grip the sample tubing quite firmly. The usual problem of rapid wear of the spinner O-ring is eliminated in this manner.

Dr. B. L. Shapiro

-2-

July 19, 1963

In research--Schiff Bases are under investigation as usual. We have strayed afield slightly, and Gert Volpp and I have a model compound for the dicarbonyl amide portion of the aureomycin molecule: The compound's NMR spectrum has the enolic hydrogen resonance (in CDCl_3) at 17.8 ppm. The large paramagnetic shift of the hydroxyl signal indicates that resonance interactions in the system are extremely large and is a nice complement to Donohue's x-ray work on aureomycin (JACS 85, 851 (1963)). We are making a few more models of this system.



Yours truly,

Gerald Dudek

GD:dl

July 17, 1963

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

Dear Dr. Shapiro:

I should like to write on the experimental determination of absolute sign of spin-coupling constant J . In this communication I will point out that the sign of J can be determined by means of the magnetic saturation.

Consider a two-spin system composed of nonequivalent nuclear spins of $1/2$, I and S . The stationary state Hamiltonian in the unit of angular frequency may be given by

$$\underline{H} = -\omega_I I_z - \omega_S S_z + J I \cdot S,$$

where the relation $|\omega_I - \omega_S| \gg |J|$ is assumed. The corresponding eigenfunctions may be written as

$$\varphi_1 = \alpha\alpha, \varphi_2 = \beta\alpha, \varphi_3 = \alpha\beta, \text{ and } \varphi_4 = \beta\beta.$$

If the spectrum is observed under the condition of constant frequency, the resonance field for the line, $a \rightarrow b$, will be denoted as $H(ab)$. The following relations are evident:

$$\begin{aligned} H(12) > H(34) \quad \text{and} \quad H(13) > H(24) \quad \text{for positive } J, \\ H(34) > H(12) \quad \text{and} \quad H(24) > H(13) \quad \text{for negative } J. \end{aligned}$$

However the appearance of the spectrum does not depend on the sign of J .

Here the nuclear relaxation times for each component line of the spectrum are considered. Two mechanisms may be assumed: (I) the dipolar interaction between the two spins, $V_d(t)$, and (II) the interaction of the spin S with the fluctuating local field resulted from the anisotropic shielding of the external field, $V_a(t)$. The relaxation times are given in terms of the probability for the transition due to these two random perturbations. The transition probability may be calculated by using the second order perturbation theory; it may be written in a symbolic manner as

$$P = (1/T_{1,2}) \sim (V_d)^2 + (V_a)^2 + (V_d V_a) + (V_a V_d).$$

No special explanation will not be needed for the terms, $(V_d)^2$ and $(V_a)^2$. The latter two cross terms are resulted from the interference between the two perturbations. Though these cross terms have been neglected in the calculation of nuclear relaxation processes, they should not generally be neglected. The correlation between these two perturbations does exist. A straightforward calculation showed that the inclusion of the cross terms made possible to determine the "absolute" sign of J .

As an example of illustration a two-spin system in the CHFRF' type compound may be taken. Assuming $C-H = 1.10A$, $C-F = 1.33A$, $\angle HCF = 109^{\circ}30'$, and the anisotropy of F shielding = 1, 000 ppm, the following relative saturation parameter is given for each component line, provided that the external field of 15, 000 gauss is used.

$S(12) = 1.732$ and $S(34) = 2.029$ for the I-spectrum,

$S(13) = 2.138$ and $S(24) = 0.494$ for the S-spectrum.

The difference among the saturation of component lines may be detected without special difficulties.

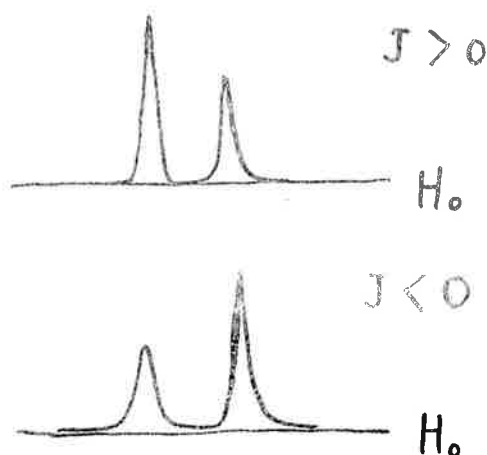
The calculation showed that the relative saturation parameters do not depend on the sign of J . Hence, the line, $1 \rightarrow 3$, is always saturated more quickly than the line, $2 \rightarrow 4$. The absolute sign of J_{HF} may be determined by observing the saturated spectrum of the fluorine resonance: if the line at a higher field side is more strongly saturated than another, the absolute sign of J is positive and, contrary to this, if the line at a lower field side is more strongly saturated, J has negative sign. A full discussion as well as the experimental results will be given in the near future.

