

Illinois
Institute of
Technology
N-M-R
Newsletter

No. 114
MARCH, 1968

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Deadline Dates: No. 115: 3 April 1968 }
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Reminder: For the period August 10, 1967 to August 15, 1968 inclusive, all Newsletter contributions, enquiries, etc., should be addressed as follows:

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

-continued on the outside back cover

UNIVERSITY OF OXFORD

Dr. Lee's Professor of Chemistry

R. L. RICHARDS, F.R.S.

Tel. Oxford (OOX2) 57757



Physical Chemistry Laboratory,

South Parks Road,

Oxford.

6 February 1968

Professor Bernard L. Shapiro,
Visiting Scholar,
Department of Chemistry,
Stanford University,
Palo Alto, California 94305.

Dear Barry,

I thought the readers of IITNN might be interested to hear about a crossed coil probe which Howard Hill and I have constructed for use in a superconducting magnet. The special features of the probe are that the sample is placed neither in the receiving coil nor in the transmitting coil, the balance is extremely stable and unaffected by mechanical disturbances, and relatively large samples can be used in the room temperature space of the superconducting solenoid.

The probe itself is shown in the diagram, and is made entirely from a thin -walled copper tube. The tube is joined at points A. to a milled aluminium block which contains matching elements for the coils and, if necessary, a preamplifier; the whole assembly then has complete cylindrical symmetry. Just above the points A. holes are drilled in opposite sides of the copper tube of about 1 cm. diameter. These two holes are joined by a very narrow slot, B, to the oval shaped holes in the copper tube S. A transmitting coil is wound with copper tape on a plastic former and fitted into the hole just above the points A. so that any RF field generated by the transmitting coil is coupled by the copper material into the space encompassed by the holes S.

Immediately above the transmitting coil is a copper disc soldered into the copper tube, and the copper disc is slotted with a very narrow slot. This disc acts as a very efficient electrostatic screen, screening the transmitting coil from the receiving coil which is immediately above it. Two further holes of about 1 cm. diameter are drilled in the copper tube above the slotted copper disc and exactly at right angles to the holes of the transmitting coil. Into this space is inserted the receiving coil. The receiving coil is wound on a plastic former which is a tight fit in the copper tube but provision is made for it to be

Contd.....

rotated a few degrees by means of a screwdriver through a small hole in the cylindrical screening can that surrounds the whole thing. The holes for the receiving coil are coupled to the sample space, S, by another pair of oval slots at right angles to those coupled to the transmitter coil. The sample is placed in a test tube and lowered into the copper tube at the top and sits with its bottom on top of the receiving coil. Radio-frequency fields are induced in the sample space by the transmitting coil coupling through the slot B, and signals from the sample are induced in the receiving coil by the transformer at right angles to the transmitting coil. Coupling between the receiving coil and the sample is extremely tight and very little filling factor is lost. Coupling between the sample space and the transmitter coil is not as strong as it would be if the coil surrounded the sample, but can easily be made up by small increase in transmitter power. When the probe is surrounded by its screening can, and the two coils are rotated to give optimum balance, it is usually possible to achieve an isolation between the two coils of 70 db. Better isolation can be obtained by adding a pair of paddles, one made of copper foil and the other of some resistive material. We have mounted these on half sections of a plastic cylinder which fit between the screening can and the copper tube and which can be moved up and down from above by means of small nylon screws. With the aid of these small paddles, the isolation between the two coils can be improved to as much as 100 db, and once adjusted is quite remarkably stable.

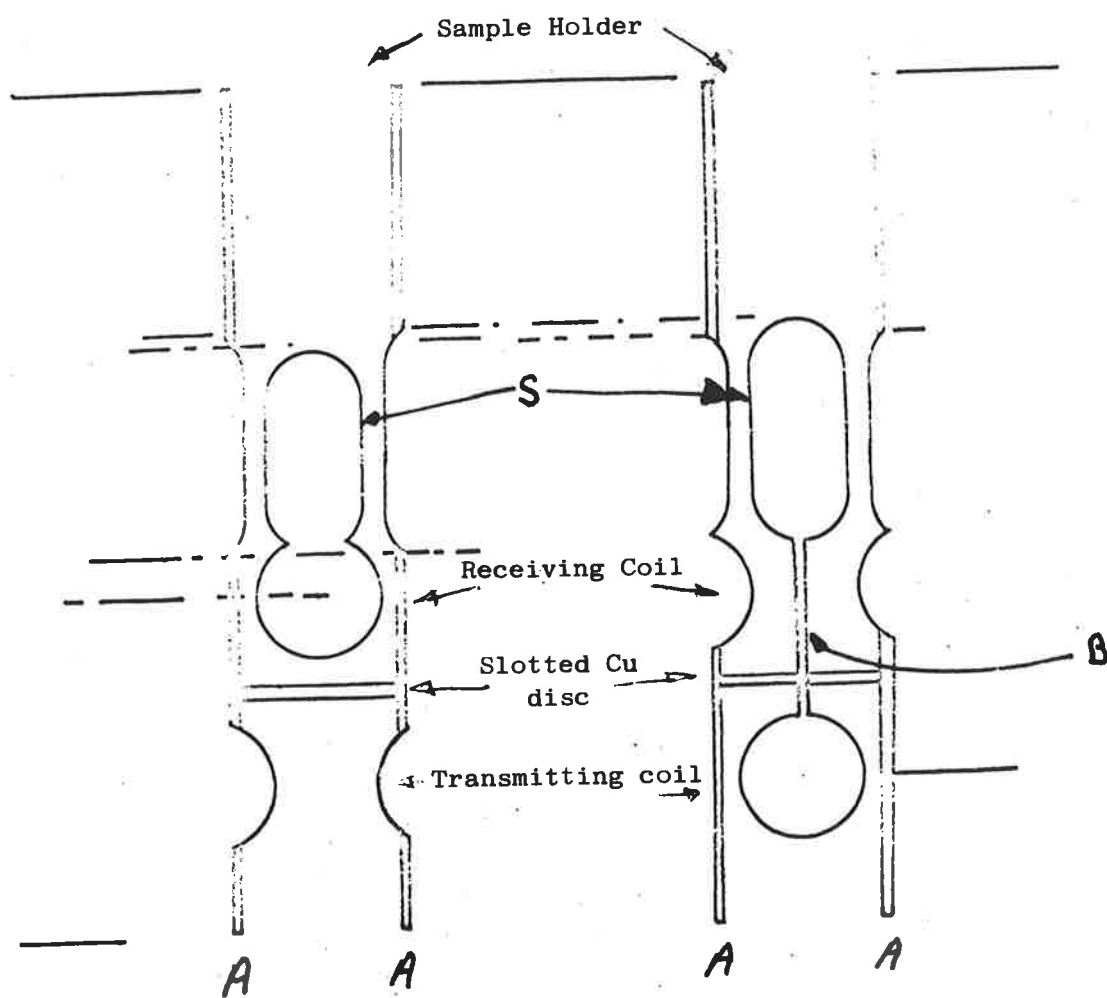
We have used this probe in a superconducting magnet and have obtained very good sensitivity with it. One disadvantage we have found, however, is that the sampling region does not have a cylindrically symmetrical sample in it. The sample ends just at the receiving coil. The result of this is that, even with diamagnetic materials, the susceptibility shifts are quite large and one also has to re-adjust the magnetic field homogeneity, if very narrow lines are being observed. We are just about to try a related scheme which does not suffer from this disadvantage, but I have nothing to report on it yet.

With best wishes,

Yours sincerely,

Rep

P.S. Short Title 'Crossed Coil Probe for Superconducting Magnets'



Crossed Coil Probe.

LABORATORIUM VOOR ORGANISCHE
SCHEIKUNDE DER UNIVERSITEIT VAN
AMSTERDAM

NIEUWE ACHTERGRACHT 129
TELEFOON 94 71 74 (5 LIJNEN)

AMSTERDAM, February 8, 1968

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

Digital spectra (using the C-1024)

Dear Dr. Shapiro,


The availability of digital spectra is extremely useful for iteration line shape analysis (T.R. Lusebrink, IITNMR 107,62). While waiting for the equipment that will produce punched tape from the spectrometer's analog signal, we decided to do some preliminary experiments using standard instruments we already had.

As a digitizer we used the Varian C-1024.

To obtain the digital information from the C-1024's store, the read-out switch is set at "decimal" and a positive going pulse is applied at P2-8. This starts a read cycle. If P2-13 is kept at -3V (conveniently obtained from P2-14), the read cycle is followed by a write cycle, thus effecting a non-destructive read out. After a $40 \mu s$ delay the content of the arithmetic register appears as a pulse train at P2-16 (repetition rate 2.5 MHz). The latter is connected to an electronic counter (HP523CR). At the end of the pulse train, i.e. when the arithmetic register has been cleared, a pulse appears at P2-15. This pulse is applied to the counter to close its gate. After a suitable delay the counter gives a print command to a digital recorder (HP561B). The standard interconnections between counter and recorder are such that at the end of a print cycle the counter is reset and its gate is opened automatically. From the "counter gate open" signal we derived the pulse that starts read-write cycle of the C-1024. Since this cycle also gives an address advance pulse, the contents of the addresses are printed out sequentially.

Remarks:

1. the counter's $.1 \mu F$ input capacitor was replaced by one of 10 pF (we lost pulses until we thought of this)
2. where to ground which shield of the cables between TAC and counter is rather critical. We found that here trial and error works faster than the electronics expert's advice.


P.J. van der Haak


K. Spaargaren

DIVISION OF PLANT INDUSTRY

P.O. BOX 109 CANBERRA CITY ACT TELEPHONE 40455 TELEGRAMS CORESEARCH CANBERRA

GFK:IVS.

Refer.:

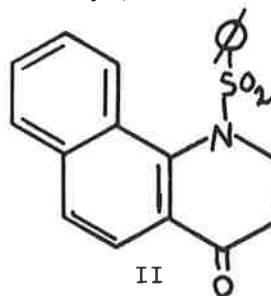
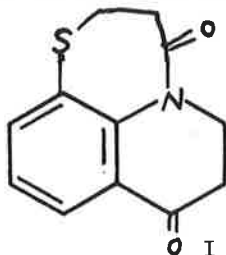
9th February, 1968.

Professor Bernard L. Shapiro,
 Department of Chemistry,
 Stanford University,
 Stanford. Calif. 94305. U.S.A.

Hindered Inversion of Nitrogen?

Dear Professor Shapiro,

We have recently looked at the spectrum of the quinolo-thiazepine I. At +20°C, the methylene protons adjacent to the nitrogen appear as a diffuse hump near 4.28 δ . Above +30°C the absorption is a triplet, which becomes fully defined at +70°C. At -30°C or lower, two principal absorptions appear at 5.2 δ and 3.3 δ with fine structure, although the latter is hidden under the signal due to the S-CH₂- protons. These signals approximate to an AB system with $J_{\text{gem}} \approx 12^2$ c.p.s. The chemical shift between these protons is large, ~ 1.9 p.p.m. arising from the different spatial arrangements relative to the amide carbonyl, this effect being averaged at +30°C or higher.



A similar effect has been observed with compounds of type II¹ and was deduced to be due to hindered inversion of the nitrogen atom, caused by steric factors. While this could be so in the present case, it would appear that the nitrogen atom should, as a first approximation, be planar. The results, then, would not be inconsistent with an essentially planar A ring, with the B ring slowly inverting at -30°C. This inversion of ring B would require a movement of the C = O bond relative to the N-CH₂- protons, and also a twisting about the N-C bond. This phenomenon is at present being investigated.

Yours sincerely,

G.F. Katekar

(G.F. Katekar)

L. Huppertz

(L. Huppertz)

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANIZATION, AUSTRALIA

1. W.N. Speckamp, V.K. Pandit, P.K. Korver, P.J. van der Haak and H.O. Huisman. Tetrahedron, **22**, 2413 (1966) and references there cited.

UNIVERSITY OF CINCINNATI

CINCINNATI, OHIO 45221

DEPARTMENT OF CHEMISTRY

February 5, 1968

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

A Detailed Analysis of the NMR Spectrum of a Bicyclo[3.1.1]-
heptene-2 System.

Dear Barry:

We have analyzed the n.m.r. spectrum of myrtenal (I), a bicyclo[3.1.1]heptene-2 system. The parameters describing the spectrum are presented in Table 1. The most striking feature is the large four bond coupling (confirmed by decoupling experiments) found between the bridgehead protons. The value of 5.78 cps falls in between the 1.5 cps found for coupling between bridgehead protons in bicyclo[2.2.1] heptanes and 18 cps found in bicyclo[1.1.1] pentane. Presumably, as the bridgeheads are brought closer together, the interaction between bridgehead protons is enhanced.

With warmest regards,

Fred Kaplan, Carl Schulz

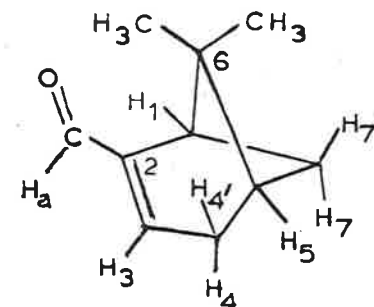
Fred Kaplan

Carl Schulz

FK:la

Table 1

Proton	δ (p.p.m)	J (+0.02 cps) ^a	
H _a	9.45	$J_{1,3} = 1.49^b$	$J_{4,4'} = 0.06^c$
H ₁	2.873	$J_{1,4} = 0.0$	$J_{4,5} = 2.77$
H ₃	6.670	$J_{1,4'} = 0.0$	$J_{4,7} = 0.0$
H ₄ , H _{4'}	2.576	$J_{1,5} = 5.78^b$	$J_{4,7'} = 0.0$
H ₅	2.200	$J_{1,7} = 0.04$	$J_{4',5} = 3.07$
H ₇	1.052	$J_{1,7'} = 5.39$	$J_{4',7} = 0.0$
H _{7'}	2.499		$J_{4',7'} = 0.0$
CH ₃	1.34, 0.74	$J_{3,4} = 3.01$	
		$J_{3,4'} = 3.05$	
		$J_{3,5} = 1.51^b$	$J_{5,7} = 0.11$
		$J_{3,7} = 0.0$	$J_{5,7'} = 5.75$
		$J_{3,7'} = 0.0$	$J_{7,7'} = -9.05$



- a. A positive value for all vicinal (3 bond) and a negative value for all geminal (2 bond) coupling interactions was assumed.
- b. The sign of the coupling constant was not determined. Variation of the sign had little effect on the calculated spectrum.
- c. A good spectral fit was also obtained when a value of -13.00 cps was used. The closeness of the chemical shifts of H₄ and H_{4'}, precluded an accurate measure of the coupling constant.

Carnegie-Mellon University

Mellon Institute
4400 Fifth Avenue
Pittsburgh, Pennsylvania 15213
[412] 621-1100

February 5, 1968

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

Dear Dr. Shapiro:

We have been performing 'NMR' experiments at 14, 092.5 Gauss on other nuclei while locked on a piggy-back water sample in the probe.

The R.F. source for our system is a General Radio-1164A Frequency Synthesizer. Locking is accomplished by synchronous detection of a water audio sideband from a V4311. The 5 MHz standard output of the GR-1164A is amplified [Figure (1)] and used to replace the 15 MHz crystal in the V4311, the oscillator stage being tuned to 15 MHz thereby acting as a tripler.

In the 2-16 MHz range, a V4210 serves as our R.F. amplifier and detector. Phase coherence between lock and sample channels is insured by replacement of the V4210 oscillator with the variable synthesizer output which is controlled by our digital sweep. Sample audio detection is accomplished by a P.A.R. lock-in amplifier.

To present, our most successful high resolution results have been obtained at frequencies less than 16 MHz, the limitation being lack of a suitable low noise preamplifier. However, we have taken high resolution phosphorous spectra at 24.3 MHz and we are working on extending the range of our system continuously to 70 MHz.

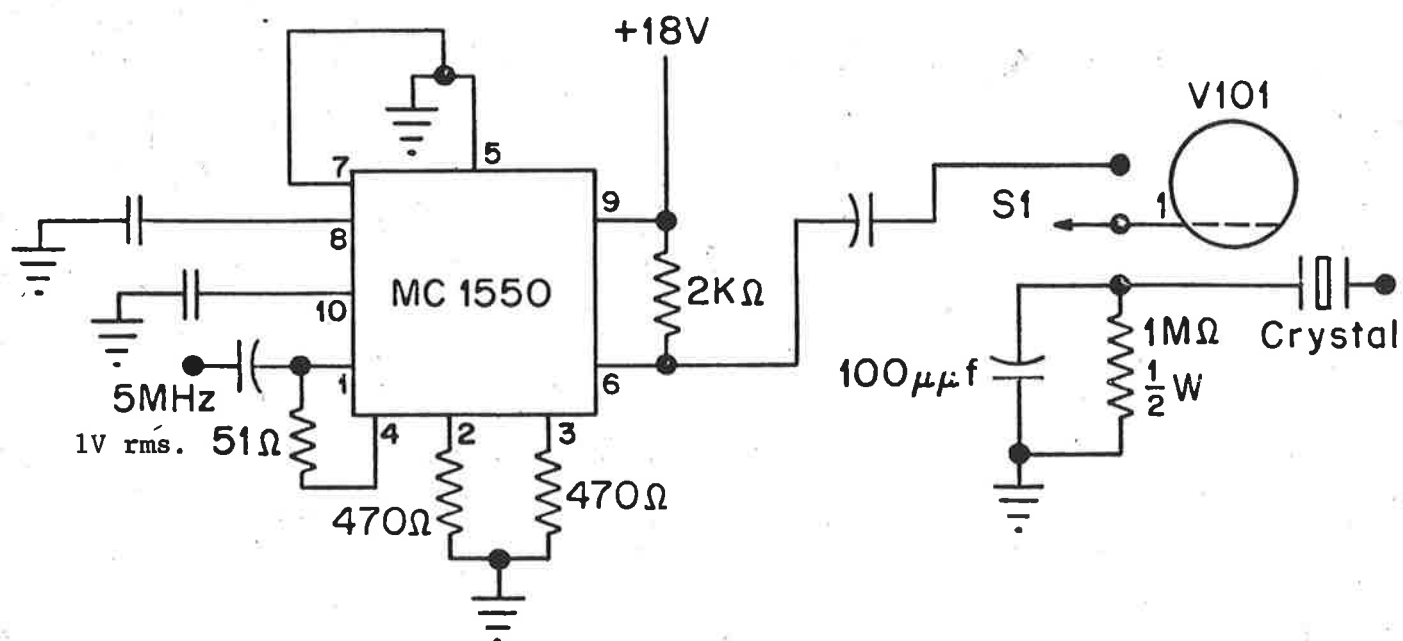
Sincerely,



DEW:sa

Dennis E. Wisnosky

FIRST STAGE 60MHz



All resistors $\frac{1}{8}$ W unless otherwise designated.

All capacitors .01μf unless otherwise designated.

Voltage gain of amplifier 10 db at 5 MHz. Tune L 101 (not shown) for maximum xmtr. output as viewed on v.t.v.m. using normal procedure.

Correct tune should insure that switching of S1 does not vary output more than 5%.

ANORGANISCH-CHEMISCHES LABORATORIUM
DER
TECHNISCHEN HOCHSCHULE MÜNCHEN

8 MÜNCHEN 2, den 9. Februar 1968
Arcisstraße 21
Ruf-Nr. 5592/330
331

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

Dear Professor Shapiro,

The Association between Paramagnetic and Diamagnetic Ions
in Solution Studied by ^1H -NMR

^1H -n.m.r. contact shifts provide a very sensitive probe for measuring unpaired spin densities on magnetically active nuclei in metal complexes. On the basis of large proton contact shifts observed for diamagnetic tetrabutyl ammonium ions in solutions of several paramagnetic complex salts, a strong ion association between diamagnetic and paramagnetic species has been postulated recently by several groups. These shifts were attributed to pseudo contact contributions, which should depend on the g-factor anisotropy of the unpaired electron and on the distance and steric position of the interacting ions. This model was used to calculate the distance between the "paired" ions in solution, however, some objections to this procedure are evident:

i) One research group studied the ion association between tetrahedral quaternary ammonium ions and the paramagnetic, regular octahedral hexacyanoferrate(III) and hexacyanochromate(III) ion, resp. All these ions are of approximately spherical symmetry, however, a preferential sterical orientation of these cations in solution with respect to the complex anions, which is the principal condition for observing pseudo contact shifts, cannot be assumed over an extended period. Despite the fact that some of the examined complexes show no g-factor anisotropy, which is another condition for pseudo contact

contributions, the dipolar interactions must be averaged out in complexes without specific steric orientation of the paramagnetic ions and the interacting magnetic nuclei, resp.

ii) In complexes with delocalized electrons pseudo contact contributions should be small or zero, so the large observed shifts in covalently bonded complexes do not appear plausible at all.

iii) The decrease in spin density along the carbon-carbon chain in the "paired" tetrabutyl ammonium salts is typical for alkyl groups in paramagnetic metal complexes, so the nitrogen atom should have the highest spin density in this system.

The above mentioned arguments lead us to postulate a direct spin transfer from the central metal ion to the nitrogen atom in ionic paramagnetic salts in solution, similar to the direct metal-proton interaction.

There is one proof for this postulate:

If direct interaction with spin transfer of positive sign from the paramagnetic to the diamagnetic ion is the reason for the observed shifts of the alkyl protons, a strong low field shift must be expected for the ^{14}N -n.m.r. signals in strict contrast to the pseudo contact shift model, which predicts high field shifts. ^{14}N -n.m.r. spectra of several complexes of the discussed type were recorded, namely $[(n\text{-C}_4\text{H}_9)_4\text{N}]_3[\text{Fe}(\text{CN})_6]$, $[(n\text{-C}_4\text{H}_9)_4\text{N}][(\text{C}_6\text{H}_5)_3\text{P}\cdot\text{CoJ}_3]$ and $[(n\text{-C}_4\text{H}_9)_4\text{N}][(\text{C}_6\text{H}_5)_3\text{P}\cdot\text{NiJ}_3]$. In all cases the ^{14}N -n.m.r. signals appear at lower fields, even using an external standard in order to estimate a paramagnetic bulk susceptibility shift in the solution. This supports our assumption of direct spin transfer between the metal ion and the nitrogen atom. On dilution the observed ^{14}N -n.m.r. shifts decrease corresponding to a decreasing ion association in the solvent.

Sincerely yours,

H. Fritz
(Heinz P. Fritz)

UNIVERSITY OF CALIFORNIA, DAVIS

BERKELEY · DAVIS · IRVINE · LOS ANGELES · RIVERSIDE · SAN DIEGO · SAN FRANCISCO



SANTA BARBARA · SANTA CRUZ

A Tribute to the People of California

DEPARTMENT OF CHEMISTRY

DAVIS, CALIFORNIA 95616

February 11, 1968

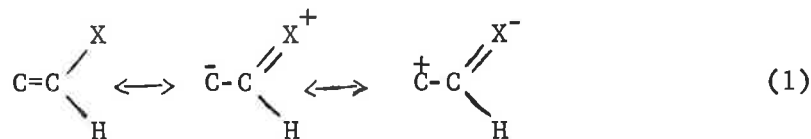
Cis/Trans Isomerism and C^{13} Shieldings in Disubstituted Ethylenes

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

Dear Barry:

Recently in collaboration with George Savitsky (Clemson University) we have taken another look at the consequences of cis/trans isomerism on C^{13} shieldings in disubstituted ethylenes. In the accompanying table we have collected most of our new and old results with such compounds.

The data on unsymmetrical compounds show that an earlier interpretation for the symmetrical case (G.B. Savitsky and K. Namikawa, J. Phys. Chem., **67**, 2754 (1963)), based on the hypothesis of steric inhibition in cis isomers of resonance of the type (1)



must not be applicable to the unsymmetrical case. In fact these data can be interpreted on the basis of a greater tendency toward charge separation and reduced bond order in the CC π -bond in cis isomers.

On the other hand, the C^{13} shieldings for the symmetrical case seem best explained on the basis of greater π -bond delocalization and CX π -bond polarizations in trans isomers. In both the symmetrical and unsymmetrical cases application of the Pople-Karplus formalism, using parametrized π MO's, is consistent with these qualitative interpretations.

Sincerely,

A handwritten signature in cursive script, appearing to read "Gary".

Gary E. Maciel
 Associate Professor
 (On leave 1967-1968)

C^{13} Chemical Shifts in $XHC^X=C^YHY$ Compounds, P.p.m. with Respect to CS_2

Substituents		δ_{C^X}		δ_{C^Y}	
<u>X</u>	<u>Y</u>	<u>cis</u>	<u>trans</u>	<u>cis</u>	<u>trans</u>
CH_3	Cl	67.0	64.3	73.6	76.0
CH_3	Br	62.8	59.0	82.3	88.0
$n-C_4H_9$	I	51.5	46.0	109.5	117.1
CH_3	CN	42.2	42.4	91.0	91.1
CH_3	CO_2H	44.5	46.1	71.8	70.8
$n-C_4H_9$	CO_2H	40.1	41.0	73.7	72.0
I	I	97.2	114.3		
Br	Br	77.3	84.3		
Cl	Cl	72.4	74.3		
CH_3	CH_3	70.4	69.2		
CN	CN	73.2	73.2		
CO_2CH_3	CO_2CH_3	65.0	61.3		
$CO_2C_4H_9$	$CO_2C_4H_9$	64.7	60.9		

**BOSTON COLLEGE***A University of 14 Colleges and Schools*

CHESTNUT HILL, MASSACHUSETTS . 02167

Phone 332-3200
Area Code 617

Department of Chemistry

February 12, 1968

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

Dear Barry:

The demonstration that the magnitudes of geminal H-H (1,) and H-F (2,3) coupling constants are affected by the orientation of an α -carbonyl group suggests the possibility of observing similar behavior in other systems.

1. N.S. Bhacca and D.H. Williams, "Applications of NMR Spectroscopy," Holden-Day, 1964, pp. 57-61.
2. D.J. Sardella, Ph.D. Thesis, Illinois Institute of Technology, Chicago 1967.
3. Y.K. Pan and J.B. Stothers, Can.J.Chem., 45, 2943 (1967)

More specifically, if we replace a proton in a methylene group by a methyl group, will the vicinal coupling constant show a conformational dependence? Although I have only preliminary results thus far, they do appear promising.

I decided to investigate the solvent-dependence of the spectrum of 3-chloro-2-butanone. Although SO_2Cl_2 chlorination of 2-butanone yields a so-far inseparable mixture of 3-chloro-2-butanone and 3,3-dichloro-2-butanone, ($\sim 50:50$), the data given below for CCl_4 and CH_3CN solutions of the product mixture seem to indicate a small, though significant, variation in $J(\text{vic})$:

Ltr to: Prof. B.L. Shapiro
February 12, 1968

pg. 2.

Solvent	CCl_4	CH_3CN
ϵ	2.24	37.45
$\tau(\text{CH}_3\text{CO})$	8.45	8.46
$\tau(\text{CH}_3\text{CH})$	7.76	7.75
$\tau(\text{CHCl})$	5.75	5.61
$J(\text{vic})$	6.838 ± 0.017	6.887 ± 0.009

Obviously, a larger variation would have been preferable, and I'm hoping that the temperature dependence of the spectra of 2-butanone and/or 3-methyl-2-butanone will yield less ambiguous results.

I hope this will cover my subscriptions for another eight months. Wouldn't it be wonderful if the publish-or-perish requirements in universities were so easily filled?

Regards to the family.

Sincerely,



Dennis Sardella

Short title: Hyperconjugative enhancement of a vicinal coupling constant?

DJS/cp

Dr. K. G. Orrell

Department of Chemistry

Tel. 77911

Stocker Road,
Exeter

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

8th February 1968.

Dear Dr. Shapiro,

Apologies for my short lapse in contributions to your news letter. I trust this will reinstate me on your mailing list.

We have recently been investigating the contact shifts of heterocyclic ligand protons (viz. pyridine and substituted pyridines) in complexes with Co(II), Ni(II) and Cu(II) trifluoroacetates. We have prepared two series of complexes, $M(\text{CF}_3\text{CO}_2)_2\text{L}_4$ and $M(\text{CF}_3\text{CO}_2)_2\text{L}_2$ where $M = \text{Co(II)}, \text{Ni(II)} \text{ and } \text{Cu(II)}$ and $L = \text{pyridine and/or substituted pyridine}$. Both series of complexes are essentially octahedral with the trifluoroacetate group being attached in a unidentate manner in the L_4 series and bidentate in the L_2 series. The complexes are very easily broken down in polar organic solvents and are insoluble in non-polar solvents. Furthermore, small amounts of the pure complex produce invisibly broad NMR lines. We have, therefore, carried out our NMR studies by dissolving very small amounts of the complexes in the free ligand and, assuming rapid exchange between free and complexed ligand, investigating the time-averaged environment of the heterocyclic ring protons⁽¹⁾. The chemical shifts for the pure complexes are obtained by extrapolation of the shifts for known concentrations of complex. The results for the γ -picoline complexes of Co and Ni are listed below (Table 1). In the case of Co, the large low field shift of the -C-H protons and the high field shifts of the CH_3 - protons have been rationalised in terms of β -spin delocalisation from ligand \rightarrow metal and α -spin back-donation from metal \rightarrow ligand. Both these mechanisms lead to $\sigma \rightarrow \sigma^*$ and $\sigma \rightarrow \pi$ interaction at the nitrogen and result in large low field shifts of the 2- and 3- position protons. The CH_3 - group can be considered as an extension to the

π -electron system of the ring and hyperconjugation between the π -orbital and the -C- CH_3 fragment places β -spin density directly on

contd/

the CH_3 protons and this causes a high field shift. In the case of Ni, the α -proton shift is relatively small compared with the Co case. This can be explained on the basis that $\pi \rightarrow \sigma$ interaction is very weak, but the reason for this is not clear at present.

