

Joseph B. Lambert

Illinois  
Institute of  
Technology  
N - M - R  
Newsletter

No. 96  
SEPTEMBER, 1966

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Deadline Dates: No. 97 - 15 October 1966 No. 98 - 15 November 1966
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A monthly collection of informal private letters from laboratories of NMR. Information contained herein is solely for the use of the reader. Quotation is not permitted, except by direct arrangement with the author of the letter, and the material quoted must be referred to as a "Private Communication".

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STANFORD UNIVERSITY  
STANFORD, CALIFORNIA

DEPARTMENT OF CHEMISTRY

August 5, 1966

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

Dear Professor Shapiro:

Suggested Title: Variable Temperature  
Broadline NMR

We are using a system as shown in the figure for doing broadline variable temperature work. The heart of the system is the sintered glass cap that fits atop the broadline dewared insert. A thermocouple well is set in the cap, and the ends of a "U" of 2 mm. glass tubing lead out through the cap to a standard taper joint on one end and an exhaust tube on the other end. The sample is poured directly into the insert and the cap put on. To heat the sample, hot air is passed through the glass "U" tube, and to cool the sample cold nitrogen gas is passed through the tube.

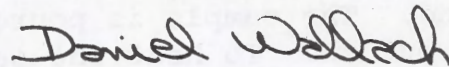
To achieve extra stability, we are also using the Varian V4341 high resolution variable temperature system. An aluminum sheet is mounted on the probe, on the sheet is set a block of bakelite, and on the bakelite is set a piece of glass tubing with standard taper joints on both ends and a "T" joint near one end that serves as a gas inlet. The Varian heater-sensor unit is placed in the glass tubing and held in place by an aluminum "L" placed snugly against it and fastened to the aluminum sheet. All exposed glass is insulated with glass wool.

For high temperature work air is blown into the "T" joint and is heated to the temperature set on the Varian control unit. The actual sample temperature is measured by a thermocouple in the thermocouple well. Temperatures up to 140°C have been achieved. About 40 minutes is required to achieve equilibrium at a new temperature. The temperature is then constant to about  $\pm 1^\circ$  C over a day and is more stable over a shorter period.

For low temperature work nitrogen gas is first passed through a "U" of copper tubing immersed in liquid nitrogen and then heated back up to the desired temperature. We have not done much low temperature work with this system and so far temperatures only down to  $-20^{\circ}\text{C}$  have been reached and held constant. One problem with the system is the tendency of the nitrogen gas to condense into small droplets in the line and cause large variations in gas pressure and therefore sample temperature. The main advantages of this system are the temperature control and stability, the ease of assembly, and the possibility of using large sample volumes.

I hope this method is of use to others doing broad line work. It is particularly useful for those who have access to a Varian high resolution temperature control unit.

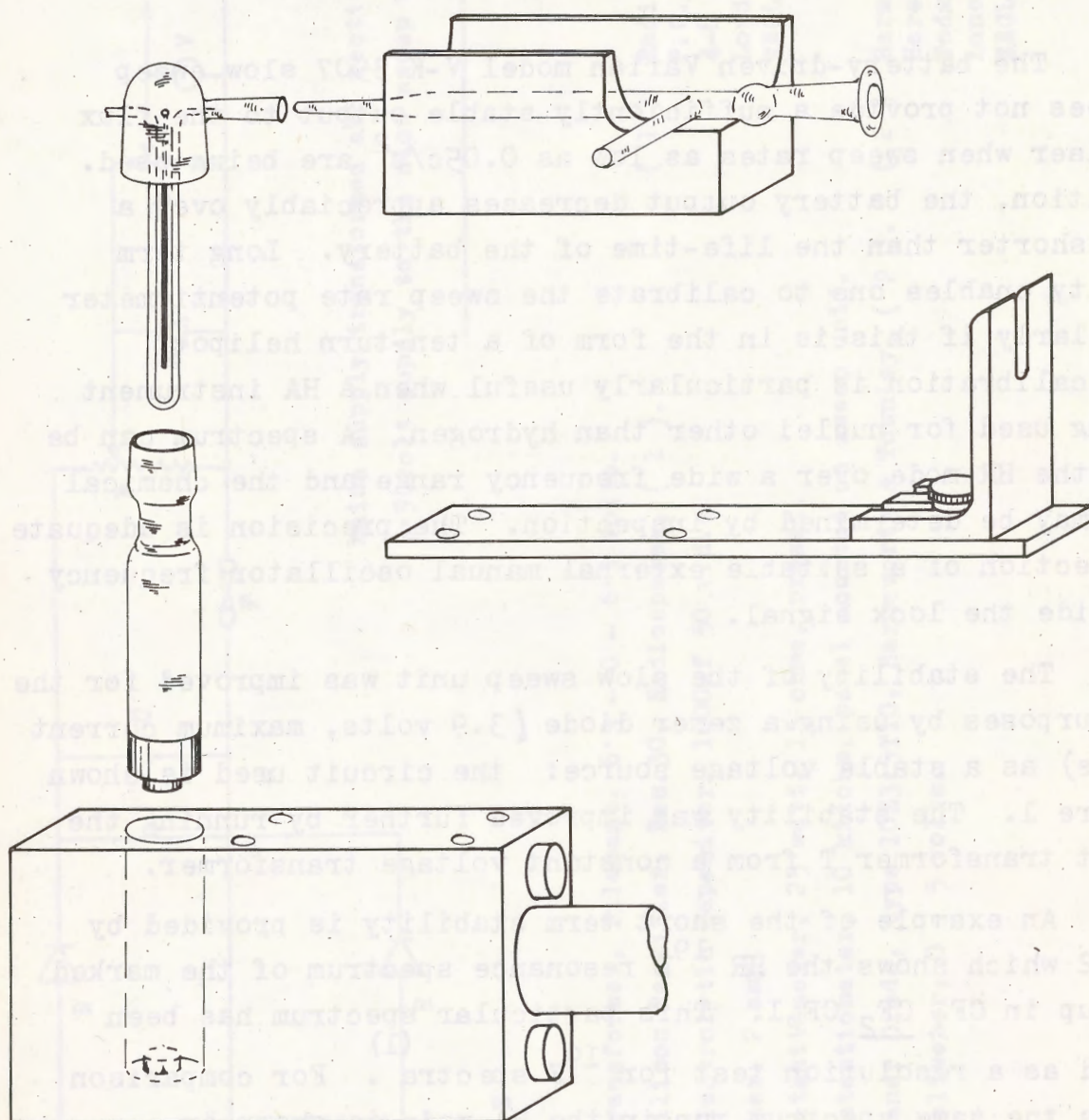
Very truly yours,



DW:la

Daniel Wallach





## MODIFICATION OF SLOW SWEEP UNIT

K.W. Jolley, D. Mallinson, L.H. Sutcliffe and S.M. Walker,  
Donnan Chemical Laboratories, The University, Liverpool.

The battery-driven Varian model V-K 3507 slow sweep unit does not provide a sufficiently stable output to the flux stabiliser when sweep rates as low as  $0.05\text{c/s}^2$  are being used. In addition, the battery output decreases appreciably over a period shorter than the life-time of the battery. Long term stability enables one to calibrate the sweep rate potentiometer particularly if this is in the form of a ten-turn helipot. Such a calibration is particularly useful when a HA instrument is being used for nuclei other than hydrogen. A spectrum can be run in the HR mode over a wide frequency range and the chemical shifts may be determined by inspection. The precision is adequate for selection of a suitable external manual oscillator frequency to provide the lock signal.

The stability of the slow sweep unit was improved for the above purposes by using a zener diode (3.9 volts, maximum current 2.5 amps) as a stable voltage source: the circuit used is shown in figure 1. The stability was improved further by running the filament transformer T from a constant voltage transformer.

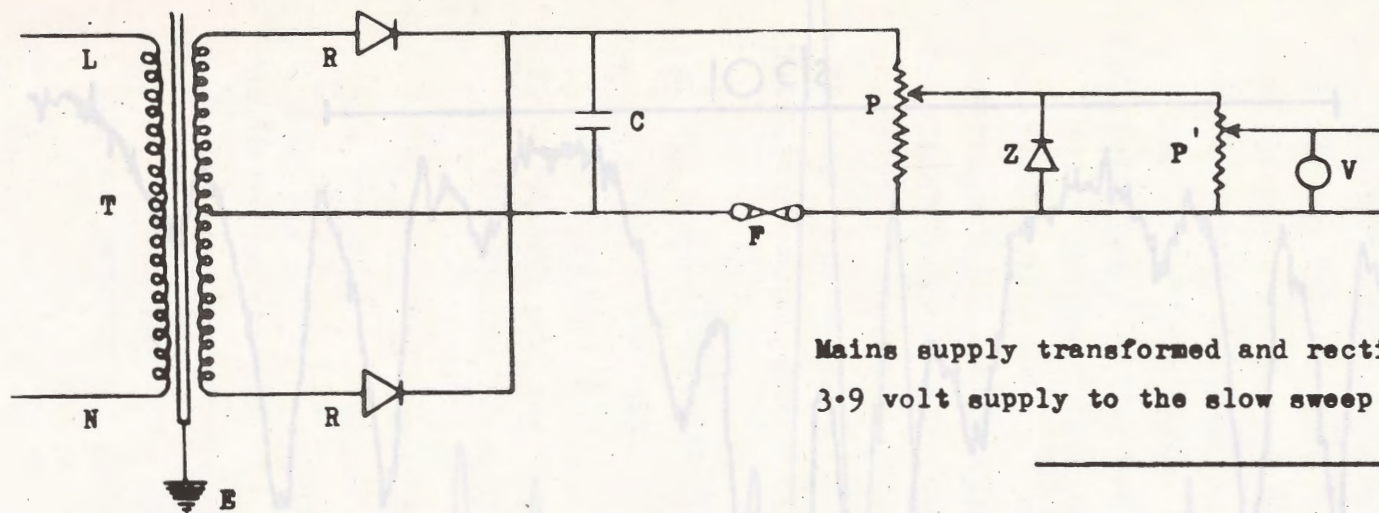
An example of the short term stability is provided by figure 2 which shows the HR  $^{19}\text{F}$  resonance spectrum of the marked  $\text{CF}_2$  group in  $\text{CF}_3\text{CF}_2\text{CF}_2\text{I}$ . This particular spectrum has been proposed as a resolution test for  $^{19}\text{F}$  spectra<sup>(1)</sup>. For comparison purposes the same spectrum run in the HA mode is shown in figure 3.

---

(1) Emsley, Feeney and Sutcliffe, "High Resolution Nuclear Magnetic Resonance Spectroscopy", Volume 1, p. 274, Oxford 1965. Note that Figure 7.6 is marked incorrectly in this reference.



figure 1



Mains supply transformed and rectified for a constant 3.9 volt supply to the slow sweep unit.

KEY.

T - Transformer, filament, 6.3 - 0 - 6.3 volts.

R - Silicon Rectifier, Rec.30, Radiospares, ( 1 ).

C - Electrolytic Capacitor, 1000µf 50 v.d.c.

F - Fuse, 2 amp.

P - Potentiometer, 25 watt, 15 ohms, preset.

P' - Potentiometer, 10 kilohm, panel mounted on sweep unit.

Z - Zener Diode, type 10Z3.9T10, Harmsworth & Townley, ( 2 ).

V - Voltmeter, 0 - 5 volts.

( 1 ) Radiospares  
P.O. Box 268,  
4-8 Maple Street,  
London W.1.  
ENGLAND.

( 2 ) Harmsworth & Townley  
Harehill,  
Todmorden,  
Lancashire,  
ENGLAND.