Yours sincerely,

Hiroshi Shimizu

Hiroshi Shimizu
Department of Chemistry
College of Arts and Sciences
Chiba University
Chiba, Japan

The F^{19} - spectrum



VOLUME MAGNETIC SUSCEPTIBILITY MEASUREMENTS BY NMR

by

Dean C. Douglass and Anthony Fratiello*
Bell Telephone Laboratories, Incorporated
Murray Hill, New Jersey

The experimental technique for measuring volume magnetic susceptibilities by NMR (1,2,3), needed for correcting chemical shifts obtained by external referencing, along with a complete theoretical analysis of the method (4), have been previously described. Although the method has demonstrated a precision of about one per cent, as well as being rapid and requiring very little sample, it is rarely used because of many seeming difficulties (3,6). Rather, susceptibilities are usually calculated leading to errors of several per cent, in some cases, in NMR shift measurements. This note describes several experimental refinements of the method which remove most of the ambiguity previously associated with the measurements.

The method, which requires the use of precision concentric sample tubes (5), consists of the following. With a sample of unknown susceptibility in the central tube, a reference compound in the annular region between tubes, and the sample not spinning, the resonance signal of the reference compound is split into a doublet. The splitting results from the nonuniform magnetic field experienced by the reference compound in this static condition. From the measured doublet

Present Address:

*Los Angeles State College, Los Angeles 32, California

separation, the reference and the glass susceptibilities, and a knowledge of the tube geometry, the unknown susceptibility can be calculated (1,2,3).

It has been observed that the reference compound signal shape and apparent peak separation often depend on the position of the sample tube in the magnetic field. However, this signal distortion can be eliminated entirely by proper adjustment of the magnetic field homogeneity. These effects are illustrated in Figs. 1a and 1b. Reading from left to right in both cases, it can be seen that the signal symmetry is destroyed by decreasing the field homogeneity in the x and z directions, respectively. Note also, the x and z field homogeneity controls regulate the peak shapes independently. With the attainment of sharp signals, and using the criterion that a spectrum is valid only when the observed signal shape and separation are unaffected by manual rotation of the sample tube through an arbitrary angle, susceptibilities can be measured with a precision of 0.5-1%.

Figures 2a and 2b illustrate how the sign of a susceptibility may be determined by this technique. In Fig. 2a, the addition of the paramagnetic Ce ion to water causes a coalescence of the reference doublet usually observed with pure water in the central tube, with the reappearance of the sharp signal only upon the dissolution of more salt. Whereas the low and high field peaks of the

- 3 -

doublet are controlled by the x and z field homogeneity controls, respectively, with a diamagnetic solution in the central tube (Figs. 1a, 1b), the reverse is true with a paramagnetic solution present, as illustrated by the spectra in 2b.

Dean Douglas
Anthony Frateello

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1. Reilly, C. A., McConnell, H. M., and Meisenheimer, R. G., Phys. Rev., 98, 264A (1955).
2. Morin, M. G., Paulett, G., and Hobbs, M. E., J. Phys. Chem., 60, 1594 (1956).
3. Scruggs, R. L., and Li, N. C., MELLONMR, ~~May (1962)~~ *No. 46*
4. Zimmerman, J. R., and Foster, M. R., J. Phys. Chem., 61, 282 (1957).
5. Wilmad Glass Company, Buena, New Jersey.
6. *L.N. Mulay, private communication*

FIGURE CAPTIONS

1. Cyclohexane reference signal as a function of field homogeneity control settings (letters and numerals) for a static coaxial tube arrangement. Pure water is the central tube sample.
2. Cyclohexane reference signal as a function of field homogeneity control settings for a static coaxial tube arrangement. Aqueous solutions of CeCl_3 are the samples in the central tube.