We have also undertaken some preliminary studies of the temperature dependence of the line widths of these spectra. In general, as the temperature is decreased from ca 50°C to ca 0°C all the bands become broader but the order of line widths remains $\alpha\text{-H} > \beta\text{-H} > \text{-CH}_3$. On

reducing the temperature still further, the $\alpha\text{-H}$ band first begins to sharpen appreciably followed by the $\beta\text{-H}$ band until at ca -50°C , in the case of $\text{Cu}(\text{CF}_3\text{CO}_2)_2 (\gamma\text{-pic})_4$, all three bands are almost as sharp as in the spectrum of the free ligand. We have attempted to explain these results on the following basis. It can be shown (2) that the line width of each band of the time-averaged spectrum is dependent on the transverse relaxation time of the proton in the pure paramagnetic complex,

$$\frac{1}{T_2} = \frac{1}{T_2^D} + \frac{[P]}{[D]} \left(\frac{1}{T_2^P} \right) \quad \text{if} \quad T_2^P \gg \tau_p$$

where $(1/T_2^D)$ is the line width of the proton in the free, diamagnetic ligand,

$(1/T_2^P)$ is the line width of the proton in the pure complex, $[P]$ and $[D]$ are the concentrations of pure complex and free ligand respectively and τ_p is the mean life-time of the paramagnetic species. $(1/T_2^P)$ differs for magnetically

inequivalent hydrogens in the complex and thus the line widths of the three bands α -, β - and CH_3 - protons will differ as is the case at higher temperatures.

On lowering the temperature, the exchange rate $P \rightleftharpoons D$ is reduced and it is postulated that it is reduced to such an extent that at the lowest temperatures, in the region of -50°C , $\tau_p > T_2^P$ and the observed line width expression

becomes

$$\frac{1}{T_2} = \frac{1}{T_2^D} + \frac{1}{\tau_D}$$

The observed line widths are now independent of the transverse relaxation time of the paramagnetic species, depending only on the life-time of the diamagnetic species in solution. Thus, the line widths are expected to be independent

contd/

8th February 1968.

of the proximity of the protons to the metal atom and thus similar to the line widths in the free ligand spectrum. This agrees exactly with the results obtained. The case of $\text{Cu}(\text{CF}_3\text{CO}_2)_2 (\gamma\text{-pic})_4$ is illustrated in Table 2 and Fig. 1.

Yours sincerely,

Keith G. Orrell

Christine A. Wrench.

K.G. Orrell.

C.A. Wrench (Miss).

References

- (1) J.A. Happe, R.L. Ward, J. Chem. Phys. 1963, 39, 1211.
- (2) C.S. Johnson, Advances in Magnetic Resonance 1965, 1, 33.

Title Contact Shifts and Line Widths of Co(II), Ni(II) and Cu(II) Complexes.

Table 1Chemical Shifts of the Pure Complexes

Metal	Complex ^(a)	α -H/ppm	β -H/ppm	CH_3^- /ppm
Co	L_4	-297	-79.6	+47.8
	L_2	-304	-91.3	+39.5
Ni	L_4	-69.0	-97.7	+34.3
	L_2	(b)	-59.1	+29.7

(a) Ligand L = γ -picoline.

(b) Band too broad to be observed.

Table 2

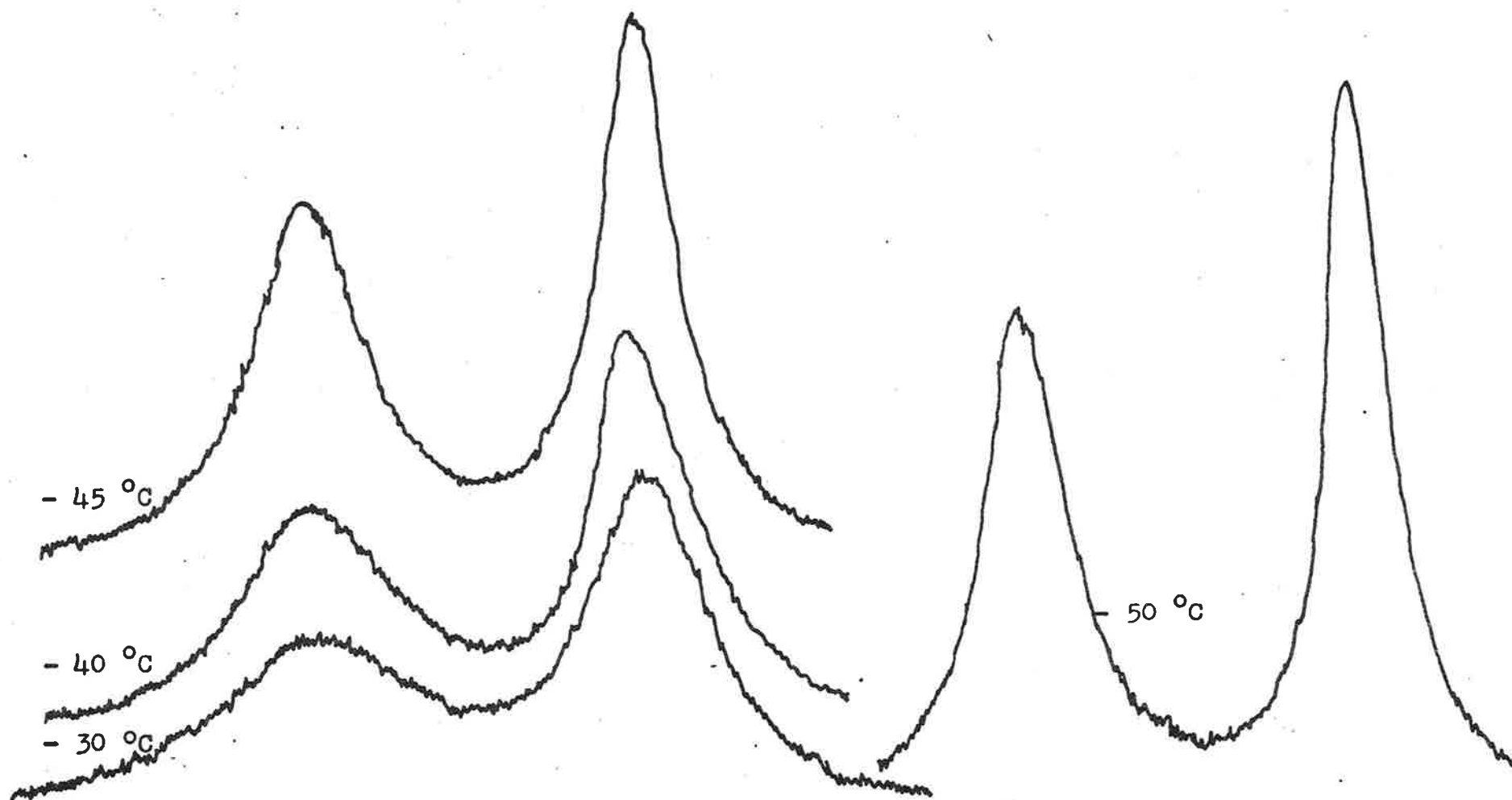
$\text{Cu}(\text{CF}_3\text{CO}_2)_2(\gamma\text{-pic})_4$ in γ -picoline (0.9997 mol fraction γ -picoline)

Temp/ $^{\circ}\text{C}$	Line width at Half Height/Hz		
	α -H	β -H	CH_3^-
+40	78?	21	2
+20	(a)	24	4
0	(a)	39	4
-20	132?	48	5
-30	71	40	6
-35	52	34	6
-50	21	15	7

(a) Unobservably broad.

Figure 1 (γ -picoline)CuTFA complex in free ligand only. Variable temperature NMR spectra of the aromatic hydrogen bands.

114-20



EMORY UNIVERSITY
ATLANTA, GEORGIA 30322

DEPARTMENT OF CHEMISTRY

February 15, 1963

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

Parameters and Solvent Effects for Fluorobenzene

Dear Barry:


In two recent issues of the IIT NMR Newsletter, T. F. Page (1) and S. Castellano (2) have reported analyses of fluorobenzene, and some discrepancies were noted between their results. We too have been working on fluorobenzene (3) and have found that our values agree quite well with those reported by Castellano, the largest deviation being 0.12 Hz in J_{15} , the meta H-H coupling across the substituent. For comparison, the three sets of values are collected in Table I.

Castellano also reported that no solvent or concentration effects could be inferred from his results. We had previously noted a concentration dependence of the H-F couplings in fluorobenzene at several concentrations in TMS (3), and decided to extend this study to several solvents. All spectra were obtained at 60 MHz with ten mole percent solutions of fluorobenzene in seven solvents. Our results are given in Table II.

The largest change, 0.30 Hz, is obtained for the ortho H-F coupling and appears to be significant since the uncertainty in these parameter, is estimated to be no greater than 0.05 Hz. The changes in the meta and para H-F couplings are not so large, and may or may not be significant. The H-H couplings showed no appreciable change with solvent except in one case, J_{15} , for which a variation of 0.22 Hz was found.

Further studies of solvent effects on H-F parameters are in progress and we hope to report the results before too long.

Sincerely,


J. H. Goldstein
J. E. Loemker
J. M. Read, Jr.

1. T. F. Page, Jr., IIT NMR Newsletter 110, 34 (Nov. 1967).
2. S. Castellano, IIT NMR Newsletter 112, 17 (Jan. 1968).
3. J. E. Loemker, J. M. Read, Jr., and J. H. Goldstein, Mol. Phys. 13, 433 (1967).

Table I. Comparison of Fluorobenzene Parameter^a

		From ref. (2)	From ref. (1)
J ₁₂	8.40	8.36	8.42
J ₁₃	1.10	1.07	1.12
J ₁₄	0.42	0.43	0.32
J ₁₅	2.65	2.77	3.13
J ₂₃	7.45	7.39	7.56
J ₂₄	1.74	1.81	2.13
J ₁₆	9.11	9.03	9.08
J ₂₆	5.74	5.74	5.82
J ₃₆	0.28	0.24	0.35
$\nu_1 = \nu_5^b$	12.44	12.45	12.00
$\nu_2 = \nu_4$	0.00	0.00	0.00
ν_3	10.58	10.57	10.46

^a For the neat liquid, in Hz.

^b At 60 MHz.

Table II. Solvent Effects on the NMR Parameters in Fluorobenzene

Parameter ^a	TMS	Acetone	Methanol	DMF	Acetonitrile	N-methyl- propionamide	CCl ₄
ν_o^b	-415.27	-424.77	-422.80	-428.68	-424.93	-423.75	-418.22
ν_m	-430.96	-442.25	-440.32	-444.50	-441.97	-440.43	-434.76
ν_p	-418.99	-429.28	-427.83	-431.24	-429.77	-427.70	-422.24
$J_{12} = J_{45}^c$	8.35	8.37	8.38	8.38	8.40	8.36	8.37
$J_{13} = J_{35}$	1.03	1.04	1.03	1.11	1.03	1.11	1.03
$J_{14} = J_{25}$	0.40	0.42	0.44	0.43	0.42	0.36	0.40
J_{15}	2.58	2.75	2.75	2.62	2.80	2.64	2.59
$J_{23} = J_{34}$	7.50	7.46	7.46	7.46	7.48	7.49	7.50
J_{24}	1.76	1.84	1.87	1.78	1.86	1.71	1.72
$J_{16} = J_{56} = J_o^{HF}$	8.90	9.31	9.28	9.38	9.44	9.02	8.64
$J_{26} = J_{46} = J_m^{HF}$	5.57	5.79	5.76	5.79	5.83	5.82	5.68
$J_{36} = J_p^{HF}$	0.20	0.34	0.33	0.51	0.39	0.24	0.29
$ \nu_o - \nu_m $	15.69	17.48	17.52	15.82	17.04	16.68	16.54
$ \nu_o - \nu_p $	3.72	4.51	5.03	2.56	4.84	3.95	4.02

^a Numbering begins with proton adjacent to fluorine

^b At 60 MHz, relative to TMS, in Hz.

^c In Hz.

ABBOTT LABORATORIES. SCIENTIFIC DIVISIONS, Chemical Physics Lab.
NORTH CHICAGO. ILLINOIS 60064 U. S. A.

February 13, 1968

Dear Barry:

DELTA SCALE ONLY, PROPOSED BY ASTM

As many of our readers know, the American Society for Testing and Materials has maintained Committee E-13 on absorption spectroscopy, one subcommittee of which is E-13.7 on NMR spectroscopy. For many years the able chairman of E-13.7 was Bill Ritchey; in 1966, this responsibility was transferred to Ed Brame. Committee members contributing very important ideas during the development of NMR spectroscopy have phased in and out. It may be interesting to see the present roster: H. Agahigian, R. H. Bible, E. G. Brame, W. S. Brey, S. Brownstein, N. F. Chamberlain, M. W. Dietrich, R. A. Friedel, H. Foster, W. Fulmor, H. S. Gutowsky, L. F. Johnson, J. E. Lancaster, P. C. Lauterbur, F. Mannis, R. W. Mattoon, N. L. McNiven, M. T. Melchior, F. Nelson, R. T. O'Connor, W. M. Ritchey, E. J. Rosenbaum, P. S. Sadtler, H. Skala, W. C. Smith, E. Stejskal, P. A. Strauss, G. V. D. Tiers, J. S. Waugh, C. W. Wilson, and J. R. Zimmerman.

The task group on referencing, standards, and nomenclature, headed by George V. D. Tiers (τ -scale), and recently by Harlan Foster, has spent much time in arriving at proposed terms, symbols, and conventions. These were published in the ASTM MANUAL. I was asked to give this broad publicity. Following is the excerpt on NMR. Some readers may wish to purchase the complete MANUAL available from ASTM.

The most important item proposed for international convention and publications on proton spectra (Section 4) is the following.

Chemical shifts should be in ppm with 0 at TMS, δ -scale, see Sections 2.3, 4.1.9, 4.3, 5.1.1, and 5.1.2.

The Committee strongly urges the τ -minority to cease using that arbitrary 10 and avoid the micron vs. wavenumber confusion in infrared. Technical comments should be addressed to Dr. Edward G. Brame, Jr., Chairman, ASTM Committee E-13.7, Building 353, Room 18, Du Pont Experiment Station, Wilmington, Delaware 19898, U. S. A.

Best hopes for universal resonance and magnetic homogeneity!

Sincerely yours,

Richard

Richard W. Mattoon
Publicity, E-13.7 on NMR

MANUAL ON RECOMMENDED PRACTICES IN SPECTROPHOTOMETRY
Sponsored by ASTM COMMITTEE E-13 on Absorption Spectroscopy

Nomenclature, Adequacy of Available Equipment, General Qualitative Analysis, Infrared Methods, Ultraviolet Methods, Complete Index to Spectrophotometric Methods

FIRST EDITION, FEBRUARY, 1966, List Price: \$3.50
Published by the AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103

DEFINITIONS RELATING TO ABSORPTION SPECTROSCOPY (E 131) 3

TERMS AND SYMBOLS RELATING SPECIFICALLY TO NUCLEAR MAGNETIC
RESONANCE (NMR) SPECTROSCOPY²

Absorption Band, NMR Band.—A region of the high-resolution NMR spectrum in

which a detectable signal exists and passes through one or more maxima.

Band Width (NMR), W .—The width of an NMR absorption band measured at one half of the maximal band-height. The recommended unit is cycles per second.

Basic Frequency (NMR) ν_0 .—The fre-

² For additional definitions relating to nuclear magnetic resonance, see the Proposed Terms, Symbols, Conventions, and References Relating to Nuclear Magnetic Resonance (NMR) Spectroscopy, which appears in this publication.

4 DEFINITIONS RELATING TO ABSORPTION SPECTROSCOPY (E 131)

quency of the radiofrequency (r-f) magnetic field, H_1 .

External Reference (NMR).—A reference compound placed in a separate container (or present in a separate phase) from that containing the sample. It may be used to establish reproducibility of the applied magnetic field external to the sample in order to make possible the study of non-specific solvent interactions and bulk magnetic susceptibility effects. External references are often used because a convenient internal reference is not available.

Field-Sweeping (NMR).—Uniformly varying the magnetic field strength, H_0 , to bring nuclei, differing in degree of shielding, successively into resonance; thereby making available an NMR spectrum consisting of signal intensity versus field strength.

Field-Sweep Rate (NMR).—The rate at which the magnetic field strength, H_0 , is varied. The recommended units are cycles per second for high-resolution NMR.

Internal Reference (NMR).—A reference compound, dissolved in the medium containing the sample, used to minimize or eliminate the effects of concentration, temperature, bulk magnetic susceptibility, and nonspecific solvent interactions upon the spectral position.

Nuclear Magnetic Resonance (NMR) Spectroscopy.—A form of spectroscopy concerned with radiofrequency-induced transition between magnetic energy levels of atomic nuclei in a magnetic field.

NMR Apparatus; NMR Equipment.—An instrument comprising magnet, radiofre-

quency oscillator, sample holder, sweep unit, and detector, capable of producing an oscilloscope image or line recording of an NMR spectrum. The instrument need not provide for quantitative measurement of spectral position or signal strength.

Reference Compound (NMR).—A compound that produces at least one clearly defined NMR absorption band, the spectral position (or intensity) of which is taken as a standard for the measurement of other spectral positions (or intensities).

Spin-Spin Coupling Constant (NMR), J .—A measure of the degree of indirect coupling between the magnetic moments of neighboring nuclei. In the simplest cases, it is the separation in frequency between adjacent component bands of a spin-spin multiplet, but in more complex cases, it must be determined by detailed calculations. It is independent of the strength of the applied magnetic field. The only recommended unit for J is cycles per second.

Spin-Spin Multiplet Band (NMR).—The group of high-resolution NMR bands that are (components) produced by a single type of nucleus under the influence of neighboring nuclei to which it is magnetically coupled. A total multiplet may consist of a group of sub-multiplets when a nucleus is coupled to several neighboring nuclei and should be designated "multi" or "m" when components are resolved but not satisfactorily analyzed. When components are unresolved, the resulting (broadened) band should be designated "broad" or "b."

PROPOSED TERMS, SYMBOLS, CONVENTIONS, AND REFERENCES RELATING TO NUCLEAR MAGNETIC RESONANCE (NMR) SPECTROSCOPY¹

These are proposed terms and symbols and are published as information only. Comments are solicited and should be addressed to the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

1. Nomenclature and Definitions

1.1 *Background (NMR)*—Apparent absorption caused by anything other than the substance under investigation or the internal or external reference.

1.2 *Carbon-13 Satellites*—Weak NMR bands, often multiplet, characteristic of the sample (or solvent) and which are spaced nearly symmetrically about the principal sample bands (the frequency spacing being independent of the basic NMR frequency). They are produced by spin-spin coupling of the observed nucleus with the carbon-13 present, usually in its natural abundance of 1.1 per cent. Thus they are generally observed only when the principal band is of high intensity. These bands always should be identified to prevent misinterpretation as impurities.

1.3 *High-Resolution NMR Spectrometer*—An NMR apparatus that is so adjusted that the NMR band width, W , for tetramethylsilane using the absorption mode, at 1 per cent concentration in carbon tetrachloride, is less than 3 cps, and which is equipped for the calibration of NMR spectra to an accuracy of 0.6 cps or better. Alternative spectrometric testing procedures that are equivalent in performance to the foregoing may be employed. Unless

stated otherwise, all definitions refer to the absorption mode.

1.4 *Modulation Sidebands*—Extraneous bands introduced, usually for purposes of calibration, into the NMR spectrum by audiofrequency modulation of the resonance signals. At lower frequencies this may be accomplished by modulation of the static magnetic field; at higher frequencies (above about 1500 cps) by frequency modulation of the basic frequency. Another technique is amplitude modulation of the basic frequency, as the sideband heights become virtually independent of modulating frequency.

1.5 *Radiofrequency Field (r-f Field) (NMR), H_1* —The amplitude of the effective rotating component of the r-f magnetic field within the sample volume. The recommended unit is milligauss.

1.6 *Resolution, Spectral (NMR)*—The narrowest observable NMR band width according to 1.3, for a single sharp band in a spectrum. When only broad or complex bands are present, it would be necessary to employ an internal reference compound, such as tetramethylsilane, to permit estimation or measurement of spectral resolution.

1.7 *Spinning Sidebands*—Extraneous spectral bands located in pairs, more or less symmetrically about the true sample bands. They are produced as a result of

¹ These proposed terms and symbols are under the jurisdiction of the ASTM Committee E-13 on Absorption Spectroscopy.

6 TERMS, SYMBOLS RELATING TO NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

mechanical modulation of the static magnetic field. The separation between a sample band and its spinning sidebands, in cycles per second, equals an integral multiple of the spin rate in revolutions per second. They are readily identified by virtue of the displacement produced upon altering the spin rates.

2. NMR Conventions

2.1 The multiplying factor used with the dimensionless shift or shielding parameter should be 10^6 (ppm).

2.2 The unit used for "raw data" (peak positions) should be cycles per second.

2.3 The dimensionless and frequency scales should have a common origin.

2.4 The standard sweep direction should be from low to high applied field (or high to low radio frequency).

2.5 The standard orientation of spectra should be with high applied field on the right.

2.6 Absorption mode peaks should point up.

3. NMR Referencing Procedures

3.1 When possible, the scale should be tied to an *internal* reference.

3.2 Capillary tubes are generally adequate.

3.3 Oxygen generally need not be removed from samples and reference liquids.

3.4 A precision of measurement of ± 0.5 cps is adequate for most published spectra.

3.5 A precision of measurement of ± 0.2 cps is adequate for most published J values.

4. NMR Data Presentation

4.1 The following should be specified whenever NMR data are published:

4.1.1 Name of solvent and concentration.

4.1.2 Name and concentration of internal reference.

4.1.3 Name of external reference.

4.1.4 Temperature of sample in probe.

4.1.5 Procedure used for measuring peak positions.

4.1.6 Radio frequency at which measurements were made.

4.1.7 Mathematical operations used to analyze the spectra.

4.1.8 Numbers on the frequency scale (if used). They should increase from high to low applied field.

4.1.9 Dimensionless and frequency scales. They should run in the same direction.

4.2 The following additional information should be given when the spectra themselves are published:

4.2.1 Sweep rate.

4.2.2 Values of both r-f fields when spin decoupling or double resonance is employed.

4.3 The shifts and couplings obtained from the spectra should be reported, the former in dimensionless units (ppm) and the latter in frequency units (cps).

5. NMR Reference Substance for ^1H Spectra

5.1 The primary internal reference for hydrogen spectra in nonaqueous media shall be tetramethylsilane (TMS).

5.1.1 The position of the tetramethylsilane resonance shall be defined as exactly 0.0 ppm.

5.1.2 The numbers on the dimensionless (shift) scale downfield from TMS should be positive.

5.2 Neither water nor benzene is recommended as either an internal or an external reference substance.

6. NMR Reference Substances for Nuclei Other Than Hydrogen

6.1 The primary internal reference for

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TERMS, SYMBOLS RELATING TO NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY 7

F^{19} spectra shall be trichloromonofluoromethane (CCl_3F).

6.1.1 The numbers on the dimensionless (shift) scale upfield from the reference should be positive.

6.2 The primary reference for C^{13} spectra shall be carbon disulfide (CS_2).

6.2.1 The numbers on the dimensionless (shift) scale upfield from the reference should be positive.

THE UNIVERSITY OF LIVERPOOL
DEPARTMENT OF ORGANIC CHEMISTRY

TELEPHONE: ROYAL 6022



THE ROBERT ROBINSON LABORATORIES,
OXFORD STREET,
LIVERPOOL 7.

16 February 1968

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

Dear Barry,

Many thanks for the back issues of I.I.T., N.M.R.N. you sent. Your generosity prompted me to write a contribution without receiving one of your reminders. I hope this is in order!

I am in the middle of writing a chapter on computer analysis of spectra for my book. This plus my year at Mellon using LAOCOON III, has prompted some thoughts about the analysis of complex (i.e. non-explicit) spectra.

What worries me is that there is appearing in the literature a number of such analyses, both by computer fits and by "visual matching" in which nobody has any idea of the errors in the resulting parameters. We had an extreme example of this at Mellon, when Dr. Castellano and I analysed an ABC spectrum recently in which, although all the transitions in the spectrum were matched to within 0.03 c.p.s., the errors in some of the coupling constants were ca 2 c.p.s! We believe (or hope) that this is an exceptional case. However, many other analyses are appearing in the literature in which the stated errors turn out to be too small by an order of magnitude. I am therefore tentatively raising the following suggestions for recording spectral analyses:

(a) For first-order or explicit spectra (e.g. ABX, A_2X_2), the inaccuracies in the molecular parameters are of the same order as the inaccuracies in the measured frequencies, apart from deceptively simple spectra. Thus the universal ± 0.1 c.p.s. could probably still stand.

(b) ...

- 2 -

(b) In spectra in which most or all of the parameters are not obtained from algebraic expressions, visual agreement between observed and calculated spectra should not constitute proof of a correct analysis, and should therefore not be publishable as such. (Obviously interesting spectra are publishable whether or not they are analysed.)

(c) In the case of the iterative computer analysis of such spectra, the following data should be recorded with every published analysis:

- i) The number of spectra recorded and the r.m.s. error of the measured frequencies.
- ii) The number of transitions used for the iteration and the theoretical number of transitions.
- iii) The r.m.s. error or mean deviation of the observed and calculated spectra.
- iv) The probable errors for all the parameters iterated.
- v) The maximum deviation of any calculated and observed line.

Also all the observable transitions should be used for the iteration excluding only those which are incompletely resolved. The two major programmes for calculating spectra, LAOCOON III and NIMRIT/NIMREN, both give all this data in their output and it seems to me that this is the only way we are going to know the accuracy of the parameters that are being obtained.

I know this procedure is routine in many establishments, but it would be of interest to know the collective view of I.I.T., N.M.R.N. readers.

Yours sincerely,



R. J. Abraham

CYANAMID

AMERICAN CYANAMID COMPANY
STAMFORD RESEARCH LABORATORIES
1937 WEST MAIN STREET, STAMFORD, CONN. 06904
AREA CODE 203 348-7331

January 31, 1968

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

Dear Barry:

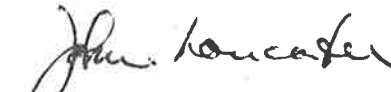
The reconstruction of our laboratory and installation of our HA-100 has been a considerable distraction of late, so we wish to apologize for having to be "reminded" again.

The attached 60 MHz spectra were obtained in connection with some work with W. K. Hausmann and D. B. Borders (of our Lederle Laboratories) on the structure of aspartocin. One of the hydrolysis products was α, β diamino butyric acid which was obtained as a crystalline compound following ion-exchange chromatography. Further resolution by preparative chromatography on an amino acid auto-analyzer yielded two diastereoisomers (erythro and threo forms) of this acid.