figure 2

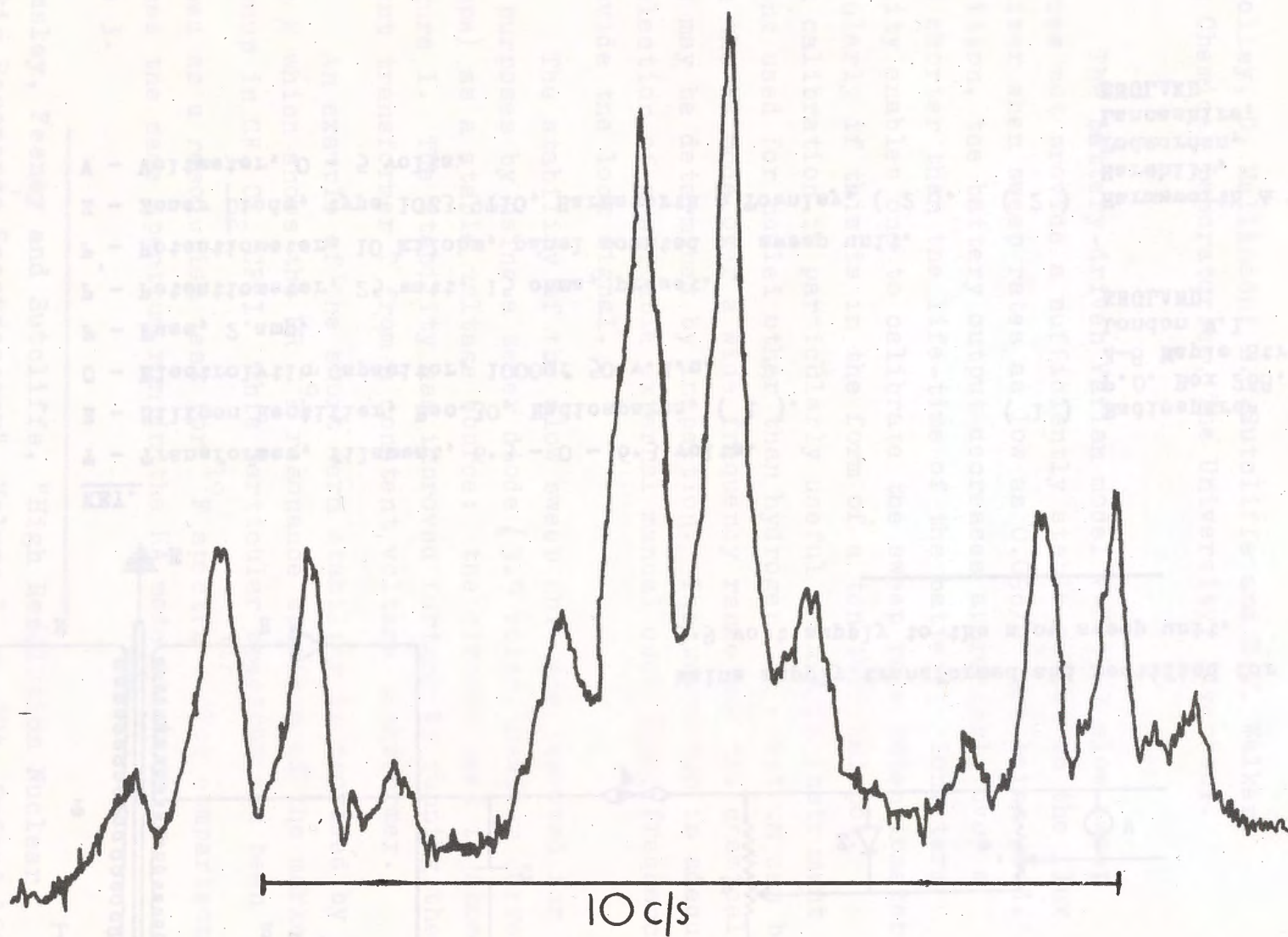
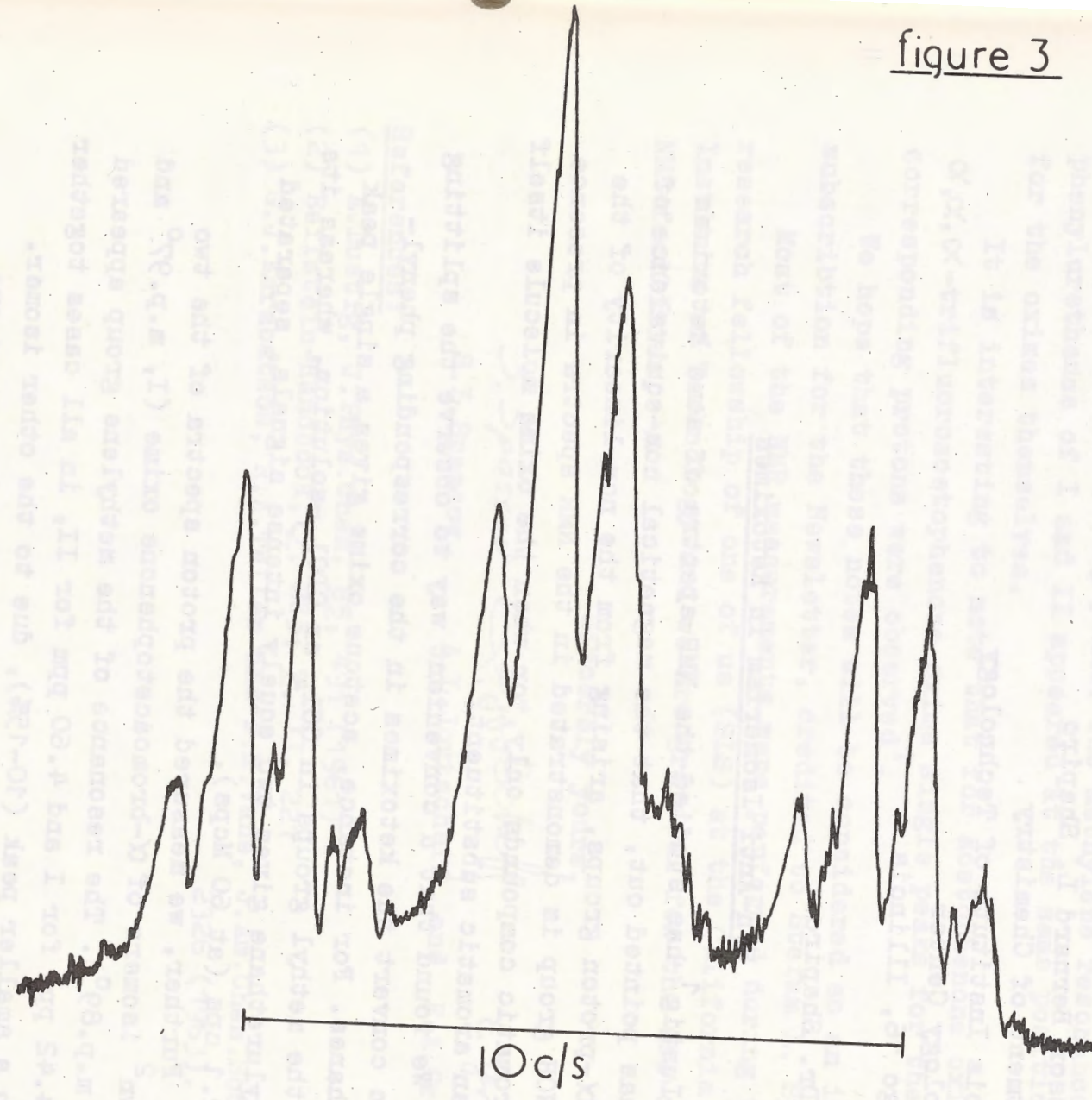




figure 3



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

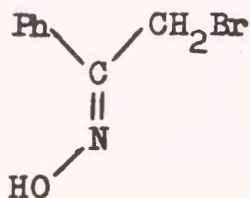
Dear Dr. Shapiro:

Syn-Anti Isomerism in Ketoximes

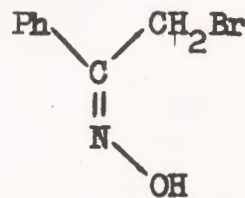
Lustig<sup>1</sup> has studied the NMR spectra of some ketoximes and has pointed out, that the magnetical non-equivalence of the  $\alpha$ -proton groups, arising from the non-linearity of the  $>C=NOH$  group is demonstrated in the NMR spectra in presence of aromatic compounds only, or when the oxime molecule itself has an aromatic substituent.

We found that a convenient way to observe the splitting is to convert the ketoximes in the corresponding phenylurethanes. For instance, acetone oxime gives a single peak for the methyl groups in  $CCl_4$  or  $CDCl_3$ -solution, whereas its phenylurethane gives two equally intense signals, separated by 2.1 cps (at 60 Mcps).

Further, we measured the proton spectra of the two known<sup>2</sup> isomers of  $\alpha$ -bromoacetophenone oxime (I, m.p.  $97^\circ$  and II, m.p.  $89^\circ$ ). The resonance of the methylene group appeared at 4.42 ppm for I and 4.60 ppm for II, in all cases together with a smaller peak (10-15%), due to the other isomer. Assuming that the protons syn to the oxime group are deshielded<sup>3</sup>, we can assign the following structures to the isomers:



I, m.p.  $97^\circ$



II, m.p.  $89^\circ$



Dr. Bernard L. Shapiro

-2-

August 9, 1966

There is no interconversion of the isomers after several hours heating above the melting point. The methylene resonance of the phenylurethanes of I and II appeared at the same positions, as for the oximes themselves.

It is interesting to note that for acetophenone oxime and  $\alpha,\alpha,\alpha$ -trifluoroacetophenone oxime single peaks for the corresponding protons were observed<sup>1</sup>.

We hope that these notes will be considered as an initial subscription for the Newsletter, credited to Stefan L. Spassov.

Most of the NMR measurements were performed during the research fellowship of one of us (SLS) at the California Institute of Technology and we are grateful for the use of the NMR-facilities.

Sincerely yours,

*S. Spassov*      *A.L. Jovtscheff*      *E. Grekova*  
S.L. Spassov,      A.L. Jovtscheff      and      E.A. Grekova

References:

- (1) E. Lustig, J. Phys. Chem. 65, 491 (1961)
- (2) Beilstein-Handbook, 7, 285; 7, II, 221
- (3) G.J. Karabatsos, R.A. Taller and F.M. Vane, J. Am. Chem. Soc. 85, 2326 (1963).

## R I C H F I E L D   O I L   C O R P O R A T I O N

RESEARCH AND DEVELOPMENT • 1900 CRESCENT AVENUE • ANAHEIM, CALIFORNIA 92803 • P. O. BOX 3883

August 12, 1966

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

Dear Dr. Shapiro:

Proton Types of Compounds from Hydrolysis of  
 $\text{AlCl}_3$ -Chloroalkane-benzene Alkylation Complex

We have been interested in identifying the compounds obtained upon hydrolysis of the  $\text{AlCl}_3$  complex obtained after alkylation of benzene with long chain alkyl chlorides. N.m.r. has proved invaluable for this purpose and some of our results are given in the accompanying table. The fraction described was obtained from the hydrolysis products by distillation and liquid chromatography.

Although complete assignment of overlapping multiplets resulting from proton spin coupling is difficult, the proton type distribution is reasonable and with supporting MS, IR and UV data leads to the structure indicated in the table. Low voltage mass spectrometry showed only  $\text{C}_n\text{H}_{2n-14}$  species with  $\text{C}_{17}\text{H}_{20}$  constituting approximately 80% of the fraction under discussion. Thus 1-methyl-6-propyl perinaphthane appears to be a constituent of the complex hydrolysate.

Please credit this letter to the subscription account of Dr. F. F. Caserio, Jr.

Sincerely yours,

ATLANTIC RICHFIELD COMPANY

*N. M. Papatsos*  
*V. G. Dunton*

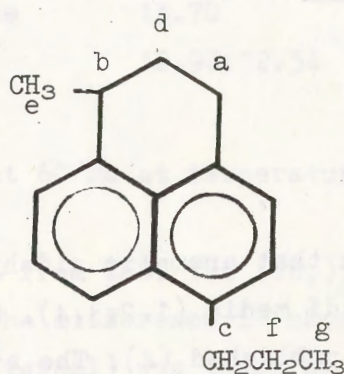
N. M. Papatsos  
V. G. Dunton  
ARCO Chemical Division

FFC:ka



## PROTON TYPE DISTRIBUTION BY N.M.R.

Proton Type	Benzenoid	a,b	c	d	e,f	g
T-Shielding (range)	2-3.2	6.7-7.3	7.3-7.8	8.0-8.5	8.5-8.9	8.9-9.3
Proton Type	4.9	3.0	1.8	2.0	4.8	3.3
Proton Type Distribution	5.0	3.0	2.0	2.0	5.0	3.0
1-methyl 6-propyl perinaphthane						



## ORGANISCH CHEMISCH LABORATORIUM, RIJKSUNIVERSITEIT LEIDEN

Hugo de Grootstraat 25, Leiden

Telefoon 26457

Afdeling voor

Theoretische Organische Chemie

Telefoon 31106

Prof. dr L. J. Oosterhoff

Leiden, August 1966.

nr.:

onderwerp: N.M.R. Spectra of Asymmetrically Substituted  
Benzophenones in  $\text{HSO}_3\text{F-SF}_5$ .

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

Dear Dr. Shapiro,

It is well known that aromatic aldehydes and ketones are protonated in strongly acidic media (1,2,3,4). NMR data of protonated acetophenones have been published (4). The study of the behaviour of aromatic ketones in strongly acidic media was started in our department several years ago (1).

The spectra of p-substituted benzaldehydes show two low field doublets due to the  $=\overset{+}{\text{O}}\text{H}$  proton and the aldehydic hydrogen ( $J=3$  cps). The captured proton in acetophenones and symmetrically substituted benzophenones causes a sharp singlet between 12.50 and 14.00 ppm from TMS, even at temperatures as low as  $-80^\circ\text{C}$ .

However, in the spectra of the ions derived from asymmetrically substituted benzophenones and recorded at sufficiently low temperatures, the  $=\overset{+}{\text{O}}\text{H}$  signal shows as a doublet. The spectrum of



TABLE  
Chemical Shift of the Captured Proton in  
Protonated Benzophenones

Starting compound	$\delta_{=\text{OH}}^*$	Signals
4-Bromobenzophenone	12.60	broadened singlet
4,4'-Dibromobenzophenone	12.47	singlet
3,5-Dibromobenzophenone	13.03; 12.83	doublet
2,6-Dibromobenzophenone	13.67	sharp singlet
2,4,6-Tribromobenzophenone	13.70	sharp singlet
4-Bromo-4'-nitrobenzophenone	13.87; 13.57	doublet
4-Bromo-4'-ethoxybenzophenone**)	13.15; 13.00	doublet
4-Chlorobenzophenone	12.50	broadened singlet
4-Chloro-4'-methoxybenzophenone	13.05; 12.95	doublet
4-Nitrobenzophenone	13.87; 13.50	doublet
2,4,6-Trimethylbenzophenone	13.70	sharp singlet
4-Ethoxybenzophenone**)	12.90; 12.54	doublet

\*) Spectra were recorded at 60 Mc at temperatures between +35° and -85°C.

$\delta_{=\text{OH}}$  in ppm down field from TMS. The  $(\text{CH}_3)_4\text{N}^+$  ion was used as an internal reference. The difference in chemical shift between the reference and TMS (external) was taken as -3.20 ppm.

The shift of the  $=\text{OH}$  proton depends on both the temperature and the acidity of the solvent, therefore no temperatures are given. The compounds were dissolved in mixtures of  $\text{HSO}_3\text{F}$  and  $\text{SbF}_5$ , containing less than 20%  $\text{SbF}_5$  (b.w.)

