

29. 8. 66

Prenos.

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

No. 94  
JULY, 1966

Little	1
Wanted: 40 MHz. RF Unit and Probe	
Koptuyug, Shubin, Rezvukhin, Korchagina, Tretyakov, Rudakov	2
Heptamethylbenzenonium Ion Rearrangement - Kinetic Studies by NMR	
Vijayaraghavan	5
Solid State Problems at TIFR using Nuclear Magnetic Resonance	
Lanzi, Mognaschi	6
Frequency Stabilization of the Varion VF-16 Oscillator	
Mislow, Raban, Bickart	8
Nomenclature for Stereochemically Nonequivalent Nuclei	
Lancaster, Neglia	10
Proton Shifts in Donor-Substituted Polycyclic Aromatic Hydrocarbons	
Homer, Huck	13
Perkin-Elmer R10 Signal to Noise Improvement	
Abramson, Wells	16
High Resolution Rotary Z-Echoes	
van der Werf	20
Slow Inversion Rate of a Sulphur Containing 7-Ring	
Caserio, M. C., Pratt, R. E.	21
Chemical Shifts and $\pi$ -Bonding in $\alpha,\beta$ -Unsaturated Organonitrogen and Organosulfur Compounds	
Richards	24
Dynamic Nuclear Polarisation	
Lambert, Mueller	26
The Inversion of Diphosphines	
Fritz, H.	28
Hindered Rotation in Trisubstituted (s)-Triazines	
Maciel	30
$C^{13}$ Chemical Shift Difference Between <u>Cis</u> and <u>Trans</u> Diiodoethylene	
Retcofsky	32
1967 Pittsburgh Conference	
Pople, Untch	33
Induced Paramagnetic Ring Currents	
Laszlo	34
Another Secondary Deuterium Isotope Effect; Rabelais and the (No, not the Franciscans!) Nomenclature for Equivalence	
Reinecke	36
NMR Spectra of Enamines of 1-Azabicycloalkanes	
Quarmby, Rowe, Wilkinson	38
Iridium Hydride Spectra on the Varian A-60A	

OVER

Willcott	
"The Principles of Nuclear Magnetic Resonance" - A Workshop at the University of Houston; 30 August - 1 September, 1966	40
Musher	
On Spin-Spin Coupling Constants	42
Colebrook	
RA-1 Spectrum Accumulator	44
Fратиello, Miller, Schuster	
Rate Studies of Dimethylformamide Complexes	46

Deadline Dates: No. 95 - 15 August 1966 No. 96 - 15 September 1966
---

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

---

June 23, 1966

Dr. Barry Shapiro  
Illinois Institute of Technology  
Chicago, Illinois

Dear Dr. Shapiro:

Neil Chapman of Varian Instruments mentioned your NMR News letter. We have been working with some NMR equipment on enamel and bone studies for the last three years, through the kindness of the Varian Co. and Joelco of Tokyo.

Recently we had turned over to our use a Varian V-16. Since we need to do  $H^1$  and  $F^{19}$  work, and it doesn't have a capacity above 13,600 G, we need a 40 mc, rf unit and probe. Since these have mostly been replaced by 60 mc systems, it is possible some group may have one tucked away in the storeroom (or attic!). We haven't heard of one yet, but hoped a help needed in the newsletter might uncover one for us. Since we're also cashless (or almost), if one is found and they're not using it, they might even lend it to us!

Ever optimistic,

*M. F. Little*

Marguerite F. Little  
Head, Biochemistry Department

MFL/mag

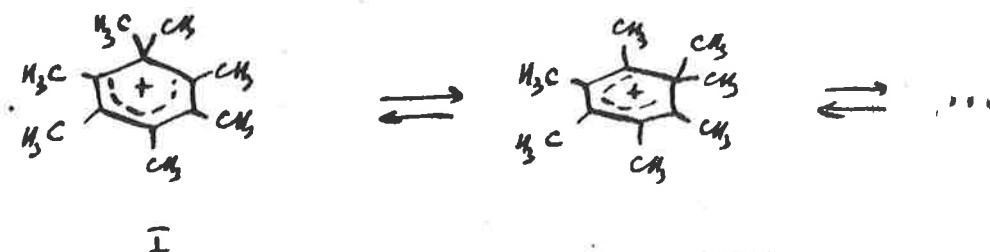
Institute of Organic Chemistry  
Siberian Division of the Academy of Sciences  
90, Novosibirsk  
U S S R

March 11, 1966

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

Dear Professor Shapiro:

We have recently found all the methyl n.m.r. peaks of heptamethylbenzenonium ion (HMB ion, I) /1/ to collapse to a single line at elevated temperatures. This has been explained by the rapid intramolecular migration of methyl group in I /2/.



As our first contribution to the IIT <sup>(NMR)</sup>Newsletter we would like to report n.m.r. measurements of the rate of this rearrangement.

The n.m.r. spectra of HMB ion in  $\text{H O}_3\text{Cl}$  have been investigated at twenty temperatures ranging from  $-10$  to  $+113^\circ$ . The rates of the rearrangement via 1,2-shifts of a methyl group have been obtained

by comparison of the observed spectra with calculated ones (Fig.1).  
By applying the method of least-squares to all the data we have  
obtained the following result:

$$k_1 = \frac{1}{\tau} = 10^{13,5 \pm 0,4} \exp \left[ -(18200 \pm 600)/RT \right] \text{sec}^{-1}$$

where  $\tau$  is the mean lifetime of the fixed structure of HMB ion.