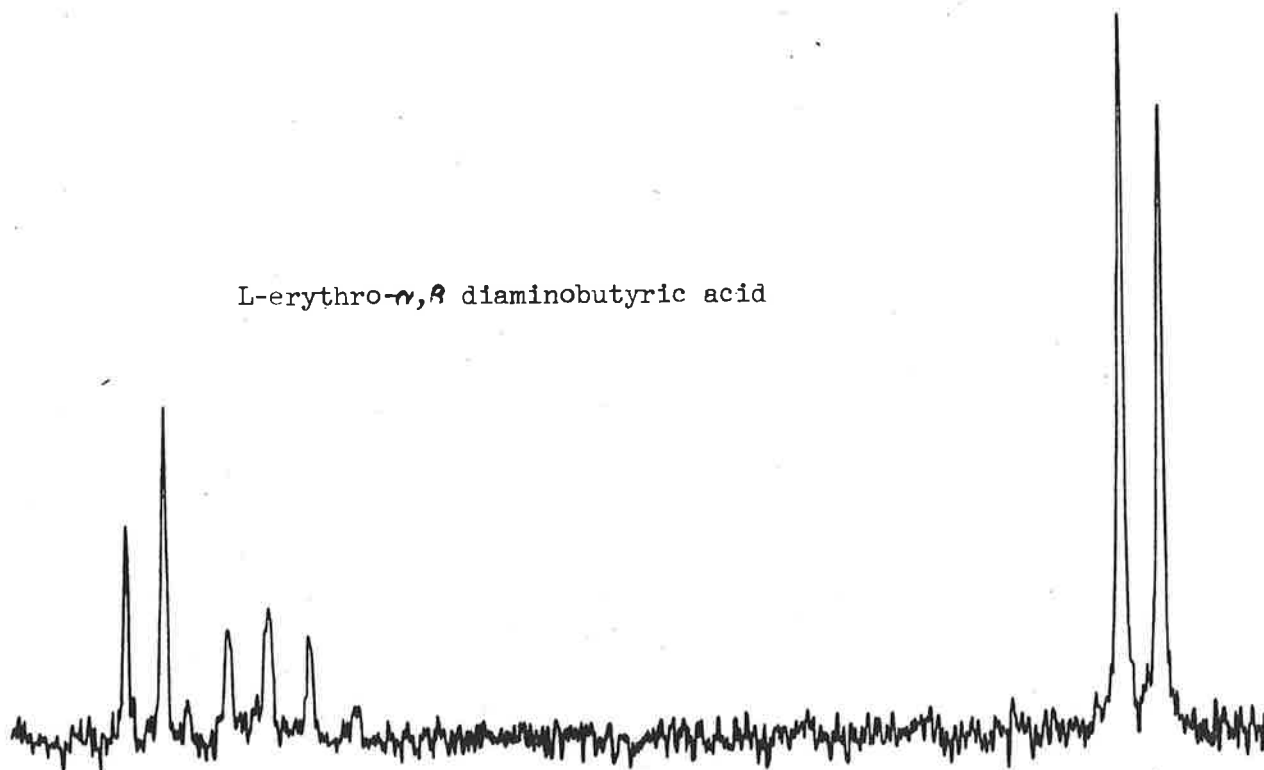
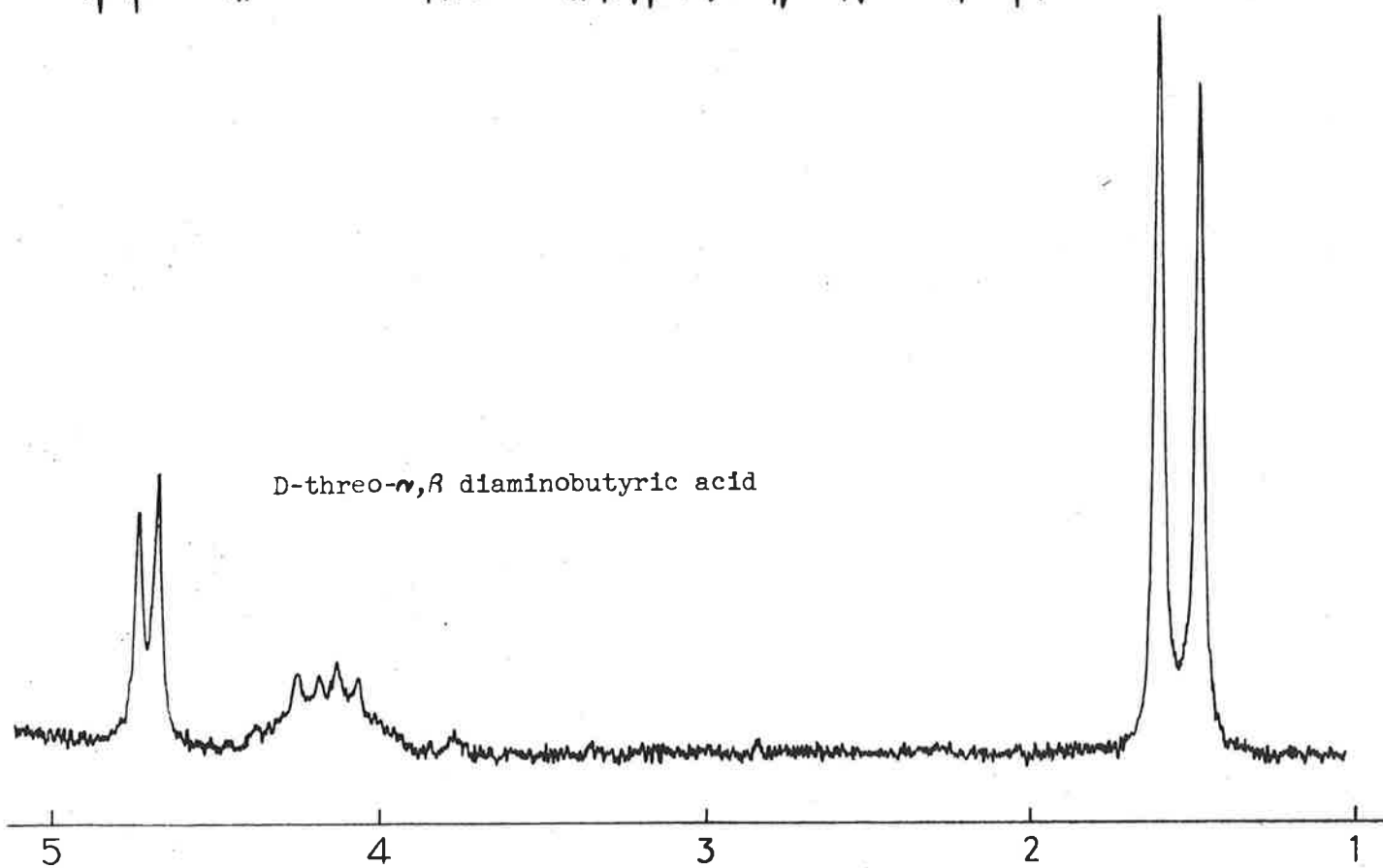
The proton spectra obtained in strongly acidified D₂O are shown in the accompanying figure. The shifts were observed to be typically pH-dependent, but these were not studied in detail. The vicinal couplings are different (3.6 vs. 6.6 Hz) in the two isomers, and were observed to be constant over the temperature range -40° to +80°C. This suggests the possibility that the rotation about the C α -C β bond is quite restricted, perhaps due mainly to the electrostatic repulsion of the two NH₃⁺ groups. If the NH₃⁺ groups are trans to each other due to electrostatic repulsion, the vicinal hydrogens would be gauche and trans to each other for the threo and erythro forms, respectively. On this basis, the spectrum with the larger coupling was assigned to the erythro isomer. An "average J" for free rotation calculated from these values of J_g and J_t is smaller than that usually observed, but increasing electronegativity of substituents is known to decrease the average J. The further assignment to L & D configurations was done by ORD studies.

The original crystalline material was found to be a 1:1 mixture of the isomers. Since it was also demonstrated that isomerization could occur under the conditions of the hydrolysis, the question of the stereochemistry of the amino acid moiety in aspartocin remains unanswered.

Very truly yours,


J. E. Lancaster

pps

L-erythro- α,β diaminobutyric acidD-threo- α,β diaminobutyric acid

M.V. Koninklijke Pharmaceutische Fabrieken N.V.
Brocades-Aheeman & Pharmacia
 RESEARCH LABORATORIUM
 POSTBUS 523
HAARLEM, THE NETHERLANDS

Subject: T_1 and J determination with the A-60-C1024 combination.

Professor Dr. B.L. Shapiro
 Dept. of Chemistry
 Stanford University
Stanford (Cal.) 94305 U.S.A.

Haarlem, February 12, 1968

Dear professor Shapiro,

Recently we learned about the existence of your NMR Newsletter. Looking through a whole pile of old issues, made us anxious to get on your mailing list.

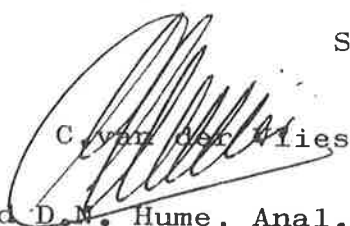
We hope you will accept the following as our first contribution to achieve such.

In your pages much has been written about CAT's and Mouses, but as far as we noticed, not yet about using the CAT with the A-60 to measure relaxation times (T_1) and small line separations. To obtain the spin-lattice relaxation time T_1 in the "direct method" ¹, the CAT is used as a fast recorder. The RAMP-plug has to be disconnected and the sample not spinning. The recorder is placed on the top of the peak and the appropriate values of band width and sweep time must be chosen. The recording of the relaxation trace is triggered in the INT. TRIGGER position, by switching the TRIGGER SOURCE from SIDEBAND to RECUR.

The unsaturated level can be recorded with a 100x longer sweep time prior to saturation and relaxation. However, with very short T_1 's, the required fast manual operation of sweep time knob and attenuator switch may give difficulties. An electronic switch (if necessary, with a time delay), to trigger, speed up and attenuate may then be the answer. The memory is read out with the A-60 recorder and T_1 is determined in the usual way (fig. 1).

To determine small separations with the wiggle beat method ² the CAT is used as a fast sweep unit (RAMP connected) and recorder. Under optimal resolution conditions, appropriate sweep width, sweep time and filter bandwidth, a nice pattern of wiggle beats is obtained in the memory. This can be read out and the separation can be determined from the number of maxima per second. (fig. 2)

Sincerely yours

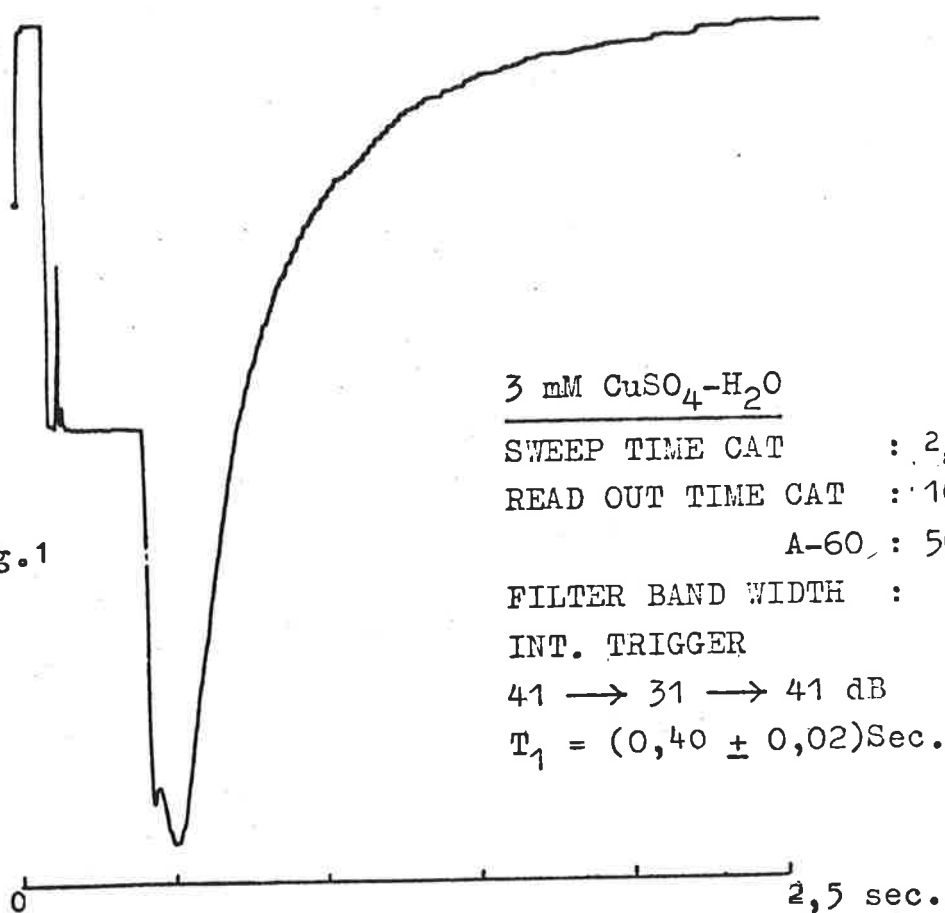

 C. van der Wries


 B.C. Caron

¹ A.L. van Geet and D.M. Hume, Anal.Chem. 37, 983 (1965)

² Pople, Schneider, Bernstein, page 76.

Fig.1



ACETALDEHYDE

CAT SWEEP TIME : 2,5 sec.
SWEEP WIDTH : 10 cps.
READ OUT CAT : 100 sec.
A-60 : 500 sec.
FILTER BANDWIDTH : 4
INT. TRIGGER
S.A. : 0,63
RF ATTENUATION : 55dB
 $J = 2,86 \pm 0,03$ cps.

Fig.2





Imperial Chemical Industries Limited

PHARMACEUTICALS DIVISION

Merceside, Alderley Park, Macclesfield, Cheshire

Telephone: ALDERLEY FDO. 2528

Cables: AVLONTEX, MACCLESFIELD

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

Your Ref:

Our Ref: GRB/DN

19th February, 1968

Dear Dr. Shapiro,

The temperature dependence of the n.m.r. spectra
of vinyllogous amides

The figure shows the 100 Mc/s proton resonance spectrum of the region 2.0 - 3.5 τ for 1-amino 2-benzoyl ethylene in CDCl₃ solution at -40°C and +60°C. At -40°C the ethylenic proton 1 is centred at τ 3.01 and is a doublet of triplets (J = 7.8, 7.8, 15.5 cps). The resonance for this proton at +60°C is centred at τ 3.08 and appears as a triplet of doublets (J = 7.8, 11, 11 cps). The outer lines of the multiplet are 31 cps apart at both -40°C and +60°C while at intermediate temperatures the inner lines are broadened. The coupling constant of 7.8 cps common to the multiplets at both temperatures was attributed by inspection and double irradiation to coupling with the ethylenic proton 2 at τ 4.3. This confirms a cis configuration of the ethylenic protons. Two NH signals are observed, at -40°C they are at 0.5 τ and 4.6 τ and at +60°C they are too broad to measure.

We interpret our findings in terms of restricted rotation of the C=N bond. At high temperatures the value of the coupling constant between the ethylenic proton 1 and the amino protons is a mean value due to rapid rotation of the C=N bond. This is supported by broadening of the NH signals. We suggest that intermolecular exchange of the amino protons is a minor factor since we would have expected considerable broadening of the whole multiplet.

The N¹⁴ chemical shift measured at 40°C by the heteronuclear decoupling method (in kind co-operation with Dr. A. Mathias and Mr. P. Hampson of I.C.I. [Dyestuffs Division] Ltd., Blackley, Manchester) is 285 \pm 5 ppm⁽¹⁾. This value is different from that normally found for amino groups attached to unsaturated units (310 - 330 ppm)⁽²⁾, but is very similar to that in amides e.g. benzamide 282 \pm 2 ppm and hence the amidic nature of the nitrogen atom is confirmed.

At -40°C , the lower field NH couples to the ethylenic proton 1 with the coupling constant 15.5 cps. This has been proved by double irradiation and is consistent with the amino proton being trans to the ethylenic proton 2 and cis to the carbonyl group. Similar results have been obtained for formamide⁽³⁾ and 3-aminoacrylic esters^(4,5). No chemical shift changes in the ethylenic signals are observed which would suggest that rotation about the ethylenic C-C bond is not occurring as observed recently for 4-pyridyl-acetaldehyde⁽⁵⁾.

The temperature dependence of the spectra of aminomethylene malonate and several differently substituted aryl derivatives of the compound described have been studied and all show similar properties. We have not yet analysed our spectra to obtain an activation energy for rotation about the C-N bond because in deuteriochloroform the coalescence temperature of the NH signals has not yet been obtained but our results⁽⁷⁾ for NN' dimethyl derivatives are in agreement with those reported⁽⁷⁾.

Yours sincerely,

G. R. Bedford

D. Greatbanks

D. B. Rogers

G. R. BEDFORD

D. GREATBANKS

D. B. ROGERS

References

- (1) The N^{14} chemical shift is quoted with respect to that of the nitrate nitrogen of a 4.5 M solution of A.R. ammonium nitrate in 3 N aqueous HCl (2).
- (2) D. Herbison-Evans and R. E. Richards, Molec. Phys., 8, 19, 1964.
- (3) L. H. Piette, J. D. Ray and R. A. Ogg, J. Mol. Spec., 2, 66, 1958.
- (4) W. Bottomley, J. N. Philips and J. G. Wilson, Tet. Letters, 1967, 31, 2957.
- (5) R. Huisgen, K. Herbig, A. Siegl and H. Huber, Chem. Ber., 99 2526, 1966.
- (6) R. J. Parry, Chem. Comm. 1967, 1294.
- (7) H. E. A. Kramer and R. Gompper, Tet. Letters, 1963, 15, 969; Zeitschrift fur Physik., 43, 292, 1964.



100

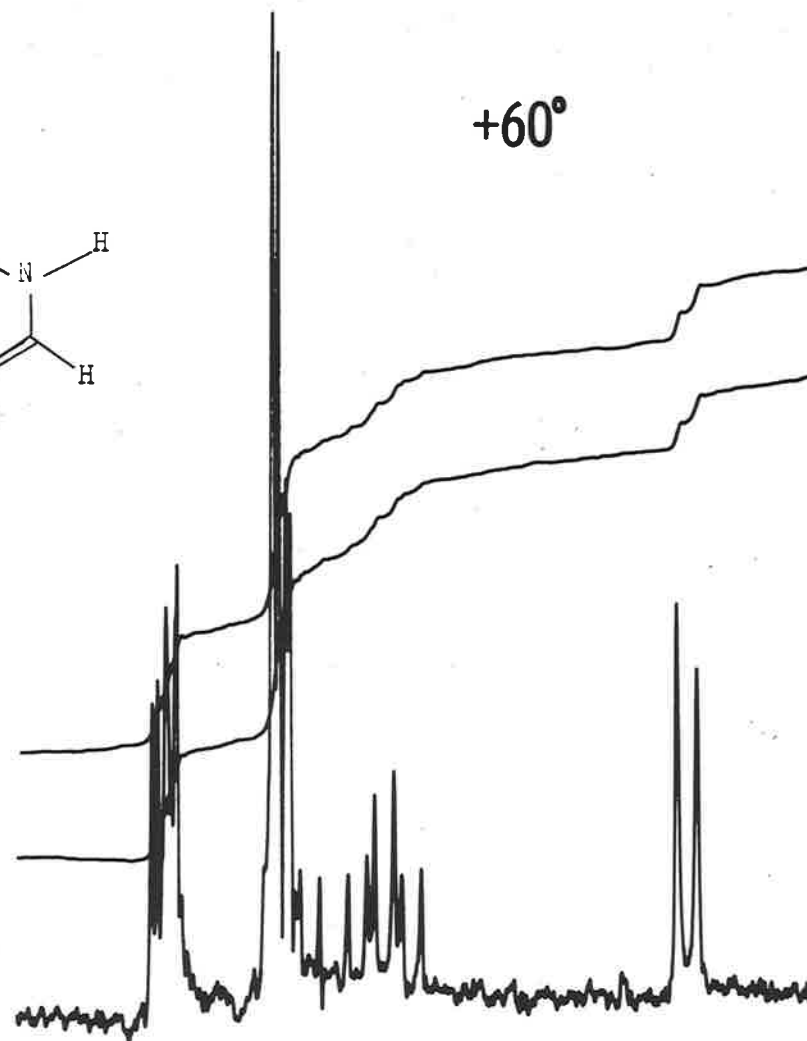
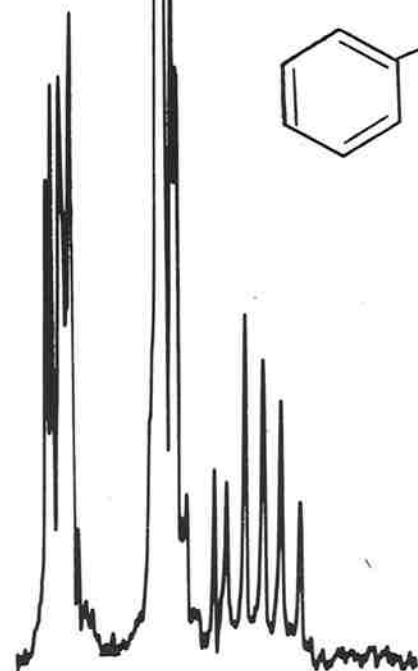
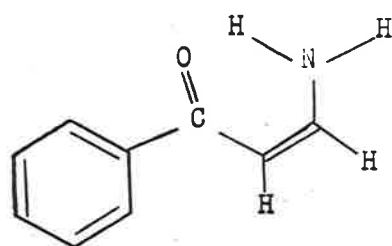


400

300

-40°

+60°



The Standard Oil Company

(An Ohio Corporation)

Research Department

4440 Warrensville Center Road

Cleveland, Ohio 44128

E. C. HUGHES
VICE PRESIDENT

February 20, 1968

Professor B. L. Shapiro
Editor, IITNMR
Illinois Institute of Technology
Chicago, Illinois 60616

Chemical Shifts of
Methylated Hydrazines

Dear Barry:

We wish to report the proton magnetic resonance chemical shifts of a series of methylated hydrazines taken in DMSO- d_6 . In all cases the methyl resonance was a single sharp band.

Regarding the data, several points may be considered. In Species I no exchange occurs between the N atoms of any group or atom \therefore the plus charge resides fully on one nitrogen. In the other species (except II) hydrogen exchange can cause plus charge delocalization between two sites resulting in an upfield shift of the N-CH₃ resonance toward the simple hydrazine analog (Species II).

Further, it might be expected that the presence of 2 methyls on 1 N atom (Species V) rather than 1 on each N atom (Species VI) might cause V to exhibit resonance at a significantly lower field because of the relative basicity of methyls. This is not the case. Species V appears at only slightly lower field than VI.

The dimethyl sulfoxide solvent may be exerting a leveling effect upon the methyl chemical shifts by acting as a base to the labile hydrogens, so that on the time average these species are much less "salt-like" than anticipated. Species IV is a possible exception owing perhaps to the few exchange sites and the relatively large number of methyl groups.

Very truly yours,

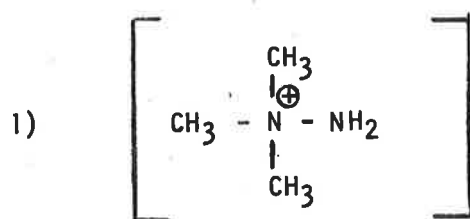


F. J. Knoll

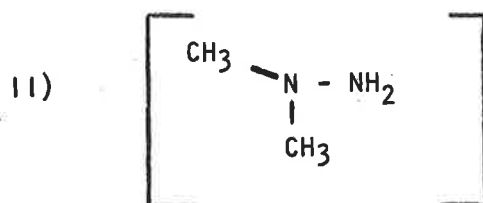


W. M. Ritchey

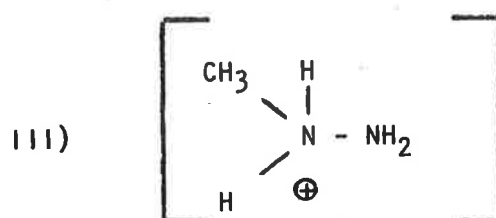
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Attachment

δ_{CH_3} 

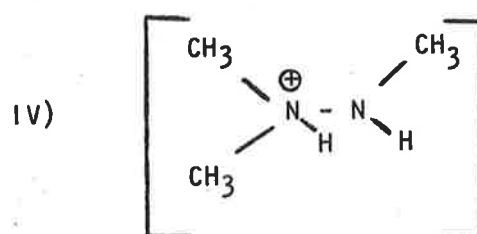
3.40



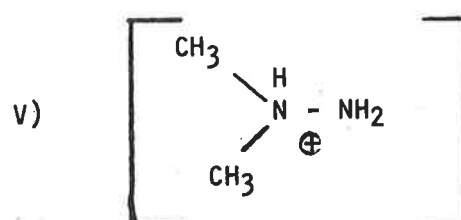
2.44



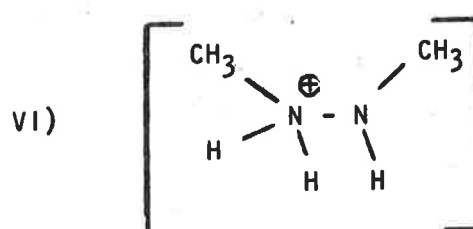
2.56



2.85



2.60



2.52

STOCKHOLM 70
SWEDEN

Stockholm, February 21, 1968

Cable address: Technology

EF/IB

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

Dear Dr. Shapiro,

Proton lattice changes in clays

In a wide line investigation of proton lattice changes in some pure clay minerals carried out by Arvid Jacobsson we have measured the bandwidth, expressed by the square root of the second moment, as a function of the temperature to which the sample is heated in vacuum before observation at room temperature. The clay minerals investigated are the water indifferent or almost hydrophobic pyrophyllite and the strongly hydrophilic montmorillonite, both belonging to the dioctahedral series. A comparison has further been made between the hydrophobic talc and the strongly hydrophilic hectorite, both belonging to the trioctahedral series.

In the observed temperature range between 80°C and 400°C, no structural changes are revealed by changes in the bandwidth of the two trioctahedral sample spectra.

Of the samples belonging to the dioctahedral series the montmorillonite is however subjected to structural changes, the bandwidth increasing from about 3 gauss at 80°C to 4 gauss at 400°C heat pretreatment. The final bandwidth observed is independent of the type of exchangeable cation present, while the paths to this level differ with the pretreatment and the type of cation.

- 2 -

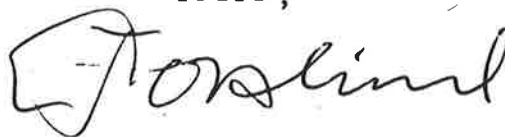
Pyrophyllite on the other hand remains as stable up to 400° C as the trioctahedral samples and shows no indications of structural rearrangements.

We conclude that the exchangeable cations influence the transformation mechanism while the final result of the transformation only depends on the intrinsic structural properties of the montmorillonite lattice.

Since it is generally believed that isomorphous substitutions in hectorite occurs only at central lattice positions of octahedral coordination, while montmorillonite shows both tetrahedral and octahedral substitution positions, it remains to investigate some other members of the trioctahedral series, which have the tetrahedral type of substitution. This should reveal whether the presence of tetrahedral substitutions can induce the transformation in the case of the trioctahedral lattice, which now appears insensitive to octahedral substitutions.

With kind regards,

Yours,

A handwritten signature in dark ink, appearing to read 'Erik Forslind', with a stylized, cursive script.