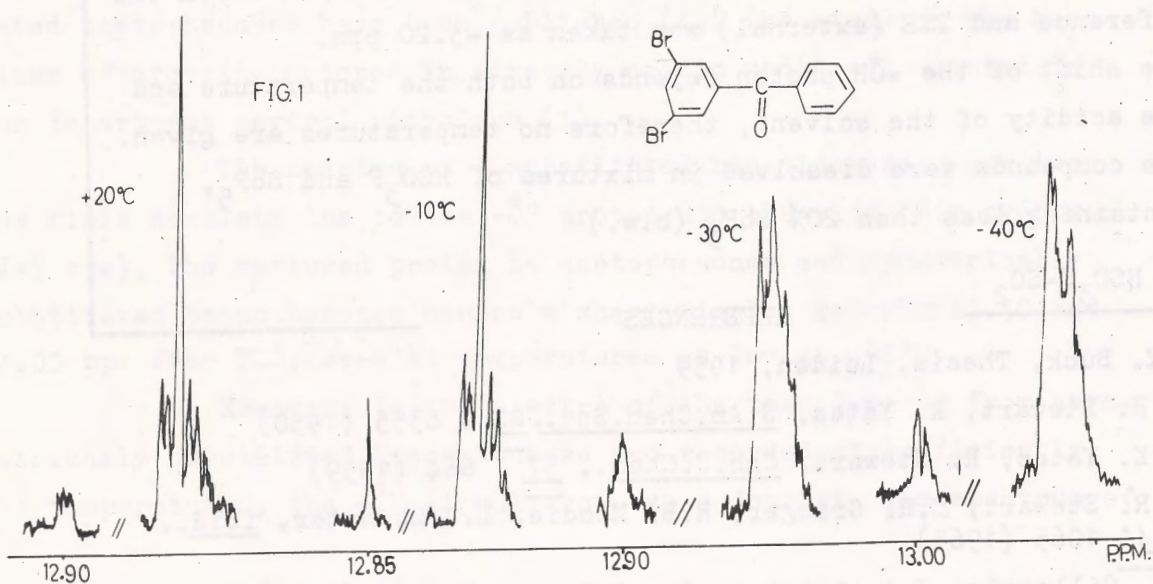
\*\* ) In  $\text{HSO}_3\text{F}-\text{SO}_2$

#### REFERENCES.

- (1) H.M. Buck, Thesis, Leiden, 1959
- (2) a) R. Stewart, K. Yates, J. Am. Chem. Soc., **80**, 6355 (1958)  
b) K. Yates, R. Stewart, Can. J. Chem., **37**, 664 (1959)  
c) R. Stewart, M.R. Granger, R.B. Moodie, L.J. Muenster, ibid., **41** 1065 (1963)
- (3) R.J. Gillespie, J.A. Leisten, Quart. Revs., **8**, 40 (1954)
- (4) T. Birchall, R.J. Gillespie, Can. J. Chem., **43**, 1045 (1965)
- (5) R.N. Jones, J. Am. Chem. Soc., **67**, 2127 (1945)

3,5-dibromobenzophenone e.g., recorded at +20°C, shows an exchange broadened singlet and at -10°C a sharp singlet; at -30°C this signal is broadened and at -40°C a doublet is observed. (Fig. 1). On raising the temperature the two lines merge into a singlet again.

As an explanation for this phenomenon we suggest the presence of an equilibrium between two conformations. In benzophenone the two phenyl groups cannot be coplanar with the carbonyl group simultaneously (5). In the asymmetrically substituted compounds there is competition for coplanarity between the two different aryl systems. In these molecules an equilibrium may occur between two conformations with different NMR spectra. For when the carbonyl group is alternately coplanar with two different aryl systems, two absorptions for the captured proton will be found. The difference in chemical shift between



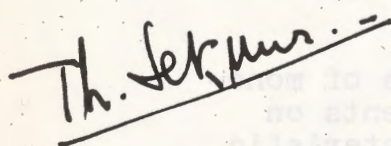


these absorptions depends on the differences between the aryl systems. The differences in intensity of both  $=\overset{+}{O}H$  signals is a measure for the position of the conformational equilibrium and presumably depends on the degree of conjugation.

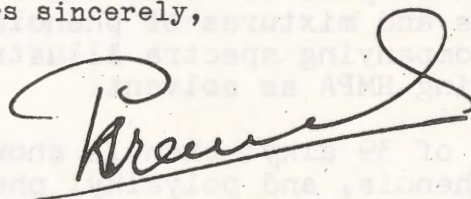
Concurring with the  $=\overset{+}{O}H$  signal, the signals of the phenyl protons broaden. At sufficiently low temperatures the  $=\overset{+}{O}H$  proton shows as a doublet, whereas at raising the temperature this doublet broadens to a singlet and at considerably higher temperatures becomes a sharp singlet. When the  $=\overset{+}{O}H$  doublet is observed the spectrum of the phenyl proton differs, in most cases, from that at higher temperatures. The protonated carbonyl group will exert a different influence on the hydrogen atoms at the ortho positions especially.

Support for our proposed explanation is also found in the fact that 4-bromobenzophenone shows a broadened  $=\overset{+}{O}H$  singlet (only under favourable conditions a doublet) and 3,5-dibromobenzophenone a sharp doublet, whereas the compounds 2,6-dibromobenzophenone and 2,4,6-tribromobenzophenone show sharp singlets. In the latter two compounds the bulky ortho substituents inhibit resonance interaction between the substituted ring and the (protonated) carbonyl group.

Yours sincerely,



Th. J. Sekuur.



P. Kranenburg.



# Monsanto

C O M P A N Y

ORGANIC CHEMICALS DIVISION

1700 South Second Street  
St. Louis, Missouri 63177  
(314) MAIN 1-4000

August 22, 1966

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

## COMPONENT IDENTIFICATION IN PHENOL MIXTURES

Dear Dr. Shapiro:

About a year ago, we began a study of phenolic materials with the aim of developing a procedure for determining the total phenolic hydroxyl content in mixtures. A number of solvents were studied to find one which would shift phenolic OH peaks below the aromatic region through hydrogen bonding. We found that hexamethylphosphoramide (HMPA) gives this shift. In most cases, comparatively sharp hydroxyl peaks are also obtained. Even such highly hindered phenols as 2,6-di-tert-butylphenol give sharp hydroxyl peaks in HMPA below the aromatic absorption region.

Spectra for phenol mixtures in HMPA were found to contain several partially resolved OH peaks. HMPA treated by the procedure included at the close of this letter gave spectra in which many phenol mixtures showed fully resolved OH peaks for each component. This was found to apply to alkyl phenol mixtures and mixtures of phenols with non-alkyl substituents. The accompanying spectra illustrate typical resolving power when using HMPA as solvent.

A study of 39 alkyl phenols showed that the isomers of mono-alkyl phenols, and polyalkyl phenols with substituents on both ortho positions, gave hydroxyl peaks in characteristic chemical shift regions. These correlations are useful in characterizing alkylphenol mixtures. A study of 28 phenols with non-alkyl substituents showed correlations of the hydroxyl chemical shift with the appropriate substituent constant. For meta and para substituted phenols, a plot



Dr. B. L. Shapiro

- 2 -

August 22, 1966

of the hydroxyl chemical shift in HMPA versus Hammett's constant  $\sigma$  was found to be linear. A similar plot for ortho substituted phenols using Taft's constant  $\sigma^*$  was also linear.

This work has been accepted for publication in Analytical Chemistry. Reprints will be sent upon request to those interested in further details.

HMPA Solvent Preparation

1. Distill HMPA under reduced pressure and collect the 50% center cut.
2. Add ca. 25 mg. of Mallinckrodt AR ortho boric acid per 10 ml. of distilled HMPA. Slight changes in the boric acid concentration may be necessary to obtain optimum resolution. We have observed a wide variation in the HMPA impurity levels even from the same supplier.
3. Store over Linde molecular sieve #4A before use.

Yours sincerely,

*Martin W. Dietrich*

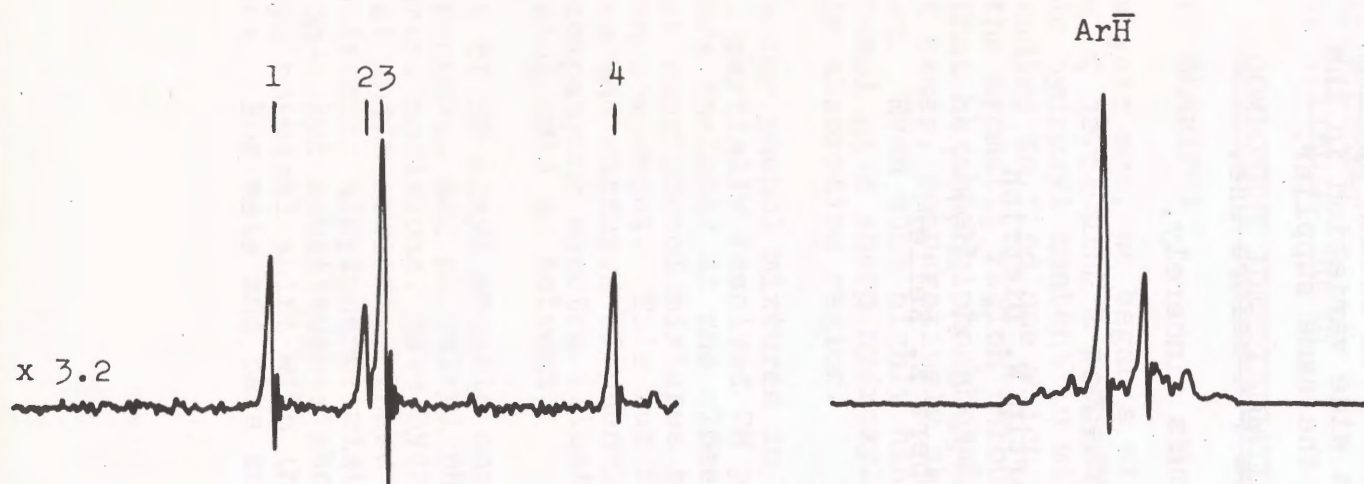
Martin W. Dietrich

*Robert E. Keller*

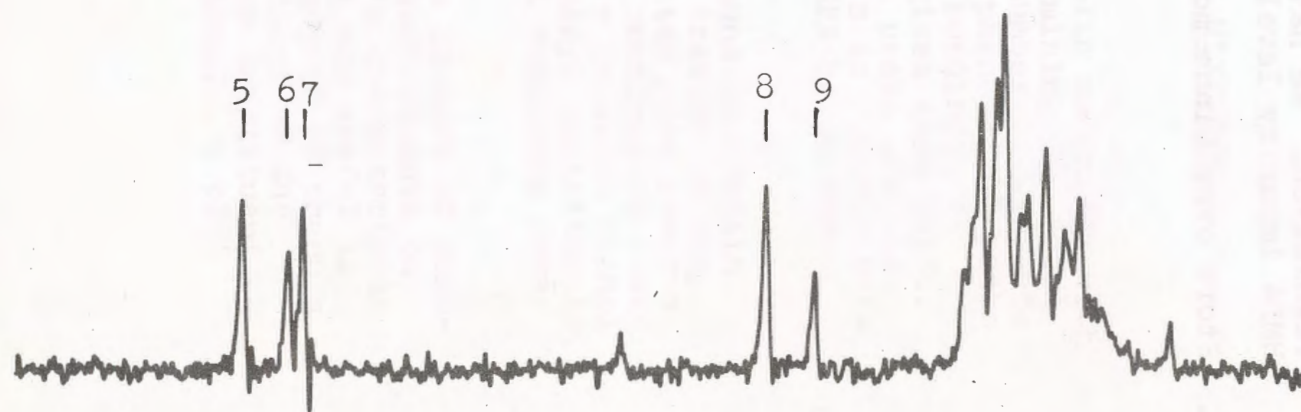
Robert E. Keller

jb

# IDENTIFICATION OF HYDROXYL PEAKS



Hydroxyl Peak	Component
1	3-Methoxyphenol
2	2-Methoxyphenol
3	4-Methoxyphenol
4	2,6-Di-Methoxyphenol



Hydroxyl Peak	Component
5	2-Tert-Butylphenol
6	3-Tert-Butylphenol
7	4-Tert-Butylphenol
8	2,6-Di-Tert-Butylphenol
9	2,4,6-Tri-Tert-Butylphenol

10.0

8.0

6.0

 $\delta$  (PPM to TMS)



## BELL TELEPHONE LABORATORIES

INCORPORATED

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582-3000

August 19, 1966

PROFESSOR B. L. SHAPIRO  
Department of Chemistry  
The Illinois Institute of Technology  
Chicago, Illinois

Dear Barry:

I am enclosing abstracts of two of our recent studies that may be of interest to IITNMR readers. The paper on polytetrafluoroethylene will be given as a talk at Canterbury next month, and we will hope to publish it shortly. The diffusion paper will be given as a talk at the ACS meeting in September, and it has been submitted to the Journal of Physical Chemistry. Complete preprints are available if anyone is interested.

Sincerely yours,

D.W.

D. W. McCALL  
Head,  
Physical Chemical Research  
and Development Department

MH-1513-DWM-LC

Enclosures (2)

## DIFFUSION IN BINARY SOLUTIONS

by

David W. McCall and Dean C. Douglass  
 Bell Telephone Laboratories Incorporated  
 Murray Hill, New Jersey

## ABSTRACT

Experimental self-diffusion results are reported for the binary systems benzene-cyclohexane, acetone-chloroform, acetone-benzene, and acetone-water. Data were recorded as a function of concentration at 25°C, using the NMR spin-echo method. The data are discussed in connection with mutual diffusion results, previously published, with particular emphasis on the equations

$$D = (\partial \ln a_1 / \partial \ln x_1)(x_1 D_2 + x_2 D_1) \quad (\text{Hartley-Crank, Darken})$$

and

$$D = D_1 (\partial \ln a_1 / \partial \ln c_1). \quad (\text{Bearman, Eyring})$$

These equations are qualitatively but not quantitatively descriptive of the experimental data.

A theoretical analysis is presented in which the mutual and self-diffusion coefficients are expressed in terms of integrals of molecular velocity correlation functions. This analysis approaches a molecular view of the three diffusion coefficients and gives some insight into the nature of the Hartley-Crank relationship.



