Illinois Institute of Technology
N-M-R Newsletter

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Please note dates!!!!

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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".
Dear Dr. Shapiro,

NMR spectra of protonated ketones (dialkylhydroxycarbonium ions)

The existence of an appreciable barrier to rotation about the partially double C-O bond in hydroxy- or alkoxycarbonium ions has recently gained increasing interest\(^1\)-\(^5\). The expectation that in protonated ketones the barrier must be high enough to render the two isomeric forms I and II observable by NMR has been borne out by the spectra (60 and 100 MHz) of a series of eight of these carbonium ions in HF:SbF\(_5\) at -20 °C. Unsymmetric ketones (\(R_1 \neq R_2\)) give two spectra, one for each isomer, the relative intensities of which vary with the combination of \(R_1\) and \(R_2\). (The isomer ratio varies from about one for ethyl isopropylketone to about 15 for methyl tert-butyketone.) Symmetric ketones (\(R_1 = R_2 = CH_3\), or \(C_2H_5\)) give spectra that show the methyl or ethyl groups to be magnetically non-equivalent.

The shielding of the OH hydrogen appears to depend exclusively on the alkyl group syn to the OH hydrogen (\(R_1\) in I, \(R_2\) in II); it increases from -14.7 ppm for \(R = CH_3\) to -14.4 for \(R = CH_3\), isopropyl, or neopentyl to -14.1 for \(R = CH_3\), tert-butyl. The shielding was found to be independent of the anti alkyl group. We believe the influence of the alkyl groups on the OH shieldings to be due mainly to steric effects on the solvation of the OH group. The shielding of hydrogens bonded to \(\text{C}_\alpha\) (the carbon attached to the carbonyl C) is greater by 0.05-0.11 ppm for alkyl groups anti to the OH hydrogen, whereas that for \(\beta\)-CH\(_3\) hydrogens is greater for syn alkyl groups by about 0.12 ppm (for \(R = CH_3\)) to about 0.04 ppm (for \(R = i-CH_2CH(\text{CH}_3)\)).

There is a weak long-range coupling between the \(\alpha\)-hydrogens of the two alkyl groups \(R_1\) and \(R_2\) ranging from 0.7 c/s in methyl-isopropyl to 1.8 c/s in methyl-ethyl hydroxycarbonium ion.

Various spectroscopic features point to the existence of preferred conformations about the OC-C\(_\alpha\) bonds. For \(\alpha\)-ethyl, isopropyl, and tert-butyl groups the most stable rotamer is probably that with \(\beta\text{CH}_3\) eclipsing the OH group (III), as found with unprotonated\(^6\) and protonated\(^5\) aldehydes. For \(\text{syn}\) alkyl groups the preferred rotamers are concluded to be as shown in IV for \(R_1 = \text{ethyl or isopropyl}, \) and similar to IV with three \(\beta\text{CH}_3\) groups for \(R_1 = \text{tert-butyl}.

Finally it is of interest to note that the existence of two distinct isomers of protonated acetaldehyde, as indicated in a recent paper by Hogeveen\(^8\), has been conclusively established. The spectrum at -20 °C in HF:SbF\(_5\), which appears to be entirely free from impurity peaks, shows not only two OH doublets but also two CH\(_2\) doublets (each pair with an intensity ratio of 4:1) and two overlapping doublets of quartets for the CH resonance.

With kind regards,

D.M. Brouwer

Amsterdam, 8th March 1967

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Dear Barry,

We are at the present time extending our nuclear electron double resonance measurements to carbon-13 and phosphorus-31 nuclear resonances. The carbon-13 resonances are sometimes enhanced and sometimes reversed but in many compounds we are able to observe quite strong signals at 3300 gauss. We have just added a noise decoupler to this machine and are obtaining sharper and stronger carbon resonances from it. We are very interested in the mechanism by which the unpaired spin density is transferred from the radical to the carbon nucleus in the compounds for which the resonance is enhanced, and for the reason for the lack of this transfer in the compounds for which the resonances are inverted. It is too early yet to be able to give a definite answer to this question.

Some of the results on phosphorus resonances are however rather more clear. Measurements of thirteen organic compounds containing phosphorus in an oxidation state of five, and seven compounds with phosphorus in an oxidation state of three have been studied at both 3300 gauss and 12,500 gauss. Very strong enhancements of the phosphorus resonance are observed in all the trivalent phosphorus compounds except diethoxy chloro-phosphine where the effect at 12,500 gauss is rather small. Most of the trivalent compounds give a negative phosphorus resonance except for some compounds which have hydrogen directly attached to the phosphorus. We thought that this effect might be due to a 'three spin effect' in which the strong negative polarisation of the protons pumps the phosphorus into a positive enhancement. Triple resonance measurements have shown, however, that this is not so in these cases. There is therefore a fairly definite correlation between the nuclear polarisation in these experiments and the valence state of the phosphorus.

The mechanism by which the spin density is transferred from the radical to the phosphorus nucleus is too complex to treat quantitatively at this stage, but Peter Atkins and Raymond Dwek have been able to show qualitatively that one would expect a much more effective spin transfer to occur by interaction with the unshared pair of the trivalent phosphorus compounds than with the pentavalent phosphorus compounds.

We have a number of other topics which I hope to report on during the next few months but which are not quite ready at the present time.