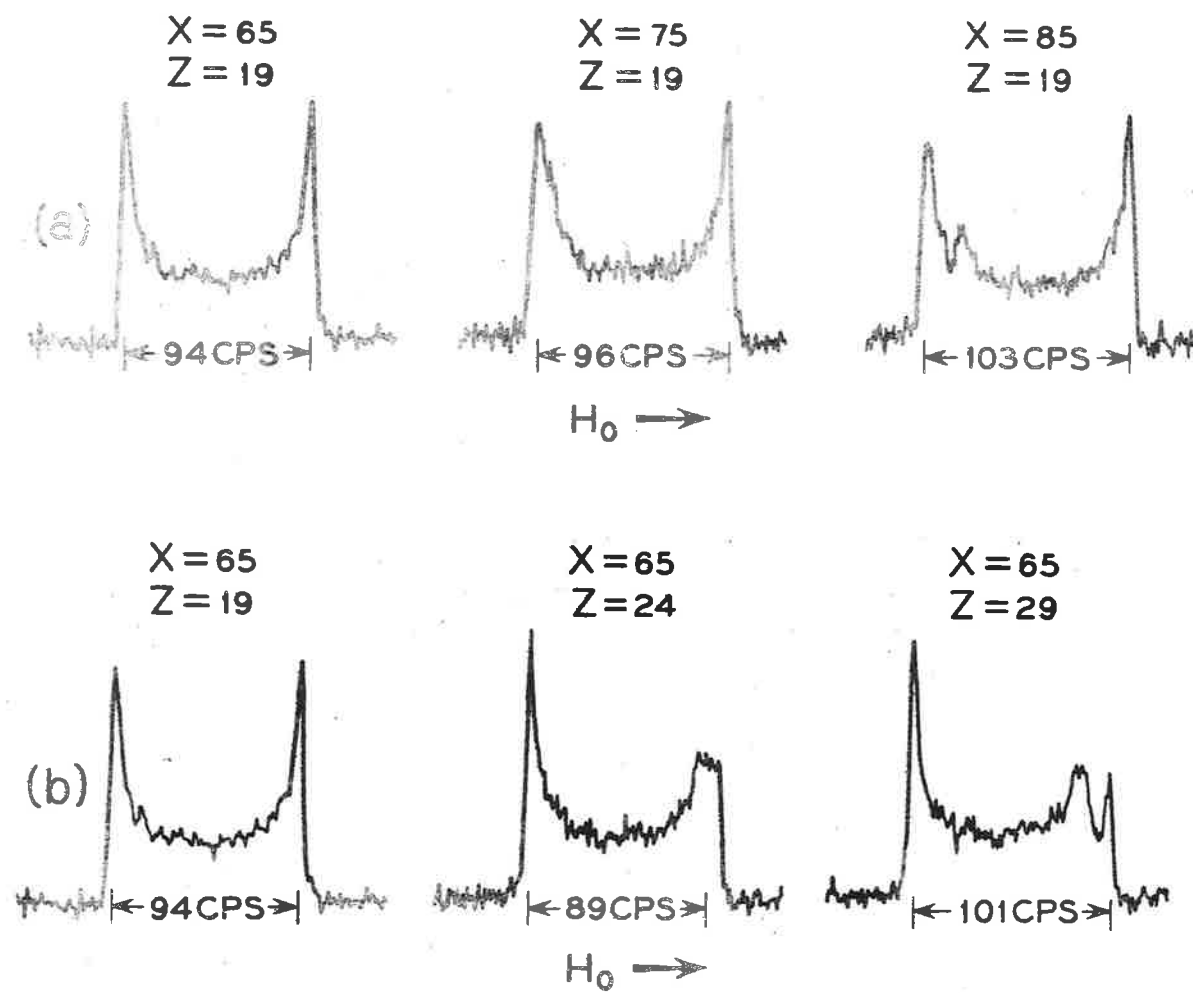


Figure 1

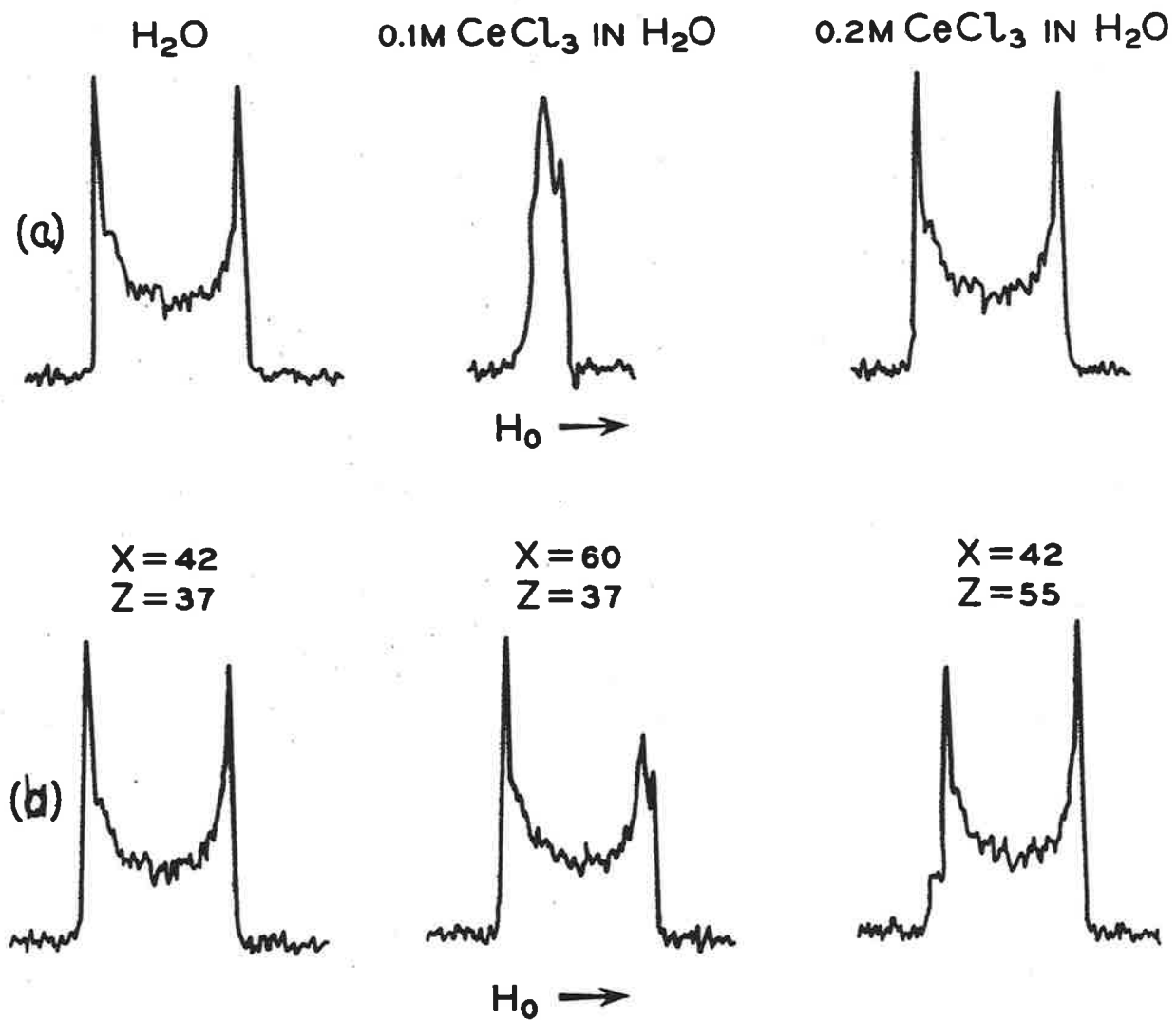


Figure 2



Staatsmijnen in Limburg

CENTRAAL LABORATORIUM GELEEN (NETHERLANDS)

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TELEPHONE GELEEN 9111
EXTENSION: 6491

TELEGRAMS: CENTRALAB GELEEN

TELEXNUMBER 11639

Dr. B.L. Shapiro
Mellon Institute
4400 Fifth Avenue

P I T T S B U R G H
(Penn.)

YOUR REF.