## NUCLEAR MAGNETIC RELAXATION IN POLYTETRAFLUOROETHYLENE

by

D. W. McCall, D. C. Douglass and D. R. Falcone  
Bell Telephone Laboratories, Incorporated  
Murray Hill, New Jersey

## ABSTRACT

Pulse methods have been employed in a nuclear magnetic resonance study of polytetrafluoroethylene.  $T_1$ ,  $T_2$  and  $T_{1\rho}$  ("rotating frame") data have been obtained as a function of temperature. The results are interpreted in terms of molecular motions and correlated with dielectric and mechanical relaxation results. In a general way the correlation is good. The low temperature  $\gamma$  transition is identified as an amorphous phenomenon and is detected in  $T_1$ ,  $T_2$  and  $T_{1\rho}$ . The crystalline transitions near room temperature are observed and evidence is presented for an unusual influence of the crystalline structure on the motions of molecules in amorphous regions. The higher temperature  $\beta$  transition is identified as a crystalline phenomenon. Molecular motional mechanisms are discussed.

96-22

UNIVERSITY OF SOUTH FLORIDA  
TAMPA, FLORIDA 33620

DEPARTMENT OF CHEMISTRY

August 23, 1966

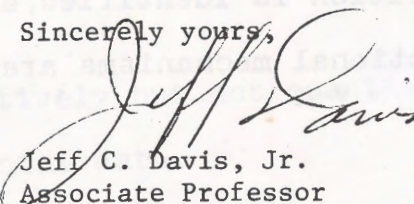
Dr. B. L. Shapiro  
Department of Chemistry  
Illinois Institute  
of Technology  
Chicago, Illinois 60616

Dear Barry,

I have learned recently that we will have funds for a post-doctoral position for the next two years beginning September 1 of this year. Although it is a little late I'd like to appeal to IITNMR readers to have any persons write me at their earliest convenience if they are interested in such a position beginning in September or perhaps January or February. The work will involve NMR, infrared, and precision dielectric constant studies of hydrogen bonding systems.

Thanks.

Sincerely yours,

  
Jeff C. Davis, Jr.  
Associate Professor

JCD:af



ORGANISCH-CHEMISCHES  
INSTITUT  
DER UNIVERSITÄT ZÜRICH

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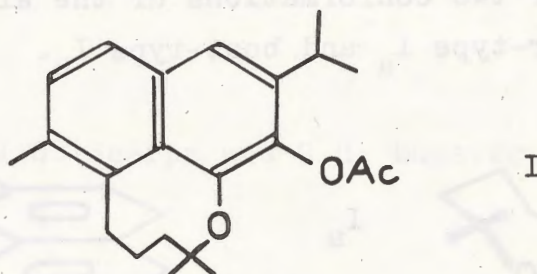
Direktion: Prof. Dr. H. Schmid

August 25, 1966

Non-equivalence of isopropyl methyl groups  
due to inherent molecular dissymmetry

Dear Barry :

Recently C.H. Eugster and coworkers <sup>1)</sup> from this department have observed a non-equivalence of the methyl protons in the isopropyl group attached to an aromatic ring in the following structure



They ascribed the non-equivalence to molecular dissymmetry since the molecule does not contain a chiral atom.

A similar case has been reported by W.D. Ollis and I.O. Sutherland <sup>2)</sup> who studied the ring inversion of tri-o-thymotide.

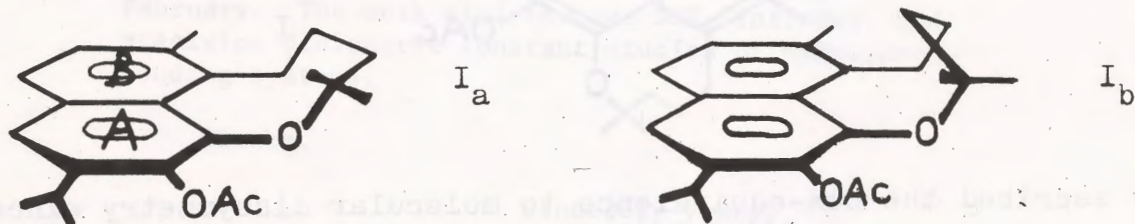
From inspection of a model of I the non-planar eightmembered ring is expected to lead to enantiomeric forms. In agreement with this the two geminal methyl groups show a large difference in chemical shift,  $\Delta\nu = 40.0$  c/s (100 Mc/s) in dichlorobenzene.

We have studied <sup>3)</sup> this novel case by measuring the spectrum of I between  $-75^{\circ}$  and  $+27^{\circ}$  C in carbon disulfide and from  $-15^{\circ}$  to  $+170^{\circ}$  C in dichlorobenzene solution. In particular, the signal of the isopropyl

methyl protons was also observed under double resonance conditions eliminating the coupling to the vicinal methine proton thus producing a doublet for the non-equivalent methyl groups. At room temperature in dichlorobenzene  $\Delta\nu$  is 5.0 c/s.

At  $90 \pm 1^\circ \text{C}$  a collapse of the four lines of the isopropyl methyl protons to a doublet and of the two non-overlapping lines under double resonance conditions to a singlet, shows the interconversion of the two chiral forms (enantiomers) of the molecule. Coalescence of the two geminal methyl groups ( $\Delta\nu = 40.0 \text{ c/s}$ ) is observed above  $150^\circ \text{C}$ .

From the method of Gutowski and Holm applied with appropriate care <sup>4)</sup> before and after the coalescence point we have obtained the energy value of  $14 \pm 2 \text{ Kcal/mole}$  and  $\log A_0 \approx 7.0$  for the exchange process. For each enantiomer two conformations of the eightmembered ring are possible, the chair-type  $I_a$  and boat-type  $I_b$ .



The magnitude and the invariance with temperature from  $-75^\circ$  to  $+70^\circ \text{C}$  of the coupling of the two benzylic protons with the two neighbouring methylene protons is well consistent with the boat-type conformation  $I_b$ . In this conformation the benzylic proton at lower field (387 c/s, and the aromatic ring A) deshielded by the ring oxygen and one vicinal methylene proton are diaxial in agreement with the measured coupling constant of  $J = 13.0 \text{ c/s}$ , the same value as  $J_{\text{gem}}$  for the two benzylic protons. The high field benzylic proton (271 c/s) shows two small  $J_{\text{vic}}$  (5.0 and 1.9 c/s). It is not possible to derive these values from averaged coupling constants due to an interconversion between the chair and

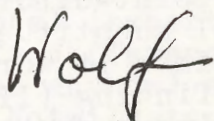


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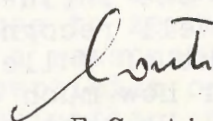
the boat-type conformation. In  $I_b$  the six methylene protons are all staggered and various other factors can be expected to cause the relative stability of the boat-form.

We hope that this contribution will neutralize the terrible but very effective admonition.

Best wishes,



W.v.Philipsborn



F.Conti

- 1) D. Karanatsios, J.S. Scarpa and C.H. Eugster, *Helv.Chim.Acta* 49, 1151 (1966)
- 2) W.D. Ollis and I.O. Sutherland, *Chem.Com.* No. 13, 402 (1966)
- 3) A full paper will be published in *Helv.Chim.Acta*.
- 4) A.Allerhand, H.S. Gutowski, J. Jones and R.A. Meinzer, *J.Amer.chem. Soc.* 88, 3185 (1966)

CHEMISCHES LABORATORIUM  
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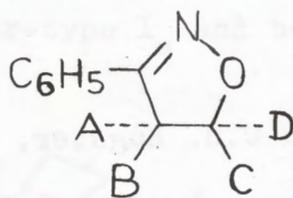
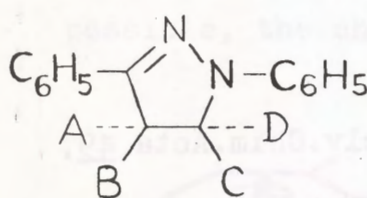
August 29, 1966

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

### A Case for the Point-Dipole Approximation

Dear Professor Shapiro:

The inherent weakness of the point-dipole approximation for predicting the influence of alkyl substituents on proton chemical shifts is well recognized. Sometimes it works, sometimes it doesn't. In the light of Muller's recent finding (IITNMRN 83-2) one might well wonder how much of its seeming success should really be called fortuitous. Be that as it may, we found that a large body of shift differences in substituted 1,3-diphenyl- $\Delta^2$ -pyrazolines (I) and 3-phenyl- $\Delta^2$ -oxazolines (II) could be consistently interpreted along these lines. Typical examples are collected in the table.



- a: A = B = C = D = H  
b: A = B = C = H; D = Me  
c: B = C = H; A = D = Me  
d: A = C = H; B = D = Me  
e: A = B = C = H; D = COOMe  
f: A = B = H; C = Me; D = COOMe  
g: A = C = H; B = Me; D = COOMe

Table. Observed and Calculated Shift Differences (ppm)

	A	B	C
Ib	-0.26	+0.29	+0.71
IIb	-0.26	+0.26	+0.57
calc	-0.16; -0.30	+0.08; +0.14	+0.32; +0.59
Ic		+0.46	+0.40
IIc		+0.30	+0.36
calc		+0.40; +0.73	+0.40; +0.73
Id	-0.05		+0.24
IIId	+0.12		+0.14
calc	+0.16; +0.29		+0.16; +0.29
If	+0.14	-0.17	
IIIf	+0.22	-0.41	
calc	+0.08; +0.14	-0.16; -0.30	
Ig	+0.34		-0.16
IIg	+0.37		-0.38
calc	+0.32; +0.39		-0.16; -0.30



The observed shift differences of Ib,c,d and IIb,c,d are relative to Ia and IIa, respectively; those of If,g and IIg relative to Ie and IIe, respectively. Two sets of calculated numbers are given. Both are obtained by considering only the CC-bond anisotropy and by assuming a planar heterocyclic ring. Set 1 is based on  $\chi_T - \chi_L = 5.5 \times 10^{-30} \text{ cm}^3$  (Bothner-By and Naar-Colin; Musher), set 2 on  $\chi_T - \chi_L = 10.0 \times 10^{-30}$  (Reddy and Goldstein).

Agreement between theory and experiment is certainly not overwhelming. In view of the idealized geometry this was not to be expected in the first place. Nevertheless, in all cases it proved possible to correctly account for the direction of the shift induced by the methyl group(s) (except for proton A in compound Id; but there the observed shift difference is so close to zero as to be of no diagnostic value anyhow) and to give a reasonable estimate of its magnitude. Notwithstanding any scepticism one might have regarding the justification of such an approximation, our results lead us to believe that this simple theoretical tool still has its value for the experimentalist and that it deserves to be tested further.

Sincerely yours,

*Reiner Sustmann*

Reiner Sustmann

*Gerhard Binsch*

Gerhard Binsch



## DOW CHEMICAL OF CANADA, LIMITED

SARNIA, ONTARIO

August 29, 1966

Prof. B.L. Shapiro  
Illinois Institute of Technology  
Technological Center  
Chicago, Illinois 60616  
U.S.A.

Dear Prof. Shapiro:

### A Thermometer for Proton Magnetic Resonance Studies of Aqueous Solutions

In a recent investigation of the proton magnetic resonance chemical shifts of aqueous nonelectrolytes between 0 and 30°C using a Varian Associates A-60 spectrometer equipped with a V-6057 thermostat attachment, it was found that the full instrumental accuracy of  $\pm 0.3$  Hz could not be fully utilized due to sample temperature cycling, which led to systematic water proton chemical shift variations of  $\pm 0.7$  Hz. Further, the recommended method (1) of sample temperature determination, by substitution of a methanol standard tube for that of the sample, was of uncertain accuracy due to the considerable time lapse of about 20 minutes between the measurements of the sample and that of its thermally equilibrated methanol thermometer substitute.

To eliminate these uncertainties, without resorting to instrumental modifications, it was required to develop a thermometer, to be contained within the normal 5 mm. sample tube, which would provide a rapid and precise definition of the sample temperature in the coil region. To achieve this with convenience, it was decided that the thermometer temperature should be defined by proton magnetic resonance chemical shift differences of two signals from an external standard, measured in the same scan as the sample signals. With such a thermometer it should then be possible to correct all chemical shifts to a standard sample temperature, thereby removing the parasitic effects of temperature fluctuations.

The study of aqueous solutions required that the region 0 ~~6.0~~ 6.0 p.p.m. should be available for sample investigation, so that the thermometer signals had to be limited to the region 6.0 ~~6.0~~ 8.3 p.p.m. Study of numerous potential liquid mixtures showed that the ternary mixture 3 mole % tetramethylsilane, 61 mole % m-chlorophenol with 36 mole % trifluoroacetic acid provided a suitable spectrum. In this mixture the m-chlorophenol aromatic protons furnish a complex multiplet signal which





- 2 -

changes with temperature but which always contains a strongest sharp signal at  $\delta = 6.86$  p.p.m. which moves little with temperature, while the trifluoroacetic acid exchanges rapidly with the phenolic protons to give a single, sharp, temperature-sensitive peak which varies in the region 7.5-8.3 p.p.m. It should be noted that the temperature sensitive phenolic peak is overlapped by the aromatic ring protons multiplet when no trifluoroacetic acid is present: the composition given is particularly suitable for the temperature range  $-10$  to  $+30^\circ\text{C}$ ; for use in higher temperature ranges additional trifluoroacetic acid can be added to prevent overlapping of the aromatic multiplet by the phenolic-acid peak at the highest temperature.

The thermometer sheath was axially symmetric and thin-walled as cold-drawn from 6 mm. diameter Pyrex tubing. It was filled with the liquid mixture via a fine capillary, then the whole was freeze-outgassed and sealed at its upper end. The final thermometer was a hemispherical bulb of 4.0 mm. diameter at the bottom gently tapering to 3.3 mm. diameter at 6 mm. height, 2.7 mm. at 12 mm., 2.3 mm. at 18 mm., 2.0 mm. at 24 mm., becoming almost parallel at 1 mm. diameter at 100 mm. to the 170 mm. height of the seal. With this tapered thermometer, relative peak height changes of the sample and thermometer signals were readily made by vertical adjustment of the sample tube position. The thermometer locates itself accurately along the sample tube spinning axis by the close fit of the bulb at the bottom and by a centrally pierced Teflon disk surrounding it at the top.