Yours sincerely,

Short title: Dynamic nuclear polarisation in phosphorus compounds.
23 April 1967

Prof. Barry Shapiro
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry:

The NMR Spectrum of 1,5-dimethylnaphthalene; or, a Deceptively Complicated Poor Girl's Decoupled Spectrum.

The analysis of the $^1$H NMR spectrum of 1,5-dimethylnaphthalene, recorded at 60 MHz on a Varian A-56/60A spectrometer, reveals an unusual high-order splitting pattern, similar to those reported by DeWolf and Baldeschwieler [1] and by Freeman and others [2], the latter most recently at the 8th ENC under the title, "A Poor Man's Spin Decoupler."

We considered the aromatic protons in 1,5-dimethylnaphthalene as two equivalent and uncoupled ABC systems, and neglected any coupling to the methyl groups. The spectrum is shown in Figure 1a (solvent, CDCl₃). This spectrum resembles neither the usual ABX (for example) pattern nor its "deceptively simple" modifications [3].

However, as indicated by DeWolf and Baldeschwieler, this pattern can be interpreted as the result of a fortuitous overlap of an A-transition with a B-transition, each of which carries the same C spin state. This overlap requires the appearance of high-order structure in the AB region of the spectrum, and the possibility of new structure (analogous to "spin-tickling" double resonance experiments) in the C region. For example, if $J_{ab}$, $J_a$, and $J_{ac}$ are all positive, a high-field line in the A spectrum has frequency $-v_0 = J_{ab}/2 + J_{ac}/2$, and a highfield line in the B spectrum, $-v_0 - J_{ab}/2 - J_{bc}/2$, where the usual notation is used. If these lines overlap,

$$-v_0 + v_0 = (J_{ac} - J_{bc})/2 = 0,$$

which is identical to the statement about matrix elements of the Hamiltonian,

$$<a^a|H|a^a> = <a^a|H|a^a> = J_{ab}/2,$$

and the appearance of unusual high-order splitting in the AB region. This condition also implies
that, in order for such an effect to occur, $\delta_{ab} = (J_{ac} - J_{bc})/2$.

In the spectrum shown, it is difficult to tell the relative sign of $J_{bc}$ by inspection because the absolute magnitude of this coupling is so small. The calculation shown in Figure lb offers a least-squares error fit to the experimental spectrum to within experimental error (approximately 0.8 hz); the calculation shown in Figure 1c, with $J_{bc}$ of opposite sign relative to $J_a$ and $J_c$ gives a least-squares error of more than twice that of calculation lb.

The spectrum of 1,5-dimethylnaphthalene in $\text{C}_6\text{D}_6$ is shown in Figure 1d. Here, the effect of the fortuitous overlap of the A and B lines is somewhat less dramatic. As Freeman has shown, in molecules where the chemical shift may be very dependent on the solvent, the "poor man's (or, for us, girl's) spin decoupling" technique may be extremely valuable in spectral assignment.

The spectrum of 1,5-dimethylnapthalene reported earlier [4] at resolution lower yet was analyzed with slightly different values of coupling constants. We acknowledge the guidance of Professor Ernst Berliner of this Department; the analysis of the NMR spectrum of 1,5-dimethylnaphthalene was originally undertaken as part of his research in aromatic substitution.

Sincerely yours,

Jean B. Kim

April 25, 1967

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

Dear Barry:

We have recently acquired a Hewlett-Packard 5100A frequency synthesizer and consider it the finest invention since the wheel. Using it to drive a V-4311 when running $^{31}$P or $^{19}$F on our old and unlocked HR-60 makes calibration easy and sideband ambiguity much less troublesome. For quick observation on the scope we use a remote programmer on the $10^{-2}$-$10^3$ decades with two banks of decade switches selected by a manual switch or a flip-flop driven by the linear sweep flyback:

![Linear Sweep](Schmitt) ![Flip-flop](5100A Selectors)

With sample and reference peaks displayed on alternate sweeps of the long-persistence CRO rapid calibration to at least $\pm 5$ Hz can be achieved by superimposition, even for very large shifts.

Further talents of this very versatile device are being explored and will be reported in these letters provided, as we all hope, that they are still extant.

Sincerely yours,

Roy W. King

WMK/1d
A computer program has been written (Mellon Institute Report February 1, 1967) to solve the simple, nevertheless time-consuming, problem of measuring NMR spectra and determining an accurate experimental frequency for each line. A brief explanation of the procedure and of the program called 'cyclist' follows.

As many as 20 scans (typically an equal number in each direction) with appropriate audio sidebands are made over the region of interest. The observed lines (max. 500 each spectrum) are assigned a number (1 to n) beginning with the line of lowest frequency. The relative position of each line with respect to the sidebands is measured in centimeters and recorded. These measurements together with the centimeter position and frequency of each sideband are punched on data cards. (Twelve measurements may be punched per card.)

The program determines a scale factor Hz/cm for each spectrum sheet, interpolates the line position with respect to the sidebands and assigns a frequency in Hz to each line. Each line then has a distribution of frequencies that corresponds to the number of spectrum sheets on which it appeared. From this data the mean frequency of each line is determined and the rms error for each distribution is obtained. The output also contains the mean rms error of all measurements.