Erik Forslind

(Spectroscopie Hertzienne)

FACULTÉ DES SCIENCES

9, Quai Saint-Bernard - 75 - PARIS 5^e

Tour 32 - 2^e Etage

Tél. : 336-25-25 - Poste 40-53

February 21 1968.-

Dr. Bernard L. SHAPIRO
Department of Chemistry
Stanford University
Stanford California 94305.-

Proposed title : Bulk magnetic susceptibilities determination by N.M.R.-

Dear Professor Shapiro,

In a previous communication ⁽¹⁾, about a work performed by Drs DAYAN and WIDENLOCHER on gases, a shift proportional to the bulk magnetic susceptibility has been observed : this was attributed to a bad gauging of the thick tube used. They turn this result to account with the view of measuring paramagnetic susceptibilities using special tubes ⁽²⁾ with smelt-in capillary (here H₂O filled). The capillary H₂O serves as external reference and all around the tube is filled with a Cl₂Ca, 2H₂O solution : they obtain v.s. bulk susceptibility the following curve. The shift of the H₂O signal is observed towards the decrease field, proportional to the solution concentrations and so to the bulk paramagnetic susceptibility of the solution. The point for which χ_v is negative (diamagnetic) corresponds to tube filled with pure water ; the air bulk susceptibility being negligible the curve goes through the origin.-

This shift can be due to the bad gauging of the capillary inside the sample tube : indeed, at the capillary the field may be representable by

$$H \simeq H_0 [1 - 2\pi (\chi_{v_2} - \chi_{v_1})(1 + \epsilon)]$$

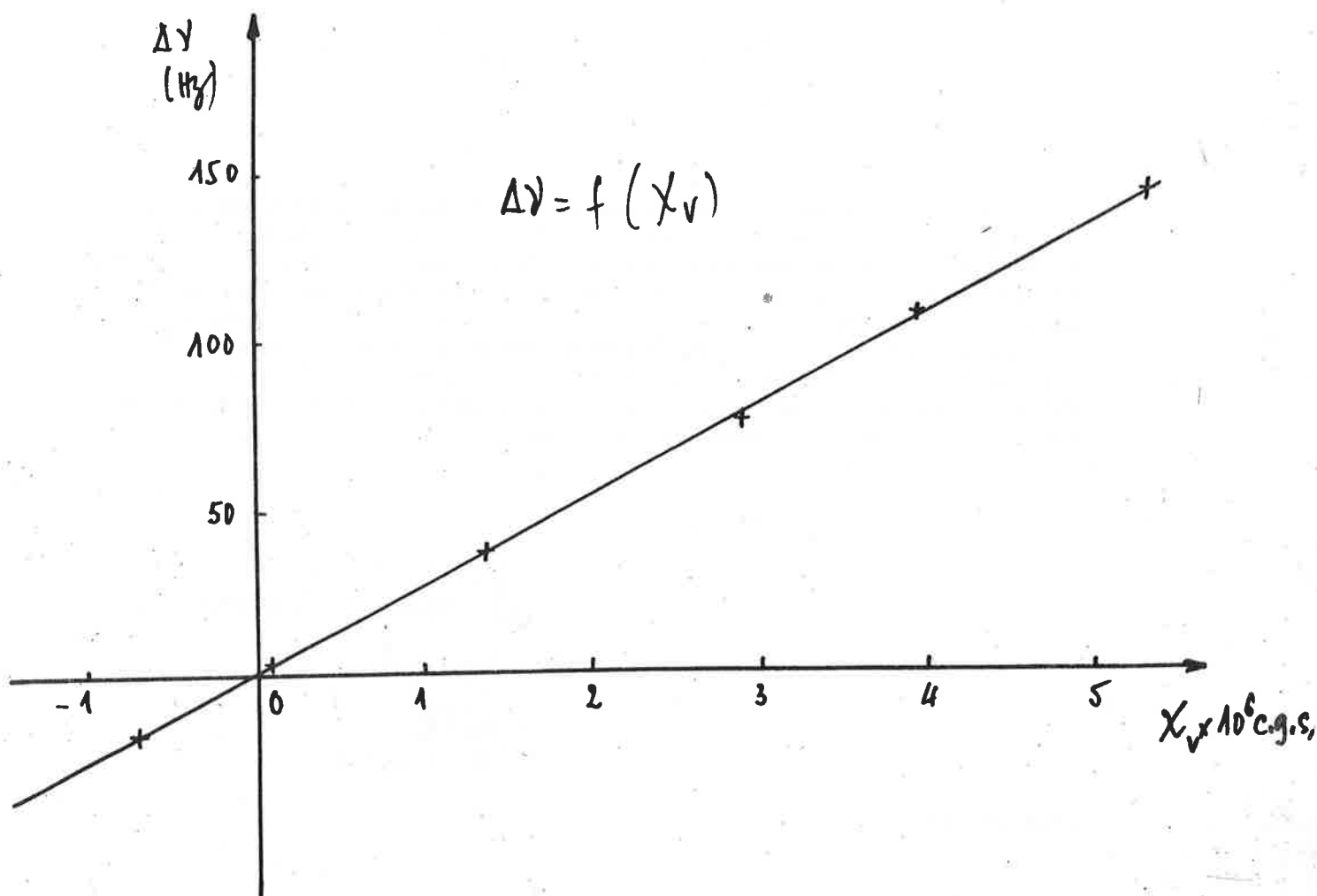
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where H_0 is the external magnetic field, χ_v and χ_v , water and paramagnetic solution, respectively, bulk magnetic susceptibilities and \mathcal{E} is a calibration coefficient. From this curve or a similar one, it is possible to determine easily the bulk magnetic susceptibility of a compound either in liquid state or powder state with some cautions.-



Sincerely yours.-
Professor R. FREYMAN

- 1) R. FREYMAN private comm. Mello N.M.R. N°58 1963
- 2) Special tubes from Glas Triebel Mannheim.-



CALIFORNIA INSTITUTE OF TECHNOLOGY

PASADENA, CALIFORNIA 91109

GATES AND CRELLIN LABORATORIES OF CHEMISTRY

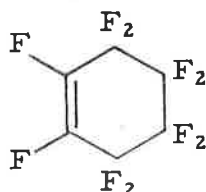
February 21, 1968

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

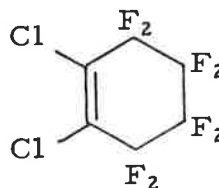
Dear Barry:

Inversion in Fluorinated Cyclohexenes

We recently had our A56/60 spectrometer modified for operation down to -150° . With this new capability, interesting information was obtained for ring inversion of I and II.



I



II

These substances give three and two groups of absorptions, respectively, at room temperature. These absorptions are essentially singlets with fine splittings due to vicinal couplings. At -150° , each singlet (except for the resonances of the vinyl fluorines in I) becomes an AB-quartet (the δ_{AB} 's are 700 to 1200 Hz and the J_{AB} 's are about 300 Hz). The coalescence temperatures for these AB-quartets are between -120 and -130° , whence the barrier to interconversion of half-chair conformations is about 6.2 kcal/mole. For comparison, cyclohexene has a barrier of 5.3 kcal/mole.

With all good wishes,

Very truly yours,

J. Edgar Anderson

J. Edgar Anderson

Jack

John D. Roberts

JEA/JDR:bi

UNIVERSITY OF CALIFORNIA, SANTA BARBARA

BERKELEY • DAVIS • IRVINE • LOS ANGELES • RIVERSIDE • SAN DIEGO • SAN FRANCISCO



SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY
SANTA BARBARA, CALIFORNIA 93106

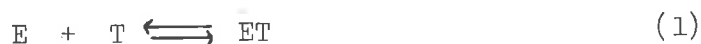
February 23, 1968

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

N.M.R. Studies of the Interaction of Tryptophan with
 α -Chymotrypsin

Dear Dr. Shapiro:

We have recently completed a preliminary study of the Michaelis complex formed by the interaction of tryptophan with the proteolytic enzyme, α -chymotrypsin, by high-resolution n.m.r. techniques. The system may be described by the equilibrium



where E and T represent the enzyme and the amino acid, respectively. The rates involved in the formation of the Michaelis complex (ET) probably approach those expected for a diffusion-controlled process and under these conditions the observed n.m.r. spectrum of T is an averaged one. The parameters which characterize the averaged spectrum are weighted means of the parameters which characterize the free and bound states of T, the contribution of each being weighted by the appropriate mole fraction.^(1,2,3) For the present system, binding of the tryptophan to the enzyme results in significant line-broadening in the spectrum of the former (Figure 1). Analysis of the data leads to the line widths for tryptophan in the bound state that are listed in Table I. As might be expected there is a considerable difference between the behavior of the D- and L-forms of the amino acid.⁽⁴⁾

Table ILine Widths of Tryptophan Bound to α -Chymotrypsin^a

Line	L-Tryptophan	D-Tryptophan
AB, alkyl	60 Hz.	85 Hz.
X, alkyl	20	33
Aromatic	28	43

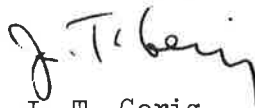
^a Estimated error, $\pm 15\%$.

If one assumes that dipolar coupling is responsible for the observed line-broadening effects, it can be estimated that the rotational correlation time, τ_c , for D-tryptophan in the complexed state is 3.6×10^{-8} sec. This value is in moderate agreement with 4.9×10^{-8} sec., the rotational correlation time of anthraniloylchymotrypsin deduced from fluorescence polarization experiments.⁽⁵⁾ It thus appears that this amino acid is bound tightly enough to the enzyme so that the overall rotational motions of the amino acid are essentially identical to those of the enzyme.

References

- (1) J. J. Fisher and O. Jardetsky, J. Am. Chem. Soc., 87, 3237 (1965).
- (2) D. P. Hollis, Biochemistry, 6, 2080 (1967).
- (3) E. W. Thomas, Biochem. Biophys. Res. Commun., 24, 611 (1966).
- (4) T. McL. Spotswood, J. M. Evans, and J. H. Richards, J. Am. Chem. Soc., 89, 5054 (1967).
- (5) R. P. Haugland and L. Stryer in "Conformation of Biopolymers", Academic Press, New York (1967), Vol. I, p. 321.

Sincerely yours,


J. T. Gerig

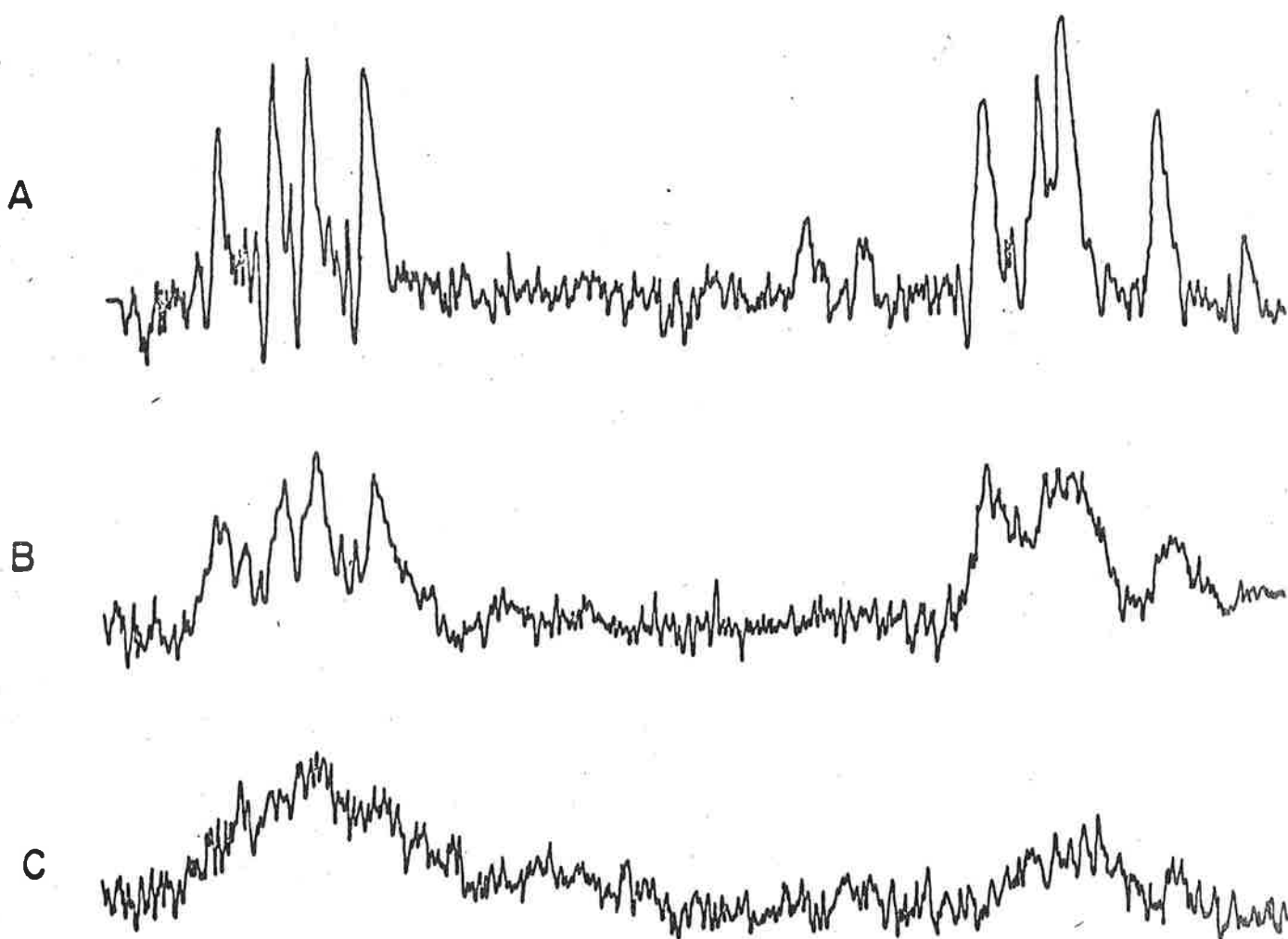


Figure 1. Observed p.m.r. spectra of the alkyl region of (A) 40 mM D-tryptophan at pH 6.9, (b) 50 mM D-tryptophan and 1.1 mM α -chymotrypsin, and (C) 40 mM D-tryptophan and 2.6 mM α -chymotrypsin at 100 MHz.

BULGARIAN
ACADEMY OF SCIENCES
INSTITUTE OF ORGANIC CHEMISTRY
Sofia 13

Sofia, the Feb. 25

1968

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

Conformational analysis of substituted ethanes using the
equation of Bothner-By

Dear Dr. Shapiro:

Thanks for the reminder; I am sorry that our contribution is late. From some time we (Prof. B. Kurtev and Dr. J. Stefanovsky from our Institute's stereochemical group and myself) are interested in the conformational analysis of some diastereomeric 1,2-diphenylethanes by the N.M.R. and other methods. The values of the vicinal coupling constants, obtained from the room-temperature spectra of some compounds of the type $C_6H_5CHXCHYC_6H_5$ are given below.

<u>X</u>	<u>Y</u>	<u>Configuration</u>	<u>Solvent</u>	<u>J_{vic} (Hz)</u>
NH ₂	OH	erythro	CDCl ₃	6.0
			DMSO-d ₆	5.5
		threo	CDCl ₃	6.2
			DMSO-d ₆	7.0
NH ₃ ⁺	OH	erythro ¹	CF ₃ COOH	5.8
		threo ¹	CF ₃ COOH	9.8
NH ₂	COOCH ₃	erythro	CDCl ₃	9.6
			DMSO-d ₆	9.8
		threo	CDCl ₃	10.2
			DMSO-d ₆	10.5

For semiquantitative determination of the rotamer populations in these and other similar cases we are using the equation of Bothner-By², taking $J_t = 11-13$ Hz and $J_g = 1-3$ Hz.

With hope to improve the accuracy of the results given by the

Dr. Bernard L. Shapiro

Page 2

equation, we are trying to determine some "Shoolery-like" additive parameters correlating J_t and J_g with the substituents, by studying some model compounds with "fixed" conformation and more or less known dihedral angle.

With thanks for the Newsletters,

Yours sincerely,

S.L. Spassov

Stefan L. Spassov

¹Before us these compounds were studied as HCl-salts in D₂O-solution by Dr. B.M. Benjamin (see J.W. Huffman and R.P. Elliot, J.Org.Chem. 30, 365 (1965)).

²A.A. Dothner-By and C. Naer-Colin, J.Am.Chem.Soc. 84, 743 (1962).

SIMON FRASER UNIVERSITY

DEPARTMENT OF CHEMISTRY



BURNABY 2, BRITISH COLUMBIA

Telephone 291-3111 Area code 604

February 26, 1968

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

Dear Barry,

Exchange Mechanism from Exchange Line Shapes.

We have recently been interested in the pseudorotation of the asymmetric puckered 5-membered rings $(PCF_3)_5$, $(AsCH_3)_5$ and $(AsCF_3)_5$. The details of the process are obscured in the F^{19} spectrum of $(PCF_3)_5$ by severe $P^{31}-F^{19}$ coupling (IITNMRN no 106, p 20) but "isotopic" substitution of P^{31} by As^{75} automatically performs a convenient heteronuclear decoupling in the liquid through the good services of the large As^{75} quadrupole moment. The basic 2:2:1 triplet of the H^1 spectrum of $(AsCH_3)_5$ below $170^\circ C$ and of the F^{19} spectrum of $(AsCF_3)_5$ from $-130^\circ C$ to $+160^\circ C$ indicates an effective plane of symmetry in both molecules perpendicular to the rings. This is ascribed to rapid ring puckering inversion with simultaneous inversion of the As atom at the puckering centre.

The collapse of the proton spectrum of $(AsCH_3)_5$ above $180^\circ C$ (Fig. 1) is taken to be due to intramolecular pseudorotation of the puckering centre, although the situation is muddled at still higher temperatures by an apparent opening of the As ring. We point out that for a cyclic permutation process like pseudo-rotation one can choose between various mechanisms by appeal to the NMR exchange line shapes. Thus we considered two limiting mechanisms: a) a "clickstop" process where the puckering advances around the ring by 1 position at a time, due to ring torsional modes, and b) a "pancake" process where the puckering drops randomly into any one of the 5 ring positions from a flat transition state. It is a trivial matter to write the Anderson-Weiss 5×5 A-matrices for the two cases and to ask the computer to invert them, thus plotting the line shape.

- 2 -

The result of this procedure is shown in Fig. 2 (labelled 3) for the two mechanisms and the two possible assignments. We conclude that the lineshapes between 170°C and 185°C are due to an intramolecular "clickstop" process, and that the subsequent broadening above 190°C is caused by intermolecular exchange between the ring compound and chain polymers.

A more detailed discussion of these weird rings has been submitted to Canadian Journal of Chemistry under the names of Dr. Louis Peterson, Dr. John Hallet, Bob Ferguson and myself.

Finally, we were all pleased and relieved recently to learn that your newsletter was not to die.

Best wishes.

Yours sincerely,

Fred Wells

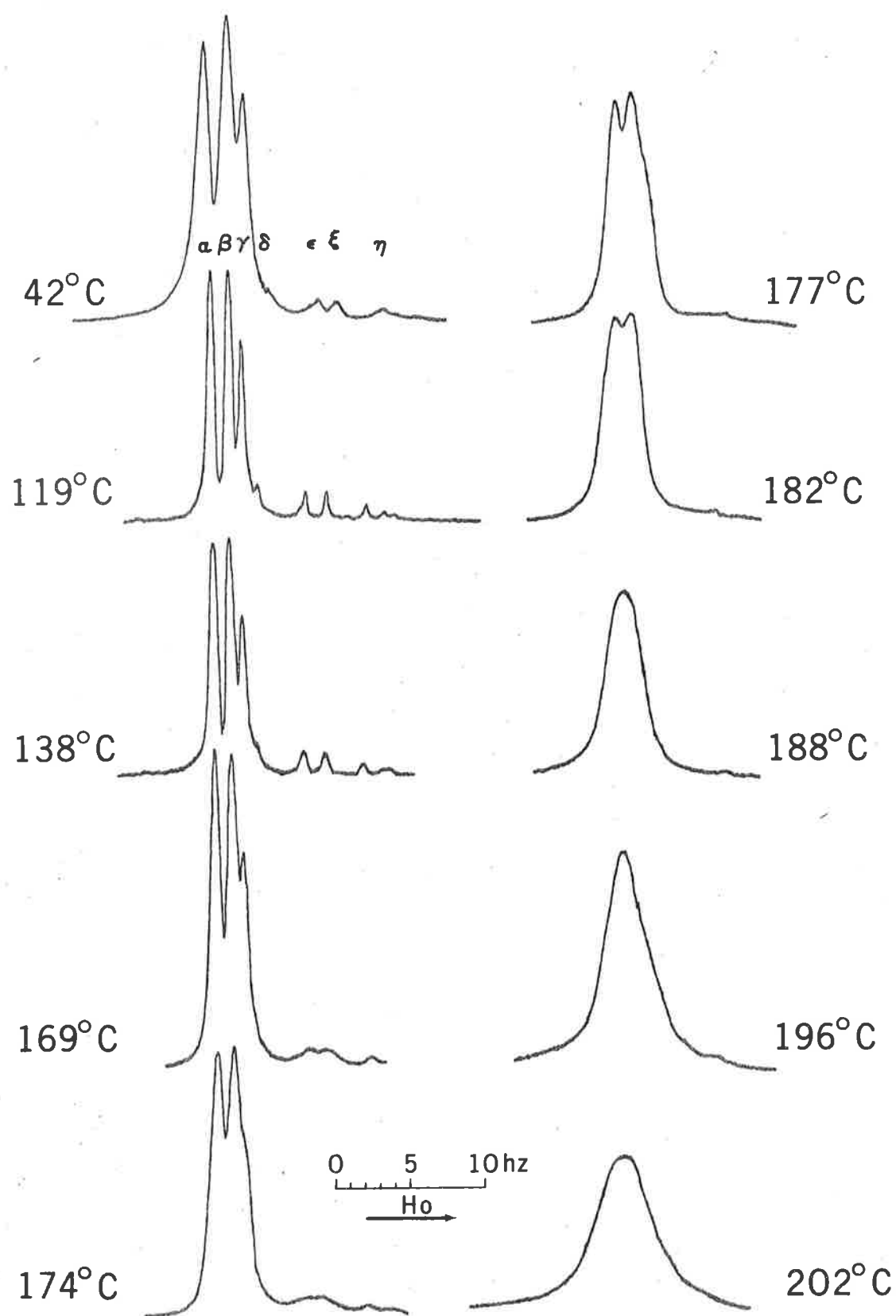


Fig. 8. Temperature dependence of 60 MHz ^1H spectrum of heat-treated indolemethane.

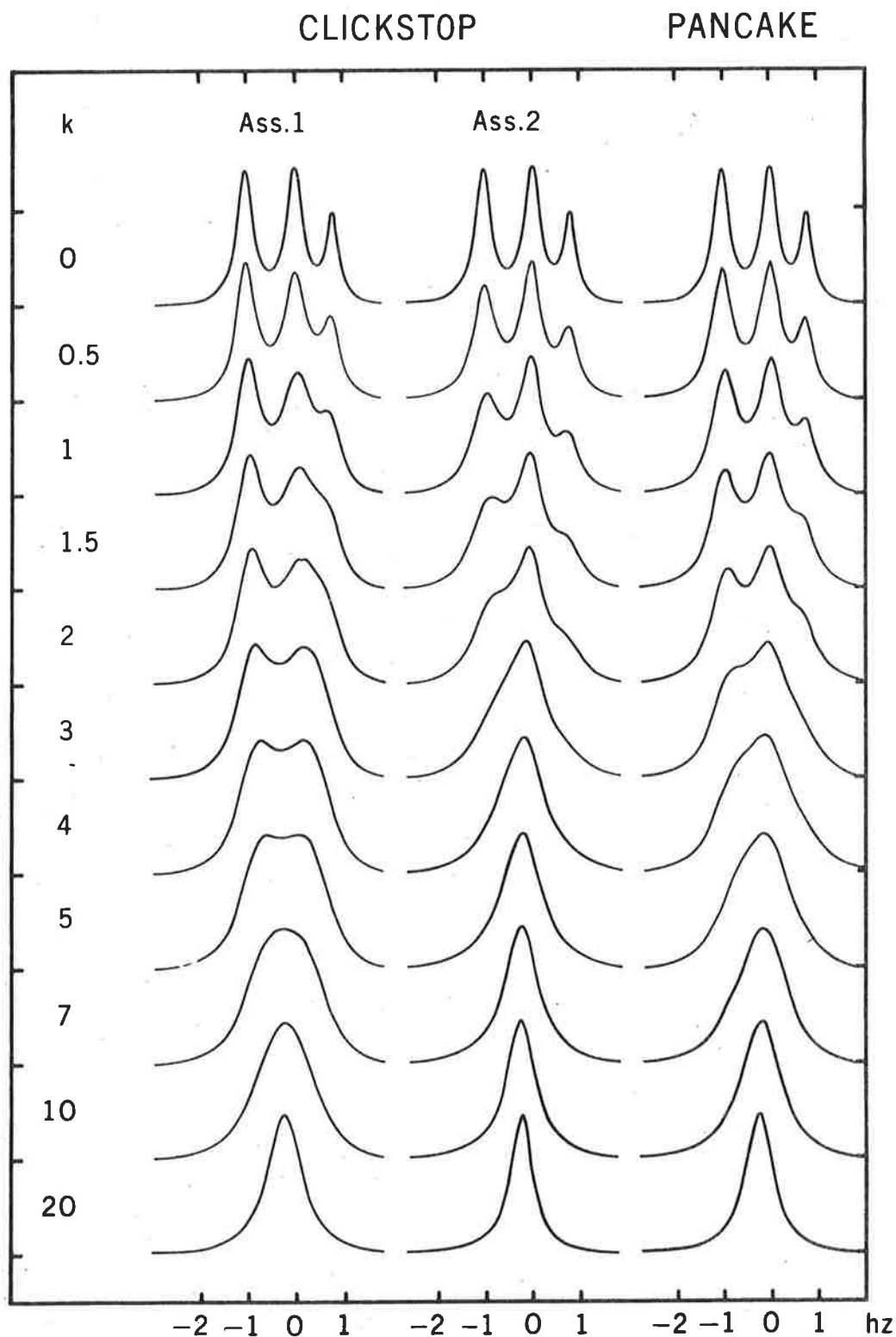


Fig.3. Comparison of computed spectra for intramolecular exchange between lines α , β , and γ of the $(AsCH_3)_5$ triplet. Assignment 1 is ($\alpha = 1,5$ methyls, $\beta = 2,4$, $\gamma = 3$) and Assignment 2 is ($\alpha = 2,4$, $\beta = 1,5$, $\gamma = 3$). The random pancake mechanism is independent of assignment. The input parameters are $\omega_\alpha = -2\pi \times 1.05 \text{ sec.}^{-1}$, $\omega_\beta = 0$, $\omega_\gamma = +2\pi \times 0.80 \text{ sec.}^{-1}$, $R_\alpha^\alpha = R_\beta^\beta = 1.32 \text{ sec.}^{-1}$, $R_\gamma^\gamma = 1.04 \text{ sec.}^{-1}$. (see footnote (27)).



BELFER GRADUATE SCHOOL OF SCIENCE

Amsterdam Avenue and 186th Street / New York, N.Y. 10033 / (212) LORraine 8-8400

February 26, 1968

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

Dear Barry,

In order to keep my subscription alive let me sketch a technique for solving first-order perturbation theory equations that we are applying to a number of problems in many-electron quantum mechanics. In a series of calculations of interest to NMR people, Professor A. Saika is using this technique to calculate chemical shifts and spin-spin coupling constants.

The usual one-electron first-order p. t. equation is of the form

$$L [\psi_1] = Q$$

and we expand ψ_1 in a finite set of functions ϕ_i which include the type of functions we consider important (for the spin-spin coupling constant this includes ψ_0/r as well as non-singular functions) as

$$\psi_1 = \sum_{i=1}^N c_i \phi_i$$

which gives

$$\sum_{i=1}^N c_i L [\phi_i] = Q.$$

Instead of choosing the coefficients c_i by multiplying on the left by ϕ_m^* for each m to give a set of N simultaneous equations for the c_i (equivalent to the variational procedure for linear parameters) we evaluate the equation at N points in the domain of ψ_1 , to give a different set of N equations for the c_i . The method is related to Gauss' method of interpolations, and it is easily seen that for $N \rightarrow \infty$ the two choices of c_i become equivalent. The present method saves evaluating the set of integral $\langle \phi_m^* | L | \phi_i \rangle$ and gives a wave function and values of χ or J to no worse accuracy than the variational method.

Best regards,


J. I. Musher

bw

Title: Method for solving perturbation theory equations

UNIVERSITY OF MARYLAND

COLLEGE PARK 20742

DEPARTMENT OF CHEMISTRY

27 February 1968

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

Dear Dr. Shapiro,

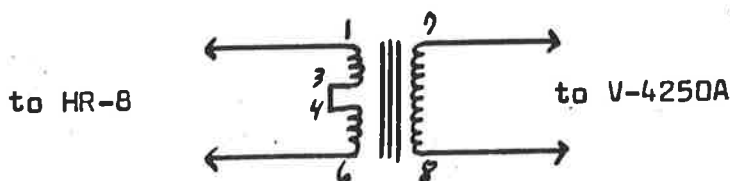
We recently completed a marginal oscillator broadband spectrometer which we grafted on to our DP-60 system. The Varian phase-sensitive detector (V-4270A output control unit) being what it is, we're using a PAR HR-8 phase-sensitive detector. It is convenient to use the Varian modulation amplitude and display controls as well as the power amplifier, but the system requires about 8 VAC input. The HR-8 will give out 1 volt of the modulation frequency with an output impedance of 500 ohms. We found that a Triad HS-1 audio input transformer (about \$25), used as shown below, provides ample output to drive the Varian modulation system. The input impedance of the transformer is 600 ohms and the turns ratio is 1:11.3, so that only a bit more than half a volt is required from the HR-8. The transformer is good to 20 KHz, well above the useful range of the Varian amplifier. We mounted the transformer in a small aluminum case with a double banana plug on the bottom which plugs into the external oscillator jack on the 4250A modulation unit.

We have a postdoctoral appointment open beginning this summer for someone interested in studying solids. I hope to have some interesting experimental results to report next time.

Sincerely yours,

Gerald R. Miller
 Gerald R. Miller
 Assistant Professor

Title: Using an HR-8 with a DP-60; Postdoctoral vacancy



Triad HS-1
 connected for
 600 ohms input
 impedance.

114-56

Dr. W. Brügel i.Fa.
Badische Anilin- & Soda-Fabrik AG · Ludwigshafen am Rhein



Luftpost

Herrn
Prof. Dr. B.L. Shapiro

Department of Chemistry
Stanford University

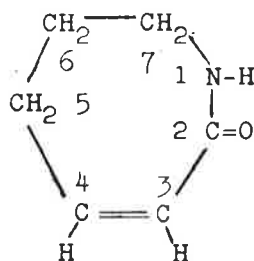
Stanford, California 94305
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Ihre Zeichen	Ihre Nachricht vom	Unsere Zeichen	Fernsprecher-Durchwahl	Telex	67 Ludwigshafen am Rhein
		Dr.Brü/Fa	(06 21) 60 100	464	27. 2. 1968

Betreff Long range coupling in lactams

Sehr geehrter Herr Professor Shapiro!

BARROW und SPOTSWOOD (Tetrah.Lett. No. 37 (1965), 3325-35) haben vor einiger Zeit über eine weitreichende Kopplung des Amidprotons in β -Lactamen mit den Protonen in 3-Stellung (Nachbarstellung zur C=O-Gruppe) berichtet. Wir haben eine ähnliche Kopplung in Δ^3 -Caprolactam beobachtet. Hier koppelt das Amidproton ebenfalls mit dem Proton in 3-Stellung ($J =$



1,7 Hz). Hingegen konnten wir eine entsprechende Kopplung in Δ^4 - und Δ^6 -Caprolactam sowie in den gesättigten Lactamen Pyrrolidon, Caprolactam und Capryllactam nicht finden. Offenbar spielt in dem von BARROW u. SPOTSWOOD angegebenen Fall die Ringspannung, in unserem Fall die Konjugation des einsamen Elektronenpaares am Stickstoff über die C=O-Gruppe mit den π -Elektronen der Doppelbindung die entscheidende Rolle.