The thermometer was calibrated using the instrument thermostat at 14 temperatures between  $-10$  and  $+30^\circ\text{C}$  by the substitution method using a methanol standard (1), which gave a standard error of  $\pm 0.84^\circ\text{C}$  on a single defined temperature. The thermometer accurately represented the calibration temperature  $t^\circ\text{C}$  by the linear expression.

$$t = 87.961 - 1.0736 (\nu_{\text{OH}} - \nu_{\text{ArH}})$$

in which  $\nu_{\text{OH}}$  and  $\nu_{\text{ArH}}$  are respectively the phenolic and the aromatic ring proton chemical shifts in Hz. The temperature sensitivity of the thermometer is 1.8 times greater than that of the methanol standard, so that with a standard error of  $\pm 0.3$  Hz on the measurement of each peak frequency, a precision of  $\pm 0.4^\circ\text{C}$  should be obtained on a single temperature measurement.

Table 1 shows the standard errors in Hz for single determinations of proton magnetic resonance chemical shifts determined on a series of dilute aqueous solutions using the thermometer between 0 and  $30^\circ\text{C}$ . The first column gives the solute mole percentage, the second the number of measurements of each chemical shift, the third the standard error on a single determination of the methylene chemical shifts (which have small temperature coefficients  $d\nu_{\text{M}}/dt = \pm 0.05$  Hz/ $^\circ\text{C}$ ), the final column the standard error on a single determination of the water proton chemical shift (which has a large temperature coefficient  $d\nu_{\text{W}}/dt = -0.55$  Hz/ $^\circ\text{C}$ ). All peaks were sharp and could be readily measured. The standard error on the methylene proton determinations provides an essentially temperature-independent blank for the overall instrument-measurement accuracy, while



the standard error on the water signal includes the temperature error component  $(d\theta_W/dt) \cdot \sigma(t)$ . It is immediately apparent that the use of the thermometer permits the water signal to be measured with an error of the same magnitude as that for the temperature insensitive methylene groups. The standard error for a single water determination is  $\pm 0.326$  Hz for the set of 104 determinations, while that for a single methylene group determination is  $\pm 0.288$  Hz for the set of 124 determinations: the square root of the variance difference of the water and methylene signals is the temperature error component, from which it is found that the thermometer follows the sample temperature with a standard error  $\sigma(t) = \pm 0.28^\circ\text{C}$ .

Over a nine month period the thermometer liquid changed colour from a light yellow to a dark red-brown, however no change of the PMR spectrum was observed and regular temperature checks by the methanol standard showed that the thermometer calibration was accurately retained. Convenient and successful use of the thermometer in our particular study of dilute aqueous nonelectrolytes for 1000 chemical shift determinations, lead us to believe that this PMR thermometric technique may have more general application to other aqueous and alcoholic solution work.

#### Literature Cited.

1. Varian Associates, Technical Information Publication 87-100-110.

TABLE I

#### STANDARD ERRORS OF METHYLENE AND WATER PMR SIGNALS USING THERMOMETER

Mole % solute	Number of observations	Methylene signal error (Hz)		Water signal error (Hz)
0	18	-		.300
0.10 ethylene oxide	10	.335		.274
4.50 " "	9	.132		.441
10.0 " "	7	.201		.399
0.05 dioxane	14	.163		.342
10.0 " "	8	.153		.266
0.20 tetrahydrofuran	17	$\alpha$ .250	$\beta$ .356	.292
4.50 " "	13	$\alpha$ .433	$\beta$ .240	.344
10.0 " "	8	$\alpha$ .399	$\beta$ .229	.304
Mean of data	-	.288		.326

D.N. Glew.

*H.D. Mak*

D.N. Glew  
H.D. Mak  
J.S. McIntyre  
N.S. Rath

/ers

*J.M. McIntyre**N.S. Rath*





LABORATORIUM  
VOOR  
ORGANISCHE CHEMIE

Dir.: Prof. Dr. F. GOVAERT

GENT, August 30th 1966.

J. Plateastraat, 22

Tel. 23.38.21

Prof. Dr. B. L. SHAPIRO,  
Department of Chemistry,  
Illinois Institute of Technology,

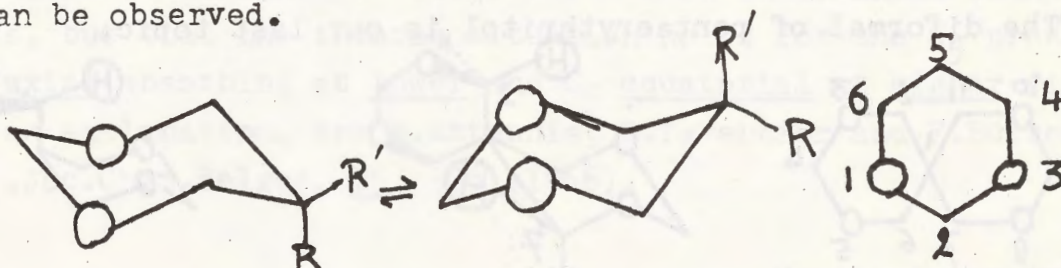
CHICAGO 60616.

Title : Some PMR features of 1,3-dioxanes.

Dear Professor Shapiro,

We are now studying 1,3-dioxanes. Here are four topics for our contribution.

- a) Generally 5,5-dialkyl-1,3-dioxanes can be frozen out and at  $-100^{\circ}$  the superimposed spectra of the two chair forms can be observed.



Two overlapping AB systems are observed for the  $C_4-C_6$  protons if  $R=Me$  and  $R' \neq Me$ , but not if  $R=R'$  or if  $R=Et$  and  $R=n.Bu$ . We think that this results from the different rotation possibilities of an alkyl group in axial and equatorial position, the influence of the alkyl group on the shift of the  $C_4-C_6$  protons then being different. More details will be found in a forthcoming paper (Tetrahedron Letters).

- b) The  $C_4-C_6$  proton pattern of cis 4,6-diisobutyl-1,3-dioxane and of some trans 4,6-dialkyl-1,3-dioxanes is complicated, compared with the more readily interpreted spectra of cis and trans 4,6-diMe-1,3-dioxane. The hindered rotation of the isobutyl side chain may cause a non-equivalence of its methylene protons, thus affecting the  $C_4-C_6$  hydrogens.

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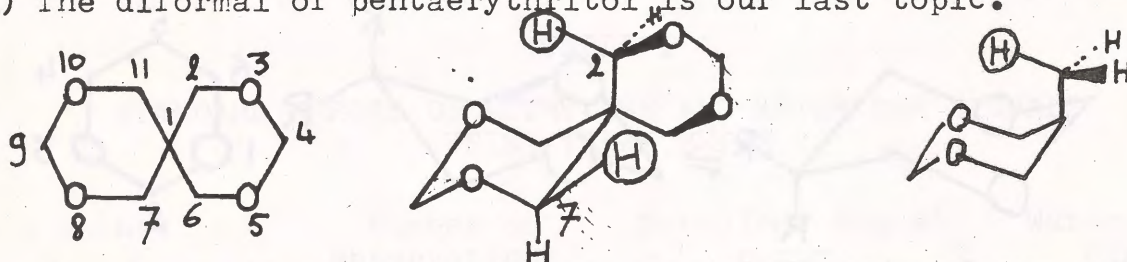


.../...

We will, however, have to wait for our new 100 Mc apparatus to establish this more clearly.

- c) The shift difference of the equatorial and axial  $C_2$  protons  $\delta_{2e} \rightarrow 2a$  of fifteen cis-4,6-dialkyl-1,3-dioxanes (both substituents equatorial) is not constant in the series, but varies with the alkyl groups. A very good correlation was obtained by the equation :  $\delta_{2e} \rightarrow 2a = 17,90 + 1,170 \sum Cno$  (in cps, 56,4 Mc)  $\sum Cno$  is the sum of the number of carbon atoms on the  $\alpha$  carbon atom of the alkyl groups on  $C_4$  and  $C_6$  (i.e.  $\sum Cno = 0$  for  $R = R = Me$  and  $\sum Cno = 6$  for  $R = R = t.Bu$ ). The variation consists mainly in a downfield shift of the equatorial  $C_2$  proton. Recently a similar effect was observed in acetates  $MeC_0OR$ . (See O. Rosado Lojo, C.H.Hancock and A.Danti; J.Org.Chem., 31, 1899 (1966)). Here the number of carbon atoms of the R group contributes significantly to the shift of the methyl group.

- d) The diformal of pentaerythritol is our last topic.



This spiro compound can be frozen out. The measurements are difficult, because the compound crystallizes out very easily. At room temperature, two sharp lines are observed : one for the  $C_4-C_9$  protons  $\tau \approx 4,70$  surface 4 and one for the  $C_2-C_6-C_7-C_{11}$  protons  $\tau \approx 3,65$  surface 8. From the low temperature spectrum it is obvious that two of the eight protons on  $C_2-C_6-C_7-C_{11}$  resonate at rather low field. There result three broad absorption regions (see table).

.../...



.../...

<u><math>\tau</math></u>	<u>Surface</u>	<u>Assignment</u>
$\approx 4,85$	2	$C_4-C_9$ equatorial protons.
$\approx 4,65$	4	$C_4-C_9$ axial protons and $C_2-C_7$ equatorial protons H. The latter hydrogens point directly into the other ring, and are situated above the oxygen atoms.
$\approx 3,35$	6	$C_2-C_7$ axial protons and $C_6-C_{11}$ protons.

We had shown earlier that the shift difference of an axial and equatorial  $C_5$  methyl group is 0.4 ppm, the axial methyl group resonating at lower field ( $\tau \approx 8.85$  ppm) and the equatorial one at higher field ( $\tau \approx 9.25$  ppm). The large shift difference is thus mainly due to the downfield shift of the hydrogen atoms of the axial methyl group, pointing into the 1,3-dioxane ring.

We have also found that the equatorial  $C_2$  and  $C_4-C_6$  protons of 1,3-dioxanes resonate at lower field than their axial counterparts, but that the inverse situation holds for the  $C_5$  protons,  $C_5$  axial absorbing at lower and  $C_5$  equatorial at higher field. For an explanation, see M. Anteunis, D. Tavernier and F. Borremans, Bull. Soc. Chim. Belges, 75, 396 (1966).

Yours sincerely,

D. Tavernier. Assoc. Prof. M. Anteunis

D. Tavernier



ECOLE DE PHYSIQUE  
UNIVERSITE DE GENEVE

Geneva, August 30th, 1966

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BOULEVARD D'YVOY 32  
TEL. (022) 25 22 10  
GENEVE

Dr. B.L. Shapiro  
Associate Professor  
Illinois Institute of  
Technology Chicago  
C h i c a g o 60616

GJB/rf

Dear Dr. Shapiro,

Direct observation of  $C_{13}$ - $H_1$  indirect coupling

in terrestrial magnetic field

With an apparatus based on the free precession spectrometer of Packard and Varian completed by a synchronous detection system and a Fourier's converter we were able to observe directly the spectral lines resulting of  $C_{13}$ - $H_1$  indirect spin spin interaction in diamagnetic mobil liquids at the natural content of  $C_{13}$  nuclei.

In order to have a good signal on noise ratio a mnemotron was used and the spectrum registered on a magnetic tape.

We give here, as an illustration

- ① - a free precession signal obtained with a prepolarisation field of 200 gauss on acidified methanol  $CH_3OH$ .
- ② - the envelope of the preceding signal in which appear the two main time constants of  $C_{13}$ - $H_1$  coupling in  $(CH_3)$  system.
- ③ - the Fourier transformation of this spectrum which gives the four components of high frequency multiplet of  $(C_{13})^3(H_1)^1$  system.

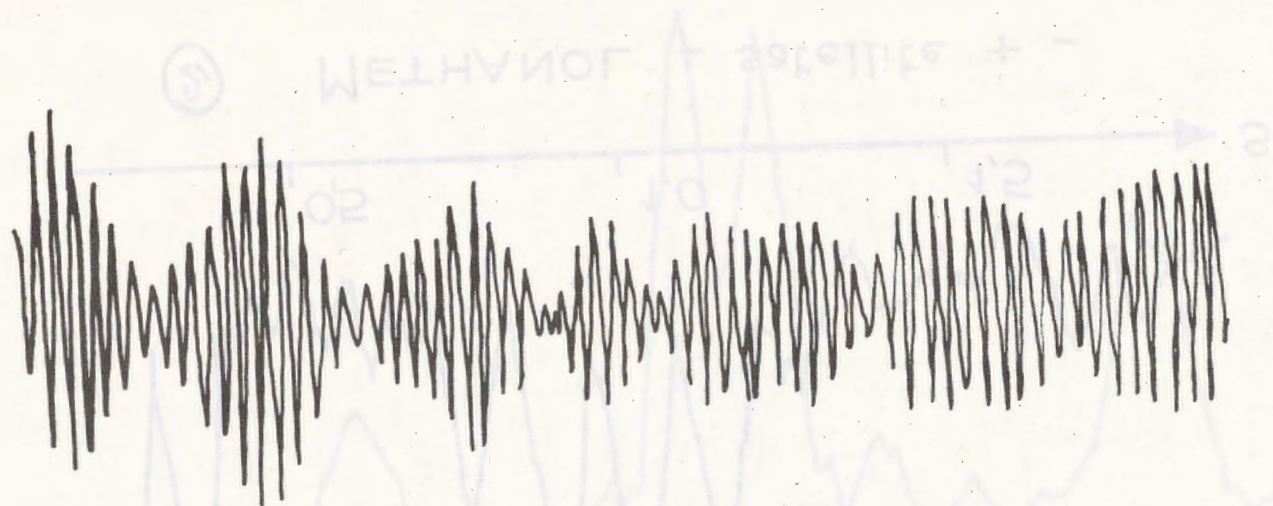
It does not seem too ambitious to work in the future on  $N^{15}$ - $H^1$  J couplings at natural content of  $N^{15}$  nuclei.