Sincerely yours,

V.A. Koptug

V.G. Shubin

A.I. Rezvukhin

D.V. Korchagina

V.P. Tretyakov

E.S. Rudakov

- /1/ W. von E. Doering, M. Saunders, H.G. Boyton, H.W. Earhart,  
E.F. Wadley, W.R. Edwards and G. Laber, Tetrahedron, 4, 178 (1958).  
/2/ V.A. Koptug, V.G. Shubin, A.I. Rezvukhin, Izv. Akad. Nauk  
S.S.S.R., Ser. Khim., 1965, 201.

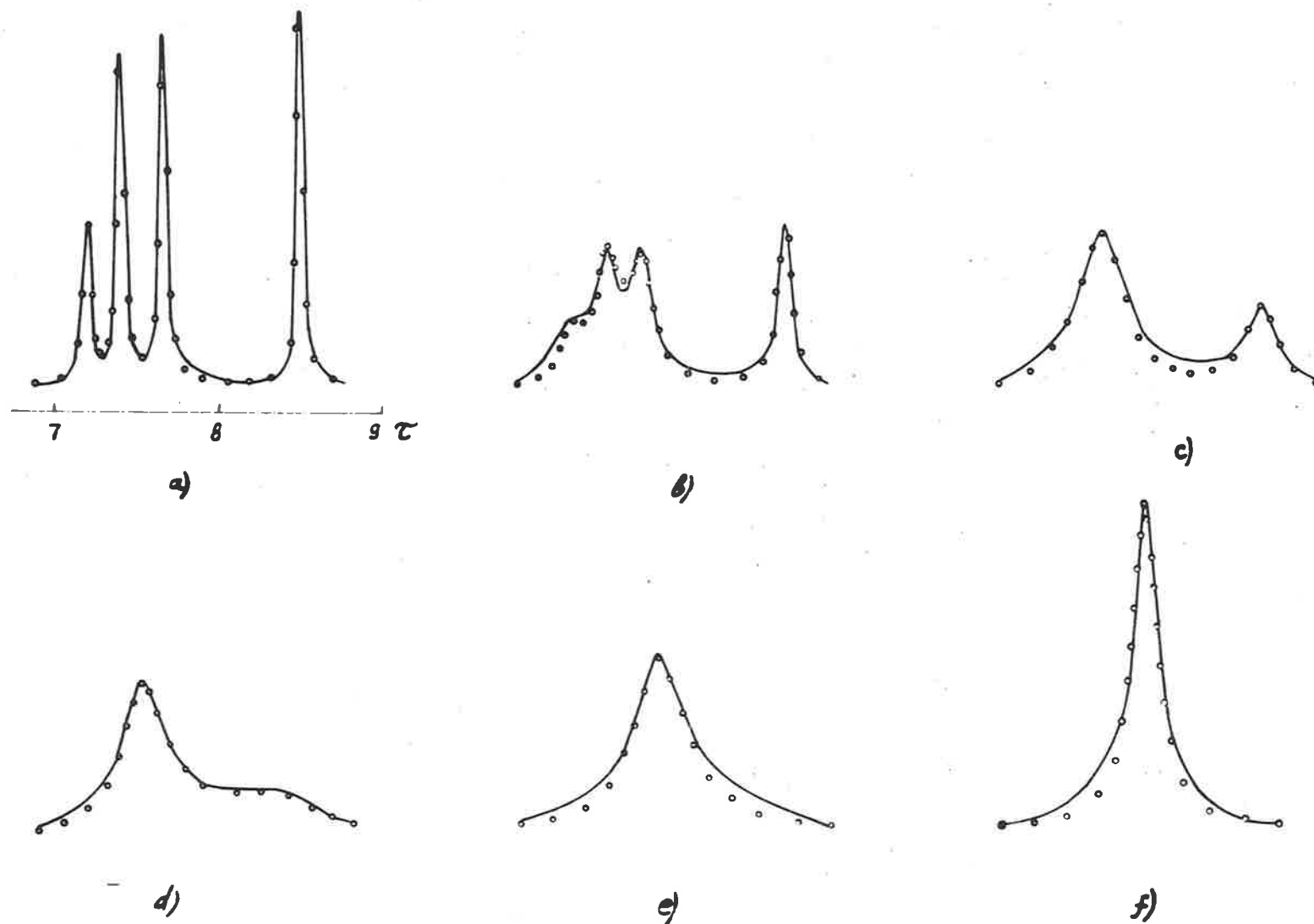


Fig.1. N.m.r. spectra (40 Mc/s) of HMB ion in  $\text{HSO}_3\text{Cl}$ . The solid lines represent the obtained spectra and the points, the calculated. a)  $t=-10^\circ$ ,  $\tau=10^2 \text{ sec}$ . b)  $t=49^\circ$ ,  $\tau=8,0 \cdot 10^{-2} \text{ sec}$ . c)  $t=68^\circ$ ,  $\tau=2,2 \cdot 10^{-2} \text{ sec}$ . d)  $t=77^\circ$ ,  $\tau=5,4 \cdot 10^{-3} \text{ sec}$ . e)  $t=96^\circ$ ,  $\tau=1,7 \cdot 10^{-3} \text{ sec}$ . f)  $t=113^\circ$ ,  $\tau=7,6 \cdot 10^{-4} \text{ sec}$ .

## TATA INSTITUTE OF FUNDAMENTAL RESEARCH

*National Centre of the Government of India for Nuclear Science and Mathematics*

Telegrams: ZETESIS

COLABA, BOMBAY 5

Telephone: 213141

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

Dear Dr. Shapiro,

We have studied the following problems.

1.  $F^{19}$  resonance in  $UF_4$  and in Rare-earth trifluorides.
2.  $Pt^{195}$  resonance in Rare-earth platinum alloys.
3. In and Bi resonances in In - Bi alloys.