Sample spectra Fig. (1) and corresponding output Fig. (2) are shown below. We have found this method to be a great improvement over the various hand and electronic approaches that we have tried in the past. The major advantages are that efficiency increases with the number of calculations involved and that consistent accuracy can be depended upon.

A complete Fortran (63) source list (53 card images) and instructions will be furnished upon request.
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Fig. 2
Dr. B. L. Shapiro  
Illinois Institute of Technology  
Chicago, 60616  
U. S. A.

Dear Barry,

We have done some studies of the $^{19}$F magnetic resonance of fluoroberyllate ions in solution. The equilibrium constant for the dissociation reaction:

$$\text{BeF}_4^- \rightleftharpoons [\text{BeF}_3(\text{OH}_2)]^- + F^-$$

was determined from intensity measurements as $K_e = 8.0 \pm 0.9 \times 10^{-2}$ moles Kg$^{-1}$. Line width studies show that in addition to the dissociation and re-association process there is fluoride ion exchange. We have analysed the reactions according to the following scheme and we give below the rough rate constants. These are correct within a factor of three at 33°C.

\begin{align*}
\text{BeF}_4^- & \overset{k_1}{\rightleftharpoons} \text{BeF}_3^- + F^- \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \ quad
The slow process is the insertion of a water molecule in the tightly bound tetrahedral arrangement in BeF$_2^-$. We also find two resonances in BeF$_2$ solutions in water and evidence seems conclusive that a non dissociative equilibrium of the type:

$$\text{BeF}_2 \rightleftharpoons (\text{BeF}_2)_{\text{tr}}$$

occurs. $^9$Be chemical shifts do not change very much as might be expected.

We are preparing a paper which will contain these conclusions as well as other aspects of the problem. We have ambitions to do a complete line shape fit so as to get better rate constants and attempt a temperature study. To this end we have been modifying in a small way the comparative Fortran IV program 'BLOKIN' written by Chuck Holm at Shell. Our aim of making this iterative is now virtually accomplished. Mr. Stewart has been largely responsible for this work. The basic program will handle up to 30 lines with simultaneous exchange occurring. The rates in equations (1)-(4) can be regarded as a simple form of a 9 site problem where the spin states of the $^9$Be nucleus produce quadruplets for BeF$_3^-$ and BeF$_4^-$. 

All Best Wishes,

L. W. Reeves
Professor

LWR/bas
April 20, 1967

Professor Bernard L. Shapiro
NMR IIT Letters
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Barry,

F\textsuperscript{19} and P\textsuperscript{31} NMR of P\textsubscript{2}F\textsubscript{4}

In cooperation with Dr. Ralph Rudolph (F. J. Seiler Research Laboratory, U. S. Air Force Academy), I've been working on the P\textsuperscript{31} and F\textsuperscript{19} spectra of P\textsubscript{2}F\textsubscript{4}, diphosphorous tetrafluoride. This is one of the few AA'X'A''XX' systems I am aware of and this one has some interesting quirks.

First, the F\textsuperscript{19} spectra are identical at 12.8, 40 and 56 Mc - thus all the F's have the same shift, as do the two P's. Second, we observe no changes in F\textsuperscript{19} spectra down to -150°C, implying the configuration is fixed.

Proceeding on these assumptions we have arrived at the constants in Table I, following Harris [Mol. Phys., 10, 437 (1966)] and Lynden-Bell [Mol. Phys., 6, 601 (1963)].

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</tbody>
</table>

The relative signs of J\textsubscript{PF} and J\textsubscript{PF}' are certain - all others appear to be indeterminate. One of the J\textsubscript{FF}'s must be $0 \pm 0.5$ cps - this leads to extensive degeneracy (only four "characteristic" lines of the eight described by Lynden-Bell), and makes the calculated spectra quite insensitive to relative signs and to the magnitude of J\textsubscript{FF} (gem). The three values in column one are all quite similar to known values - the three in the second column appear to be the first of their kind.
IR and Raman spectra indicate the structure is "trans" i.e. $\psi \approx \pi$. We have tentatively assigned $J_{FF} = 34.5$ to the 'trans' coupling (dihedral angle $\sim 180^\circ$) and $J_{FF} = 0$ to the 'cis' ($\sim 80^\circ$), although this is admittedly rather speculative.

Sincerely,

Fred

Frederic A. Johnson
(205) 876-9037

Figure 1 - $^{19}F$ Low Field Half-Spectrum of $P_2F_4$ at 40 Mc
Dear Professor Shapiro,

C-C and C-H Bond Diamagnetic Anisotropies

Thank you for your reminder that a subscription to the IIT N-M-R newsletter is due from us. Recently, we have been commissioning a new laboratory to accommodate both our existing Perkin-Elmer R10, together with our new addition, the last of the A.E.I. R.S2 spectrometers. Nevertheless, we have found sufficient time to do some NMR research, and would like to inform you of some of our findings.

Considerable interest has been shown in bond diamagnetic susceptibility parameters, and the possibility of using these in the calculation of proton chemical shifts. Because limited success in predicting shifts had been achieved by calculating the secondary fields arising from the anisotropy in bond diamagnetic susceptibilities, we have attempted to calculate the chemical shifts for some compounds in which we are interested (bicyclo[2.2.1] heptanes). Using the various literature values that are available for $\Delta \chi_{CC}$ and $\Delta \chi_{CH}$ we have only achieved moderate success when account is taken of C-H contributions, but we have obtained quite good agreement with observed shifts in calculations which account only for C-C contributions to shielding.