Ein anderer Befund der genannten Autoren kann von uns ebenfalls erweitert werden. Sie fanden in β -Lactamen keine Kopplung zwi-

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Badische Anilin- & Soda-Fabrik AG

Empfänger

Prof.Dr. B.L. Shapiro

Unsere Zeichen

Dr.Brü/Fa

67 Ludwigshafen

27.2.1968

Blatt

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Betreff

schen dem Amidproton und den Protonen in der (unmittelbar benachbarten) 4-Stellung. Das gilt nach unseren Beobachtungen auch noch für die erwähnten ungesättigten Caprolactame mit der Doppelbindung in 3-, 4- und 6-Stellung. In diesen Fällen besteht jeweils eine Kopplung mit einer Kopplungskonstante von ungefähr $J = 5.5$ Hz.

Mit den besten Grüßen

Ihr sehr ergebener



(Dr. W. Brügel)

IOWA STATE UNIVERSITY

of Science and Technology



AMES, IOWA 50010

Department of Chemistry

February 28, 1968

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

Dear Barry,

We have been measuring a number of ^{29}Si shifts by the INDOR method using a hp 5100A/5110A synthesiser and an HR-60 modified for internal lock. The ^1H centre band frequency is obtained from the 15 MHz crystal oscillator in a V4311 synchronised by an injected harmonic of the 1 MHz standard crystal frequency of the 5110A; the 2 KHz field modulation oscillator is similarly synchronised by a 1 KHz frequency obtained from the 1 MHz standard by division. This results in a very stable lock if the error signal is fed to both the super stabiliser and the voltage-control jack of the 5110A.

Since most of the compounds examined contained a $\text{CH}_3\text{-Si}$ group, benzene is used to give a lock signal. A modified hp 200CD oscillator provides a reasonably stable modulation frequency to sit on a $^{29}\text{Si-CH}_3$ peak while the attenuated output from the synthesiser applied to the probe transmitter coils is swept through the silicon resonance by a motor-driven potentiometer applied to the search oscillator input. Fine structure from proton splitting is usually visible in the INDOR spectrum.

Silicon resonances can be quoted in MHz relative to the protons of internal TMS at 60.000000 MHz (original idea courtesy of L. M. Jackman) or in p.p.m. relative to the silicon resonance of internal TMS. No appreciable solvent shifts of this resonance have yet been observed. Some results for the chloro- and methoxy-methyl silanes are shown below. The ^{29}Si shifts are believed accurate to ± 0.1 p.p.m. and proton-silicon couplings to ± 0.05 Hz.

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Dr. Shapiro
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	ν_{CH_3}	J_{HCSi}	ν_{Si} (TMS=60 MHz)	δ_{Si}
Me_4Si	0 Hz	6.60 Hz	11.920312 MHz	0 p.p.m.
Me_3SiCl	-22.7	6.95	11.920666	-29.7
Me_2SiCl_2	-44.9	7.65	11.920729	-35.0
$MeSiCl_3$	-63.2	9.10	11.920455	-12.0

	ν_{CH_3Si}	J_{HCSi}	ν_{CH_2O}	J_{HCOSi}	ν_{Si}	δ_{Si}
Me_4Si	0	6.60	---	---	11.920312	0
$Me_3Si(OEt)$	-3.4	6.65	- 216	3.10	11.920485	-14.5
$Me_2Si(OEt)_2$	-1.85	7.10	- 222	3.00	11.920241	+ 5.9
$MeSi(OEt)_3$	-0	8.20	- 224	3.10	11.919787	+44.0
$Si(OEt)_4$	---	---	- 226	3.05	11.919327	+82.6

These results are in general agreement with those reported by Reeves at the 8th. E. N. C. from direct observation of the silicon resonances. Further data-gathering and attempts at rationalisation are in progress.

Sincerely,

Roy W. King

RWK/ld



UNIVERSITY OF SASKATCHEWAN REGINA CAMPUS

DIVISION OF NATURAL SCIENCES

REGINA, SASKATCHEWAN

March 1, 1968

Dr. B. L. Shapiro,
Department of Chemistry,
Stanford University,
Stanford, California 94395,
U.S.A.

Dear Dr. Shapiro:

Molecular Structure of Tri-substituted Olefins by NMR

I was happy to notice (1) that new forces have joined the battle in a new attempt to crack an old and embarrassing problem.

I would like to point out that I did also study the NMR spectra of the API standard samples (2). There follow some of my results on τ -values of the olefinic protons (API cis/trans labelling of the 1st and 3rd pair has been reversed following my recommendation from IR studies; see ref. 3).

			$\Delta\tau_{t-c}$ obs	$\Delta\tau_{t-c}$ calc.
3-Me-pentene-2	cis	4.84	+0.03	+0.03
	trans	4.87		
3-Me-hexene-3	cis	4.90	+0.08	+0.03
	trans	4.98		
3,4-di-Me-pentene-2	cis	4.74	+0.16	+0.08
	trans	4.91		

The "calculated" $\Delta\tau_{t-c}$ refer to anisotropic shielding by c-c bonds, assuming free rotation and using a point-dipole approximation. At least these results are consistent and in line with my IR results.

The shifts of the α - and β -alkyl groups are not a great help. Often there are two nearly equivalent groups; signal assignment could only be attempted if we were sure of the molecular structure! For the few cases where unambiguous assignments could be made I found that $\tau(\text{cis}) > \tau(\text{trans})$. The difference is usually about 0.05 ppm, but for the α -CH group in 3,4-di-methyl-pentene-2 the difference is close to 1.0 ppm, while in the same pair of molecules the trend for the β -CH₃ groups is in the opposite direction!

Dr. B. L. Shapiro

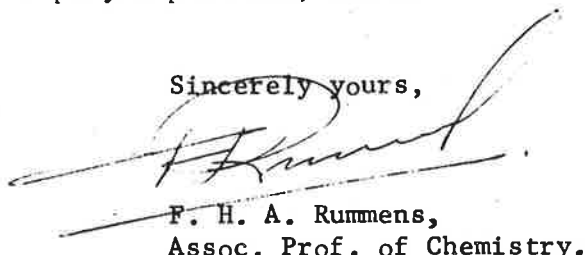
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March 1, 1968

Simple considerations of shielding effects of C=C, C-C and C-H bonds offer no way out. It seems clear that sterical interactions and thereby preferred spatial orientation of the substituents play a major role. The complexity of the problem at least caused me to temporarily retract to simpler situations (4).

In my opinion we need first of all definite information on vicinal, allylic and homoallylic coupling constants in tri-substituted olefins (Dr. Ravage's statement that trans $\text{H} \cdots \text{CH}_3$ couplings are larger than cis is, unfortunately enough, not generally correct(4)). Knowledge on coupling constants then should be turned into information on spatial orientation of the alkyl substituents. This all being done one might try concepts like bond anisotropy to explain the shifts of α - and β - substituents. And after all that we might be permitted to look at polyisoprenoids, but no sooner!

Sincerely yours,



F. H. A. Rummens,
Assoc. Prof. of Chemistry.

FHAR:sfm

References

- (1) D. K. Ravage IIT-NMR 113, (1968), 16.
- (2) F. H. A. Rummens. D. Sc. Thesis, University of Eindhoven, University Press 1963. (Copies still available for gentle-minded readers)
- (3) F. H. A. Rummens. Rec. Trav. Chim Pays Bas 84, 5, 1003, (1965).
- (4) F. H. A. Rummens IIT-NMR 101 (1967), 56.



CANISIUS COLLEGE

BUFFALO, NEW YORK 14208

DEPARTMENT OF CHEMISTRY

March 1, 1968

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

Dear Dr. Shapiro:

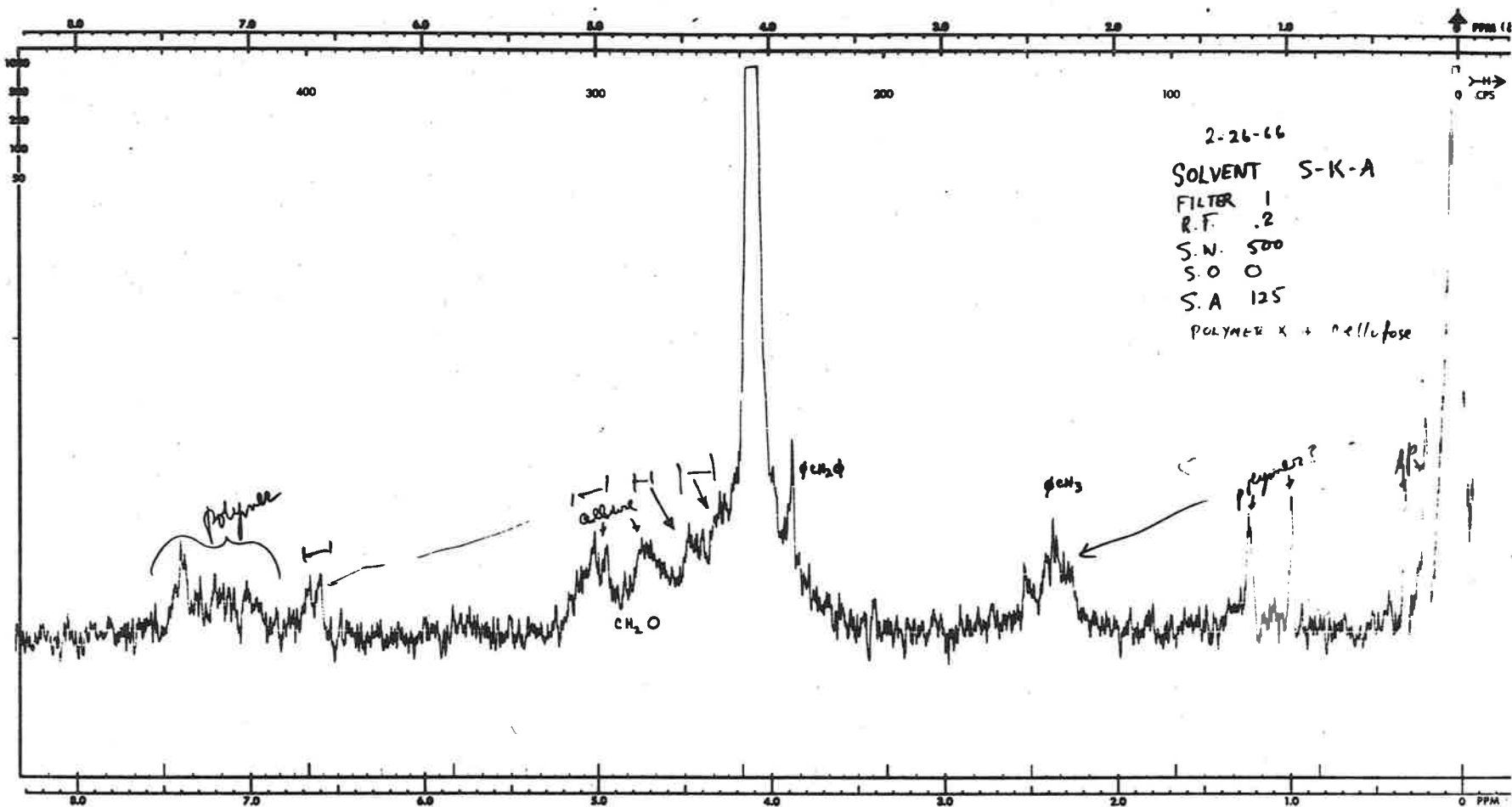
As my contribution to the Newsletter, I would like to submit the following technique. Phenol formaldehyde resins cured on paper are difficult to analyze since most solvents merely soften the resin and do not attack the paper. We have found that a 50-50 mixture of AsCl_3 and SbCl_3 plus 5% KOH gives a solvent system which will dissolve both the resin and paper giving a solution for NMR measurements. Using the same solvent for paper only and determining the solution spectra of it we can identify peaks due to the cellulose and eliminate these from the resin plus paper spectra. The spectrum shown in the attached figure is that of the resin plus paper solution. We identify peaks for cellulose near 6.75, 5.15, 4.8 and 4.4 ppm from TMS. The remaining peaks are assigned to the resin except for the strong line at 4.15 ppm which represents all the exchangeable OH protons. The resin lines are weak as the resin is very intractable.

Title: Dissolving Phenol-formaldehyde Resins and the Paper they are coated on.

Sincerely,

Dr. Herman A. Szymanski
Chairman
Department of Chemistry

Has/am
Encl.



UNIVERSITY OF VIRGINIA

DEPARTMENT OF CHEMISTRY

CHARLOTTESVILLE, VIRGINIA 22901

March 4, 1968

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

Dear Dr. Shapiro:

Downfield Shifts Upon Deprotonation in Dipeptides

Dr. Vito J. Morlino has completed a comparative study of the proton magnetic resonance spectra of DL and LL-phenylalanylvalines. The valyl resonances of the phenylalanylvalines are shown in the table in ppm downfield from DSS as an internal standard in D₂O.

<u>Charge</u>	<u>α-CH</u>		<u>Methyls</u>	
	<u>DL</u>	<u>LL</u>	<u>DL</u>	<u>LL</u>
+ 0	4.18	4.23	0.73	0.93
+ -	3.95	4.05	0.76	0.92, 0.90
0 -	3.97	4.03	0.65	0.85

The table shows a downfield shift for the α -CH resonance of the valyl residue upon ionization of an ammonium hydrogen in the DL-dipeptide but no such downfield shift in the LL-diastereomer. Thus only the DL-dipeptide shows the downfield shift similar to that previously reported for the high field glycyl hydrogen resonance upon ammonium ionization in L-phenylalanylglycine (V. J. Morlino and R. B. Martin, J. Am. Chem. Soc. 89, 3107 (1967)).

The DL-dipeptide also exhibits a downfield shift of the average of the split valyl methyl resonances upon ionization of the carboxylic acid group. No such shift is observed in the LL-dipeptide, which does, however, exhibit chemical shift non-equivalence for the methyl groups in the dipolar ion form, in contrast to the DL-dipeptide. Though some dipeptide conformations can be ruled out, a sufficient range of possibilities still exists so that it is difficult to pinpoint the reasons for this interesting contrast in chemical shift behavior upon deprotonation in the pair of diastereomeric dipeptides.

Sincerely yours,

Bruce Martin

R. Bruce Martin
 Professor of Chemistry

RBM:era



NMR DATA COMPILATION

A new cooperative program associated with the
National Standard Reference Data System and the
National Bureau of Standards

The National Standard Reference Data System (NSRDS) is a nation-wide effort to make available reliable reference data in the physical sciences. As part of this effort, serious consideration has been given to a long-term program for data compilation and related information services in NMR Spectroscopy of interest to chemists. Recently, needs for quality criteria, present data resources, user requirements, format of data presentation and instrumentation developments have been reviewed by an advisory panel under the auspices of the National Research Council Office of Critical Tables.

Currently, the program plans call for:

1. Intensive scrutiny and interlaboratory comparisons of a small group of selected molecules, with the purpose of determining the currently existing limits of precision and accuracy of NMR frequency measurements and related parameters;
2. Continuing compilation of tables of carefully evaluated, high precision chemical shifts and coupling constants, appropriately referenced, based on retrospective and new data. This compilation will stress quality before comprehensiveness. Major emphasis will be on proton NMR, but other nuclei will definitely be included;
3. A coordinating body which can act as a focus of attention on new developments, and as a nucleus for cooperative efforts among all bodies concerned with NMR spectroscopy.

Present efforts under this program are on a volunteer basis. At a later date, some funding may be available to support some phases of the work.

Anyone who is interested in participating, or in following the progress of the program, should communicate with any one of the people named below.

Dr. B. L. Shapiro,* Chairman
Ad hoc Subpanel on NMR Spectroscopy
Advisory to NSRDS through NRC-OCT
Department of Chemistry
Stanford University
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Washington, D. C. 20234

STANFORD UNIVERSITY
STANFORD, CALIFORNIA

DEPARTMENT OF CHEMISTRY

March 6, 1968

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

Subjects: Correction of Nonlinearity of HA-100 Sweep;
Postdoctoral Position

Dear Barry:

Other HA-100 owners may welcome the news as we did that the nonlinearity which exists in the sweep circuit of some units can be minimized considerably by relatively minor surgery.

In our particular case, the nonlinearity showed up as a correction of 4 Hz to be added in the mid-scale chart readings of the 1000 Hz scale even though the end-to-end calibration was adjusted to within less than 0.5 Hz of the chart value. Corrections on other scales varied in such a way that separate correction curves were required for each scale. On the 100 Hz scale a 10.0 cm interval taken at different locations along the chart varied from 9.9 to 10.5 Hz in a nonlinear manner.

The specifications published in one version of the Mars Recorder Manual (Varian Publication No. 87-117-001-A-1067, section 3.2.3) would seem to allow for rather large deviations. The tolerances given for all zero and maximum chart positions is ± 2.5 Hz (2500 ± 2.5 at zero and 2500 + chart width ± 2.5 Hz at the other end). Mid-scale tolerances are given as ± 2.5 Hz for the 50, 100 and 250 Hz scales and as ± 3 Hz for the other two. While these tolerances represent 0.1% or better on the actual frequencies measured, it is the difference between two frequencies which is of interest in measurements utilizing the precalibrated chart paper for obtaining either chemical shift or coupling data. If any mention is made of what can be expected in this frame of reference, it somehow escaped me.

A plaintive cry to a Varian Service Engineer that these corrections were a nuisance brought back the reply that things need not be so bad. With his competent aid we were able to reduce the deviations on all scales far within the published specifications. Most of the remedy involved selecting the optimum value for a capacitor (C-3301) which is located on the sweep pot (R-3304A). The calibration must be readjusted each time the capacitor is changed to ascertain if an improvement has been obtained. Apparently the

Dr. B. L. Shapiro

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value required may be either larger or smaller than the 0.002 μ f capacitor which was supplied. On one scale it was necessary to change the range of the tuning coil by removing one or two turns of conductor--an operation which we preferred to have the Service Engineer perform. We stopped with the results tabulated below, but further improvement was probably attainable with more effort. Frequencies were measured by 10-second counts using a V-4315 frequency counter. Both the difference between the actual frequencies and the "DIFF 1" method of counting produced the same results.

Scale	1000	500	250	100	50
Mid-scale Corr.	+0.3	-0.5	-0.4	-0.4	+0.85
End-scale Corr.	+0.8	-0.4	-0.05	-0.2	+0.65
Max. Corr.	+0.8	-0.75	-0.4	-0.55	+0.85

(Corrections to be added to frequency read on chart to equal counter)

In August we will have a one-year appointment available at the postdoctoral level. The work will be principally concerned with the application of the HA-100 to the wide variety of problems which arise in our nmr service operation. Most of our routine work is carried out on our A-60, so that the problems left for 100 MHz work usually present some special challenge. Some experience with HR or HA type instruments is desirable. A background in organic chemistry would be helpful, though not essential. Interested parties should contact me at their earliest convenience.

Sincerely yours,



Lois J. Durham

LJD:pjc

CARNEGIE-MELLON UNIVERSITY
BIBLIOGRAPHY

- "Nuclear Magnetic Resonance Studies of Organometallic Exchange Processes"
T. L. Brown
Accounts Chem. Res. 1, 23 (1968)
- "Signal Distortion in the Saturation Recovery Method for the Measurement of Nuclear Spin Lattice Relaxation Times"
A. L. Van Geet
Anal. Chem. 40, 304 (1968)
- "N-Persubstituted 3,5-Diaminophenols and 1,3,5-Benzenetriamines and their Protonation"
F. Effenberger and R. Niess
Angew. Chem., Intern. Ed. 6, 1067 (1967)
- "Cycloadditions of Derivatives of Methylenetetrahydrocyclohexane and of Quadricyclanone (Bishomocyclobutadiene)"
H. Prinzbach and J. Rivier
Angew. Chem., Intern. Ed. 6, 1069 (1967)
- "Cycloadditions to Derivatives of Oxaquadricyclanes (Bishomofuran)"
H. Prinzbach, M. Argüelles, P. Vogel, and W. Eberbach
Angew. Chem., Intern. Ed. 6, 1070 (1967)
- "Preparation of S-Aminoisothioureas by Nucleophilic Substitution of S-Chloroisothiocarbamoyl Chlorides"
G. Ottmann and H. Hooks, Jr.
Angew. Chem., Intern. Ed. 6, 1072 (1967)
- "Preparation of 5-Alkyl-1,3-thiazolin-2-ones by Reaction of S-Chloroisothiocarbamoyl Chlorides with Aldehydes and Vinyl Ethers"
G. Ottmann, H. Hoberrecht, and H. Hooks, Jr.
Angew. Chem., Intern. Ed. 6, 1073 (1967)
- "Synthesis of a Morphinan Derivative by Oxidative Ring Closure"
B. Franck, G. Dunkelmann and H. J. Lubs
Angew. Chem., Intern. Ed. 6, 1075 (1967)
- "Reactions of Silylphosphines. Formation of Lithium Tris(diethylphosphinyl)silicide"
G. Fritz and G. Becker
Angew. Chem., Intern. Ed. 6, 1078 (1967)
- "Stereoselektive Synthesen, III. Die Umsetzung von (S)- α -Phenyläthylamin und Isobutyraldehyd mit Benzoesäure und tert.-Butyl-isocyanid als Modellreaktion für stereoselektive Peptid-Synthesen mittels Vierkomponenten-Kondensationen"
I. Ugi, K. Offermann, H. Herlinger und D. Marquarding
Ann. Chem. 709, 1 (1967)
- "Neuere Ergebnisse der Chemie von Insektensexuallockstoffen. Synthesen von D, L-10-Acetoxy-hexadecen-(7-cis)-ol-(1), 12-Acetoxy-octadecen-(9-cis)-ol-(1) ('Gyplure') und 1-Acetoxy-10-propyl-tridecadien-(5-trans-9)"
K. Eiter, E. Truscheit und M. Boneß
Ann. Chem. 709, 29 (1967)
- "Die Dreikohlenstoffautomerie von Nitroolefinen"
G. Hesse, R. Hatz und Helmut König
Ann. Chem. 709, 79 (1967)
- "Reaktionen mit Diazocarbonylverbindungen, XXV. Umsetzung von o-Chinondiaziden mit Ketenen in Alkoholischer Lösung"
W. Ried und P. Junker
Ann. Chem. 709, 85 (1967)
- "Untersuchungen über Heterocarbene, IX. Zur Selektivität des Phenoxycarbens bzw. Phenoxycarbenoids"
U. Schöllkopf und H. Göth
Ann. Chem. 709, 97 (1967)
- "Über den Mechanismus der Wasserstoffübertragung mit Pyridinnucleotiden, XXIX. Synthese und Reaktionen von N-Methyl-3,4,5-tricyanpyridiniumperchlorat"
K. Wallenfels und W. Hanstein
Ann. Chem. 709, 151 (1967)
- "Synthese von Enoläthern der Brenztraubensäure"
F. Lingens und B. Sprößler
Ann. Chem. 709, 173 (1967)
- "Konfiguration und Umsetzungen von 20-stereoisomeren 20-Chlor-16 β -hydroxy-pregnanen. Heterolytische Fragmentierung zu 16.17-Secosteroidaldehyden"
G. Adam und K. Schreiber
Ann. Chem. 709, 191 (1967)
- " $\Delta^{24(28)}$ -Dehydro-ergosterin, Isolierung und chemische Reaktionen"
K. Petzoldt, M. Föhne, E. Blanke, K. Kieslich und E. Kaspar
Ann. Chem. 709, 203 (1967)
- "Protonenrelaxation in Mn²⁺-Lösungen, Einfluß der Hyperfeinkopplung. II. Experimente"
H. Sprinz
Ann. Physik 20, 168 (1967)
- "Rearrangements of Some Dihydroanthracene Derivatives"
K. G. Flynn and G. Bergson
Arkiv för Kemi 27, 393 (1967)
- "Proton Magnetic Resonance Spectra, Configuration, and Conformation of 4-Substituted Prolines"
R. H. Andreatta, V. Nair, and A. V. Robertson
Australian J. Chem. 20, 2701 (1967)
- "Extractives of Australian Timbers. VIII. Enmolactone, a Bishnor-triterpene"
R. A. Eade, J. Ellis, and J. J. H. Simes
Australian J. Chem. 20, 2737 (1967)
- "Diterpenoids. XIV. 4-Epidehydroabietic Acid from the Oleoresin of *Callitris Columnaris* F. Muell."
T. M. Carman and H. C. Deeth
Australian J. Chem. 20, 2789 (1967)
- "(+)-N-Benzoyl[2-Hydroxy-2-(4'-Methoxyphenyl)] Ethylamine from *Clausena Brevistyla* Oliver (Family Rutaceae)"
S. R. Johns, J. A. Lamberton, and J. R. Price
Australian J. Chem. 20, 2795 (1967)
- "Paramagnetic Proton NMR Shifts in Methemoglobin, Metmyoglobin and Some Hemin Derivatives"
R. J. Kurland, and D. G. Davis
Biophys. J. (Soc. Abstr.) 8, A104 (1968)
- "Stéréochimie du perhydrindane. VI. Relations structurales entre amino-5 trans-perhydrindanes et trans-perhydrindanols-5"
R. Granger, J.-P. Girard, J.-C. Rossi et J. Boussinesq
Bull. Soc. Chim. France 4035 (1967)
- "Sur quelques aspects de la réactivité des phénylhalogénogermanes"
P. Rivière et J. Satgé
Bull. Soc. Chim. France 4039 (1967)
- "Introduction d'une insaturation dans un système tétrahydrofurannique"
J. Cleophax, J. Hildesheim et S. D. Gero
Bull. Soc. Chim. France 4111 (1967)
- "Recherches en série hétérocyclique. XII. Synthèse de bromothiophènes substitués"
P. Fournari, R. Guillard et M. Person
Bull. Soc. Chim. France 4115 (1967)

"On the Reaction of Maleic and Citraconic Anhydrides with Acetylacetone and with Ethyl Acetoacetate"
E. Berner and P. Kolsaker
Tetrahedron 24, 1199 (1968)

"Chemical Investigations on Putranjiva Roxburghii. The Structure of a New Triterpene, Putranjivadiene"
P. Sengupta and A. K. Chakraborty and A. M. Duffield, L. J. Durham and Carl Djerassi
Tetrahedron 24, 1205 (1968)

"Mycotoxins Produced by Fusarium Nivale (Fries) Cesati Isolated from Tall Fescue (Festuca Arundinacea Schreb.) Synthesis of 4-Acetamido-4-Hydroxy-2-butenic Acid- γ -lactone"
H. J. Burkhardt, R. E. Lundin and W. H. McFadden
Tetrahedron 24, 1225 (1968)

"Studies on Lactams. VIII. The Conformation of N-aryl Lactams"
M. S. Manhas, S. Jeng and A. K. Bose
Tetrahedron 24, 1237 (1968)

"Anisatinic Acid and Isoanisatinic Acid Isomerization Products of Anisatin"
K. Yamada, S. Takada and Y. Hirata
Tetrahedron 24, 1255 (1968)

"Facile Acetylation of a Tertiary Hydroxyl Group and an Unusual Deshielding Phenomenon by an Acetoxyl Group in NMR Spectra"
K. Yamada, S. Takada, S. Nakamura and Y. Hirata
Tetrahedron 24, 1267 (1968)

"Stereochemistry in the β -Lactam Series. III.
J. L. Luche, H. B. Kagan, R. Parthasarathy, G. Tsoucaris, C. de Rango, and C. Zelwer
Tetrahedron 24, 1275 (1968)

"Some Derivatives of 5H-Dibenz[b,f]azepine"
E. D. Bergmann, M. Rabinovitz and A. Bromberg
Tetrahedron 24, 1289 (1968)

"The Assignment of Configuration to the Photoaddition Products of Unsymmetrical Carbonyls to Furan using Pseudocontact Shifts"
E. B. Whipple and G. R. Evanega
Tetrahedron 24, 1299 (1968)

"The Structure of Gentioflavine, a New Alkaloid of Some Gentiana Species"
N. L. Marekov and S. S. Popov
Tetrahedron 24, 1323 (1968)

"New Metabolites of Gibberella Fujikuroi. XIV. Gibberellin A₁₆ Methyl Ester"
R. H. B. Galt
Tetrahedron 24, 1337 (1968)

"Bis(trifluoromethyl)ketene. VIII. Reaction with Unsaturated Compounds"
Yu. A. Cheburkov, N. Mukhamadaliyev and I. L. Knunyants
Tetrahedron 24, 1341 (1968)

"Diterpene. IV. Über eine Epimerisierung der 6-Hydroxygruppe am Trisnor-labdangerüst im Verlauf des Sauer katalysierten Ringschlusses zu Tricyclischen Athern"
K. Bruns and W. Sandermann
Tetrahedron 24, 1357 (1968)