Briefly, the results are that  $F^{19}$  shift in the first half of R.E. $F_3$  compounds is positive and in the second half negative. There is evidence for diffusion in lighter rare earths and inequivalence for fluorine sites. Pt resonance in RE  $Pt_5$ , gives two lines, one of them, in some alloys, is negative and the other positive.

All the results are being sent for publication.

Yours sincerely,

R. Vijayaraghavan  
R. Vijayaraghavan.

UNIVERSITÀ DI PAVIA

ISTITUTO DI FISICA "A. VOLTA"

TELEF. 04.241/2/3/4

GRUPPO SPETTROSCOPIA A RADIOFREQUENZA

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

Pavia, 14th June, 1966

We submit to the attention of the readers the system we use to synchronize the radiofrequency oscillator of the VF-16 Varian Associates NMR spectrometer. The synchronization has as a consequence a far greater frequency stability and hence an improvement of performances.

We use as oscillation source the output of the Hewlett & Packard Mod. 5245L electronic counter. This instrument provides some standard output frequencies among which there are 10 MHz and 1 MHz. The 10 MHz output can be directly used to synchronize the Varian oscillator at this frequency. The 1MHz output, owing to the content of harmonics, can be used to synchronize directly the oscillator at 2,3 and 4 MHz. For higher harmonics of 1 MHz it is necessary to use a little two-stage transistorized amplifier tuned at the selected harmonic. A schematic of such amplifier is enclosed.

The frequency stability achieved in this way, is that of the electronic counter internal standard i.e. 3 parts in  $10^9$  per 24 hours.

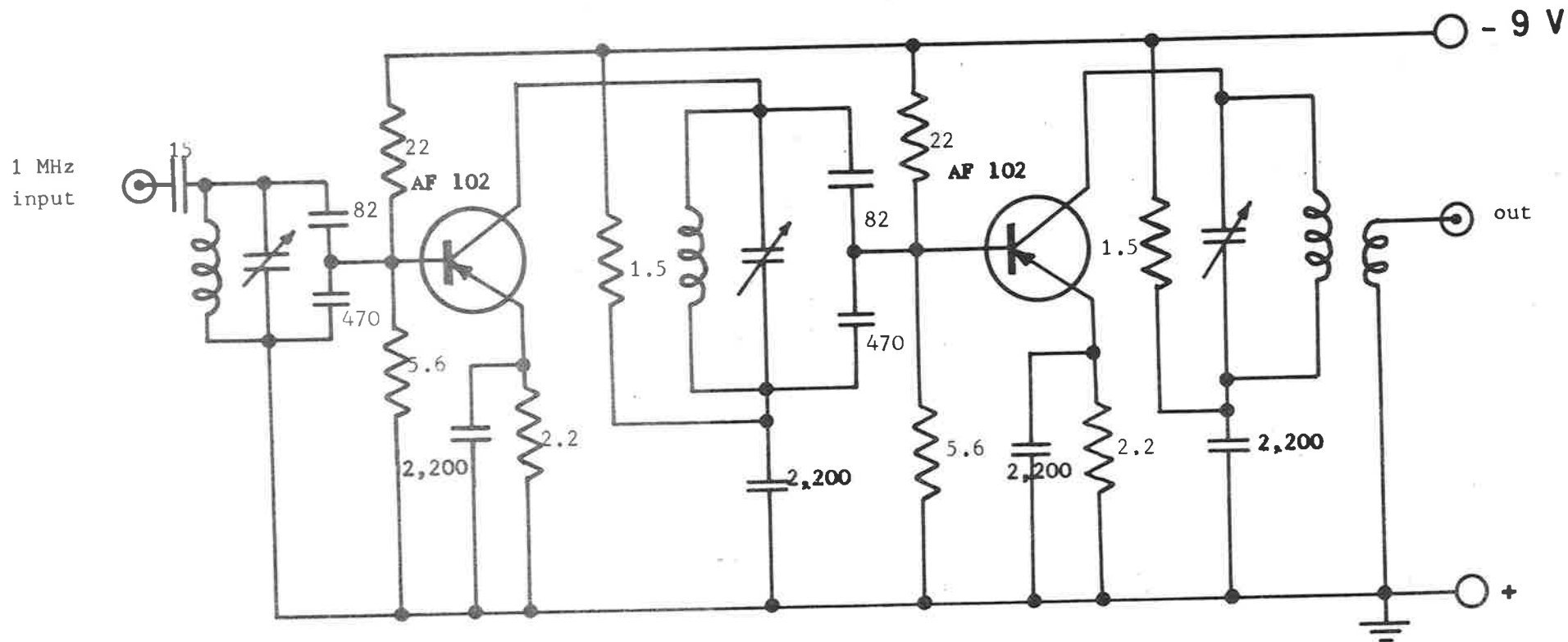
We hope this short note entitles us to be placed on your mailing list.