This stimulated us to review the bond anisotropy situation. As a result, it was evident that the literature values for $\Delta \chi_{CC}$ and $\Delta \chi_{CH}$ were by no means consistent. This appeared in part to be due to the apparently misplaced necessity for making compatible the results of NMR and magnetic birefringence measurements. We have, therefore, deduced $\Delta \chi_{CC}$ and $\Delta \chi_{CH}$ purely from NMR measurements. Our results are certainly incompatible with magnetic birefringence measurements, and from our work it would appear that the reason for this could be due to a fallacy in the method used for comparing continued...
the data from the two techniques. The generally accepted method of comparison is essentially dependent on the assumption that bond magnetic susceptibility components are truly additive. We consider that unlike some other bond parameters this is not the case for bond susceptibilities.

We have devised a rigorous method of estimating both the magnitudes of the C-C and C-H bond anisotropies, and the point along the C-H bond at which the induced point dipole acts, assuming that the C-C point dipole acts from the centre of the C-C bond. \( \Delta \chi_{\text{CC}} \) is found to be \(+4.3 \times 10^{-6} \text{ cm}^3 \text{ mole}^{-1}\) and \( \Delta \chi_{\text{CH}} \) is zero, or less likely to have a small negative value not exceeding \(-1.2 \times 10^{-6} \text{ cm}^3 \text{ mole}^{-1}\), in which case the point dipole acts from the carbon atom. The negative sign is surprising, but Zurcher \(^1\) has previously found a value of \(-0.74 \text{ cm}^3 \text{ mole}^{-1}\) for \( \Delta \chi_{\text{CH}} \). These values, which incidentally agree very favourably with those obtained by Moritz and Sheppard \(^2\) and other workers who have based their calculations only on NMR data, enable relative shifts for a wide range of hydrocarbons to be predicted.

Yours sincerely,

J. Homer

D. Callaghan

---


April 26, 1967

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

Dear Doctor Shapiro:

An Example of Long-range Coupling Across Four Single Bonds

We wish to report on an interesting example of long-range coupling across four single bonds. This coupling \( J_{1,3} \) is appreciably larger than is normally observed.\(^1\)

We believe that this large long-range coupling \( J_{1,3} \) occurs by the "Meinwald" type mechanism, that is, direct overlap between the small orbitals of the carbon atoms involved.\(^2\)

The initial approximate coupling constants and chemical shifts for the cyclobutane protons were refined by computer analysis.\(^3\) The structure and refined coupling constants are as follows:

\[
\begin{align*}
J_{1,2} &= +8.2 \text{ cps} \\
J_{1,3} &= +3.5 \text{ cps} \\
J_{1,4} &= +0.6 \text{ cps} \\
J_{2,3} &= +5.9 \text{ cps} \\
J_{2,4} &= +9.1 \text{ cps} \\
J_{3,4} &= -12.9 \text{ cps}
\end{align*}
\]


Very truly yours,

G. O. Morton and W. Fulmor
April 28, 1967

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

Dear Barry:

The 2kHz modulation frequency of the V3521 integrator can be varied approximately ±5% by making the following modification:

(1) Remove C201 and C202 from the V3521.

(2) Install the following circuit:

```
  C1A     C1B     → To Junction, R201-R203
  •       •      
  C2      C2      → To Junction, R201-V201 (pin 1)
                  → To R202 (Free End)
```

C1 - 3.6 to 52 pf (Hammarlund type HFD-50)  
C2 - Approximately 1000 pf, silver mica, selected to give center frequency of 2000 Hz.

We have found this useful in eliminating interferences between centerband peaks in an F19 spectrum and their sideband responses when their chemical shifts are accidentally some multiple of the modulation frequency. Homo-nuclear spin decoupling is also facilitated when the modulation can be set exactly to 2000 Hz.

Sincerely,

A. F. Barney  
J. E. Lancaster
magnetic non-equivalence/equivalence in methylene groups.

Dear Dr. Shapiro,

I wish to report an example of magnetic equivalence of methylene protons in a case where non-equivalence would have been expected.

Investigating the product distribution from the reaction:

\[ \text{II} \rightarrow \text{I} \quad \text{or} \quad \text{III} \]

\[ \text{I} \quad \text{II} \quad \text{III} \]

\[ A = \text{Me, Et and i-Pr.} \]

I have found that the \( \alpha \)-methylene protons in II exhibit magnetic non-equivalence when \( A = \text{Me and i-Pr} \), but, surprisingly, not when \( A = \text{Et} \).

The data are given in the table:

<table>
<thead>
<tr>
<th>A</th>
<th>( \gamma )</th>
<th>( \delta )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Me</td>
<td>7.68 doublet</td>
<td>7.16 doublet</td>
</tr>
<tr>
<td>Et</td>
<td>7.51 singlet</td>
<td>7.51 singlet</td>
</tr>
<tr>
<td>i-Pr</td>
<td>7.58 doublet</td>
<td>7.57 doublet</td>
</tr>
</tbody>
</table>

An explanation might be, that \( \alpha \)-neighbouring \( \alpha \)-carbon-atom is "less asymmetric" and substituted with ethyl, butyl, or isopropyl, butyl - but I have found nothing in the literature confirming this explanation.

I would like to hear your comments.