YOUR LETTER OF

OUR REF

GELEEN, P.O. BOX 18

3239 CL - N 8

17th July 1963

RE

Dear Dr. Shapiro

Using the Varian Variable Temperature N.M.R. Probe Accessory the required temperature is established by means of the heater assembly and the heater control unit.

If Wide Line measurements are to be performed between -100°C and -200°C , a stream of 35-40 l/min of dry nitrogen (cooled in a copper coil immersed in a liquid nitrogen filled Dewar vessel) must be passed through the Dewar insert.

To reduce this large amount of nitrogen to a considerable degree, we replaced the heater assembly by a Dewar inlet tube of equal dimensions the double wall of which reaches into the liquid nitrogen.

It is possible now to reach a temperature of -195°C in the insert, using a nitrogen flowrate of 7-8 l/min.

By controlling the nitrogen current any desired temperature between $+20^{\circ}\text{C}$ and -196°C can be established.

Following a change in the nitrogen flowrate, the temperature becomes constant again within 10 minutes.

Temperature adjustment in the range between $+20^{\circ}$ and $+200^{\circ}\text{C}$ is done by means of the normal Varian heater.

Yours truly

(W. v. Raayen)

THE UNIVERSITY OF BRITISH COLUMBIA

VANCOUVER 8, CANADA

DEPARTMENT OF CHEMISTRY

19th July, 1963.

Dr. Barry Shapiro
Mellon News
Mellon Institute
4400 5th Avenue
Pittsburgh 13, Pa.

Dear Barry:

We were very interested to read the letter by David Grant and Karl Kuhlmann in Mellon No. 57 which arrived simultaneously on my desk with experimental results on the same topic taken in our laboratory.

We are investigating salts which contain fluorine in the anion by changes in proton resonance - line width and chemical shift - in solvent, fluorine resonance in the ion and several other parallel classical chemical measurements. The data for NaBF_4 is shown in the figure. One line represents the change in chemical shift of sodium borofluoride F^{19} resonance with an external saturated solution of sodium borofluoride as reference at 56.4 Mc. The diamagnetic corrections are negligible. One of the measurements we have made is the concentration dependence of the volume susceptibility by the method of Frei and Bernstein (J. Chem. Phys. 37, p. 1891 (1962)). The points can be fitted to the same type of equation as used by Grant and Kuhlmann.

$$\delta_e = \delta_o a + \delta_p (1-a) \quad \text{--- (1)}$$

We have used the activity coefficients for NaClO_4 for direct comparison. The parameters obtained from equation (1) are

$$K = 0.21 \pm 0.7 \text{ moles/1000 grams}$$

$$\begin{array}{l} \delta_p = +160 \pm 10 \text{ cps} \\ \delta_o = -225 \pm 9 \text{ cps} \end{array} \quad \left. \begin{array}{l}) \\) \end{array} \right\} \text{ at } 56.4 \text{ cps}$$

The value of δ_o obtained is in close agreement with the extrapolated value -228 cps.

As a by-product of our work we also obtained the concentration dependence of J_{BF}^{19} . The chemical shift change between ion pair and free ion solvated only by water is therefore 385 cps. This is presumably on any one F atom $1/4$ of the total effect since any one of 4 atoms can be associated with a sodium ion. Thus the chemical shift change on one fluorine atom should be 1540 cps.

Dr. Barry Shapiro

-2-

19th July, 1963.

We have not yet processed our data for the following salts KF , NH_4F , KHF_2 and NaF but the chemical shift measurements should obey the same relationships. We have not used any other salts to investigate the effect of other ions on the F^{19} chemical shifts, but it seems that this should also be possible. The problem of distorting the spherically symmetric F^- ion in the electric field of the ion pair might yield some theoretical results. At present we can only note that there are remarkable line width changes with concentration changes in the F^{19} resonance of NH_4F , KHF_2 and KF solutions. This suggests the use of our spin-echo spectrometer to measure relaxation times at varied temperature and concentration.