Sincerely yours

G. Lanzi  
G. Lanzi

E. R. Mognaschi  
E.R. Mognaschi





ALL RESISTOR VALUES ARE IN K-OHMS AND  $\frac{1}{2}$  WATT  
 ALL CAPACITOR VALUES ARE IN  $\mu\mu\text{F}$

## PRINCETON UNIVERSITY

DEPARTMENT OF CHEMISTRY  
PRINCETON, NEW JERSEY 08540

Frick Chemical Laboratory

June 23, 1966

Professor B. L. Shapiro  
Department of Chemistry  
Illinois Institute of Technology  
Chicago, Illinois 60616Nomenclature for Stereochemically Nonequivalent Nuclei

Dear Professor Shapiro:

In an earlier Newsletter (MELLONMR 57-27, June 1963), we had proposed the terminology "diastereomeric protons" and "enantiomeric protons" with reference to nuclei which reside in diastereomeric and enantiomeric environments, respectively; these terms were discussed in a subsequent publication (K. Mislow, M. A. W. Glass, H. B. Hopps, E. Simon and G. H. Wahl, Jr., J. Am. Chem. Soc., 86, 1710 (1964)) and more fully in our text (K. Mislow, "Introduction to Stereochemistry", W. A. Benjamin, 1965). However, the spatial relationship of atoms and groups of atoms are properly the relationships of their environments, and the above terminology, though clear in context, does not in itself distinctly convey the intended attribute. After consideration of suitable alternative adjectives such as "diastereoscopic" and "enantioscopic" (suggested to us at the Euchem. Conference on Stereochemistry at Bürgenstock, May, 1966), and "diastereoperistatic" and "enantiooperistatic" (suggested to us by Professor S. D. Atkins, Department of Classics, Princeton University), we have adopted "diastereotopic" and "enantiotopic" as the most apt and concise terms to convey our meaning. The terms "diastereomeric" and "enantiomeric" will be retained to describe groups which are inherently stereoisomeric, i.e. even when removed from their environments.

A convenient classification which is based on symmetry rather than observational properties is the following:

1. Equivalent atoms or groups of atoms: those which reside in stereochemically indistinguishable environments and whose positions can be interchanged by a symmetry operation of the first kind ("performable"), i.e. simple rotation around an axis of rotation  $C_n$  ( $n > 1$ ). Example: the protons in methylene fluoride. Molecules belonging to point groups  $C_{\infty v}$ ,  $C_1$ ,  $C_s$  and  $C_1$  cannot contain equivalent (as here defined) atoms or groups.
2. Enantiotopic atoms or groups of atoms: Those which reside in enantiomeric environments and whose positions can be interchanged only by a symmetry operation of the second kind ("non-performable"), i.e. rotation-reflection around an alternating axis  $S_n$ . Example: the protons in fluorochloromethane. Molecules belonging to the linear ( $C_{\infty v}$ ,  $D_{\infty h}$ ) or chiral ( $C_n$ ,  $D_n$ ,  $T$ ,  $O$ ,  $I$ ) point groups cannot contain enantiotopic atoms or groups.

Professor B. L. Shapiro

-2-

June 23, 1966

3. Diastereotopic atoms or groups of atoms: those which reside in stereoisomeric (diastereomeric) environments and whose positions cannot be interchanged by any symmetry operation. Example: the geminal methylene protons in glycerol (an achiral molecule) or in 2-butanol (a chiral molecule).

Equivalent and enantiotopic nuclei must be isochronous, i.e. have identical chemical shifts, in achiral media; in chiral media enantiotopic, but not equivalent, nuclei are anisochronous. For a recent demonstration of this effect, see W. H. Pirkle, J. Am. Chem. Soc., 88, 1837 (1966); the enantiotopic fluorine atoms in this case resided in enantiomeric molecules. Incidentally, we support the suggestion by G. Binsch (IITNMRN 87-32) that the term "isochronous" (introduced by A. Abragam, "The Principles of Nuclear Magnetism", Oxford, 1961, p. 480) and "anisochronous" be generally adopted.

Diastereotopic nuclei are always anisochronous. Note that the chemical shift difference between diastereotopic protons is sometimes not large enough to be observed. Such nuclei are nevertheless anisochronous since the chemical shift nonequivalence must still in principle exist, and one should in such cases speak of apparent chemical shift equivalence since a change in the conditions of measurement (change of magnetic field, solvent, etc.) may yet reveal the nonequivalence. Just such a case (doubly bridged biphenyls) was discussed in our 1963 letter.


We emphasize that in the foregoing analysis the time-scale of measurement must be kept firmly in mind at all times. Thus, whereas the fluorine atoms in 1,1-difluorocyclohexane are diastereotopic on the n.m.r. time scale at low temperatures and hence anisochronous, they are equivalent above room temperature and hence isochronous.

Although nuclei in spin systems in which spin coupling nonequivalence is observed have been termed "magnetically nonequivalent" or "magnetically nonequivalent in the spin coupling sense", this terminology may be misleading. Nuclei such as the protons in 1,1-difluoroethene are stereochemically equivalent and must show equivalence in all of their properties. It is the geometries of their interactions which are nonequivalent, and from which the nonequivalence of their coupling constants arises, as can be recognized by replacing one of the fluorine atoms by a chlorine atom: the two protons now are diastereotopic. Such a "substitution criterion" serves to distinguish spin systems where diastereomeric interactions are present and where spin coupling nonequivalence may occur.

These concepts will be more fully discussed as part of a paper on "Stereoisomeric Relationships of Groups in Molecules". We thank the conferees of the Bürgenstock Conference, in particular Professors V. Prelog, D. Arigoni and P. Laszlo, and Dr. G. Binsch for valuable and stimulating discussions.

Sincerely yours,

  
Kurt Mislow

  
Morton Raban

  
Paul Bickart

CYANAMID

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

June 2, 1966

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

Dear Barry:

Dr. Arnold Zweig of our laboratories has prepared a number of methoxy-, dimethylamino-, and methylthio-substituted polycyclic aromatics. We have examined the proton spectra of 5 mole % solutions in  $\text{CCl}_4$  and have made assignments where possible. All of the data obtained are listed in the accompanying table, which, together with some discussion of the relation of the shifts observed to factors such as  $\pi$ -electron distribution, ring currents, "peri" effect, etc., is being submitted shortly for publication.