Yours sincerely,

[Signature]

[Name, Institution, Date]
May 4, 1967

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

Dear Barry:

This is in reply to your letter of April 17th reminding me that I should pay for my subscription.

For this purpose I wish to report the results of a recently completed fluorine spin-echo study of internal rotation in liquid 1,1-difluoro-1,2-dibromodichloroethane. This work was actually done by Bob Vold.

Carr-Purcell spin-echo (CPSE) trains were observed from -115° to +100°C with rf pulse spacings of 0.4 msec to 32 msec. Modulation of the echo trains was observed at temperatures below -60°C, and the modulation frequencies were found to agree quantitatively with predictions of a general theory for the combined effects of scalar coupling and chemical exchange in spin-echo experiments. At -100°C, effects of the internal rotation are negligible and dipole-dipole interactions give different relaxation rates for fluorines in different rotamers. Above -60°C the echo trains decay exponentially. The dependence of the observed decay rates on pulse spacing was used to obtain values of the free energies of activation for exchange between rotamers and values of the fluorine chemical shifts in the different rotamers. The free energies of activation are in agreement with values obtained by steady state NMR, and by analysis of CPSE trains at lower temperatures, but the apparent chemical shift values are about 25% too large.

I hope this will fill the bill for the time being at least.

Sincerely yours,

[Signature]

H. S. Gutowsky


Subject: Spin-Echo Studies of Internal Rotation in CF₂BrCCl₂Br.
Subject: Rotational Isomerism in $\alpha$-Oximino-Esters

Dear Dr. Shapiro:

We studied the NMR-spectra of the syn- and anti-forms of some $\alpha$-oximino-esters:

$\begin{align*}
\text{syn} & : R^1 = \text{C}_6\text{H}_5\text{CH}_2 \text{ (I), } \text{C}_2\text{H}_5 \text{ (II), } \text{m-C}_4\text{H}_5, \text{ etc.} \\
\text{anti} & : R^1 = \text{OH, } \text{HO-CH}_2, \text{ etc.}
\end{align*}$

The spectra of the anti-isomers are quite trivial, but for the syn-isomers are observed double signals for the most proton-containing groups, as $\text{CH}_2$ and $\text{CH}_3$O in syn-I, (see the figure), $\text{C}_6\text{H}_5$ and $\text{CH}_3$O in syn-II, etc. Almost no changes are noted in the spectra of the syn-forms in various solvents, and also in the temperature interval $30$ to $130^\circ\text{C}$.

We propose that the syn-$\alpha$-oximino-esters exist as mixtures of two rotational isomers, stabilized by hydrogen bonding and resonance effects:
From the chemical shift difference of 2.3 Hz (at 60 MHz) between the CH$_3$O-signals of syn-II at 150°C, the calculated lower limit of the rotational barrier $\Delta F^\ddagger$ is 24 kcal./mole. The magnitude of the barrier suggests that some syn-α-oxirano-esters might be separated into rotational isomers, as in the cases of substituted amides$^2$) and nitrosoamines$^3$).

Our investigations, which are carried in collaboration with scientists from Germany and Czechoslovakia include besides NMR, also dipole moment measurements and calculations, H-D exchange, IR, and chemical studies. The results are already sent to the Monatshefte für Chemie$^4$), and further work is in progress.

Yours sincerely,

Stefan Spassov

Stefan L. Strassov

3) A.Mennschreck, H.Münsoh, and A.Mattheus, Angew. Chem. 77, 751 (1965).
Adaptation of LAOCOON-II for the IBM-1620 Computer

May 4, 1967

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

Dear Barry:

A recently completed project of ours may be of interest to a number of your readers. For the past several months we have been involved in adapting the Bothner-By-Castellano program, LAOCOON-II, for use on the relatively small IBM 1620 computer. The resulting 1620-SPS-III program, which we call PROSPECT-1, should permit 1620 users to perform many of the sophisticated spectral analyses that previously required a large computer such as the IBM 7090.

PROSPECT is capable of both computing spectra from a set of trial parameters and also making the iterative refinements of these parameters in order to produce a "best" fit between the calculated and observed spectra. Like version two of LAOCOON, PROSPECT is written in two parts. Part 1 will calculate the theoretical spectrum of any system of from three to seven 1/2-spin nuclei. Output of the theoretical spectrum may then be obtained on either the standard print unit or an online curve plotter. Part 2, which performs the iterative calculations, will operate on any system from 3 to 5 spins in size. In the future we hope to extend part 2 to even larger systems.

We feel that PROSPECT could be of great value to those persons who either do not have access to large computers or, like us, find the smaller system more convenient. Accordingly we would like to make this program available to interested parties. However, there are some problems associated with distributing the program. First, since we do not have the facilities to prepare and ship the card decks, this will have to be done by a local computer center at some small cost to the user. By far the more serious problem concerns the wide variety of 1620 configurations available. There will be quite a few 1620 systems on which PROSPECT will not operate and we ask the potential user to carefully check the machine requirements listed in the next paragraph so that he may be certain the program will (or will not) work on his 1620.
For execution PROSPECT requires a 1620 with a 40 K (or larger) memory, a 1311 disk drive, floating point hardware indirect addressing and the special instructions transmit numeric strip, transmit numeric fill and move flag. In addition one disk pack is needed for program and intermediate data storage. We suggest that this disk not be used for other programs. The input/output configuration is flexible. The program as written is for paper tape/typewriter I/O. However, in the near future we do plan to generate the card decks for both card/typewriter and card/line printer I/O. The use of the 1627 plotter is strictly optional. The lack of a plotter will not affect normal program operation.