"Reaktionen mit Phosphororganischen Verbindungen. XIV. zur Konstitution von γ -Acylpropenylphosphoniumsalzen und deren Oxydation mit Derivaten der Salpetrigen Säure"
E. Zbiral und L. Berner-Fenz
Tetrahedron 24, 1363 (1968)

"Stereochemistry of Electrophilic Substitution of (+)-3-carene. Prins and Friedel-Crafts-Acetylation Reactions"
P. J. Kropp, D. C. Heckert and T. J. Flautt
Tetrahedron 24, 1385 (1968)

"Unsaturated Macrocyclic Compounds. LII. The Synthesis of 1,8-Diethynylnaphthalene and Its Oxidation to a Conjugated Fourteen-membered Ring System"
R. H. Mitchell and F. Sondheimer
Tetrahedron 24, 1397 (1968)

"Solvent Effects in NMR Spectroscopy. Solvent Shifts of Methoxyl Resonances in Flavones Induced by Benzene; an Aid to Structure Elucidation"
R. G. Wilson, J. H. Bowie and Dudley H. Williams
Tetrahedron 24, 1407 (1968)

"Axial and Equatorial Thiols. III. 17 α and 17 β -Mercapto Derivatives of Androstane and Androstan-3 β -ol"
D. A. Swann and J. H. Turnbull
Tetrahedron 24, 1441 (1968)

"The Reaction of Isophorone Dienamines with Certain Dienophiles"
H. Nozaki, T. Yamaguti, S. Ueda and K. Kondo
Tetrahedron 24, 1445 (1968)

"The Chemistry of Cryptopine—I. The Epicryptopines"
S. F. Dyke and D. W. Brown
Tetrahedron 24, 1455 (1968)

"Lignans of *Ulmus Thomashii* Heartwood—I. Thomasic Acid"
M. K. Seikel, F. D. Hostettler and D. B. Johnson
Tetrahedron 24, 1475 (1968)

"Tetanortriterpenoids—IV. [Bicyclononanolides II] The Constitution and Stereochemistry of Mexicanolide"
J. D. Connolly, R. McCrindle and K. H. Overton
Tetrahedron 24, 1489 (1968)

"Tetanortriterpenoids—V. [Bicyclononanolides III] The PMR Spectrum of Mexicanolide"
J. D. Connolly, R. McCrindle and K. H. Overton
Tetrahedron 24, 1497 (1968)

"Tetanortriterpenoids—VI. [Bicyclononanolides IV] Swietenolide—The Functional Groups"
T. Chakraborty, J. D. Connolly, R. McCrindle, K. H. Overton and J. C. P. Schwarz
Tetrahedron 24, 1503 (1968)

"Tetanortriterpenoids—VII. [Bicyclononanolides—V] The Constitution and Stereochemistry of Swietenolide"
J. D. Connolly, R. McCrindle, K. H. Overton and W. D. C. Warnock
Tetrahedron 24, 1507 (1968)

"Tetanortriterpenoids—VIII. The Constitution and Stereochemistry of Nimbin"
M. Harris, R. Henderson, R. McCrindle, K. H. Overton and D. W. Turner
Tetrahedron 24, 1517 (1968)

"Tetanortriterpenoids—IX. The Constitution and Stereochemistry of Salannin"
R. Henderson, R. McCrindle, A. Melera and K. H. Overton
Tetrahedron 24, 1525 (1968)

"Diels-Alder Adducts of Dicyanoacetylene and Their Electronic Spectra"
R. C. Cookson, J. Dance and M. Godfrey
Tetrahedron 24, 1529 (1968)

"Reactions of Amines. XVI. 1-*t*-Butyl-3,3-Diphenyl-aziridinone"
H. E. Baumgarten, R. D. Clark, L. S. Endres, L. D. Hagemeier, and V. J. Elia
Tetrahedron Letters 5033 (1967)

"Some π -Molecular Complexes Showing no Charge Transfer Spectra Formed by *p*-Toluenesulfonic Esters"
M. D. Bentley and M. J. S. Dewar
Tetrahedron Letters 5043 (1967)

"Terpenoids CXIV: Lyratol, A New C₁₀ Alcohol from Cyathocline Lyrata"
O. N. Devgan, M. M. Bokadia, A. K. Bose, M. S. Tibbetts, G. K. Trivedi and K. K. Chakravarti
Tetrahedron Letters 5337 (1967)

"On the Unexpected Identity of Theasapogenol D (=Camelliagenin A), A Minor Sapogenol of Tea Seeds, with Dihydropriverogenin A"
I. Yosioka, T. Nishimura, N. Watani, and I. Kitagawa
Tetrahedron Letters 5343 (1967)

"Reaction of 6-Methylbenz [d]-1,3-Oxazepine-2-Carbonitrile with Amines"
C. Kaneko and I. Yokoe
Tetrahedron Letters 5355 (1967)

"3 β -Methoxy-5 α -Cholestane-4 β ,5 α -Diol and Related Compounds"
T. H. Campion and G. A. Morrison
Tetrahedron Letters 1 (1968)

"Stereospecific Selective Hydrogenation of 1,2-Diene Phosphonic Esters"
A. A. Petrov, B. I. Ionin and V. M. Ignatyev
Tetrahedron Letters 15 (1968)

"Protolysis of trans-2-Chlorovinyl Mercuric Chloride in Dimethyl Sulphoxide Solution"
P. J. Banney, W. Kitching and P. R. Wells
Tetrahedron Letters 27 (1968)

"Formation of Pyrimido[1,2-b]pyridazines, a New Bicyclic Triazaheterocyclic System"
B. Stanovnik and M. Tisler
Tetrahedron Letters 33 (1968)

"Conformation and NMR of Lactone-Bridged Cyclohexanes"
E. Dunkelblum and J. Klein
Tetrahedron Letters 55 (1968)

"Classification of Steroid Alcohols by NMR Spectroscopy"
I. R. Trehan and C. Monder
Tetrahedron Letters 67 (1968)

"Triphenyldiethoxyphosphorane and Tributyl-diethoxyphosphorane"
D. B. Denney, D. Z. Denney and L. A. Wilson
Tetrahedron Letters 85 (1968)

"Sur les Reactions des Quinones avec l'Anhydride Acetique en Presence d'Acetate de Sodium. Partie III. Sur les Reactions de la 2,6-Dimethoxy-p-Benzoquinone"
M. Lounasmaa
Tetrahedron Letters 91 (1968)

"New Triterpenes of Alisma Plantago-Aquatica L. Var. Orientalis Samuels"
T. Murata, M. Shinohara, T. Hirata, K. Kamiya, M. Nishikawa and M. Miyamoto
Tetrahedron Letters 103 (1968)

"A Non-Aldolic Dimerization of N-Phenyl Acetone D-Cylceraldimine"
M. Funabashi, H. Ando and J. Yoshimura
Tetrahedron Letters 115 (1968)

"Syntheses of Diastereoisomeric Racemates of Melanthoidine for Determination of its Structure by Mass Spectrometry"
T. Kametani and S. Takano
Tetrahedron Letters 121 (1968)

"Experiments Directed Toward the Total Synthesis of Polycyclic Terpenes. Part VI. Stereoselective Oxidation of Methyl Group"
T. Ogawa, K. Mori and M. Matsui
Tetrahedron Letters 125 (1968)

"Photolysis of Hindered Spiro[2.5]Octa-4,7-Dien-6-Ones"
W.H. Pirkle and G. F. Koser
Tetrahedron Letters 129 (1968)

"The Isolation and Characterization of Hellebrigenin 3-Acetate and Hellebrigenin 3,5-Diacetate, Bufadienolide Tumor Inhibitors from Bersama Abyssinica"
S. M. Kupchan, R. J. Hemingway, and J. C. Hemingway
Tetrahedron Letters 149 (1968)

"Zur Rotationsbehinderung bei Substituierten Benzophenonen und Benzilen"
D. Lauer und H. A. Staab
Tetrahedron Letters 177 (1968)

"Über Triterpene XXIII. Über Struktur und Chemie der Priverogenine"
R. Tschesche, B. T. Tjoa und G. Wulff
Tetrahedron Letters 183 (1968)

"The Structure of Polyoxin C"
K. Isono and S. Suzuki
Tetrahedron Letters 203 (1968)

"NMR Spectra of Co-ordination Compounds of Trialkyl Aluminums with Donors"
K. Hatada and H. Yuki
Tetrahedron Letters 213 (1968)

"A Facile Synthesis of Indoline-systems Involving an Intramolecular Cyclisation via Arylcarbenes"
R. Garner
Tetrahedron Letters 221 (1968)

"Quaternization of 1,2-Disubstituted 6,7-Dimethoxy-1,2,3,4-Tetrahydroisquinolines"
G. Bernath, J. Kobor, K. Koczka, L. Radics and M. Kajtar
Tetrahedron Letters 225 (1968)

"Optically Active Gossypol from Thespesia Populnea"
T. J. King and L. B. de Silva
Tetrahedron Letters 261 (1968)

"Callitrisic Acid: a New Diterpenoid"
L. J. Gough
Tetrahedron Letters 295 (1968)

"Syntheses dans la Serie des 2,5-Anhydrides de Sucres. Solvolyses d'Esters Sulfoniques"
J. Defaye et J. Hildesheim
Tetrahedron Letters 313 (1968)

"The Selective Deuteration of the Vinylic Protons of Cyclopropene"
E. A. Dorko and R. W. Mitchell
Tetrahedron Letters 341 (1968)

"The Structure and Configuration of Caseamine and Caseadine Two Novel Tetrahydroprotoberberines from Corydalis Caseana A. Gray"
C.-Y. Chen, D. B. MacLean, and R. H. F. Manske
Tetrahedron Letters 349 (1968)

"The Structure of Bakkenolide-A"
N. Abe, R. Onoda, K. Shirahata, T. Kato, M. C. Woods, and Y. Kitahara
Tetrahedron Letters 369 (1968)

"Structure of Pterosterone. A Novel Insect-Moulting Substance from Lastrea Thelypteris and Onoclea Sensibilis"
T. Takemoto, S. Arihara, Y. Hikino, and H. Hikino
Tetrahedron Letters 375 (1968)

"4-tert-Butylcyclohexyllithium"
W. H. Glaze and C. M. Selman
J. Organometal. Chem. 11, P3 (1968)

"A Hexafluoro(para-bonded Benzene) Platinum Complex"
B. L. Booth, R. N. Haszeldine and N. I. Tucker
J. Organometal. Chem. 11, P5 (1968)

"NMR Studies of Ligand Exchange Reactions Involving
Diolefin Complexes of Rh^I and Ir^I"
K. Vrieze and H. C. Volger
J. Organometal. Chem. 11, P17 (1968)

"Cyclopentadienyl-molybdenum(0)-cycloheptatrienyl"
E. O. Fischer and H. W. Wehner
J. Organometal. Chem. 11, P29 (1968)

"Organosilicon Chemistry. I. Octaarylspiro[5.5]
Pentasiloxanes"
T. C. Wu and C. A. Hirt
J. Organometal. Chem. 11, 17 (1968)

"Rearrangement of β -Silyl Ketones into O-Silyl-
Substituted Enols"
I. F. Lutsenko, Yu. I. Baukov, O. V. Dudukina and E.
N. Kramarova
J. Organometal. Chem. 11, 35 (1968)

"Polyhalo-organometallic and -organometalloidal
Compounds. XI. Some Organosilicon Derivatives of
1,2,3,4-Tetrafluorobenzene"
I. Haiduc, and H. Gilman
J. Organometal. Chem. 11, 55 (1968)

"The Preparation of Group IV Organometallic Compounds
Containing gem-Dihalocyclopropyl Groups"
D. Seyferth, T. F. Julia, H. Dertouzos and M. Pereyre
J. Organometal. Chem. 11, 63 (1968)

"Synthese und Reaktionen von Lithium-trimethylgerma-
nium-methylamid"
I. Schumann-Ruidish and B. Jutzi-Mebert
J. Organometal. Chem. 11, 77 (1968)

"Action des Alcoxytrialkylétains sur les Esters
D'énols: le problème de l'Obtention de Composés C-Ou
O-Stanniques"
M. Pereyre, B. Bellegarde, J. Mendelsohn et J. Valade
J. Organometal. Chem. 11, 97 (1968)

"Die ¹H-NMR-Spektren der Alkylhalogenstannane"
J. Lorberth und H. Vahrenkamp
J. Organometal. Chem. 11, 111 (1968)

"Synthesis and Proton Magnetic Resonance Study of Some
Tetraalkyllead Derivatives"
G. Singh
J. Organometal. Chem. 11, 133 (1968)

"Observations of the Proton Magnetic Resonance Spectra
of Certain Group IVB Derivatives of Sulphur,
Selenium and Arsenic"
E. W. Abel and D. B. Brady
J. Organometal. Chem. 11, 145 (1968)

"Electronic Transmission in Some Organometallic
Derivatives of Cobalt(III) Bae and S len Complexes"
H. A. O. Hill, K. G. M. Rallee, G. Pellizer, G.
Mestroni and G. Costa
J. Organometal. Chem. 11, 167 (1968)

"Recherches par résonance magnétique nucléaire sur les
composés organométalliques. IV. Magnésiens
aromatiques para-substitués"
G. J. Martin, B. Mechin, and M. L. Martin
J. Organometal. Chem. 11, 177 (1968)

"Reactions of tri-n-butyltin hydride with diorganotin
dichlorides"
A. K. Sawyer, J. E. Brown, and G. S. May
J. Organometal. Chem. 11, 192 (1968)

"Synthese, Eigenschaften, Schwingungsspektren und
Protonenresonanzspektren von Organoquecksilber-
aziden"
K. Dehnicke und D. Seybold
J. Organometal. Chem. 11, 227 (1968)

"¹H-NMR-Spektroskopische Untersuchungen Zum Substituenten-
und Kettenlängeneinfluss Auf Die SiOSi-Bindungsstruktur
in α,ω -Disubstituierten Polydimethylsiloxanen"
G. Engelhardt
J. Organometal. Chem. 11, 243 (1968)

"An Investigation of Methyltin-Lithium Compounds
Preparation and Properties of Tris(trimethylstannyl)
Stannyllithium Tris(tetrahydrofuran)"
W. L. Wells and T. L. Brown
J. Organometal. Chem. 11, 271 (1968)

"Reaction of Diphenyltin with Alkyl Halides Nuclear
Magnetic Resonance Spectra of Methyltin and Ethyltin
Compounds"
K. Sisido, T. Miyazaki, K. Nabika and S. Kozima
J. Organometal. Chem. 11, 281 (1968)

"Some N-Triaryl tin and -Lead Derivatives of Certain
Purines"
E. J. Kupchik and E. F. McInerney
J. Organometal. Chem. 11, 291 (1968)

"Study on Triorganostibine Sulfide and Its Complexes"
M. Shindo, Y. Matsumura and R. Okawara
J. Organometal. Chem. 11, 299 (1968)

"Organometallic π -Complexes XI. Vinylmetallocenes"
M. D. Rausch and A. Siegel
J. Organometal. Chem. 11, 317 (1968)

"Iridium Dicarbonyl β -Diketonates"
F. Bonati and R. Ugo
J. Organometal. Chem. 11, 341 (1968)

"Nuclear Magnetic Resonance Studies in Coordination
Chemistry. III. Kinetic Studies of Reactions
Occurring in the System $[(\pi-C_4H_7)PdCl]_2 + Triphenyl-$
phosphine"
K. Vrieze, P. Cossee, A. P. Praat and C. W. Hilbers
J. Organometal. Chem. 11, 353 (1968)

"Ferricinium Salts"
I. J. Spilners
J. Organometal. Chem. 11, 381 (1968)

"Nuclear Magnetic Resonance of Oxygen-17 and Chlorine-
35 in Aqueous Hydrochloric Acid Solutions of Cobalt(II).
I. Line Shifts and Relative Abundances of Solution
Species"
A. H. Zeltmann, N. A. Matwiyoff, and L. O. Morgan
J. Phys. Chem. 72, 121 (1968)

"The Proton Magnetic Resonance Spectra of Diethyl
Vinylphosphonate and Substituted Vinylphosphonates"
M. P. Williamson, S. Castellano, and C. E. Griffin
J. Phys. Chem. 72, 175 (1968)

"Solvent-Dependent H-H Couplings in Hexachloro-
bicyclo[2.2.1]heptenes"
S. L. Smith and R. H. Cox
J. Phys. Chem. 72, 198 (1968)

"Carbon-13 Nuclear Magnetic Resonance Studies of 3-
Substituted Pyridines"
H. L. Retcofsky and R. A. Friedel
J. Phys. Chem. 72, 290 (1968)

"Integerressin und Integerrenin, zwei Peptid-Alkaloide aus *Ceanothus integerrimus* Hook. et Arn."
R. Tschesche, J. Rheingans, H.-W. Fehlhaber und G. Legler
Chem. Ber. 100, 3924 (1967)

"Alkaloide aus Rhamnaceen, III. Frangulatin, ein Peptid-Alkaloid aus *Rhamnus frangula* L."
R. Tschesche, H. Last und H.-W. Fehlhaber
Chem. Ber. 100, 3937 (1967)

"Darstellung, IR- und ¹⁹F-KMR-Spektren von Pentafluorphenylmercapto-Metallkomplexen"
W. Beck, K. H. Stetter, S. Tadros und K. E. Schwarzhaus
Chem. Ber. 100, 3944 (1967)

"¹H-NMR-Spektroskopische Untersuchungen an 2.3-disubstituierten Derivaten des Hexachlorbicycloheptens"
K. Ballschmiter und H. Singer
Chem. Ber. 101, 7 (1968)

"¹H-NMR-Spektroskopische Untersuchungen an 1.3-disubstituierten Derivaten des 4.5.6.7.8.8-Hexachlor-1.3.3a.4.7.7a-hexahydro-4.7-methano-isobenzofurans"
H. Singer und K. Ballschmiter
Chem. Ber. 101, 17 (1968)

"Säureamid-Reaktionen, L; Orthoamid, I. Darstellung und Eigenschaften der Amidacetale und Aminalester"
H. Brederick, G. Simchen, S. Rebsdorf, W. Kantlehner, P. Horn, R. Wahl, H. Hoffmann und P. Grieshaber
Chem. Ber. 101, 41 (1968)

"Dimere des 1.2-Dimethyl-cyclobutadiens"
R. Criegee, W. Eberius und H.-A. Brune
Chem. Ber. 101, 94 (1968)

"Darstellung und Valenzisomerisierung von Cyclobutenen mit 'seitlich' ankondensierten Ringen"
R. Criegee und H. G. Reinhardt
Chem. Ber. 101, 102 (1968)

"Elektrophile und Nucleophile Substitutionsreaktionen an Verbindungen des pentakoordinierten Arsens"
D. Hellwinkel und G. Kilthau
Chem. Ber. 101, 121 (1968)

"Über Aromatenkomplexe von Metallen, CIII. Trirhodium-tetracyclopentadienyl-hydrid Rh₃(C₅H₅)₄H"
E. O. Fischer und H. Wawersik
Chem. Ber. 101, 150 (1968)

"Cyclopentadienyl-alkyl- bzw. -arylmethoxycarben-dicarbonylrhenium-Komplexe und Cyclopentadienyl-methylhydroxycarben-dicarbonylrhenium"
E. O. Fischer und A. Riedel
Chem. Ber. 101, 156 (1968)

"Schwefel-Stickstoff-Verbindungen, II. Darstellung und Untersuchung von Fluorsulfurylverbindungen"
H. W. Roesky und A. Hoff
Chem. Ber. 101, 162 (1968)

"Carboxoniumverbindungen in der Kohlenhydratchemie, IV. Acetoxonium-Umlagerungen der D-Xylose"
H. Paulsen, F. G. Espinosa, W.-P. Trautwein und K. Heyns
Chem. Ber. 101, 179 (1968)

"Carboxoniumverbindungen in der Kohlenhydratchemie, V. Eine einfache Synthese von D-Talose durch Acetoxonium-Umlagerung von D-Galaktose"
H. Paulsen, F. G. Espinosa und W.-P. Trautwein
Chem. Ber. 101, 186 (1968)

"Carboxoniumverbindungen in der Kohlenhydratchemie, VI. Untersuchungen über Reaktionen der D-Idose. Acetoxonium-Umlagerung der D-Idose zu D-Galaktose"
Chem. Ber. 101, 191 (1968)

"Heterocyclen durch Diensynthese. Pyridylpyrrole aus Diels-Alder-Addukten von Nitrosoverbindungen"
J. Firl
Chem. Ber. 101, 218 (1968)

"Eine neue Synthese wichtiger Pyrrollderivate"
H. Plöninger, P. Hess und J. Ruppert
Chem. Ber. 101, 240 (1968)

"Chemie nucleophiler Carbene, XIII. Über die Bildung eines Cyanins bei der HJ-Abspaltung aus N-Methylbenzoxazolium-jodid"
H.-W. Wanzlick und H. Steinmaus
Chem. Ber. 101, 244 (1968)

"Olefin-Palladiumchlorid-Komplexe, X. π-Allyl-Komplexe durch Decarboxylierung von ungesättigten Malonsäurederivaten"
R. Hüttel und H. Schmid
Chem. Ber. 101, 252 (1968)

"Di-π-cyclopentadienyl-titan(IV)-pentaselenid und -pentasulfid, zwei Hetero-cyclohexachalkogene in fixierter Konformation"
H. Köpf, B. Blöck und M. Schmidt
Chem. Ber. 101, 272 (1968)

"Einfache Synthese von 1,2-Dithioliumsalzen aus Sulfanen und Diketonen"
M. Schmidt und H. Schulz
Chem. Ber. 101, 277 (1968)

"Reaktionen von Inaminen mit Carbonsäure- und N-Acyl-aminosäureanhydriden sowie Anlagerung an Oxazolone-(5) und Pseudooxazolone-(5)"
W. Steglich, G. Höfle, W. König und F. Weygand
Chem. Ber. 101, 308 (1968)

"Organische Schwefelverbindungen, LI. Einwirkung von Diphenyldiazomethan und von Thionylchlorid auf 1-Thio-α- und -γ-pyrone"
A. Schönberg und R. von Ardenne
Chem. Ber. 101, 346 (1968)

"Organische Schwefelverbindungen, LII. Bildung von Thionaphthenderivaten aus 1-Thio-α-bzw. -γ-pyrenen. Darstellung von α-Pyrenen aus γ-Pyrenen"
A. Schönberg und R. von Ardenne
Chem. Ber. 101, 356 (1968)

"Reaktionen mit N-Sulfinylverbindungen, VIII. Bildung und Fragmentierung von N-Arylsulfonyl-imidchloriden"
G. Kresze und W. Wucherpfennig
Chem. Ber. 101, 365 (1968)

"Reaktionen mit N-Sulfinylverbindungen, IX. Notiz über die Sulfinylierung o-difunktionaler Aniline"
W. Wucherpfennig
Chem. Ber. 101, 371 (1968)

"High Resolution n.m.r."
R. F. M. White
Chem. Britain 4, 20 (1968)

"Reactions of Tetrakis(triphenylphosphine)palladium(0) with Olefins bearing Electron-withdrawing Substituents"
P. Fitton and J. E. McKeon
Chem. Commun. 4 (1968)

"Oxidative Additions of Palladium(0)"
P. Fitton, M. P. Johnson and J. E. McKeon
Chem. Commun. 6 (1968)

"Aluminium-Olefinic Double Bond Interaction in Alkenyl-aluminium Compounds"
G. Hata
Chem. Commun. 7 (1968)

"Recherches en série hétérocyclique. XIII. Synthèses d'iodothiophènes substitués"
R. Guillard, P. Fourmari et M. Person
Bull. Soc. Chim. France 4121 (1967)

"La chimie des carbazoles. II. L'orientation dans les substitutions électrophiles chez les diéthyl-3,9 et triéthyl-3,6,9 carbazoles"
N. P. Buu-Hoi, G. Saint-Ruf et J.-C. Perche
Bull. Soc. Chim. France 4126 (1967)

"Réactivité en série thiazolique. Action du *n*-butyllithium sur le méthyl-2 thiazole"
J. Crousier et J. Metzger
Bull. Soc. Chim. France 4134 (1967)

"Configurations et conformations des γ -lactones étudiées par résonance magnétique nucléaire"
D. Savostianoff et M. Pfau
Bull. Soc. Chim. France 4162 (1967)

"Action des agents nucléophiles sur différents phosphonates γ -halogéno β -éthyléniques"
G. Lavielle, G. Sturtz et H. Normant
Bull. Soc. Chim. France 4186 (1967)

"Spectrométrie de RMN en série benzothiazolique. Étude structural de bases, de sels quaternaires, de spiropyranes benzothiazoliques ainsi que de dérivés carbonyles aromatiques"
R. Guglielmetti, E.-J. Vincent et J. Metzger,
Bull. Soc. Chim. France 4195 (1967)

"Résonance magnétique de ^{19}F : étude des fluorures de benzyle substitués"
C. Béguin
Bull. Soc. Chim. France 4214 (1967)

"Contribution à l'étude de la thiéno- α 2,3-d α pyridazine et de la thiéno- α 3,4-d α pyridazine"
M. Robba, B. Roques et Y. Le Guen
Bull. Soc. Chim. France 4220 (1967)

"Acide fluorométhane phosphonique et quelques dérivés"
E. Gryszkiewicz-Trochimowski
Bull. Soc. Chim. France 4289 (1967)

"Alcaloïdes stéroïdiques. LXV. Préparation de dérivés du nor-18 cyano-13 β androstane, à partir de la conessine; nor-18 cyano-13 β testostérone"
M.-M. Janot, P. Milliet, X. Lusinchi et R. Goutarel
Bull. Soc. Chim. France 4310 (1967)

"Alcaloïdes stéroïdiques LXVII. Structure de l'holaline, alcaloïde mineur des écorces de l'Holarrhena floribuda, (G. Don) Dur. et Schinz"
M.-M. Janot, P. Devissaguet, Q. Khuong-Huu et R. Goutarel
Bull. Soc. Chim. France 4315 (1967)

"Alcaloïdes stéroïdiques LXVIII. Transpositions de Westphalen à partir de dérivés de la conessine. Alloconessine"
M.-M. Janot, P. Devissaguet, M. Pais, Q. Khuong-Huu, F.-X. Jarreau et R. Goutarel
Bull. Soc. Chim. France 4318 (1967)

"Alcaloïdes stéroïdiques LXIX. Transposition de la conessine en milieu acide. Structure de l'isconessine"
M.-M. Janot, P. Devissaguet, M. Pais, Q. Khuong-Huu, F.-X. Jarreau et R. Goutarel
Bull. Soc. Chim. France 4323 (1967)

"Les cétones α -alléniques et les réactions de Diels-Alder"
M. Bertrand et J. Le Gras
Bull. Soc. Chim. France 4336 (1967)

"Reaction Mechanism Studies. 4. The Diaxial \rightarrow Diequatorial Rearrangement of β -chlorothioethers"
J. F. King, K. Abikar, D. M. Deaken, and R. G. Pews
Can. J. Chem. 46, 1 (1968)

"Reaction Mechanism Studies. 5. The Mechanism of the Diaxial \rightarrow Diequatorial Rearrangement of β -chlorothioethers"
J. F. King and K. Abikar
Can. J. Chem. 46, 9 (1968)

"The Alkaloids of *Lycopodium alopecuroides* L."
W. A. Ayer, B. Altenkirk, S. Valverde-Lopez, B. Douglas, R. F. Raffauf, and J. A. Weisbach
Can. J. Chem. 46, 15 (1968)

"Preparation and Base-catalyzed Reactions of Some β -halohydroperoxides"
K. R. Kopecky, J. H. van de Sande, and C. Mumford
Can. J. Chem. 46, 25 (1968)

"Cyclohexane Compounds. VII. Nucleophilic Scission of the Stereoisomeric 3-Methoxycyclohexene Oxides"
R. A. B. Bannard, A. A. Casselman, E. J. Langstaff, and R. Y. Moir
Can. J. Chem. 46, 35 (1968)

"Photochemical Rearrangement and Dimerization of 1,1-disubstituted Indenes"
J. J. McCullough
Can. J. Chem. 46, 43 (1968)

"Photochemical Synthesis. 19. The Flash Photolysis of Unsaturated Sultones"
J. L. Charlton and P. de Mayo
Can. J. Chem. 46, 55 (1968)

"Preparation of Unsaturated Carbohydrates. A Facile Synthesis of Methyl 4,6-O-Benzylidene-D-hex-2-enopyranosides"
R. U. Lemieux, E. Fraga, and K. A. Watanabe
Can. J. Chem. 46, 61 (1968)

"Thiohydantoins. VI. Hydrolysis of 1-Acyl-2-Thiohydantoins: Reported Anomalies"
J. T. Edward and J. K. Liu
Can. J. Chem. 46, 71 (1968)

"The Effect of Tetra-*n*-butylammonium Salts on the Nuclear Magnetic Resonance of Nitrobenzene"
J. B. Hyne and A. R. Fabris
Can. J. Chem. 46, 73 (1968)

"Steroid 16,17-Furoxan Derivatives"
H. Reimann and H. Schneider
Can. J. Chem. 46, 77 (1968)

"Reactions of Nitro Sugars. VII. A Study on Acetylation and Some Chemical Properties of Acetates"
H. H. Baer, F. Kienzle, and F. Rajabalee
Can. J. Chem. 46, 80 (1968)

"Two-step Synthesis of *cis,cis*-1,6-cyclodecadiene"
S. N. Sharma, R. K. Srivastava, and D. Devaprabhakara
Can. J. Chem. 46, 84 (1968)

"A Rapid, Mild Procedure for the Preparation of Alkyl Chlorides and Bromides"
J. Hooz and S. S. H. Gilani
Can. J. Chem. 46, 86 (1968)

"Polyacetylenverbindungen, CXLII. Synthese verschiedener natürlich vorkommender Polyinsäureisobutylamide"
F. Bohlmann und R. Miethe
Chem. Ber. 100, 3861 (1967)

"Ditertiäre Carbinole des Dewar-benzols und Prismsans"
R. Criegee, R. Askani und H. Grüner
Chem. Ber. 100, 3916 (1967)

"Di-isotacticity of Poly(methyl Propenyl Ether) and Double-Bond Opening of Methyl Propenyl Ether in Cationic Polymerization.