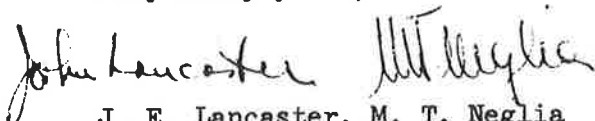
If position 1 of the naphthalene ring carries one of the three groups mentioned above, the proton at position 8 is always considerably shifted to lower field. This "peri" effect, noted earlier by Dudek (Spectrochimica Acta, 19, 691 (63)) is observed in eleven of the compounds listed. The effect is about the same for  $-\text{OCH}_3$  and  $-\text{N}(\text{CH}_3)_2$ , but larger for  $-\text{SCH}_3$ . In 9,10-bis(methylthio)anthracene, where the  $-\text{SCH}_3$  is presumably constrained to lie out of the plane of the rings, the effect is largest of all.

Protons ortho and para to  $-\text{OCH}_3$  and  $-\text{N}(\text{CH}_3)_2$  groups are usually shifted to higher field, and one can see the superposition of these effects with the "peri" effect on  $\text{H}_8$  in the 1,5 and 1,7 disubstituted naphthalenes (four in all), which results in partial cancelling of the "peri" effect.

Comparison of the  $\text{H}_3$  resonance position in 2,6 and 2,7 disubstituted naphthalenes shows that it is sensitive to the position of the  $\text{OCH}_3$  on the second ring, and can be explained by the existence of greater inter-ring resonance interaction in one case than the other.

I hope this will renew our subscription to IITNMR in good order.

Very truly yours,



J. E. Lancaster, M. T. Neglia  
 Nuclear Magnetic Resonance Group  
 Research Service Department

amb  
 Att.

Proton Chemical Shifts of Donor  
Substituted Polycyclic Aromatic Hydrocarbons  
 (τ-values)

	<u>Methyl Resonance</u>	<u>Ring Position</u>	<u>Ring Proton Resonance</u>
1-Methoxynaphthalene	6.04	2 8	3.30 1.80
2-Methoxynaphthalene	6.14	1	3.00 <sup>a</sup>
1,3-Dimethoxynaphthalene	6.07 6.16	2 4 8	3.61 3.40 1.91
1,4-Dimethoxynaphthalene	6.11	2,3 5,8 6,7	3.47 1.87 2.60
1,5-Dimethoxynaphthalene	6.03	2,6 3,7 4,8	3.27 2.73 2.24
1,6-Dimethoxynaphthalene	6.08 6.15	2 8 ?	3.46 1.92 2.83 <sup>a</sup>
1,7-Dimethoxynaphthalene	6.05 6.10	5 <sup>a</sup> a a	3.05 <sup>a</sup> 3.33 2.40
1,8-Dimethoxynaphthalene	6.11	2,7 3,6 4,5	3.28 2.78 2.78
2,3-Dimethoxynaphthalene	6.11	1,4 5,8 6,7	3.06 2.43 2.79
2,6-Dimethoxynaphthalene	6.15	1,5 3,7 4,8	3.04 2.96 2.47
2,7-Dimethoxynaphthalene	6.14	1,8 3,6 4,5	3.05 3.13 2.45
1,4,5,8-Tetramethoxynaphthalene	6.21	2,3,6,7	3.28
1-Methylthionaphthalene	7.52	8	1.72
2-Methylthionaphthalene	7.50	1,3-8	2.2-2.9 <sup>b</sup>
1,4-bis-(Methylthio)naphthalene	7.53	2,3 5,8 6,7	2.71 1.71 2.50

(continued)

-2-

(Table Continued)

	<u>Methyl Resonance</u>	<u>Ring Position</u>	<u>Ring Proton Resonance</u>
1,5-bis-(Methylthio)naphthalene	7.52	2,6 3,7 4,8	~2.61 ~2.61 1.88
1,8-bis(Methylthio)naphthalene	7.56	2-7	2.3-2.9 <sup>b</sup>
2,3-bis(Methylthio)naphthalene	7.53	1,4 5,8 6,7	2.52 2.42 2.71
2,6-bis(Methylthio)naphthalene	7.48	1,5 3,7 4,8	2.52 2.75 2.48
2,7-bis-(Methylthio)naphthalene	7.52	1,8 3,6 4,5	2.62 2.82 2.46
1-Dimethylaminonaphthalene	7.17	2 8 a	3.10 1.88 2.33
2-Dimethylaminonaphthalene	7.07	1 3	3.23 3.02
1,5-bis(Dimethylamino)naphthalene	7.17	2,6 3,7 4,8	3.06 2.71 2.12
2,6-bis(Dimethylamino)naphthalene	7.10	1,5 3,7 4,8	3.28 3.06 2.60
2,7-bis(Dimethylamino)naphthalene	7.06	1,8 3,6 4,5	3.31 3.22 2.55
9,10-Dimethoxyanthracene	6.01	1,4,5,8 2,3,6,7	1.79 2.63
9,10-bis(Methylthio)anthracene	7.64	1,4,5,8 2,3,6,7	1.02 2.47
1,6-bis(Dimethylamino)pyrene	7.03	2,7 3,8 5,10 4,9	2.40 2.07 1.75 2.16

a. Assignment not certain

b. All ring H's in these limits



# THE UNIVERSITY OF ASTON IN BIRMINGHAM

GOSTA GREEN . BIRMINGHAM 4.

TELEPHONE 021-359 3611

The Department of Chemistry.

Head of Department: Professor W. G. Parker, Ph.D., F.R.I.C., A.F.R.Ae.S.

Our Ref JH/DBB/CHEM

Your Ref

Telephone Ext.