Those persons wishing to obtain either card decks of paper tape copies of PROSPECT should address inquiries to J. H. Goldstein, Chemistry Department, Emory University, Atlanta, Ga. 30322.

John M. Read, Jr.                     J. H. Goldstein
May 5, 1967

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

Dear Barry:

We have been interested in calculating $^1H$ and $^{13}C$ shifts in heterocycles particularly pyridine. Using the formulas of Pople and Karplus in conjunction with Mataga and Nishimoto's SCF wave function we calculated diamagnetic and paramagnetic terms both for a purely covalent and partially ionic sigma framework. These shifts are listed in the table below. Also shown are the direct $\pi$-electron density shifts obtained from $160$ ppm/electronic charge for $^{13}C$ and $10$ ppm/electronic charge for $^1H$. All the shifts in the table are in ppm with respect to benzene.

It is seen that it doesn't matter much whether the sigma bonds are ionic or not. The sums of the diamagnetic and paramagnetic terms correlate very badly with the observed shifts. On the other hand if the direct $\pi$-density term is included the correspondence between observed and calculated values is remarkable.

In principle one should not have to include the $\pi$-density effect as a separate term. It is not clear why the above treatment works so well. Perhaps certain effects are undervalued due to approximations in the theory. We are trying other wave functions.

With best regards.

Sincerely yours,

Gideon Fraenkel

Tadashi Tokuhiro
### Calculated and Observed Shifts for Pyridine

<table>
<thead>
<tr>
<th></th>
<th>$^{13}\text{C}$</th>
<th></th>
<th>$^1\text{H}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>calc dia + para</td>
<td>calc dia + para</td>
<td>calc dia + para</td>
</tr>
<tr>
<td></td>
<td>calc charge</td>
<td>$\Delta$ calc</td>
<td>obs</td>
</tr>
<tr>
<td>covalent</td>
<td>$\nabla$</td>
<td>-7.23</td>
<td>-13.8</td>
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<td>covalent</td>
<td>$\nabla$</td>
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<td>+2.8</td>
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<tr>
<td>ionic</td>
<td>$\nabla$</td>
<td>-9.58</td>
<td>-13.8</td>
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<td>covalent</td>
<td>$\nabla$</td>
<td>+3.66</td>
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<tr>
<td>ionic</td>
<td>$\nabla$</td>
<td>-1.75</td>
<td>-7.3</td>
</tr>
</tbody>
</table>

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

Dear Professor Shapiro:

MODIFICATION OF THE VARIAN HA-60-EL PROTON STABILIZER FOR USE WITH OTHER NUCLEI

The Varian NMR External Reference Proton Stabilization Controller has been adapted for stabilization of other nuclear resonances which can be observed at 14.1 kG. Field stabilization is achieved by mounting the Varian proton control probe (the "piggyback" probe) on the analytical probe of the nucleus of interest and sweeping the resonance frequency of the "piggyback" sample with a 60 MHz signal derived from a commercially available voltage controlled crystal oscillator (VCO). Sweeping the resonance frequency of the "piggyback" sample has the same effect as sweeping the current through the set of small coils which surround only this "piggyback" sample (the latter being the procedure in the conventional system), since the field stabilization system maintains the "piggyback" reference sample on resonance by adjusting the field whenever there is a shift in either field or frequency. This compensating field variation is "seen" by both the "piggyback" control and analytical samples so the sweep of the control sample resonance frequency results in a field sweep over the analytical sample resonance.

The use of a VCO is a compromise between a costly and highly stable frequency synthesizer, and a less expensive and relatively unstable conventional swept oscillator. A block diagram of the system is shown in Figure 1. The 15 MHz VCO is swept ± 5KHz by ±5V. A linear sweep voltage is provided by a Spectromagnetic Industries Model 9011 Sweep Generator. At the end of each sweep the voltage returns to zero in a time which depends on the length of the sweep. For most sweep times used, the feedback is sufficiently slow for the field stabilization system to remain locked to the proton resonance.

In order to provide an adjustable starting voltage for the sweep, a biasing voltage is added to the sweep voltage in an operational amplifier. The sum of these two voltages controls the frequency of the 15MHz oscillator. The VCO output is amplified to a 15V peak to peak level and fed into the Varian 60 MHz V-4311 RF unit at the junction of R107 and R105, which is the input to the first frequency doubler. The V101 oscillator tube in the V-4311 unit is removed.
The system was tested on a $^{31}\text{P}$ resonance at 24.3 MHz. The limiting factor on the stability of the system is the relative drift between the 60 MHz frequency of the control probe and the 24.3 MHz $^{31}\text{P}$ resonance frequency. Figure 2 shows the low field quintet of diethyl phosphite taken at a sweep rate of about 1 Hz/sec for which the coupling constant is 8.0 Hz.

I wish to acknowledge the encouragement of my thesis advisor, Professor Julian H. Gibbs, and the excellent technical assistance of Mr. Henry Klos, Mr. George Carnegis and Mr. Fred Bargoot.