Yours sincerely,

*Leonard W. Reeves.**Rizwanul Haque*

Leonard W. Reeves

Rizwanul Haque

LWR/amm



UNIVERSITY OF CALIFORNIA

DEPARTMENT OF CHEMISTRY
LOS ANGELES 24, CALIFORNIA

July 22, 1963

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

Dear Barry,

In a recent letter,¹ Reeves and Wells report that they found the coupling constants in $(\text{CH}_3)_2\text{SnH}_2$ to be 3% lower than those reported by us.^{2,3} We were induced therefore to recheck our measurements upon the receipt of a Hewlett-Packard 5232L Electronic Counter (not available for our previous studies). At the present, we report on our results for CH_3SnH_3 , which we happened to have on hand. However, as our measurements on the series of methylstannanes were obtained using identical techniques throughout, and the precision of the measurements within the series was checked by comparing different members of the series in the same sample tube, we believe the measurements now presented for CH_3SnH_3 also serve to check those formerly reported for the entire series, and for $(\text{CH}_3)_2\text{SnH}_2$ in particular.

Using the new electronic counter to calibrate the side bands as obtained before from a Hewlett-Packard 200D audiofrequency oscillator coupled to a Varian A-60 Spectrometer, we have obtained the following values:

for CH_3SnH_3	<u>J</u>	<u>c.p.s. \pm 2 c.p.s.</u>
	$^{119}\text{Sn-H}$	1,854
	$^{117}\text{Sn-H}$	1,771

These are in excellent agreement with our previously reported results,³ and confirm that the coupling constants published are accurate to 1% or better, as claimed

Sincerely yours,

- 1) MELLONMR No. 57 (1963).
- 2) MELLONMR No. 53 (1963).
- 3) J. Am. Chem. Soc., 85, 1377 (1963).

Herbert Kaesz *N. Flitcroft*

Herbert Kaesz and Neville Flitcroft

LABORATOIRE DE SPECTROSCOPIE HERTZIENNE

ANNEXE DU

LABORATOIRE DE RECHERCHES PHYSIQUES A LA SORBONNE

1, RUE VICTOR COUSIN, PARIS V*

1148/2/RF/MNF

PARIS, LE 19 juillet 1963

ODÉon : 24-13 POSTE 263

Dr. B. L. SHAPIRO
MELLON INSTITUTE
4.400 Fifth Avenue
PITTSBURGH 13,
Pennsylvania, U. S. A.

Dear Dr. SHAPIRO,

I wish to acquaint you with the details of a technique perfected by Messrs. E. DAYAN and G. WIDENLOCHER at the Laboratory of High Pressure (Director Pr. VODAR), and at my own laboratory. This technique enables the study of gas under pressure in Nuclear Magnetic Resonance.

To date two methods were used :

- a) The gas was condensed at a temperature inferior to that of liquefaction, then the sample was sealed (1) (2).
- b) The gas was obtained under pressure by a chemical reaction occurring in the sealed tube (3).

The pressure of the gas was only known within a 10% margin ; moreover, the same tube could only be used for one value of pressure, therefore necessitating correction, as will be shown further on.

In order to avoid the above inconveniences, they have studied an apparatus (fig. 1) composed of a pyrex tube with a flange, capped with a needle valve made of non-magnetic metal (titanium, beryllium copper).

The above apparatus offers the following advantages :

- A. Systematic study of the mechanical resistance of the pyrex tubes of different interior diameters in function of the pressure, in particular, determination of the average pressure of explosion (°).
- B. Easy adaptation to an instrument fitted with taps and a manometer (fig. 2) ; thus the pressure can be read precisely.

The gas used is either obtained directly from a bottle sold commercially, or from a compressor for the higher pressures.

C. Because of a reserve enabling the mixture of gases, the perturbations of one gas provoked by another are easily studied.

D. Utilization, within a range of given pressures, of one same pyrex tube (°°)

Yours sincerely,



R. FREYMANN

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(°) Explosion tests were made on pyrex tubes with outside diameters of 5mm :

Inside diameter	3.2 mm ;	explosion pressure	80 \pm 5 bars
" "	2.0 mm ;	" "	250 \pm 10 bars

This preliminary study has enabled a coefficient of security to be established sufficient to undertake experiments in Nuclear Magnetic Resonance with a spectrometer Varian type A 60.

(°°) In the process of measuring, an important reason for possible errors was found : for the same pressure, the resonance signal shifts ($\Delta\delta$) according to the tube used. Further detailed measurements showed that this deviation depends on the bulk susceptibility (χ_v) of the body under study (fig. 3). This can be attributed to a bad gauging of tube used or to irregular thickness of the glass. The tubes supplied by Varian Associates were used as reference.