7th June, 1966

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

Dear Professor Shapiro,

We apologise for failing to contribute to the I.I.T.N-M-R newsletter in accordance with your schedule for subscriptions and hope that this contribution will reinstate us to your mailing list.

Based on various ideas reported previously in I.I.T.N-M-R we have developed what we consider to be a versatile 'mouse-type' system for the Perkin-Elmer R10 spectrometer. It is a plug-in unit consisting of a series of timer operated relays which allow successively (i) integral collection (C1) (ii) reading of the integrated signal (C2) (iii) field scan and synchronised chart movement (C3) and (iv) integral cancellation (C4). The unit can be operated in a variety of ways but in the stepped scan spectral point integration mode the pen remains on the chart continuously and records the accumulated signal by moving to a new position only during operation (ii).

The four relays are incorporated in separate commercial timing units (Venner Electronics Ltd. Timing Units Type ETDR with fast reset time of 0.1 secs) connected in a loop which after initiation continues in sequence operation until interrupted. The device is plugged in permanently and the switches S1 to S4 allow the various stages to be by-passed either individually or completely so increasing the versatility of the system.

Figure 1 is the schematic circuit in which all manual switches are shown in their positions for point integration and the timer relays shown in their normal positions. Figure 2(b)

Continued.....

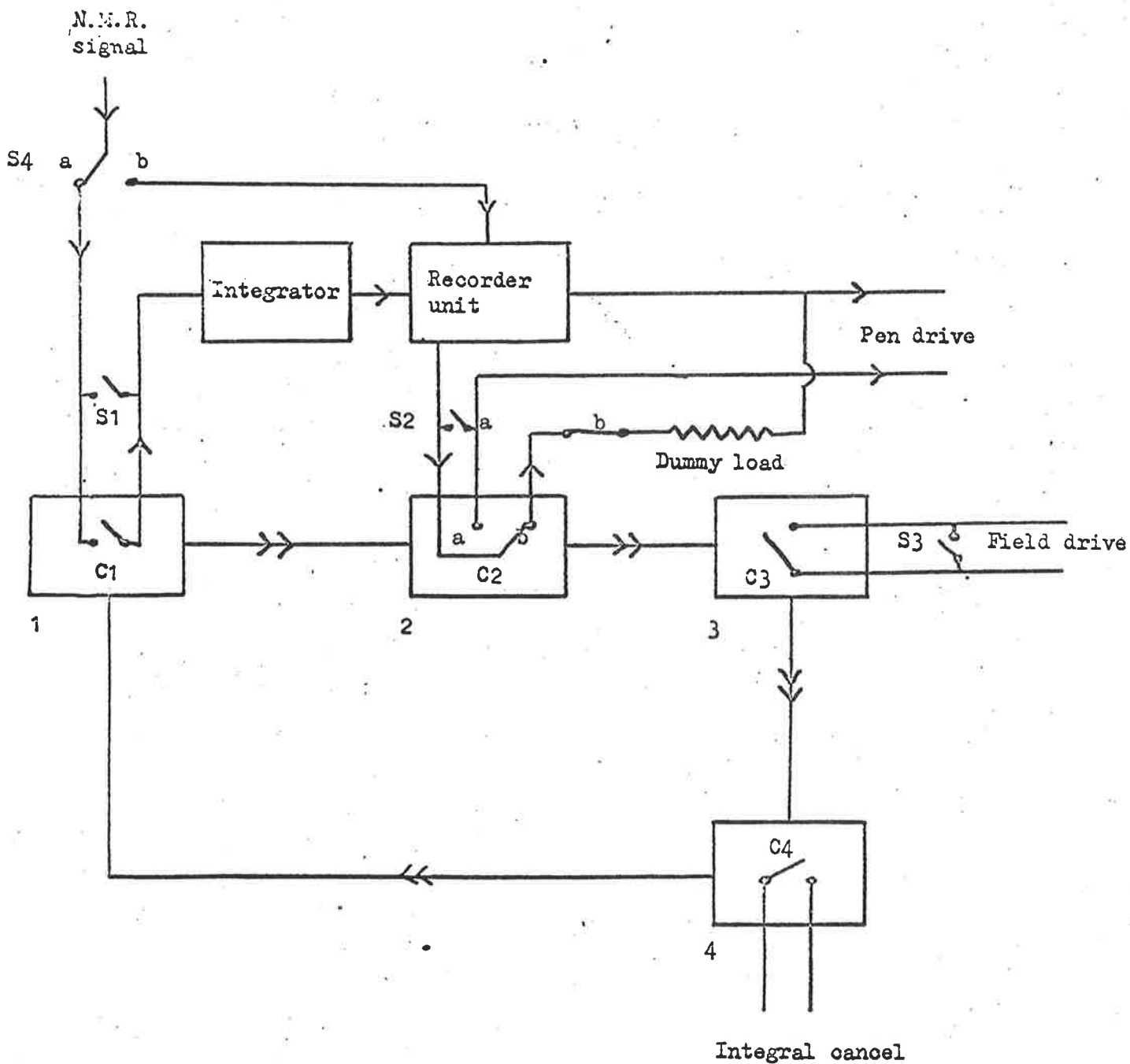


FIG. 1.