This work was supported in part by U.S.P.H.S. grant GM-10906(06), Air Force grant AFOSR 1027-66, Army contract DA-18-035-AMC-274, A.E.C. contract AT(30-1)1983, the Advanced Research Projects Agency, and by a N.A.S.A. Traineeship in Chemistry.

Sincerely yours,

Stanley Kaufman
Figure 1.
Block diagram of the modified field stabilization system.

Figure 2.
The low field $^{31}P$ spectrum of diethyl phosphite in a 5mm spinning sample tube, with a sweep rate of 1 Hz/sec.
May 5, 1967

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

Dear Barry,

I regret that we have been out of touch with IIT NMR Newsletter so long. To rectify this situation, I wish to submit a simple but useful modification of an HA-100 system.

A Vernier Control for the HP 200AB Audio Oscillator

The Varian HA-100 NMR spectrometer can include a Hewlett Packard model HP 200AB audio oscillator which is used to generate H_2, the RF sideband used for double resonance experiments. The existing vernier on the oscillator is much too coarse for the fine adjustments needed in NMR work, particularly for spin tickling experiments.

We have added a 10K potentiometer to the resistance network on the range switch at a point where it will be effective only when the instrument is used in the X100 range. The potentiometer was mounted on the left side of the front panel above the range switch. A log taper pot was found to provide an output frequency shift reasonably linear with angular displacement of the control. This circuit shifted the oscillator by 3 Hz when the pot was at maximum resistance and provided a range of 9 Hz (the additional shift at zero resistance). There was no detectable distortion of the sine wave and with reasonable lead dress this modification did not affect the stability of the oscillator. The range of the adjustment could easily be increased by lowering the value of the 1.5K resistor.

Sincerely yours,

Milton I. Levenberg
Chemical Physics Laboratory
May 12, 1967

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

Dear Barry,

Please include in IITNMRN (LONG MAY IT LIVE!) the following two announcements:

1. Position Available in Instrumentation Section

   Qualifications - M.S. or Ph.D. with n.m.r. background (physical or organic). Some knowledge of electronics and computer techniques desirable; at least interest in these areas is important. Applicants for this Civil Service position must be U.S. citizens.

   Type of Work
   a) Research initiated by staff members of n.m.r. group and related to problems and substances of current or potential significance to FDA's mission.
   b) Research on organic molecules (structure, rate processes, etc.) originated by other groups working in organic and biological chemistry.
   c) Development of instrumentation, and of instrumental and interpretative techniques.

   Publication is encouraged.

   Equipment
   a) Available now: HA-100, A-60, and C-1024.
   b) To be built: double-resonance apparatus based on frequency synthesizer (components on order).
   c) Computer facilities: IBM 360-30 (being installed in FDA building; 360-40 available early 1968), access to CDC 3800; search capability for Preston n.m.r. abstract cards.

(over)
2. Fourier Transform Spectrometry

We should like to explore who is interested in having his high-resolution n.m.r. spectrometer adapted to Fourier transform spectrometry*. We assume that the present price for such an adaptation may come down substantially, if and when several parties join in approaching Varian on this.

Any steps taken in this direction at this stage by anyone (including us) should and will not imply any commitment whatsoever.

I should appreciate any response from the readers of this newsletter. Please indicate what type of spectrometer is involved.


Best regards.

Sincerely yours,

Ernest Lustig
Division of Food Chemistry
Bureau of Science
Dear Sir/Madam,

Your subscription demand letter caught me in one of those periods when it is very difficult to write of anything which would interest a wide audience. I have computer programs which are about to produce interesting results, but which have not done so yet, hence I wonder if I can get away with an appeal for other people to tell me what they are doing? I am writing a review on fluorine chemical shifts together with Laurie Phillips of Imperial College, and I would very much appreciate any preprints or chemical shift data which is in the process of publication. We are particularly interested in attempts to calculate chemical shifts, and studies of medium effects (including complex formation) of fluorine.

Yours ever,

Jim Emsley.
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"The Absolute Configuration of Paeonanthine and Hirustine"
W. F. Trager, C. M. Lee, J. D. Phillips and A. W. Beckett
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The IIT NMR Newsletter will continue unchanged, at least for the next several months. Those recipients (academic as well as industrial, research institutes, government labs, etc.) who might be able to help out with a contribution toward the costs of the Newsletter are encouraged to consider the possibility of doing so at some time in the not-too-distant future.

B. L. Shapiro

29 May 1967


"The Nuclear Magnetic Resonance Spectra of Some Related Alkylene-Substituted Diethylamines"
M. Freifelder. R. W. Nafziger and H. Kriese

"The Examination of Lobina line and Some Degradation Products by Mass Spectrometry"
D. M. Clugston and D. B. MacLean and R. H. F. Manske

"Dimerization of an Intermediate During the Sodium in Liquid Ammonia Reduction of L-Tryptophane-4-
Carbonyl Acid"
P. Blondeau, C. Berge, and D. Gravel

"Photochemistry of Nitroso Compounds in Solution. V. Photolysis of N-Hitrosodialkylamines"
Y. L. Chow

"Synthesis of Some Disubstituted Naphthazarins"
P. C. Aroca and P. Brassard

"Oxalic Acid Analouges. The Synthesis and Identifidica-
tion of 4-Loropyl-3,5-Dihalogeno-2-Pyrroli dine"
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"The Reaction of all-cis Cyclopentadetetrahydroxy-
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Y. P. Mattoon and R. K. Kriese