+ 2 figures

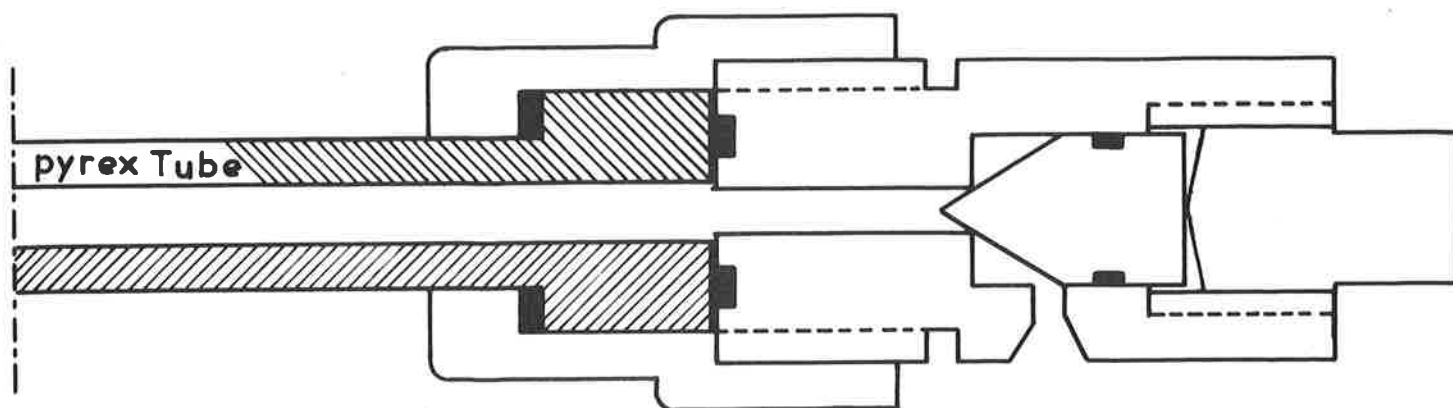


Fig. 1

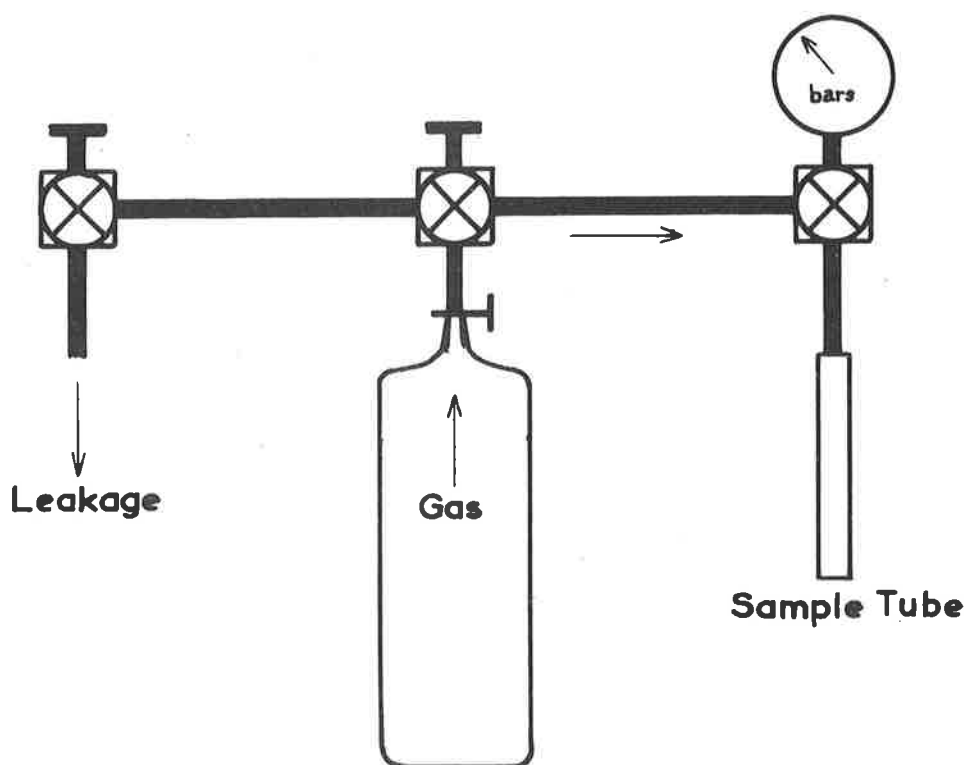