Professor B.L. Shapiro.7th June, 1966

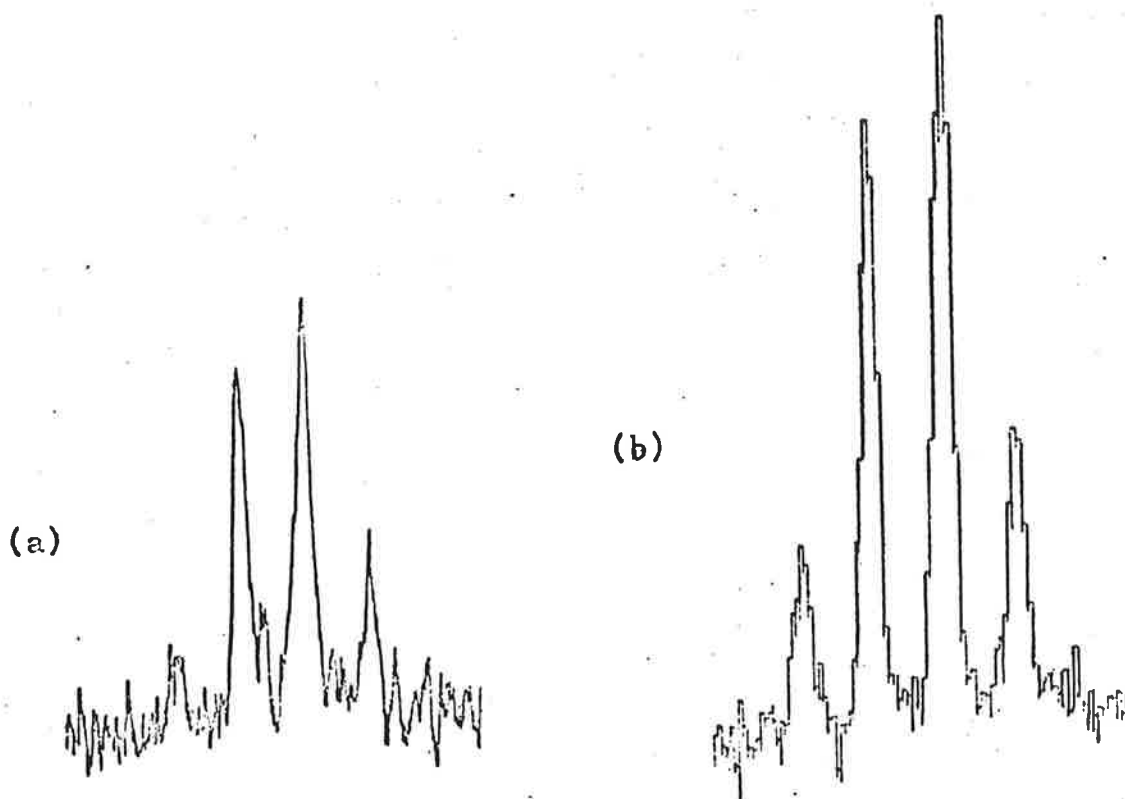
demonstrates the sort of S/N improvement that can be obtained relative to 2(a) which was obtained using normal spectrometer operation.

Yours sincerely,

*John Homer*

*P. J. Huck*

John Homer and P. J. Huck.



Methylene proton resonance of 1% ethyl benzene in  $\text{CCl}_4$

FIG. 2

## SIMON FRASER UNIVERSITY

DEPARTMENT OF CHEMISTRY



BURNABY 2, BRITISH COLUMBIA

Telephone 291-3111 Area code 604

24 June 1966

Professor B.L. Shapiro  
 Department of Chemistry  
 Illinois Institute of Technology  
 Technology Centre  
 Chicago, Illinois 60615

Dear Barry,

High Resolution Rotary Z-Echoes

Thank you for your reminder. I hope the following will put us back in the blue for a little while.

Ray Freeman has pointed out that Solomon's rotary echo experiment regains the selectivity lost in the normal high power pulse experiment. However Solomon's method suffers two disadvantages:

a) the  $H_1$  amplitude may change in the phase switching process, and b) errors in the phase switching angle give rise to cumulative errors in the refocussed magnetization in the rotating frame.

A simple alternative scheme for refocussing the magnetization which avoids these limitations is to sit at resonance and gate  $H_1$  on. The magnetization precesses about  $H_1$  in the  $yz$  plane and dephases in the inhomogeneous  $H_1$  field. At later times  $\tau, 3\tau, 5\tau, \dots$  we swing the magnetization through  $180^\circ$  in the rotating frame by a succession of positive or negative pulses applied to the dc  $H_0$  field. The magnetization refocusses along the  $z$  axis at  $2\tau, 4\tau, 6\tau, \dots$  and the maximum signal is observed at  $\{2n\tau + \pi/(2\gamma H_1)\}$  when the magnetization again dips through the  $xy$  plane.  $H_1$  is constant in the rotating frame, and the error blow-up due to inaccuracies in the pulse widths is not as severe as in Solomon's experiment. The magnetization trajectory is sketched in Fig. 1.

Fig. 2 shows the rotary Z-echoes obtained by this scheme for degassed benzene on our modified Varian A56/60.  $H_1$  was about  $5 \times 10^{-4}$  gauss (attenuated output full up) corresponding to ca.  $10^{-3}$  of the earth's field, and a

..2../

- 2 -

Professor B.L. Shapiro  
Chicago, Illinois 60615  
24 June 1966

precession frequency of 2 Hz, which can be clearly seen. The pulse repetition time  $2\tau$  was 2.5 secs and the pulse width  $15 \mu\text{s}$ , corresponding to a pulse off-resonance by about 8 gauss or 33 kHz. The time scale is 5 secs/division.

It is our experience that for the A56/60, the coupling between the fields at the receiver sample and the control sample is loose enough that  $180^\circ$  pulses can be applied to the receiver sample up to a repetition frequency of 100 Hz before the frequency lock is tripped.