Y. Ohsumi, T. Higashimura, S. Okamura, R. Chujo and T. Kuroda

J. Polymer Sci.: Pt. A-1, 5, 3009 (1967)

"Polymerization of Alkylene Oxides by Dialkylzinc-Lewis Base Systems"

J. Furukawa, N. Kawabata, and A. Kato

J. Polymer Sci.: Pt. A-1, 5, 3139 (1967)

"NMR Data on the Stereoregularity of Polypropylene"

A. Zambelli, A. L. Segre, M. Farina and G. Natta

Makromol. Chem. 110, 1 (1967)

"Copolymerization of N-Carboxy- γ -Benzyl-L-Glutamate Anhydride and Alkylene Oxides by Organometallic Compounds"

T. Tsuruta, S. Inoue, M. Yoneyama, and N. Yamada

Makromol. Chem. 110, 133 (1967)

"High Resolution NMR Studies on Sequence Distribution of Vinylidene Chloride Copolymers"

K. Ito, S. Iwase, and Y. Yamashita

Makromol. Chem. 110, 233 (1967)

"Reaktion von Isobutylenmethyläther mit Diazoessigsäure. 20. Mitt.: Zur Kenntnis cyclischer Acylale. 6. Mitt.: Zur Kenntnis organischer Lewisäuren"

H. Peham, O. E. Polansky und F. Wessely

Monatsh. Chem. 98, 1665 (1967)

"Zur Strukturklärung der Isomeren von α -Oximino-carbonsäureestern"

S. L. Spassov, G. Heublein, A. Jovtscheff, H.

Reinheckel und V. Jehlicka

Monatsh. Chem. 98, 1682 (1967)

"Zur Kenntnis cyclischer Acylale, 18. Mitt.: Über die Einwirkung von Diazomethan auf substituierte Benzylidenmethyläther und ähnliche Verbindungen"

P. Schuster, F. Wessely und A. Stephen

Monatsh. Chem. 98, 1772 (1967)

"Synthesen von Heterocyclen, 97. Mitt." (Chinolin und Indolizin, III): Über die Reaktion der 2-Pyridyläthyläthyläther mit Brombenzyläthyläther

Th. Kappe

Monatsh. Chem. 98, 1858 (1967)

"Ringschlussreaktionen zu methoxysubstituierten Benzothien-3-essigsäuren"

F. Sauter und P. Stätz

Monatsh. Chem. 98, 1962 (1967)

"Stereochemie von Metallocenen, 10. Mitt.: Chemische Korrelation von α - und β -substituierten Methylferrocenen; die absolute Konfiguration optisch aktiver, β -substituierter Methyl- und 1,1'-Dimethylferrocene"

G. Haller und K. Schlögl

Monatsh. Chem. 98, 2044 (1967)

"The Anisotropy of the Nuclear Magnetic Resonance in Thallium Metal"

J. J. Schratte and D. L. Williams

Phys. Letters 26A, 79 (1967)

"Approach to High-Resolution nmr in Solids"

J. S. Waugh, L. M. Huber, and U. Haeberlen

Phys. Rev. Letters 20, 180 (1968)

"Spin-Lattice Relaxation in Manganese Ammonium Tutton Salt and How it is Influenced by Impurities"

A. J. De Vries, J. W. M. Livius, D. A. Curtis, A. J.

van Duynveldt and C. J. Gorter

Physica 36, 65 (1967)

"Spin-Lattice Relaxation in a Few Hydrated Paramagnetic Sulphates"

A. J. De Vries, D. A. Curtis, J. W. M. Livius, A. J.

van Duynveldt and C. J. Gorter

Physica 36, 91 (1967)

"Mycotoxins Produced by *Fusarium Nivale* Isolated from Tall Fescue (*Festuca Arundinacea* Schreb.)"

S. G. Yates, H. L. Tooke, J. J. Ellis and H. J. Burkhardt

Phytochem. 1, 139 (1968)

"Anisotropy of Nuclear Magnetic Resonance Spectra of Polyacrylonitrile Fibres"

A. I. Kol'tsov, S. Kamalov and M. V. Vol'kenshtein

Polymer Sci. USSR, (English Transl.) 9, 143 (1967)

"NMR Studies of Solvent-Solute Interactions of OH, SH and CH Protons in Dimethyl Formamide and Dimethyl Acetamide"

C. R. Kanekar, C. L. Khetrapal, K. V. Ramiah, and C. A.

Indirachary

Proc. Indian Acad. Sci. Sect. A, 64, 189 (1967)

"N.M.R. and I.R. Studies of 1, 1, 1 Trifluoroacetone in Proton Donor Solvents"

C. L. Khetrapal, M. M. Dhingra and V. B. Kartha

Proc. Indian Acad. Sci. Sect. A 64, 196 (1967)

"Configuration, Conformation and Spin in Radical Pairs"

P. D. Bartlett and J. M. McBride

Pure Appl. Chem. 15, 89 (1967)

"Synthesis and Reactions of 6-Phenyl-2:3-Benzobicyclo[3.1.0]hex-2-en-4-one"

M. Popovici, V. Ioan, M. Elian and C. D. Nenitzescu

Rev. Roumaine Chim. Acad. Rep. Socialiste Roumaine

12, 583 (1967)

"Synthesis of Oxazolidinic Dispirans"

N. Bartulescu, C. Greff and C. Balaita

Rev. Roumaine Chim. Acad. Rep. Socialiste Roumaine

12, 705 (1967)

"The High Resolution Nuclear Magnetic Resonance Spectrum of Acridine-N-Oxide"

H. H. Mantsch, W. Seiffert and V. Zanker

Rev. Roumaine Chim. Acad. Rep. Socialiste Roumaine

12, 1137 (1967)

"Free-Induction Decay in a Magnetically Dilute Solid"

T. M. Wu

Solid State Commun. 5, 935 (1967)

"Relaxation and Nuclear Magnetic Resonance of Polarized β -Active Nuclei"

A. D. Gulko, S. S. Trostin, and A. Hudoklin

Soviet Phys. JETP (English Transl.) 25, 998 (1967)

"Magnetic Relaxation of Nuclei in Crystals Containing Magnetic Impurities"

G. R. Khutsishvili

Soviet Phys. JETP (English Transl.) 25, 1050 (1967)

"Synthesis of Cyclopropanes by the Reaction of Olefins with Dialkylzinc and Methylene Iodide"

J. Furukawa, N. Kawabata and J. Nishimura

Tetrahedron 24, 53 (1968)

"Gibberelline—XII. Zur Umlagerung von 8,15-Epoxy-Gibberellinsäure"

K. Schreiber, G. Schneider und G. Sembdner

Tetrahedron 24, 73 (1968)

"Protonenresonanz-Spektroskopische Untersuchungen zur Kenntnis der Bindungseigenschaften des Siliciums in Organo-silicium-Verbindungen"

H. A. Brune

Tetrahedron 24, 79 (1968)

"NMR Studies of Rate Processes and Conformations—IX. Conformational Rate Processes in Bridged Bicyclic Tetrahydropyridazine and Piperidazine Systems"

J. E. Anderson and J. M. Lehn

Tetrahedron 24, 123 (1968)

"The Formation of Alkylplatinum Carbonyl Chlorides by Treatment of a Tetrachloroplatinate with Olefins and Formic Acid"
E. Lodewijk (in part) and D. Wright
J. Chem. Soc., A, Inorg. Phys. Theor. 119 (1968)

"Polyfluoroacetone Complexes of Transition Metals"
B. Clarke, M. Green, R. B. L. Osborn and F. G. A. Stone
J. Chem. Soc., A, Inorg. Phys. Theor. 167 (1968)

"Chemistry of the Metal Carbonyls. Part XLI. Synthesis of Complexes with C_6F_5S or $p-RS \cdot C_6F_4$ ($R=Me$ or Ph) Groups as Ligands"
J. Cooke, M. Green, and F. G. A. Stone
J. Chem. Soc., A, Inorg. Phys. Theor. 170 (1968)

"Chemistry of the Metal Carbonyls. Part XLII. Reactions between Carbonylmethyl Anions and Perfluoropyridine, Perfluoropyridazine, or Cyanuric Fluoride"
J. Cooke, M. Green and F. G. A. Stone
J. Chem. Soc., A, Inorg. Phys. Theor. 173 (1968)

"Chemistry of the Metal Carbonyls. Part XLIII. Perfluoroallenyl-rhenium and -iron Complexes"
R. J. Goodfellow, M. Green, N. Mayne, A. J. Rest and F. G. A. Stone
J. Chem. Soc., A, Inorg. Phys. Theor. 177 (1968)

"The Chemistry of Ottonia vahlii, Kth. Part I. The Volatile Constituent"
A. R. Pinder and S. J. Price
J. Chem. Soc., C, Org. 2597 (1967)

"The Synthesis of Pyrido[4,3-d]pyrimidin-4(3H)-ones from 4-Aminonicotinic Acid"
A. G. Ismail and D. G. Wiberley
J. Chem. Soc., C, Org. 2613 (1967)

"The Thermal Decomposition of n-Alkyltriarylphosphonium Alkoxides"
A. Majid Hamid and S. Trippett
J. Chem. Soc., C, Org. 2625 (1967)

"Aromatic Polyfluoro-compounds. Part XXXIX. The Synthesis of Some Fluoroaromatic Acetylenes"
P. L. Coe, J. C. Tatlow and R. C. Terrell
J. Chem. Soc., C, Org. 2626 (1967)

"Electrochemical Reactions. Part II. The Structure of an Unusual Product from the Reduction of 1-Acetylnaphthalene"
J. Grimshaw and E. J. F. Rea
J. Chem. Soc., C, Org. 2628 (1967)

"Polypeptides. Part V. The Use of t-Butyl 2,4,5-Tri-chlorophenyl Carbonate in the Synthesis of N-t-Butoxycarbonyl Amino-acids and their 2,4,5-Tri-chlorophenyl Esters
W. Bradbent, J. S. Morley and B. E. Stone
J. Chem. Soc., C, Org. 2632 (1967)

"The Action of Sulphur Monochloride on 2-Acyl-1,1-dimethylhydrazines; the Formation of Tetrazans and Oxadiazolines"
P. Hope and L. A. Wiles
J. Chem. Soc., C, Org. 2636 (1967)

"Solvent Effects in Nuclear Magnetic Resonance Spectroscopy. Part XIII. Solvent Shifts Induced by Benzene in some α,β -Unsaturated Compounds. An Aid to the Determination of Stereochemistry"
J. Ronayne and D. H. Williams
J. Chem. Soc., C, Org. 2642 (1967)

"Synthesis of Some N-Substituted 4-Piperidones"
J. D. Baty, G. Jones, and C. Moore
J. Chem. Soc., C, Org. 2645 (1967)

"The Abnormal Reaction between Methyl 1-Phenylpyrazole-4-carboxylate and Phenylmagnesium Bromide"
I. L. Finar and D. M. Rackham
J. Chem. Soc., C, Org. 2650 (1967)

"The Oxidative Dimerisation of 1,2,3,4-Tetrahydro-6-hydroxy-7-isopropyl-naphthalene"
C. P. Falshaw, A. W. Johnson, T. J. King and S. I. Rodrigo
J. Chem. Soc., C, Org. 2652 (1967)

"Butadiene Sulphone Chemistry. Part IV. Evidence for the Structures of Some Butadiene Sulphone Derivatives from Proton Magnetic Resonance Measurements"
J. A. Elvidge
J. Chem. Soc., C, Org. 2656 (1967)

"Organometallic Reactions. Part IX. The Preparation and Reactions of Trialkyl-lead Alkoxides"
A. G. Davies and R. J. Puddephatt
J. Chem. Soc., C, Org. 2663 (1967)

"The Reaction of 2,4-Diethoxycarbonyl-5-hydroxy-5-methyl-3-phenylcyclohexanone with 2,4-Dinitrophenylhydrazine"
T. D. Binns and R. Brettell
J. Chem. Soc., C, Org. 2676 (1967)

"Dienone-Phenol Type Rearrangements. Part II. Synthesis of a Bicyclic Phenol"
B. R. Davis and L. J. Forrest
J. Chem. Soc., C, Org. 2677 (1967)

"Organophosphorus Compounds. Part V. Cationic Reactions of Vinyl Acetate with OO-Dialkyl Phosphorodithioates"
T. Nishiwaki
J. Chem. Soc., C, Org. 2680 (1967)

"The Synthesis of D-Psicose Derivatives using Recent Oxidative Procedures"
K. James, A. R. Tatchell, and (in part) P. K. R. y
J. Chem. Soc., C, Org. 2681 (1967)

"Elimination-Addition. Part XIV. Addition of Carboxylate Ions to Allenes and Acetylenes and Use of the Adducts in Acylation"
G. D. Appleyard and C. J. M. Stirling
J. Chem. Soc., C, Org. 2686 (1967)

"The Addition of Polyhalogenomethanes and Related Compounds to Norbornadiene"
D. I. Davies and L. T. Parfitt
J. Chem. Soc., C, Org. 2691 (1967)

"Reactions of Phenylglyoxal Hydrate and Some Aromatic Amines"
G. R. Proctor and M. A. Rehman
J. Chem. Soc., C, Org. 2696 (1967)

"2-Alkoxy-5-amino- and -5-arenesulphonamido-1,3,4-thiadiazoles and Related Compounds"
R. Clarkson and J. K. Landquist
J. Chem. Soc., C, Org. 2700 (1967)

"Periodate Oxidation of 4,4,6,6-Tetramethylcyclohexane-1,2,3-triol"
J. S. McConaghy, jun., and J. J. Bloomfield
J. Chem. Soc., C, Org. 7 (1968)

"A Synthesis of 1-O-Octadec-cis-1'-enyl-L-glycerol"
J. Gigg and R. Gigg
J. Chem. Soc., C, Org. 16 (1968)

"Studies in Mycological Chemistry. Part XXII. Total Synthesis of (+)-Aflatoxin-B₂"
J. C. Roberts, A. H. Sheppard and (in part) J. A. Knight and P. Roffey
J. Chem. Soc., C, Org. 22 (1968)

"Benzocyclo-octatetraenes. Part I. Bromo-derivatives Formed in Addition Reactions of Biphenylene"
J. W. Barton and K. E. Whitaker
J. Chem. Soc., C, Org. 28 (1968)

"NMR Studies of Rate Processes and Conformations—X. Conformational Rate Processes in Di-, Tetra- and Hexa-Hydropyridazine Systems"
J. E. Anderson and J. M. Lehn
Tetrahedron 24, 137 (1968)

"The Structures of Anisatin and Neoanisatin Toxic Sesquiterpenes from *Illicium Anisatum* L."
K. Yamada, S. Takada, S. Nakamura and Y. Hirata
Tetrahedron 24, 199 (1968)

"The Biosynthesis of the Gibberellins—II"
B. E. Cross, R. H. B. Galt and K. Norton
Tetrahedron 24, 231 (1968)

"Synthesis of Pyridazine Derivatives—XV. Some Electrophilic Substitutions on Imidazo[1,2-b]-Pyridazines"
J. Kobe, B. Stanovnik and M. Tisler
Tetrahedron 24, 239 (1968)

"Rearrangements of 3-Methylcar-4-en-2-one"
A. J. Bellamy and G. H. Whitam
Tetrahedron 24, 247 (1968)

"Elimination of the 4-Hydroxyl Group of the Alkaloids Related to Morphine—IX. Synthesis of 3-Methoxy-N-Methylisomorphinan Derivatives"
Y. K. Sawa, M. Horiuchi and K. Tanaka
Tetrahedron 24, 255 (1968)

"Elimination of the 4-Hydroxyl Group of the Alkaloids Related to Morphine—X. Structure of the Ketal Derivatives Prepared from 7-Oxo-Dihydrothebainone"
Y. K. Sawa, K. Okabe and T. Miyamoto
Tetrahedron 24, 261 (1968)

"Proton Magnetic Resonance Studies of Cyclic Compounds—VI. *cis*- and *trans*-2,6-Dimethylpiperidine and *cis* (2,4), *cis* (4,6), *cis* (2,6)-2,4,6-Trimethylpiperidine"
H. Booth, J. H. Little, and J. Feeney
Tetrahedron 24, 279 (1968)

"Revised Structures for the Adducts of Ergosteryl Acetate with Maleic Anhydride"
D. N. Jones, P. F. Greenhalgh and I. Thomas
Tetrahedron 24, 297 (1968)

"Die Konstitution der Valepotriate. 3. Mitteilung über die Wirkstoffe des Baldrians"
P. W. Thies
Tetrahedron 24, 313 (1968)

"The Addition of Aryl Azides to Unstrained Olefins"
P. Scheiner
Tetrahedron 24, 349 (1968)

"The Stereochemistry of the Michael Addition"
R. A. Abramovitch and D. L. Struble
Tetrahedron 24, 357 (1968)

"Reactions of α -Hydroxyimino Derivatives of Carbonyl Compounds with Bases—I. Derivatives of α,β -Dihydroxyimino-Butyric Acid"
G. Alimenti, M. Grifantini, F. Gualtieri and M. L. Stein
Tetrahedron 24, 395 (1968)

"1,2-Dihydroisoquinolines—VI. The Benzylation of Isoquinoline Derivatives"
M. Sainsbury, S. F. Dyke, D. W. Brown and W. G. D. Lugton
Tetrahedron 24, 427 (1968)

"The Constituents of *Cacalia Decomposita* A. Gray—IV. Structure of Decompositin"
L. Rodriguez-Hahn, A. Guzmán and J. Romo
Tetrahedron 24, 477 (1968)

"Natural Chromenes—III. Colouring Matters of Wars: The Structure of Flemingins A, B, C and Homoflemingin"
G. Cardillo, L. Merlini and R. Mondelli
Tetrahedron 24, 497 (1968)

"Configurational Analysis of Rhynchophylline-Type Oxindole Alkaloids. The Absolute Configuration of Ciliaphylline, Rhynchociline, Specionoxeine, Isospecionoxeine, Rotundifoline and Isorotundifoline"
W. F. Trager, C. M. Lee, J. D. Phillipson, R. E. Haddock, D. Dwuma-Badu and A. H. Beckett
Tetrahedron 24, 523 (1968)

"Organic Reactions with Polyphosphoric Acid VII. Wagner-Meerwein Rearrangement in the Intermolecular Acylation of Cycloheptene"
S. B. Kulkarni and S. Dev
Tetrahedron 24, 545 (1968)

"Organic Reactions With Polyphosphoric Acid VIII. Intramolecular Acylation with Lactones (Further Extension), Hydroxy Acids and Esters"
S. B. Kulkarni and S. Dev
Tetrahedron 24, 553 (1968)

"Magnetic Anisotropy and the Interpretation of the NMR Spectra of Lactams—I. N-Substituted Piperidones"
H. Baumann, N. C. Franklin, and H. Möhrle, U. Scheidegger
Tetrahedron 24, 589 (1968)

"On the Reaction of Bicyclobutanes With Carbonyl Compounds"
G. Just and E. Lee-Ruff
Tetrahedron 24, 601 (1968)

"The Reaction of Benzotropenium Ion, 1,2-Benzocycloheptatriene-7-Methyl Ether and 3,4-Benzocycloheptatriene-7-Methyl Ether with Methyl and 2-Propyl Grignard Reagents"
D. J. Bertelli and W. J. Rossiter
Tetrahedron 24, 609 (1968)

"A New Synthesis of 4,4'-Dihydroxydiphenyl(2-Pyridyl) Methane"
G. Pala, E. Crescenzi and G. Bietti
Tetrahedron 24, 619 (1968)

"Components of the Root of *Lindera Strychnifolia* VILL.—XIII. Structure of Isogemafurene and Linderoxide"
H. Ishii, T. Tozjo, M. Nakamura and K. Takeda
Tetrahedron 24, 625 (1968)

"The Synthesis of Oligoribonucleotides—IV. Preparation of Dinucleoside Phosphates from 2',5'-Protected Ribonucleoside Derivatives"
B. E. Griffin, M. Jarman and C. B. Reese
Tetrahedron 24, 639 (1968)

"Triterpenoids from *Bupleurum Falcatum* L.—III. Isolation of Genuine Sapogenins, Saikogenins E, F, and G"
T. Kubota and H. Hinoh
Tetrahedron 24, 675 (1968)

"Hypohalite-Induced Oxidative Decarboxylation of α -Amino Acids"
E. E. van Tamelen, V. B. Haarstad and R. L. Orvis
Tetrahedron 24, 687 (1968)

"Some *cis* and *trans*-Decahydroisoquinoline Derivatives and Their NMR Spectra. An Interesting Stable Twist-Boat Fused Cyclohexane"
R. A. Abramovitch and D. L. Struble
Tetrahedron 24, 705 (1968)

"O-Methylsterigmatocystin, A New Metabolite from *Aspergillus Flavus*, Link Ex Fries"
H. J. Burkhardt and J. Forgas
Tetrahedron 24, 717 (1968)

"Small Ring Compounds—XVII. The Methanolysis of *trans*- $\alpha,\alpha,\alpha',\alpha'$ -Tetramethyl-1,2-Cyclopropanedimethanol"
T. Shono, T. Yoshimura and R. Oda
Tetrahedron 24, 729 (1968)

"The Rearrangement of Ethyl 2-Hydroxyindoxyl-2-carboxylate to Ethyl 3-Hydroxyoxindole-3-carboxylate"
R. M. Acheson and S. R. G. Booth
J. Chem. Soc., C, Org. 30 (1968)

"Bacterial Degradation of Riboflavin"
E. C. Owen and D. W. West
J. Chem. Soc., C, Org. 34 (1968)

"Iron Carbonyl Complexes from Schiff Bases"
M. M. Bagga, W. T. Flannigan, G. R. Knox, and P. L. Pauson, F. J. Preston and R. I. Reed
J. Chem. Soc., C, Org. 36 (1968)

"Amino-acids and Peptides. Part XXVII. Further Studies on Esters of 1-Hydroxypiperidine"
J. H. Jones and G. T. Young
J. Chem. Soc., C, Org. 53 (1968)

"Pteridine Studies. Part XXXIV. Nucleophilic Addition Reactions of Pteridine-2-thiol and 2-(methylthio) pteridine"
A. Albert, and J. J. McCormack
J. Chem. Soc., C, Org. 63 (1968)

"Photochemical Behaviour of Bridged Compounds. Part II. Photolytic and Some Ground State Reactions of Carbonyl-bridged Anhydrides and their Decarbonylation Products"
B. Fuchs
J. Chem. Soc., C, Org. 68 (1968)

"The Biosynthesis of Phenols. Part XV. Some Metabolites of Penicillium Citrinum Related to Citrinin"
R. F. Curtis, C. H. Hassall and M. Nazar
J. Chem. Soc., C, Org. 85 (1968)

"Synthetic Hydroxyrotenoids"
D. J. Ringshaw and H. J. Smith
J. Chem. Soc., C, Org. 102 (1968)

"Bromination of Creatine, Creatinine, and Creatine Ethyl Ester"
I. P. Rudakova, T. A. Pospelova, and A. M. Yurkevich
J. Gen. Chem. USSR (English Transl.) 37, 313 (1967)

"Reaction of Polyhalogenated Ethers with Bromine Fluorides"
V. S. Yuminov, L. N. Pushkina, and S. V. Sokolov
J. Gen. Chem. USSR (English Transl.) 37, 350 (1967)

"Reactions of Hydride Alkoxyalkylsilanes with 1,2-Epoxy-4-Vinylcyclohexane"
K. A. Andrianov, G. P. Bochkareva, and M. A. Golubenko
J. Gen. Chem. USSR (English Transl.) 37, 372 (1967)

"Reactions of Compounds Containing Mobile Halogen III. Reactions of Ethanesulphenyl Chloride with Alkenylphosphonic Esters"
S. Z. Ivin, V. K. Promonenkov, and B. I. Tetel'baum
J. Gen. Chem. USSR (English Transl.) 37, 454 (1967)

"Hydrogen Bonds in Phosphonium Salts Studied with the Aid of IR and NMR Spectra"
L. B. Senyavina, V. I. Sheichenko, Yu. N. Sheinker, A. V. Dombrovskii, M. I. Shevchuk, L. I. Barsukov, and L. D. Bergel'son
J. Gen. Chem. USSR (English Transl.) 37, 469 (1967)

"The Synthesis of Seneciophyllic Acid"
J. D. Edwards, Jr., T. Hase, and N. Ichikawa
J. Heterocyc. Chem. 4, 487 (1967)

"1,4-Disubstituted Pyridazino[4,5-d]pyridazines"
L. C. Dorman
J. Heterocyc. Chem. 4, 491 (1967)

"Base Catalyzed Acylative Decarboxylation. Part III. Acylation of Homophthalic Acid"
G. G. Smith, C. W. Delong, W. H. Wetzel and V. P. Muralidharan
J. Heterocyc. Chem. 4, 501 (1967)

"Chlorination of Isothiocyanates. VIII. Reaction of S-Chloroisothiocarbonyl Chlorides with Aliphatic and Cycloaliphatic Ketones"
G. Ottmann, G. D. Vickers and H. Hooks, Jr.
J. Heterocyc. Chem. 4, 527 (1967)

"Synthesis and Properties of Fluorine-Containing Heterocyclic Compounds. V. Trifluoromethyl-1,8- and 1,10-Phenanthrolines"
E. B. Nyquist and M. M. Joullie
J. Heterocyc. Chem. 4, 539 (1967)

"Friedländer Syntheses with o-Aminoaryl Ketones. III. Acid-Catalyzed Condensations of o-Aminobenzophenone with Polyfunctional Carbonyl Compounds"
E. A. Fehnel
J. Heterocyc. Chem. 4, 565 (1967)

"as-Triazine Chemistry. V. The Oxidative Ring-opening of 5,6-Diphenyl-as-triazine-3(2H)-ones with Organic Peracids"
T. Sasaki and K. Minamoto
J. Heterocyc. Chem. 4, 571 (1967)

"A New Synthesis of Thiazolo[3,2-a]pyrimidinones"
H. F. Andrew and C. K. Bradsher
J. Heterocyc. Chem. 4, 577 (1967)

"PMR Spectra and Conformation of some Four-membered Rings Containing One Nitrogen Atom. 2-Azetidinones, 2-Azetidinethiones and 1-Azetines"
A. Vigevani and G. G. Gallo
J. Heterocyc. Chem. 4, 583 (1967)

"1-Substituted Phenothiazine Derivatives"
R. H. Nealey and J. S. Driscoll
J. Heterocyc. Chem. 4, 587 (1967)

"Some Hydroxylated Derivatives of Chlorpromazine"
H. M. Grotta, T. F. Page, Jr., C. J. Riggle, and A. A. Manian
J. Heterocyc. Chem. 4, 611 (1967)

"1-Azetines"
G. Pifferi, P. Consonni, G. Pelizza and E. Testa
J. Heterocyc. Chem. 4, 619 (1967)

"The Reaction of Imidazole and Acrylonitrile"
T. J. Schwan
J. Heterocyc. Chem. 4, 633 (1967)

"New 2,3-Dihalopyridines. Potential Precursors for 2,3-Dehydropyridine"
W. J. Link, R. F. Borne, and F. L. Setliff
J. Heterocyc. Chem. 4, 641 (1967)

"Reactions of 2,2,4,4-Tetramethyl-1,3-Cyclobutanedione with Aziridines and Other Nucleophiles"
G. R. Hansen and T. E. Burg
J. Heterocyc. Chem. 4, 653 (1967)

"A Stable α -Lactam Derived from Adamantane"
E. R. Talaty, A. E. Dupuy, Jr., and A. E. Cancienne, Jr.
J. Heterocyc. Chem. 4, 657 (1967)

"Synthesis and Tautomeric Behavior of Dihydropyrido [2,3-b] [1,4]diazepinones"
M. Israel, L. C. Jones and E. J. Modest
J. Heterocyc. Chem. 4, 659 (1967)

"The Structures and Arundoin, Cylindrin and Fernenol"
K. Nishimoto, M. Ito, S. Natrioi and T. Ohmoto
Tetrahedron 24, 735 (1968)