Details of these experiments will be presented at the XIV Colloque Ampère Ljubljana (Yougoslavia) September 5th - 10th, 1966 and published in the proceedings of this meeting.

With best regards,

  
Prof. G.J. Béné

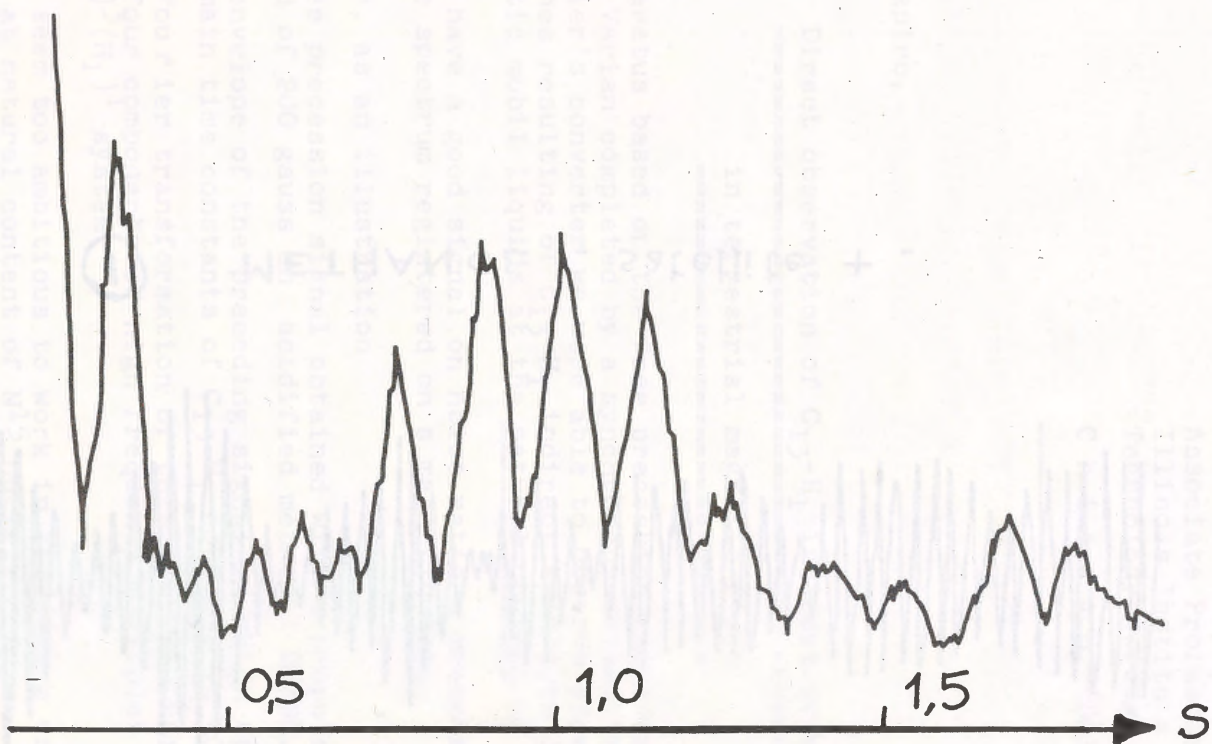




①

METHANOL - satellite + -

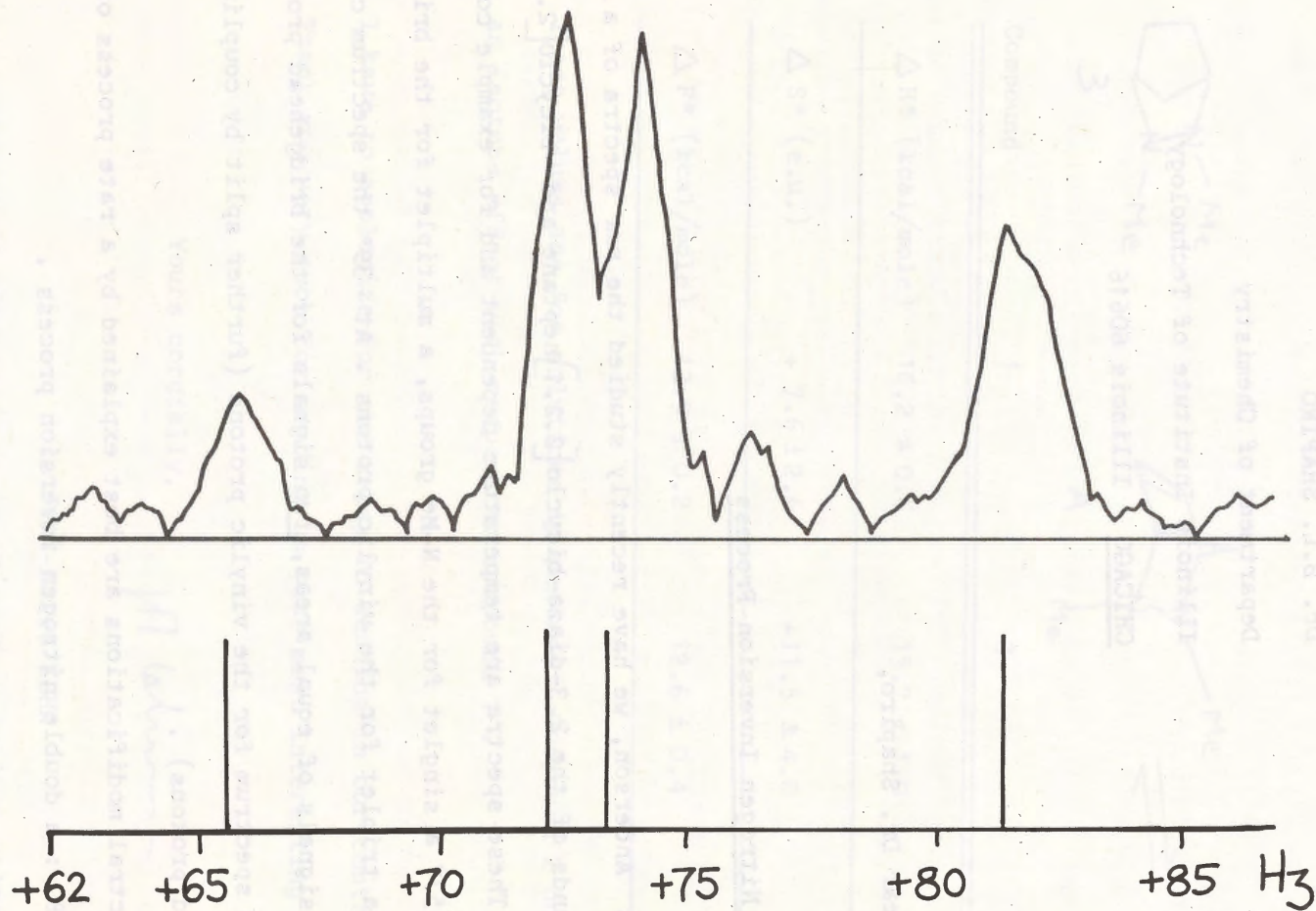
③ CH<sub>3</sub>OH



② METHANOL - satellite + -



③  $\text{CH}_3\text{OH}$  -



UNIVERSITÉ DE STRASBOURG  
Faculté des Sciences

# INSTITUT DE CHIMIE

Boîte postale 296  
Téléphone 36.63.51 à 53

STRASBOURG, le August 31<sup>st</sup> 1966  
1, Rue Blaise PASCAL

Dr. B.L. SHAPIRO

Department of Chemistry

Illinois Institute of Technology

CHICAGO Illinois 60616

Dear Dr. Shapiro,

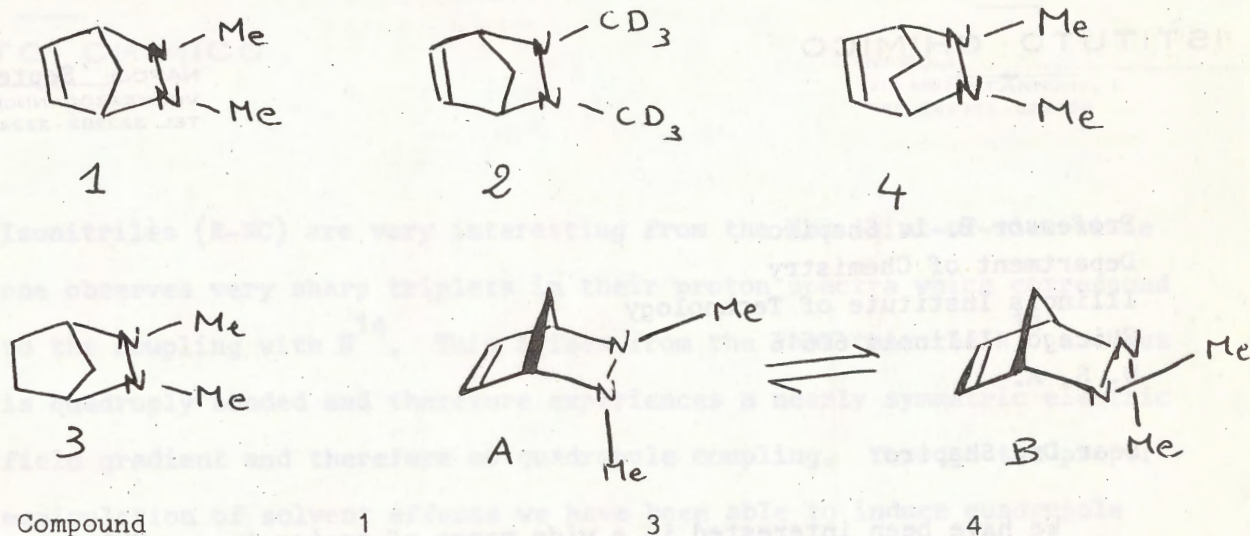
## A Double Nitrogen Inversion Process

With J.E. Anderson, we have recently studied the nmr spectra of a number of compounds of the 2,3-diaza-bicyclo[2.2.1]heptane and -bicyclo[2.2.2]octane series. These spectra are temperature dependent and for example compound 1 shows at 57° a singlet for the N-Me groups, a multiplet for the bridgehead protons and a triplet for the vinylic protons. At -39° the spectrum contains two N-Me signals of equal areas, two signals for the bridgehead protons and an AB spectrum for the vinylic protons (further split by coupling to the bridgehead protons).

These spectral modifications are best explained by a rate process of the type  $A \rightleftharpoons B$  : a double nitrogen inversion process.

The activation parameters have been calculated for the compounds listed and are given below.





Compound	1	3	4
$\Delta H^*$ (kcal/mole)	$16.2 \pm 0.6$	$15.7 \pm 1.0$	$12.4 \pm 0.6$
$\Delta S^*$ (e.u.)	$+7.6 \pm 2.6$	$+11.5 \pm 4.0$	$+2.8 \pm 2.5$
$\Delta F^*$ (kcal/mole)	$13.9 \pm 0.2$	$12.6 \pm 0.4$	$11.8 \pm 0.3$

These values are for solutions in pentane. Compound 2 does not show an isotope effect with respect to compound 1.

I have to apologize for the delay in sending in our contribution to IITNN.

Thank you very much for sending us your most interesting Newsletter.

Yours cordially,

Jean-Marie LEHN

UNIVERSITÀ DI NAPOLI  
ISTITUTO CHIMICO

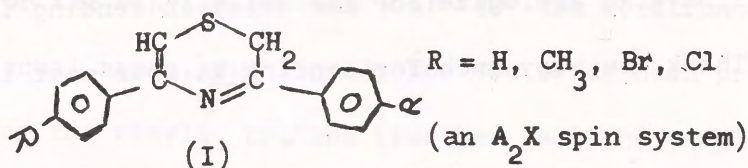
NAPOLI, September 1, 1966  
VIA MEZZOCANNONE, 4  
TEL. 323382 - 323488

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

Dear Dr. Shapiro:

We have been interested in a wide range of topics in our NMR laboratory during the past eight months. Let me now give you a brief summary of some of our current results. I will follow this up with more detailed descriptions of these results after I return from the Colloque Ampere conference in Yugoslavia.

1. We have been able to use NMR to determine the absolute screw sense of some  $\alpha$ -helices in solution. In particular, we have been interested in poly-L-benzylglutamate, poly-L-benzylaspartate, and poly-L-methylaspartate. Measurements of the  $\alpha$ -CH chemical shifts in both helix and random coil proves to be a very clean cut way of determining the helical screw sense. Preprints of this work are available should anyone be interested.
2. We have looked at the NMR spectra of a series of 2H-1,4-triazines(I).



The long range coupling constants show some interesting trends with substitution on the benzene ring (Table I).

Table I

R	$\nu_A$	$\nu_X$	J
—	—	—	—
H	202.2	387.4	1.3
CH <sub>3</sub>	202.3	383.1	1.2
Br	203.0	389.1	1.0
Cl	201.5	386.9	1.0



UNIVERSITÀ DI NAPOLI  
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VIA MEZZOCANNONE, 4  
TEL. 323382 - 323488

3. Isonitriles (R-NC) are very interesting from the NMR point-of-view since one observes very sharp triplets in their proton spectra which correspond to the coupling with  $N^{14}$ . This arises from the fact that the  $N^{14}$  nucleus is quadruply bonded and therefore experiences a nearly symmetric electric field gradient and therefore no quadrupole coupling. Through the proper manipulation of solvent effects we have been able to induce quadrupole coupling and therefore collapse the triplet into a broad singlet (one might call this solvent spin decoupling). We are presently writing this work up for publication and I hope to have preprints available shortly.

I hope this contribution to your very valuable newsletter will bring my subscription up to date.