"Alkaloids of Aspidosperma vargasii A. DC."
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"The Radiation Chemistry of Dihydromyrcene"
J. L. Brash and M. A. Golub

"Charge Distribution in Substituted Cyclopentadienyl-
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C. J. A. Corbune

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"Etude Quantitative des Réactions d'ozonolyse. IV. Ozonation de l'indène"
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"C.n.m.r. Studies. IX. 13C Spectra of Some Substituted Methyl Benzoylates"
K. S. Dhawan and J. B. Stothers

"C.n.m.r. Studies. X. 13C Spectra of Some Substituted Methyl Benzoylates"
K. S. Dhawan and J. B. Stothers

"Mechanism of the α-Silylcarbinol to Silyl Ether Rearrange-
ment"
A. G. Brook, G. E. LeGrouve, and D. M. MacRae

"A New Synthesis of Twistanol"
J. Gauthier, and P. Deslongchamps

"Lignans of Western Red Cedar (Thuja plicata Donn). VI. Dihydroxythujaplicatin Methyl Ether"
H. MacLean and E. Nakamoto

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D. E. McGreer and W. S. Wu

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M. Tanabe and D. F. Crowe

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P. A. J. Gorin and T. Tabakawa
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"The Structure of Jegosapogenol"

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A. Novelli and A. De Santis

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"Modification of Protopanaxadiol; Acid-Catalysed Epimerisation of C-20 Hydroxyl of Betulafolienetriol, Protopanaxadiol, and Their Derivatives"

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"On the Reaction of Nitrochlorination of Alkenylmethyl- 
xioloxanes"
Academician K. A. Andrianov, V. I. Sidorov, and L. M. 
Khamarashvili 

"On the Structure of Certain O, 8-Unsaturated Ketones 
and Their Oxides"
Sanin, and L. K. Yul’kheva 

"A General Method for the Synthesis of 3-Nitro- 
isoazoles"
V. A. Tartakovskii, A. A. Otsabekho, I. E. Chlennov 
and B. S. Rovikov 

"Die Konstitution von Roridin A"
B. Böhrer and Ch. Tamm 

"Die Konstitution von Roridin D"
B. Böhrer and Ch. Tamm 

"Stoffwechsel-Endprodukte von Phyllochinon, Menachinon- 
4), Ubichinon-9) und Hexahydroplastochinon-4 (Phyl 
plastocho 

U. Gloor J. Wursch, H. Mayer, O. Isler und O. Wiss 

"Ceolchlorin"
J. J. Dugan, M. Hesse, U. Renner und H. Schmid 

"Weitere Alkaloide von Aspidosperma 
A. A. 
Geiger, E. Weiss und T. Reichstein 

"Strogsid, Strukturbestimmung"
U. P. Geiger, E. Weiss und T. Reichstein 

"Über die photochemische Cyclisierung von 2-Allylph 
V. J. Dastoor, A. A. Gorman und H. Schmid 

"Über die photochemische Cyclisierung von 2-Allylph 
G. Frater und H. Schmid 

"19-nor-Steroide V. Neue Synthese von 6-Dehydro-19-nor-
Steroiden"
J. Kalvoda und G. Anner 

"Über eine außergewöhnliche Stereospezifität bei der 
Hydroborierung der diastereomeren (1R)-Isopulegole 
it Diboran" 
K. H. Schulte-Elte und G. Ohloff 

"Organische Phosphorverbindungen. XXVII. Die Direkte 
Synthese von Tetramethylphosphoniumhalogeniden"
L. Maier 

"Über die optische Reihenfolde und die Chiralität der 
enantiomeren α, α-Benzyllamine" 
H. Gerlach 

"Die Konstitution von Roridin B"
B. Böhrer und Ch. Tamm 

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"Die Konstitution von Roridin B"
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"The Structure of Olivin"
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Chaprunova, and Academician M. M. Semenya 

"Sintesl e propria della H-1,4-didro-2,3-benzossassine" 
G. Pifferi, P. Comessoni ed E. Testa 

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Sintesi dell'1,5-difenil-3,7-diaza-10-tio-adamantan-9- 
one, 10-ossido e 10,10-diossido" 
B. Misiti e E. Chiavarelli 

"Photolyse von 2,2,4,4-Tetramethylcyclobutanon-Derivaten" 
H. U. Bostatter 

"Stereospezifische Synthese und Isomerisierung der 10- 
Chlor-decahydroisochino 1 ine" 
C. A. Grob und R. A. Wohl 

"Synthese und Abbau von 1-(5-Nitro-2-thiazolyl)-2- 
imidazolidinon und Derivaten" 
M. Wilhelm, F.-H. Marquardt, Kd. Meier und F. Schmidt 

"Steroide und Sexualhormone. Die Synthese von N-Acetyl- 
3-aza-A-homo-5β, 10β-androstan und seines 5α-Iso 
J. G. Eggart und H. Wehrli 

"Kinetische Phasenverbindungen. XXVII. Die Direkte 
Synthese von Tetramethylphosphoniumhalogeniden"
L. Maier 
"Alkylation of o- and p-Tolunitriles with Halides by Means of Sodium Amide in Liquid Ammonia"  
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T. L. Cob (introduced by J. D. Memory) and J. D. Memory


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