"The Amaryllidaceae Alkaloids; Transformation of
Lycorine into Hippeastrine"
K. Kotera, Y. Hamada and R. Nakane
Tetrahedron 24, 759 (1968)

"Total Synthesis of Anthrasteroids. I. Formylation
of Certain Cyclic α,β - and β,γ -Unsaturated Ketones"
K. Wiedhaup, A. J. H. Nolllet, J. G. Korsloot and
H. O. Huisman
Tetrahedron 42, 771 (1968)

"Total Synthesis of Anthrasteroids. II. Preparation of
Some Anthraestrone and Anthraequilenine Derivatives"
K. Wiedhaup, F. H. Kesselaar and H. O. Huisman
Tetrahedron 24, 779 (1968)

"Total Synthesis of Anthrasteroids. III. The Preparation
of Some 3-Phenyl-2,4-Diaza-Anthraestrans"
K. Wiedhaup and H. O. Huisman
Tetrahedron 24, 789 (1968)

"The Diterpenes of Goodenia Ramelii F. Muell"
P. Coates, A. K. Goh, P. R. Jefferies, J. R. Knox and
T. G. Payne
Tetrahedron 24, 795 (1968)

"Conformation of *o*-Methylated Amylose and Cyclodextrins"
B. Casu, M. Reggiani, G. G. Gallo and A. Vigevani
Tetrahedron 24, 803 (1968)

"The Structure and Stereochemistry of Two New Dihydro-
flavonol Glycosides"
K. R. Markham and T. J. Mabry
Tetrahedron 24, 823 (1968)

"Conformational Isomerization in Hexahydro-s-triazines
and Hexahydropyrimidines"
R. F. Farmer and J. Hamer
Tetrahedron 24, 829 (1968)

"Pleidene Systems. VI. Further Studies in the Methyl-
pleiadene Series"
M. P. Cava and W. S. Lee
Tetrahedron 24, 837 (1968)

"Aza Steroids. VII. Synthesis of Ring A-oxygenated
6-Aza Steroids"
J. P. Kutney, G. Eigendorf and J. E. Hall
Tetrahedron 24, 845 (1968)

"Acid-catalyzed Cyclization of Farnesol and Nerolidol"
C. D. Gutsche, J. R. Maycock and C. T. Chang
Tetrahedron 24, 859 (1968)

"Polyfluoroheterocyclic Compounds. X. 2,3,5,6-Tetrafluoro-
4-methylpyridine and Related Compounds"
R. D. Chambers, B. Iddon, W. K. R. Musgrave and L. Chadwick
Tetrahedron 24, 877 (1968)

"Synthetic Studies in Steroidal Sapogenins and Alkaloids.
III. The Synthesis and Stereochemistry of Isomeric
16-Hydroxy- and 16-Oxo-5,17(20)-pregnadien-3 β -ols"
S. V. Kessar and A. L. Rampal
Tetrahedron 24, 887 (1968)

"On the Aromatic Character of 4-Pyrones"
H. C. Smitherman and L. N. Ferguson
Tetrahedron 24, 923 (1968)

"Synthesis of Grifolin, and Antibiotic from a Basidio-
mycete"
M. Isobe and T. Goto
Tetrahedron 24, 945 (1968)

"Studies on Conformation and Reactivity. V. A Nuclear
Magnetic Resonance Study of 4-Ethylthio-4-en-3-oxo
Steroids and Their Analogs: The Unique Deshielding
Effect of a Polar Thio-Function"
M. Tomoeda, M. Inuzuka, T. Furuta and M. Shinozuka
Tetrahedron 24, 959 (1968)

"Propellanes. III. Synthesis of Carbocyclic and Hetero-
cyclic Compounds"
J. Altman, E. Babad, J. Pucknat, N. Reshef and
D. Ginsburg
Tetrahedron 24, 975 (1968)

"Rearrangement of *o*-Arenesulfonyl Phenylhydroxylamines"
G. T. Tisue, M. Grassmann and W. Lwowski
Tetrahedron 24, 999 (1968)

"Synthesis of Quinolino Heterocycles"
T. George and R. Tahilramani
Tetrahedron 24, 1007 (1968)

"Die Umsetzung von Diazoverbindungen mit Azoverbindungen.
VII. Die Struktur der bei der Umsetzung von Diazo-
verbindungen mit Azodibenzoyl oder Azodiacetyl
Entstehenden Produkte"
E. Fahr, K. Doppert, K. Konigsdorfer und F. Scheckenbach
Tetrahedron 24, 1011 (1968)

"Haschisch. XIV. zur Kenntnis der Inhaltsstoffe des
Haschisch"
U. Claussen, F. von Spulak and F. Korte
Tetrahedron 24, 1021 (1968)

"The Diels-alder Reaction between Styrene and trans-trans
Hexa-2,4-Diene. Structure and Conformation of Two
Isomeric Adducts"
O. Korver, Kwa T. L. and C. Boelhouwer
Tetrahedron 24, 1025 (1968)

"Additionsreaktionen der Nitrosogruppe. IX. Die Diels-
Alder-reaktion von 1-Mono-BZW. 1,4-Disubstituierten
Dienen mit Nitrosoverbindungen"
G. Kresze und J. Firl
Tetrahedron 24, 1043 (1968)

"Triterpenoids. XXIX. The Structure of Barringtonol B-
A New Triterpenoid Sapogenin from Barringtonia
Acutangula Gaertn"
A. K. Barua and S. P. Dutta and B. C. Das
Tetrahedron 24, 1113 (1968)

"Narciclasine and Narciprimine"
F. Piozzi, C. Fuganti, R. Modelli and G. Ceriotti
Tetrahedron 24, 1119 (1968)

"Diborane as a Reducing Agent. II. The Reduction of
Indole and Pyrrole Carbonyl Derivatives"
K. M. Biswas and A. H. Jackson
Tetrahedron 24, 1145 (1968)

"The Kolbe Electrolysis as a Source of Free Radicals
in Solution. V. The Electrolysis of Potassium Acetate
in the Presence of 3,3-Dimethyl-1-butene"
W. B. Smith and Y. Hyon Yuh
Tetrahedron 24, 1163 (1968)

"Propriétés Physico-chimiques de Dérivés Aromatiques
Pontés: [2,2], [2,2,2] et [2,2,2,2] Metacyclophanes"
R. Flammang, H. P. Figeys et R. H. Martin
Tetrahedron 24, 1171 (1968)

"The Synthesis of Some 2,10-Epoxy-pinanes"
J. M. Coxon, E. Dansted, M. P. Hartshorn and K. E. Richards
Tetrahedron 24, 1193 (1968)

- "Rearrangement of 2,4-Diphenylthietan Dioxides to 3,5-Diphenyl-1,2-oxathiolan 2-Oxides"
R. M. Dodson, P. D. Hammen, and R. A. Davis
Chem. Commun. 9 (1968)
- "Reactions of Phenacyl Bromide with Nickel Carbonyl"
E. Yoshisato and S. Tsutsumi
Chem. Commun. 33 (1968)
- "The Conformations of δ -Lactones"
R. C. Sheppard and S. Turner
Chem. Commun. 77 (1968)
- "The Absolute Stereochemistry at C-2 of Tiazolidines derived from R-Penicillamine and Aldehydes"
I. McMillan and R. J. Stoodley
Chem. Commun. 11 (1968)
- "Structural Studies on Bromoglycosyl Fluorides"
J. C. Campbell, R. A. Dwek, P. W. Kent, and C. K. Prout
Chem. Commun. 34 (1968)
- "A Useful Homologation Reaction for the Preparation of Fluoro-ketones"
P. Crabbe, H. Carpio, A. Cervantes, J. Iriarte, and L. Tökes
Chem. Commun. 79 (1968)
- "4 α -Methyl-24-methylene-24,25-dihydrozymosterol, a New Sterol of *Saccharomyces cerevisiae* of Possible Importance in the Biosynthesis of Ergosterol"
D. H. R. Barton, D. M. Harrison and D. A. Widdowson
Chem. Commun. 17 (1968)
- "Nuclear Magnetic Resonance Study of 2-Halogeno-2-deoxyhexopyranosyl Fluorides"
L. D. Hall and J. F. Manville
Chem. Commun. 37 (1968)
- "Coupling and 'Virtual' Coupling in the Nuclear Magnetic Resonance Spectra of Phosphine Complexes"
A. Pidcock
Chem. Commun. 92 (1968)
- "Temperature Dependence of Orientation in the Photosensitized Addition of Maleic Anhydride to Methylbenzenes"
D. Bryce-Smith and A. Gilbert
Chem. Commun. 19 (1968)
- "The Structure of Rishitin, a New Antifungal Compound from Diseased Potato Tubers"
N. Katsui, A. Murai, M. Takasugi, K. Imaizumi, and T. Masamune
Chem. Commun. 43 (1968)
- "Stereoselectivity in the Synthesis of *cis*- and *trans*-4,4a-Dimethyl-2-octalone Derivatives"
R. M. Coates and J. E. Shaw
Chem. Commun. 47 (1968)
- "Structures of Chiograsterol A and B"
K. Takeda, H. Minato and A. Shimaoka
Chem. Commun. 104 (1968)
- "A Transition-metal Complex of Benzyne"
E. W. Gowling, S. F. A. Kettle, and (Miss) G. M. Sharples
Chem. Commun. 21 (1968)
- "Spectroscopic Studies on Potassium Nitrobenzenide"
J. M. Gross and J. D. Barnes
Chem. Commun. 50 (1968)
- "Structure of Liguloxide, Liguloxidol, and Liguloxidol Acetate, Sesquiterpenes of Antipodal Guaiane Type"
H. Ishii, T. Tozyo, and H. Minato
Chem. Commun. 106 (1968)
- "Isolation of a Substance that suppresses Feeding in Locusts"
J. H. Butterworth and E. D. Morgan
Chem. Commun. 23 (1968)
- "Electronic Transmission in Schiff-base Complexes of Cobalt(III)"
R. E. Ball and J. F. Endicott
Chem. Commun. 51 (1968)
- "Isohistamine [2-(2-Aminoethyl)imidazole]"
G. J. Durant, M. E. Footitt, C. R. Ganellin, J. M. Loynes, E. S. Pepper and A. M. Roe
Chem. Commun. 108 (1968)
- "The *cis*-Molozonide"
L. J. Durham and F. L. Greenwood
Chem. Commun. 24 (1968)
- "Synthesis of 'Homoerythrinadienone' by Phenolic Oxidative Coupling"
T. Kametani and K. Fukumoto
Chem. Commun. 26 (1968)
- "Sulphur-containing Esters of Perfluoropinacol"
M. Allan, A. F. Janzen, and C. J. Willis
Chem. Commun. 55 (1968)
- "Variations in Sign for the Coupling Constants between Directly-bonded Phosphorus Nuclei"
E. G. Finer and R. K. Harris
Chem. Commun. 110 (1968)
- "Optical Rotatory Dispersion and Absolute Configuration of Flavanones, 3-Hydroxyflavanones, and Their Glycosides"
W. Gaffield, and A. C. Waiss, Jun.
Chem. Commun. 29 (1968)
- "Synthesis of (+)- α -Chamigrene"
A. Tanaka, H. Uda and A. Yoshikoshi
Chem. Commun. 56 (1968)
- "Isolation of Ocotillol, Acetovanillone, Androsin and a New Glycoside from the Cactus *Neolloydia texensis*"
X. A. Dominguez, R. Carrero, R. González, P. Rojas and R. Ketcham
Chem. Ind. (London) 2147 (1967)
- "Structure and Chemical Identity of Diadinoxanthin and the Principal Xanthophyll of *Euglena*"
K. Aitzetmüller, W. A. Svec, J. J. Katz and H. H. Strain
Chem. Commun. 32 (1968)
- "The Synthesis of D-Arcanose"
G. B. Howarth, W. A. Szarek, and J. K. N. Jones
Chem. Commun. 62 (1968)
- "Skeletal Rearrangements of Atisine Derivatives"
S. W. Pelletier and A. Ichihara
Chem. Ind. (London) 2149 (1967)

"Trifluoromethylsulfinyl Fluoride"

E. W. Lawless and L. D. Harman
Inorg. Chem. 7, 391 (1968)

"Contact Shifts of Acetonitrile Solutions of Cupric Chloride and Titanium Trichloride"

C. C. Hinckley
Inorg. Chem. 7, 396 (1968)

"Tautomerism and Geometric Isomerism in Arylazo-Phenols and Naphthols. Part III. Orthohydroxy Derivatives and Their Reversible Photochemical Reactions"

G. Gabor, Y. Frei, D. Gegiou, M. Kaganowitch and E. Fischer
Israel J. Chem. 5, 193 (1967)

"Oxygen - 17 NMR Chemical Shifts in Quinones"

E. Rotlevi
Israel J. Chem. 5, 28p (1967)

"N.M.R. Studies of Ion Solvation - Cobaltous and Titanous Ions"

A. M. Chmelnick and D. Fiat
Israel J. Chem. 5, 32p (1967)

"Proton and Oxygen-17 NMR Studies of the Rare-Earth Aquo-Ions"

J. Reuben and D. Fiat
Israel J. Chem. 5, 33p (1967)

R } "Measurement of the N¹⁴ Spin-Lattice Relaxation Times in Liquid NH₃ and ND₃"

Y. Margalit and A. Loewenstein
Israel J. Chem. 5, 34p (1967)

"Proton Magnetic Resonance Studies of Water as Hydrogen Donor to Tributyl Phosphate"

S. Nishimura, C. H. Ke, and N. C. Li
J. Am. Chem. Soc. 90, 234 (1968)

"Tentative Interpretation of ¹³C and ¹H Nuclear Magnetic Resonance Chemical Shifts in Simple Methyl and Ethyl Derivatives"

P. Bucci
J. Am. Chem. Soc. 90, 252 (1968)

HF } "Long-Range Proton-Fluorine Coupling in a Rigid System. The Nuclear Magnetic Resonance Spectra of 5-Substituted Difluorotetrachlorobicyclo[2.2.1]heptenes"

K. L. Williamson and J. C. Fenstermaker
J. Am. Chem. Soc. 90, 342 (1968)

"Neighboring-Group Effects in Free-Radical Reactions. The Thermal Decomposition of 1,1',2,2'-Tetra-phenylazoethane and 1,1',2,2'-Tetraphenylazoethane-1,1'-d₂"

S. E. Scheppele and S. Seltzer
J. Am. Chem. Soc. 90, 358 (1968)

"The Solvolysis of 4(5)-(2-Amino-3-bromopropyl)imidazole"

D. A. Usher
J. Am. Chem. Soc. 90, 363 (1968)

"Stable Carbonium Ions. LIX. Protonated Alkyl Carbamates and Their Cleavage to Protonated Carbamic Acids and Alkylcarbonium Ions"

G. A. Olah and M. Calin
J. Am. Chem. Soc. 90, 401 (1968)

"Stable Carbonium Ions. LX. Protonated α,β -Unsaturated Carboxylic Acids and Their Cleavage to Alkenyloxocarbonium Ions"

G. A. Olah and M. Calin
J. Am. Chem. Soc. 90, 405 (1968)

"Reaction of Ninhydrin with Cytosine Derivatives"

R. Shapiro and S. C. Agarwal
J. Am. Chem. Soc. 90, 474 (1968)

"The Effect of Alcohol on Proton Exchange Reactions in Dimethyl Sulfoxide. I. Low Alcohol Concentrations"

J. I. Brauman, N. J. Nelson, and D. C. Kahl
J. Am. Chem. Soc. 90, 490 (1968)

"The Effect of Alcohol on Proton Exchange Reactions in Dimethyl Sulfoxide. II. High Alcohol Concentrations"

J. I. Brauman and N. J. Nelson
J. Am. Chem. Soc. 90, 491 (1968)

"Resonance-Stabilized Carbanions. II. Rotational Barriers in Phenylallyl Alkali Metal Salts"

V. R. Sandel, S. V. McKinley, and H. H. Freedman
J. Am. Chem. Soc. 90, 495 (1968)

"Aziridines. XI. Nitrogen Inversion in N-Haloaziridines"

S. J. Brois
J. Am. Chem. Soc. 90, 506 (1968)

"Aziridines. XII. Isolation of a Stable Nitrogen Pyramid"

S. J. Brois
J. Am. Chem. Soc. 90, 508 (1968)

"Restricted Rotation about the N-N Single Bond. Linear Correlation of Rate with Substituent"

N. P. Marullo, C. B. Mayfield, E. H. Wagener
J. Am. Chem. Soc. 90, 510 (1968)

"The Photorearrangement of 1,4-Epoxy-1,4-dihydronaphthalene to Benz[f]oxepin"

G. R. Ziegler and G. S. Hammond
J. Am. Chem. Soc. 90, 513 (1968)

"The Nuclear Overhauser Effect, a Unique Method of Defining the Relative Stereochemistry and Conformation of Taxane Derivatives"

M. C. Woods, H.-C. Chiang, Y. Nakadaira, and K. Nakanishi
J. Am. Chem. Soc. 90, 522 (1968)

"Pyrrolo[2,3-d]pyrimidine Nucleoside Antibiotics. Total Synthesis and Structure of Toyocamycin, UnamycinB, Vengicide, Antibiotic #-212, and Sangivamycin (BA-90912)"

R. L. Tolman, R. K. Robins and L. B. Townsend
J. Am. Chem. Soc. 90, 524 (1968)

"A Dinaphth[10]annulene"

R. H. Mitchell, and F. Sondheimer
J. Am. Chem. Soc. 90, 530 (1968)

"Oxygen-17 Magnetic Resonance Studies of Ion Solvation. The Hydration of Aluminum(III) and Gallium(III) Ions"

D. Fiat and R. E. Connick
J. Am. Chem. Soc. 90, 608 (1968)

"Hindered Ligand Systems. *cis,cis*-Triaminocyclohexane and Its Complexes with Trivalent Cobalt and Rhodium"

R. A. D. Wentworth and J. J. Felten
J. Am. Chem. Soc. 90, 621 (1968)

"Molecular Asymmetry. VIII. *trans*-Bicyclo[8.2.2]tetradeca-5,10,12,13-tetraene"

A. C. Cope and B. A. Pawson
J. Am. Chem. Soc. 90, 636 (1968)

"Hindered Rotation in Substituted Paracyclophanes"

G. M. Whitesides, B. A. Pawson and A. C. Cope
J. Am. Chem. Soc. 90, 639 (1968)

"Substituent Effects and Aryl Participation in β -(*syn*-9-Benzonorbornenyl)ethyl *p*-Bromobenzenesulfonate Solvolyses"

R. Muneyuki and H. Tanida
J. Am. Chem. Soc. 90, 656 (1968)

"Conformational Analysis. XIII. The Validity of the Nuclear Magnetic Resonance Method of Establishing Conformational Equilibria"
E. L. Eliel and R. J. L. Martin
J. Am. Chem. Soc. 90, 682 (1968)

"Conformational Analysis. XIV. Conformational Equilibria of Cyclohexyl Halides"
E. L. Eliel and R. J. L. Martin
J. Am. Chem. Soc. 90, 689 (1968)

C₁₃ | "Carbon-13 Magnetic Resonance. X. The Six-Membered Nitrogen Heterocycles and Their Cations"
R. J. Pugmire and D. M. Grant
J. Am. Chem. Soc. 90, 697 (1968)

"New Heteroaromatic Compounds. XXVII. Boron-11 Chemical Shifts of Some Heteroaromatic Boron Compounds"
F. A. Davis, M. J. S. Dewar, and R. Jones
J. Am. Chem. Soc. 90, 706 (1968)

"Nucleosides. XLIV. Long-Range Proton-Fluorine Spin-Spin Coupling in 5-Fluoropyrimidine Nucleosides"
R. J. Cushley, I. Wempen, and J. J. Fox
J. Am. Chem. Soc. 90, 709 (1968)

"Proton Magnetic Resonance Spectra of Ethylene Phosphites and Ethylene Sulfite"
P. Haake, J. P. McNeal and E. J. Goldsmith
J. Am. Chem. Soc. 90, 715 (1968)

"New Anionic Rearrangements. VIII. 1,4-Anionic Rearrangement of Silylethylenediamines"
R. West, M. Ishikawa and S. Murai
J. Am. Chem. Soc. 90, 727 (1968)

"Unsaturated Macrocyclic Compounds. LV. The Synthesis of 1-Bromocyclotrideca-1,2-diene-4,8,10-triyne, a Highly Unsaturated 13-Membered Ring Cyclic Allene"
C. C. Leznoff and F. Sondheimer
J. Am. Chem. Soc. 90, 731 (1968)

"Synthesis of Bis(as-indacenyl)liron"
T. J. Katz, V. Balogh, and J. Schulman
J. Am. Chem. Soc. 90, 734 (1968)

"Carbodiimide-Sulfoxide Reactions. VII. Synthesis of Stabilized Sulfonium Ylides"
A. F. Cook and J. G. Moffatt
J. Am. Chem. Soc. 90, 740 (1968)

"Nucleophilic Substitutions at Pentavalent Phosphorus. Reaction of 2,2,2-Trialkoxy-2,2-dihydro-1,3,2-dioxaphospholenes with Alcohols"
F. Ramirez, K. Tasaka, N. B. Desai, and C. P. Smith
J. Am. Chem. Soc. 90, 751 (1968)

"The Stereochemistry of the Catalytic and Light-Induced Reduction of Thymidine to Dihydrothymidine and Ureido Alcohols"
Y. Kondo and B. Witkop
J. Am. Chem. Soc. 90, 764 (1968)

"Photoreduction of Uridine and Reduction of Dihydro-uridine with Sodium Borohydride"
P. Gerutti, Y. Kondo, W. R. Landis, and B. Witkop
J. Am. Chem. Soc. 90, 771 (1968)

"Photocyclizations of Tyrosines, Tyramines, Catecholamines, and Normesalinaline"
O. Yonemitsu, T. Tokuyama, M. Chaykovsky and B. Witkop
J. Am. Chem. Soc. 90, 776 (1968)

"Chlorophyll-Ligand Interactions from Nuclear Magnetic Resonance Studies"
J. J. Katz, H. H. Strain, D. L. Leussing, and R. D. Dougherty
J. Am. Chem. Soc. 90, 784 (1968)

"The Ionic Character of Allyllithium"
P. West, J. I. Purmort, and S. V. McKinley
J. Am. Chem. Soc. 90, 797 (1968)

"The Photochemistry of 4,4-Dimethyl-1-methylene-2-cyclohexene, a Methylene Analog of a Cyclohexenone"
W. G. Dauben, and W. A. Spitzer
J. Am. Chem. Soc. 90, 802 (1968)

"Diamagnetic Susceptibility Exaltation as a Criterion of Aromaticity"
Hyp J. Dauben, Jr., J. D. Wilson and J. L. Laity
J. Am. Chem. Soc. 90, 811 (1968)

"The Structure of Thamnosin. A Novel Dimeric Coumarin System"
J. P. Kutney, T. Inaba and D. L. Dreyer
J. Am. Chem. Soc. 90, 813 (1968)

"A Benzohomotropylium Cation"
W. Merk, and R. Pettit
J. Am. Chem. Soc. 90, 814 (1968)

"Nuclear Magnetic Resonance Study of Molecular Motions in Hexamethylbenzene"
P. S. Allen and A. Cowking
J. Chem. Phys. 47, 4286 (1967)

"Additivity of Proton Magnetic Resonance Solvent Shifts in the Azine Series"
P. Laszlo and J. L. Soong, Jr.
J. Chem. Phys. 47, 4472 (1967)

"Molecular Motion and ¹⁹F Relaxation in the Liquid Hexafluorides of Molybdenum, Tungsten, and Uranium"
P. Rigny and J. Virlet
J. Chem. Phys. 47, 4645 (1967)

"Proton Resonance Study of the Hydrides of S, Se, and Te"
J. C. Hindman, A. Svirnickas, and W. B. Dixon
J. Chem. Phys. 47, 4658 (1967)

R | "Spin-Lattice Relaxation in Multilevel Systems"
R. L. Vold and H. S. Gutowsky
J. Chem. Phys. 47, 4782 (1967)

"Zinc and Magnesium Derivatives of Decaborane"
N. N. Greenwood and N. F. Travers
J. Chem. Soc., A, Inorg. Phys. Theor. 15 (1968)

"Azomethine Derivatives. Part IV. The Action of Some Organozinc Compounds on Diphenylketimine, Phenyl Cyanide, and t-Butyl Cyanide"
I. Pattison and K. Wade
J. Chem. Soc., A, Inorg. Phys. Theor. 57 (1968)

"The Transmission of Mesomeric Effects through Palladium and Platinum: Pyridine Proton Shifts in Complexes of the Type trans-[PtCl(py)L]"
J. Chatt and A. D. Westland
J. Chem. Soc., A, Inorg. Phys. Theor. 88 (1968)

"Some Reactions of Cobalt Phthalocyanines"
P. Day, H. A. O. Hill and M. G. Price
J. Chem. Soc., A, Inorg. Phys. Theor. 90 (1968)

"The Reaction of 1,3-Dienes with Phenylmanganese Carbonyl"
M. Green and R. I. Hancock
J. Chem. Soc., A, Inorg. Phys. Theor. 109 (1968)

"Nucleic Acid Components and Their Analogues. CII.
N-Alkyl Derivatives of 2- and 5-Substituted 4-
Hydroxy-6(1H)-Pyrimidinones"
M. Prystas
Collection Czech. Chem. Commun. 32, 4241 (1967)

"IR.-spektroskopische Untersuchungen in der
Chinoptalon-Reihe"
F. Kehrler, P. Niklaus, und B. K. Manukian
Helv. Chim. Acta 50, 2200 (1967)

"Photoisomerisierung von Pyrazolen und Indazolen zu
Imidazolen bzw. Benzimidazolen und 2-Amino-
benzonitrilen"
H. Tiefenthaler, W. Dörscheln, H. Göth und H. Schmid
Helv. Chim. Acta. 50, 2244 (1967)

"Reduktive Photoalkylierung des Flavinkerns und
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W. H. Walker, P. Hemmerich und V. Massey
Helv. Chim. Acta 50, 2269 (1967)

"Die Cardenolide von *Acokanthera oppositifolia* (Lam.)
CDD. 3. Mitteilung. Isolierung weiterer Cardenolide
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Helv. Chim. Acta 50, 2299 (1967)

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Helv. Chim. Acta 50, 2322 (1967)

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Helv. Chim. Acta 50, 2362 (1967)

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Helv. Chim. Acta 50, 2387 (1967)

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Helv. Chim. Acta 50, 2397 (1967)

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Helv. Chim. Acta 50, 2403 (1967)

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Helv. Chim. Acta 50, 2421 (1967)

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Helv. Chim. Acta 50, 2440 (1967)

"Synthese von α - und β -Sinensal"
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Helv. Chim. Acta 50, 2445 (1967)

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Helv. Chim. Acta 50, 2481 (1967)

"Photochemische Umwandlungen. XV. Photochemische
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3,7-dien-Systems"
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Helv. Chim. Acta 50, 2490 (1967)

"Beitrag zur Synthese von Tetracenchinonen"
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Helv. Chim. Acta 50, 2497 (1967)

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Helv. Chim. Acta 50, 2501 (1967)

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Helv. Chim. Acta 50, 2504 (1967)

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Helv. Chim. Acta 50, 2542 (1967)

"Recherches sur la formation et la transformation des
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Helv. Chim. Acta 50, 2563 (1967)

"Potassium Germyltrihydroborate"
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Inorg. Chem. 7, 213 (1968)

"Deuterium Isotope Studies on 2,3-Dicarbahehexaborane(8)"
J. R. Spielman, R. Warren, G. B. Dunks, J. E. Scott, Jr.
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Inorg. Chem. 7, 216 (1968)

"The Reactions of 1-Isopropenylcarborane with Dinitrogen
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Inorg. Chem. 7, 222 (1968)

"Boron Heterocycles. V. Preparation and Characteriza-
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Inorg. Chem. 7, 225 (1968)

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Inorg. Chem. 7, 306 (1968)

"Organometallic Compounds with Metal-Metal Bonds. X.
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Inorg. Chem. 7, 310 (1968)

"Cyclooctatetraene-Rhodium(I) Complexes"
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Inorg. Chem. 7, 321 (1968)

"Organosulfur Derivatives of the Metal Carbonyls. X.
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 π -Cyclopentadienyl and cis-1,2-Ethylenedithiolate
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Inorg. Chem. 7, 340 (1968)

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Inorg. Chem. 7, 345 (1968)

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Inorg. Chem. 7, 382 (1968)

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Tetrahedron Letters 5071 (1967)

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"Studies on Pyrimidine Derivatives and Related Compounds. LII. Reaction of Thiamine with Phosphites"
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Tetrahedron Letters 5113 (1967)

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Z. Anorg. Allg. Chem. 354, 113 (1967)

"Reaktionen von Phosphor(III)-Verbindungen mit
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E. Fluck und H. Binder
Z. Anorg. Allg. Chem. 354, 139 (1967)

"Advances in Chemical Physics"
Hirschfelder, J. O., ed.
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