Sincerely yours,

*James A. Ferretti*  
James A. Ferretti

TITLE: Absolute helical screw senses by NMR; long range coupling constants in substituted thiazines; and solvent spin decoupling in alkyl isonitriles

## LEDERLE LABORATORIES



A Division of AMERICAN CYANAMID COMPANY

PEARL RIVER, NEW YORK 10985

AREA CODE 914 735-5000

August 5, 1966

Dr. B. L. Shapiro  
 Illinois Institute of Technology  
 Technology Center  
 Chicago 16, Illinois

Dear Dr. Shapiro:

An example of deshielding on the Z axis of a double bond

In the course of our n.m.r. work on compounds of the tetrahydro-6,14-endoethenothebaine series<sup>1</sup> (Fig. 1) we had occasion to examine compounds with the C-17,18 double bond and with this double bond saturated.

We readily located H<sub>9a</sub> at  $3.13\delta \pm 0.04$  PPM in structures where the C-17,18 double bond was present. In these compounds where the C-17,18 double bond was saturated, the lines due to H<sub>9a</sub> were found at  $2.65\delta \pm 0.05$  PPM (a  $\Delta\delta$  of  $+0.5$  PPM). These results are in accordance with Jackman's<sup>2</sup> predictions and in agreement with the findings of Yamaguchi<sup>3</sup> and co-workers.

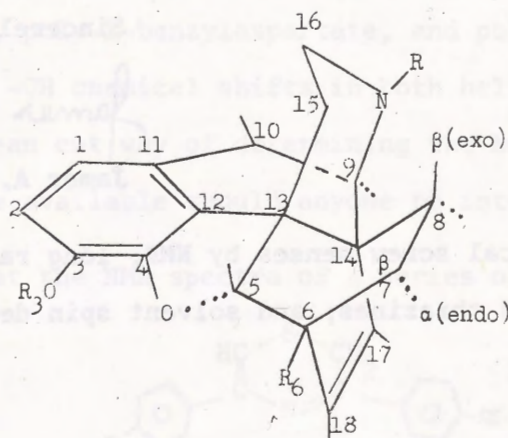


Figure 1

Very truly yours,

*W. Fulmer G. O. Morton*  
 W. Fulmer and G. O. Morton  
 Organic Chemical Research Section

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# VARIAN associates

611 HANSEN WAY

PALO ALTO, CALIFORNIA

326-4000

September 1, 1966

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

Dear Barry,

We have had some success lately in obtaining  $P^{31}$  spectra at 40.5 MHz using the internal lock technique with an HA-100 spectrometer. This success is attributed to the use of  $P_4O_6$ <sup>1</sup> (an intense sharp resonance)<sup>2</sup> as a field-frequency control signal. The  $P_4O_6$  is contained in a sealed melting point capillary of 1.6 mm O.D., 1.2 mm I.D. This capillary is held centered<sup>3</sup> in a standard 5 mm O.D. sample tube and occupies about 15% of the total cross-sectional area.

The  $P_4O_6$  resonance occurs near signals of other trivalent phosphorous compounds and one can use completely normal techniques for recording spectra of these compounds. Signals of many pentavalent phosphorous compounds occur some 100-150 ppm upfield of  $P_4O_6$ . (For example, the signal of 85%  $H_3PO_4$  is 112.5 ppm or 4556 Hz upfield of  $P_4O_6$  at 40.488 MHz.) This presents something of a problem since it requires a sideband frequency for the  $P_4O_6$  lock signal of up to 9 kHz. To get around this problem, it is only necessary to lock to the upper field sideband of the  $P_4O_6$  (as usual) and use the lower field sidebands of the signals of interest. This reduces the lock signal sideband frequency by an amount equal to two times the observing sideband frequency (thus 5-7 kHz) and this brings us back to the standard frequency range of the HA-100 oscillators. However, two things happen, 1) the signals of interest are inverted, and 2) the recorded sweep is reversed (high field at the left, low field at the right). To compensate for these minor difficulties, one inserts the chart paper in the recorder bed upside down and sweeps from right to left.

We have also found it possible to lock to the signal from a capillary of 85%  $H_3PO_4$  but this signal is weaker and being broader does not respond well to the action of the AUTOSHIM\* unit as does the  $P_4O_6$  lock.

- 
1. Available from Gallard Schlesinger Chemical Mfg. Corp., 584 Mineola Ave., Carle Place, L.I., N.Y. 11514, or Albright and Wilson, Ltd., London, England.
  2. A.C. Chapman, J. Homer, D.J. Mowthorpe, and R.T. Jones, Chem.Comm., No. 7, 121 (1965).
  3. Any number of methods may be used - the simplest is to wrap tape around the top of a two-inch capillary until its O.D. is a good fit in the standard 4.2 mm I.D. sample tube. The bottom of the capillary will stay centered in hemispherical bottom of the sample tube.



**VARIAN**  
associatesProf. B.L. Shapiro  
Illinois Inst. of Technology

- 2 -

September 1, 1966

Of course, the main reason for all of this is to obtain linear reproducible sweeps which will allow signal-to-noise enhancement with a time-averaging computer. Figure 1 shows the spectrum of 0.1M ATP at pH 10 obtained with the benefit of the  $P_{406}$  lock. Two hundred scans were accumulated by the C-1024 and the figure shows the final result as well as the single scan input to the C-1024. Theoretical improvement in signal-to-noise was achieved. Since the automated sweeping of the HA-100 recorder runs slow to the right and fast-return to the left, the data were obtained as high to low field sweeps (actually high to low frequency sweeps). The inversion of the signals was reversed by using the SUBTRACT mode of the C-1024 rather than the normal ADD mode. The complete conformist could obtain low to high field sweeps by modifying the recorder sweep switching logic.

By narrowing the sweep width to a 50-100 Hz region of interest, and using a sweep rate of 2 Hz/sec, it should be possible to get useful signals from solutions as dilute as 0.01M (4  $\mu$ moles/0.4 ml.).

Best regards.

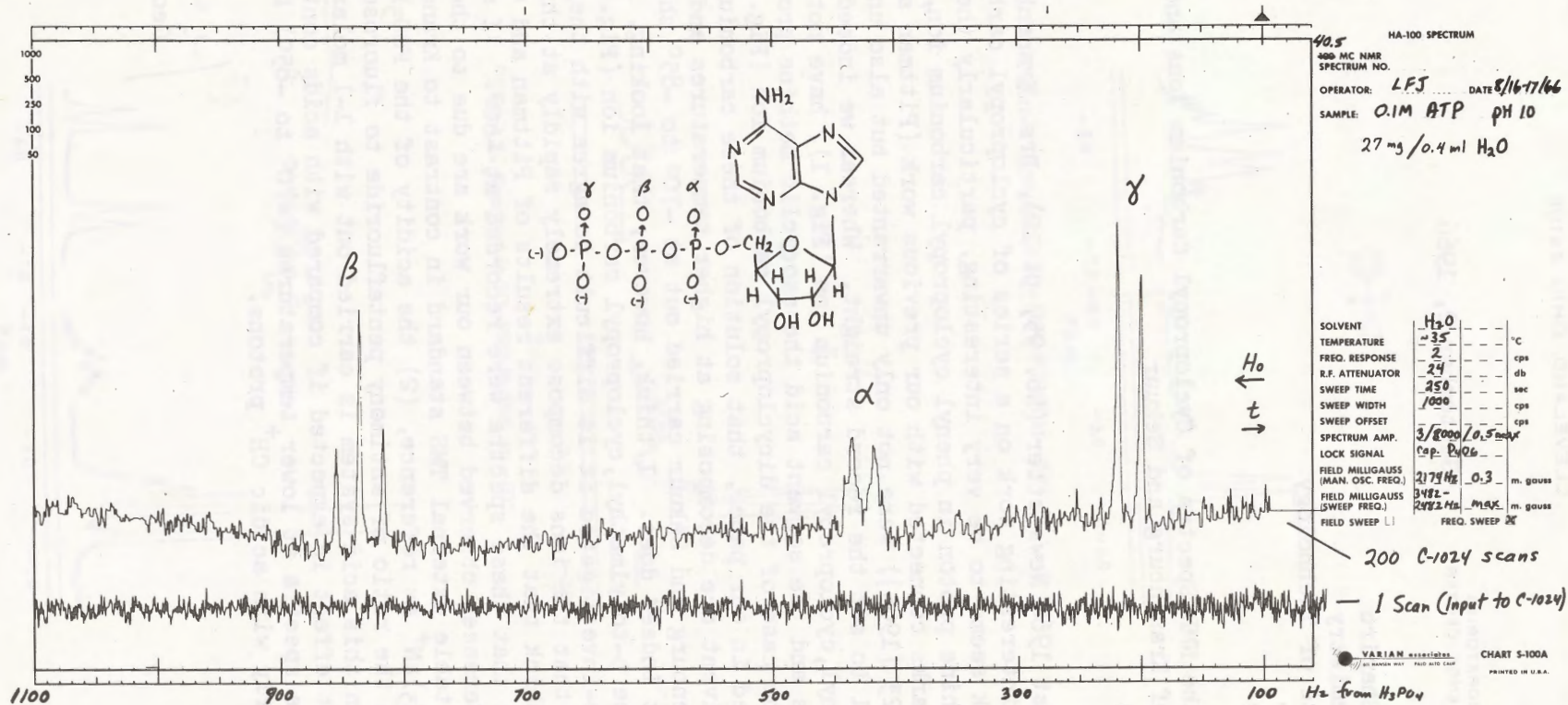
Sincerely yours,

\*Trademark

*Roy*LeRoy F. Johnson, Manager  
Spectroscopy Applications Laboratories  
Analytical Instrument Division  
Varian Associates

LFJ:jls





WESTERN RESERVE UNIVERSITY  
CLEVELAND, OHIO 44106

MORLEY CHEMICAL LABORATORIES  
JOHN SCHOFF MILLIS SCIENCE CENTER

September 2, 1966

TELEPHONE: 231-7700  
AREA CODE 216

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

Remarks on the NMR Spectra of Cyclopropyl Carbonium Ions and the recent  
Newsletter of Kranenburg and Sekuur

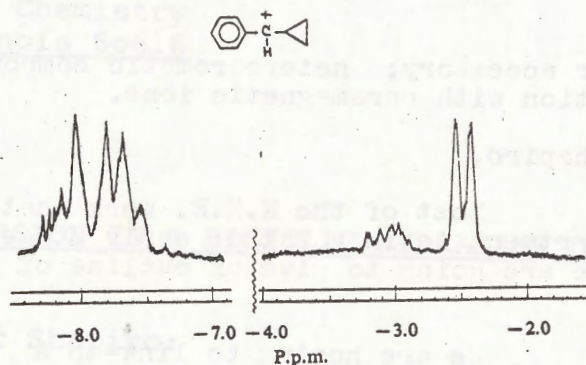
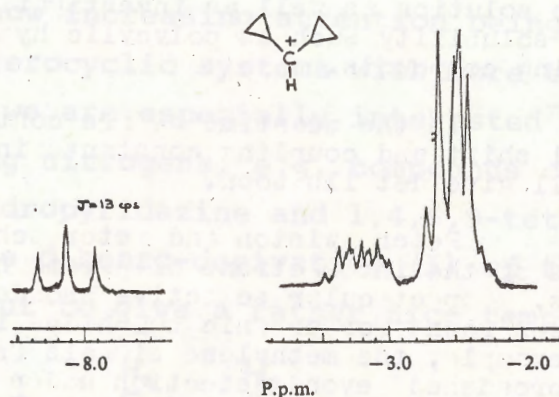
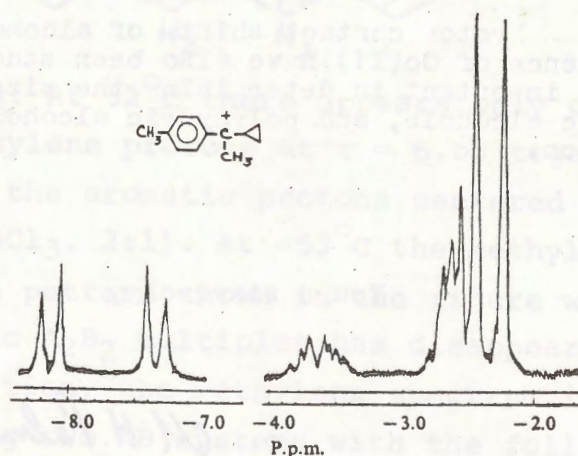
Dear Barry:

In your August 1966 Newsletter (No. 95, p. 12), Drs. Kranenburg and Sekuur reported on some interesting work on a series of cyclopropyl carbonium ions. Whereas their work seems to be very interesting, particularly the observation of the exocyclic methine proton in phenyl cyclopropyl carbonium ion, I feel that some of their remarks connected with our previous work (Pittman and Olah, J. Am. Chem. Soc. 87, 5123 [1965]) were not only unwarranted but also unfounded. I think it would be useful to set the record straight. Whereas we indeed, in the case of the secondary phenyl, cyclopropyl carbonium ion (Fig. 1), have not resolved from the phenyl protons and the solvent acid the exocyclic methine proton, we reported  $J_{HH} = 13$  cps in the case of the dicyclopropyl carbonium ion (Fig. 2). It is obvious, and stated in our paper, that solution of these carbonium ions in our  $\text{FSO}_3\text{H}-\text{SbF}_5-\text{SO}_2$  solvent are decomposing at higher temperatures and therefore the work of Drs. Kranenburg and Sekuur carried out at  $-70^\circ$  to  $-85^\circ$  should show further improvement, as it indeed does. I think, however, that looking, for example, at the spectrum of the p-tolylmethyl, cyclopropyl carbonium ion (Fig. 3) which we reported and they re-investigated it is difficult to agree with their statement, "it is our experience that the ions decompose extremely rapidly at this temperature (e.g.  $-60^\circ$ ). We think that the different results of Pittman and Olah find their origin in the fact that these spectra were recorded at  $-60^\circ$ ." I myself feel much more that the differences observed between our work are due to the fact that (1) we used a less suitable external TMS standard in contrast to Kranenburg and Sekuur using internal  $(\text{CH}_3)_4\text{N}^+$  as reference, (2) the acidity of the  $\text{FSO}_3\text{H}-\text{SbF}_5$  system is quite dependent on the ratio of antimony pentafluoride to fluorosulfonic acid. The bulk of our work in this acid system is carried out with 1-1 molar composition and therefore a solvent effect is expected if compared with acids containing 200%  $\text{SbF}_5$ , and (3) running the spectra at lower temperatures ( $-70^\circ$  to  $-85^\circ$ ) helps to slow down exchange particularly with acidic  $\text{CH}^+$  protons.