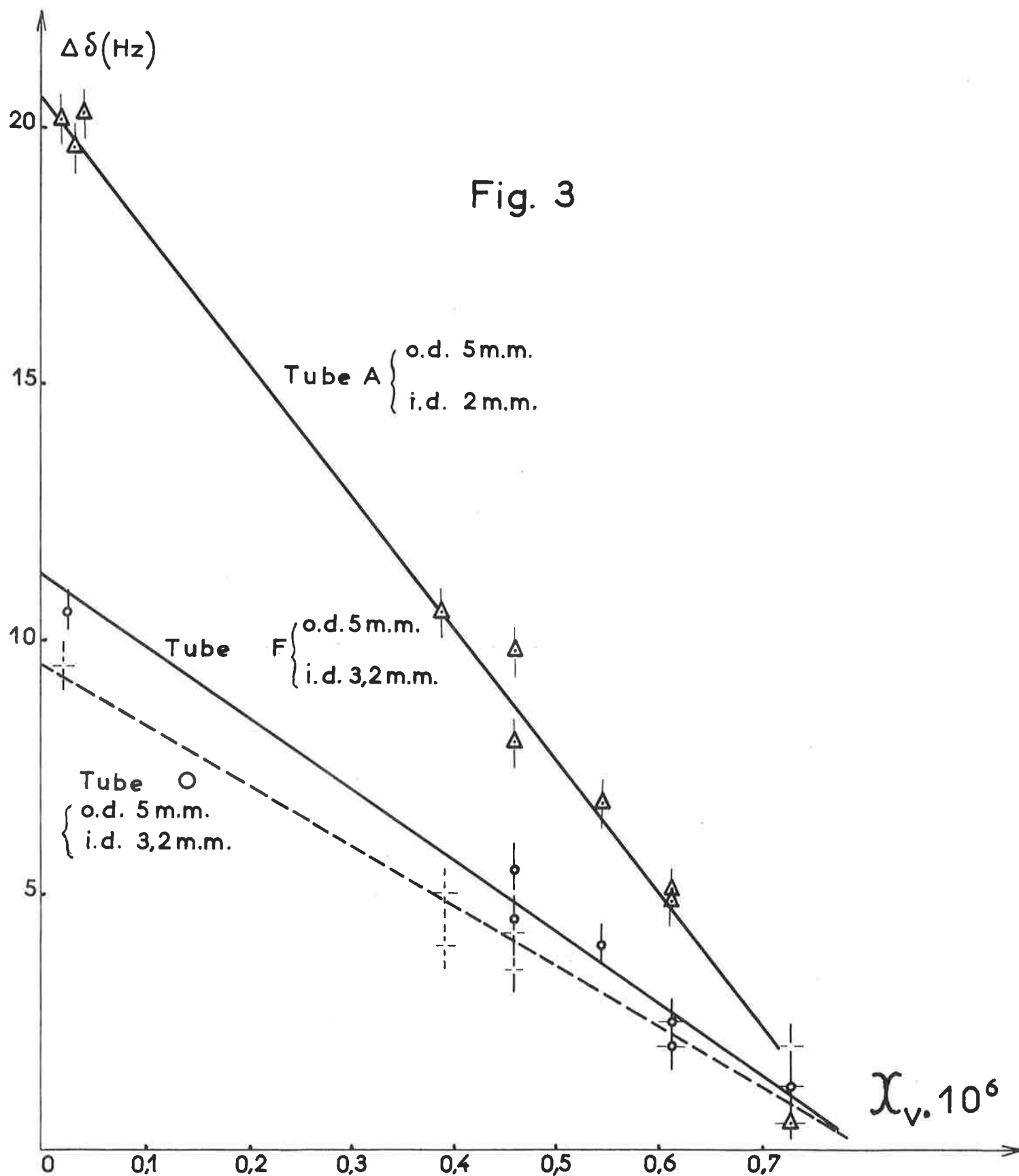


Fig. 2



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