*George A. Olah*  
George A. Olah

GAO:lm



Figure 1. Spectrum in  $\text{SO}_2\text{-SbF}_5\text{-FSO}_3\text{H}$  at  $-60^\circ$ .Figure 2. Spectrum in  $\text{SO}_2\text{-FSO}_3\text{H-SbF}_5$  at  $-60^\circ$ .Figure 3. Spectrum in  $\text{SO}_2\text{-SbF}_5\text{-HSO}_3\text{F}$  at  $-60^\circ$ .



DEPARTMENT OF CHEMISTRY

WEST MAINS ROAD

EDINBURGH, 9.

9th September, 1966.

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

Title:- Computer accessory; heteroaromatic compounds;  
interaction with paramagnetic ions.

Dear Professor Shapiro,

Most of the N.M.R. work that is being done in this Department is in a rather early stage of development so we are going to give an outline of several of the topics.

We are hoping to link-up a Northern Scientific NS 54 Computer accessory plus Perkin Elmer interface to our P.A. R10 soon. There seem to be problems of import duty etc. If any readers have experience of this particular set-up or related arrangement we would like to hear from them, so that we can compare notes on any problems that may arise. We are hoping to use the computer for work on chemical shift changes in dilute solution as well as investigations on compounds of low solubility such as polycyclic hydrocarbons and naturally occurring compounds.

In the meantime we are continuing with a study of chemical shift and coupling constants in heteroaromatic compounds and will give details soon.

Peter Swinton and Peter Schwarz are exploring aspects of the interactions of paramagnetic ions with organic molecules. Spectacular selective broadening of proton resonances has been found for organic sulphides in the presence of Cu(II); for example, the methylene signals from diethylsulphide can be broadened beyond detection under conditions which leave the methyl signal as a recognisable triplet. We know of only one previous case in which multiplicity is retained in such circumstances (Johnson and Tully, J. Chem. Phys., 1964, 40, 1744) and would like to hear of others.

Proton contact shifts of alcohols and polyols in the presence of Co(II) have also been studied. Steric hindrance is important in determining the size of the shift for monohydric alcohols, and polyhydric alcohols show evidence of chelation.

Yours sincerely,

*M.H. Palmer.*

M.H. Palmer.



ORGANISCH-CHEMISCHES INSTITUT  
DER UNIVERSITÄT

69 HEIDELBERG, September 12, 1966  
Tiergartenstraße  
Tel. 483/2411  
bei Durchwahl 483/.....

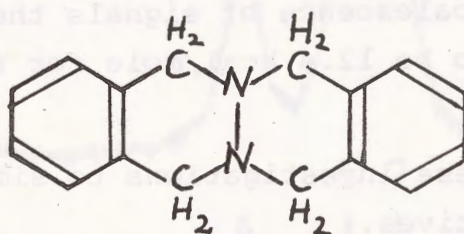
Dr. B. L. Shapiro  
Illinois Institute of Technology  
Department of Chemistry  
Chicago, Illinois 60616

INVERSION IN A BICYCLIC HYDRAZINE DERIVATIVE

Dear Professor Shapiro:

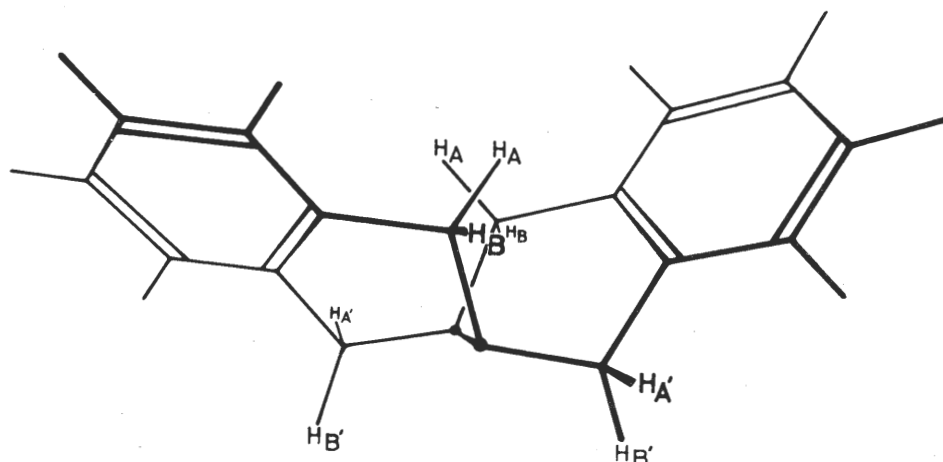
Please excuse that we are such awfully slow payers of our IIT-NMR-Newsletters subscription price. We hope you will accept it without an extra charge.

There is now increasing attention being paid to conformational problems of heterocyclic systems with more than one nitrogen atom. In this field, we are especially interested in compounds with two neighbouring nitrogens, e.g. compounds of the 1,2-dialkyl-1,2,3,6-tetrahydropyridazine and 1,4,6,9-tetrahydro-10-azaquinolizine type. The dibenzo-derivative (I) of the last mentioned system turned out to give a rather nice temperature dependence



I

of NMR spectra: At 32°C there appears only one sharp singlet for the eight methylene protons at  $\tau = 6.03$  together with an  $A_2B_2$  multiplet for the aromatic protons centered around  $\tau = 2.11$  (in  $d_5$ -pyridine/ $CDCl_3$ , 2:1). At -53°C the methylene absorption is changed to the pattern shown in the figure whilst the symmetry of the aromatic  $A_2B_2$  multiplet has disappeared. As indicated by the reconstruction, the methylene spectrum is very well accounted for by assuming two AB systems with the following parameters:  $\tau_A = 6.51$ ,  $\tau_B = 6.22$ ,  $\tau_{A'} = 6.22$ ,  $\tau_{B'} = 5.22$ ,  $J_{AB} = 17$  cps,  $J_{A'B'} = 17$  cps. This points to a structure with a cis-a,e-linkage of the two heterocyclic rings:



The assignment of the protons is based on an approximate evaluation of anisotropic effects of aromatic rings and nitrogen lone pairs. By simple ring inversion only the two groups of four protons cis to each other ( $H_A$ ;  $H_{A'}$  and  $H_B$ ;  $H_{B'}$ , resp.) might become equivalent. The observed equivalence of all eight methylene protons needs additional inversion on the nitrogen bridgeheads to occur. Formally, inversion on one of the nitrogens results in a more strained trans-e,e-system. By simultaneous or subsequent inversion on the second nitrogen a cis-a,e-structure is restored resulting in an interchange of the corresponding A- and B-protons. Careful analysis of NMR spectra in the intermediate temperature range gave no evidence for the ring inversion and nitrogen inversion processes to occur independently. From the coalescence of signals the free energy of activation is estimated to be 12.4 kcal/mole for the combined processes.

We hope to extend these investigations to similar mono- and bicyclic hydrazine derivatives.

Sincerely yours

*B. Junge*

Bodo Junge

*H. A. Staab*

Heinz A. Staab



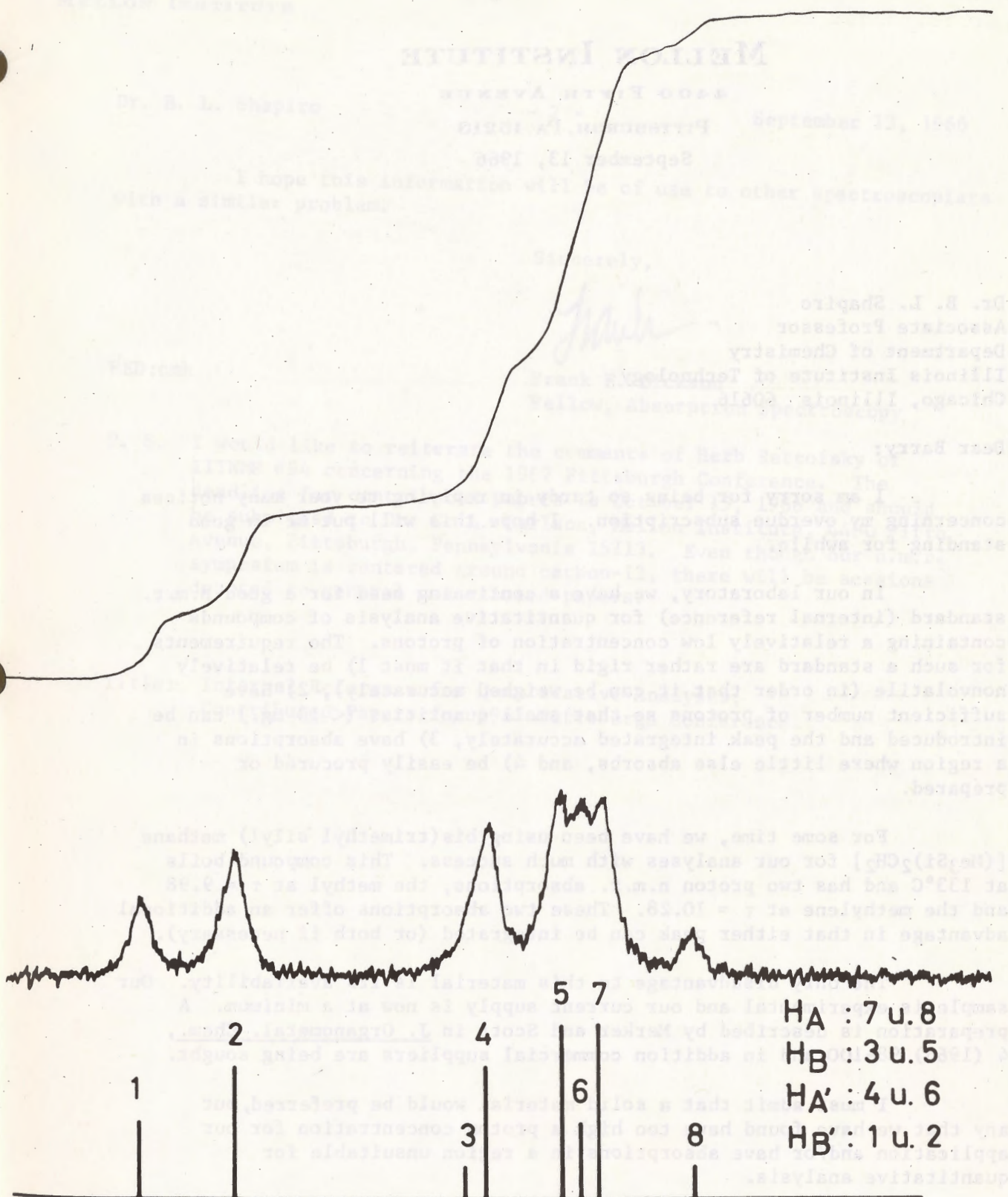


Fig.: NMR of methylene protons of compound I  
(-53°C, d<sub>5</sub>-pyridine/CDCl<sub>3</sub>, 2:1)

## MELLON INSTITUTE

4400 FIFTH AVENUE

PITTSBURGH, PA. 15213

September 13, 1966

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

Dear Barry:

I am sorry for being so tardy in replying to your many notices concerning my overdue subscription. I hope this will put me in good standing for awhile.

In our laboratory, we have a continuing need for a good n.m.r. standard (internal reference) for quantitative analysis of compounds containing a relatively low concentration of protons. The requirements for such a standard are rather rigid in that it must 1) be relatively nonvolatile (in order that it can be weighed accurately), 2) have sufficient number of protons so that small quantities (< 10 mg.) can be introduced and the peak integrated accurately, 3) have absorptions in a region where little else absorbs, and 4) be easily procured or prepared.

For some time, we have been using bis(trimethyl silyl) methane  $[(\text{Me}_3\text{Si})_2\text{CH}_2]$  for our analyses with much success. This compound boils at  $133^\circ\text{C}$  and has two proton n.m.r. absorptions, the methyl at  $\tau = 9.98$  and the methylene at  $\tau = 10.28$ . These two absorptions offer an additional advantage in that either peak can be integrated (or both if necessary).

The only disadvantage to this material is its availability. Our sample is experimental and our current supply is now at a minimum. A preparation is described by Merker and Scott in J. Organometal. Chem., 4 (1965) 98-100 and in addition commercial suppliers are being sought.

I must admit that a solid material would be preferred, but any that we have found have too high a proton concentration for our application and/or have absorptions in a region unsuitable for quantitative analysis.



MELLON INSTITUTE

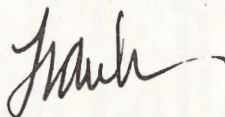
Dr. B. L. Shapiro

- 2 -

September 13, 1966

I hope this information will be of use to other spectroscopists with a similar problem.

Sincerely,



FED:cmk

Frank E. Dickson  
Fellow, Absorption Spectroscopy

P. S. I would like to reiterate the comments of Herb Retcofsky of IITNMR #94 concerning the 1967 Pittsburgh Conference. The deadline for contributed papers is October 15, 1966 and should be submitted to Dr. G. L. Carlson, Mellon Institute, 4400 Fifth Avenue, Pittsburgh, Pennsylvania 15213. Even though our n.m.r. symposium is centered around carbon-13, there will be sessions devoted to general contributed papers.

Title: Internal Reference for Quantitative Analyses;  
Contributed Papers for 1967 Pittsburgh Conference



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