With best wishes,

*K. H. Abramson*

K.H. Abramson

*E.J. Wells*

E.J. Wells

EJW/mln

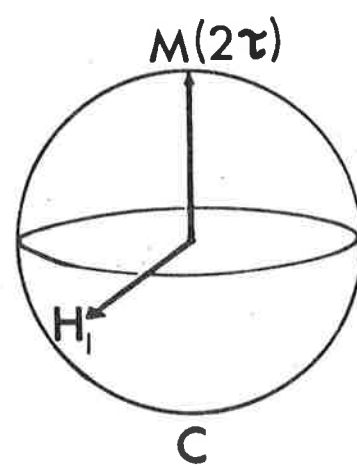
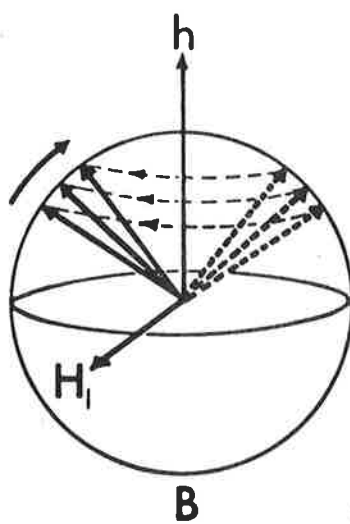
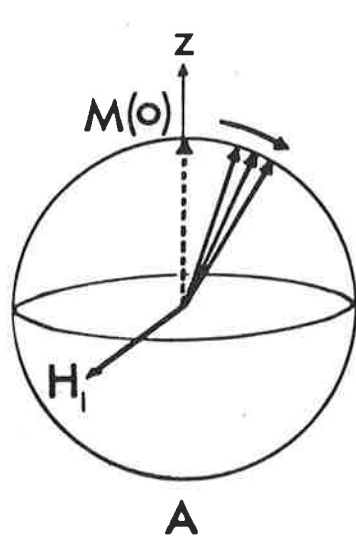
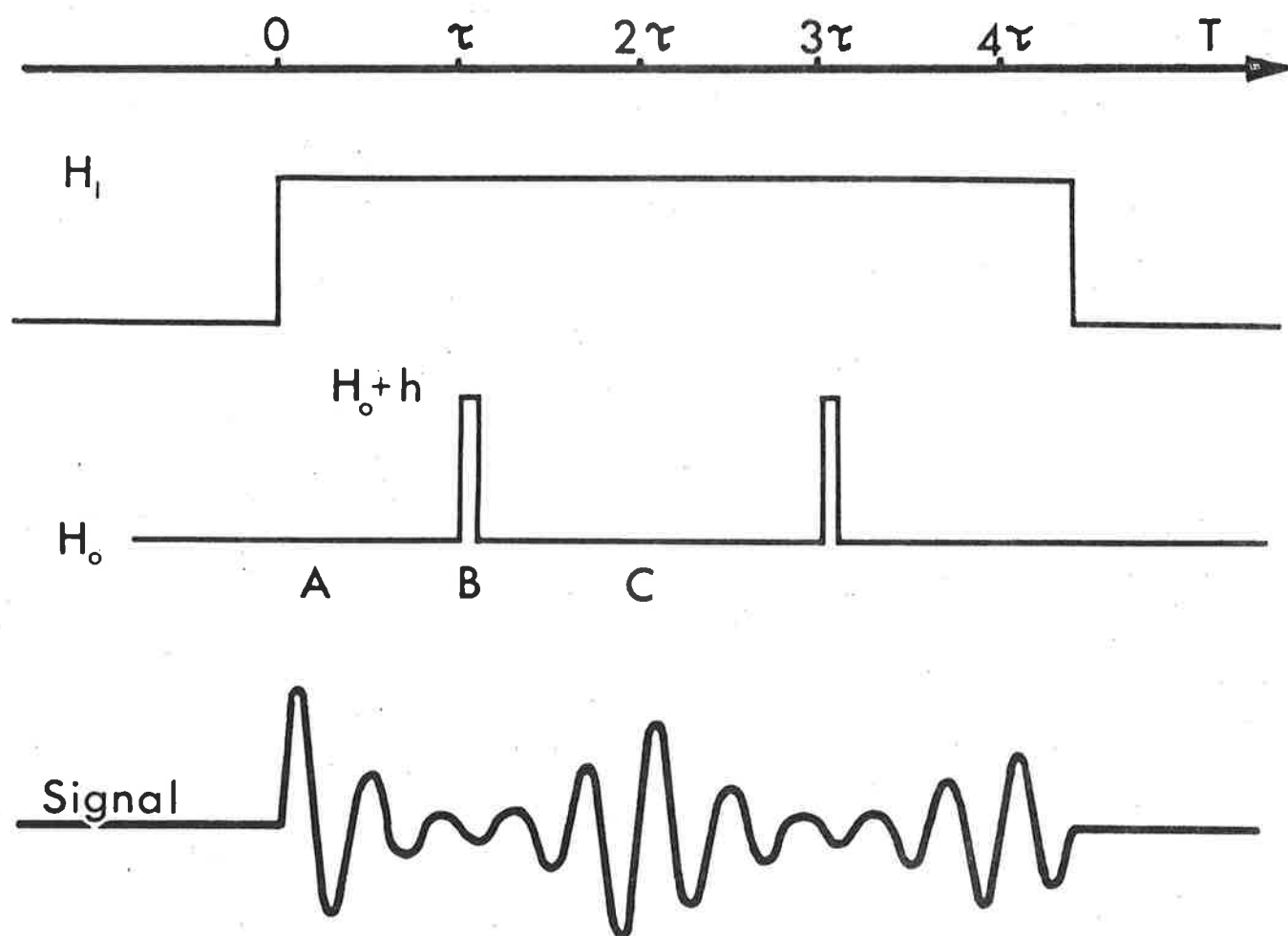


Fig 1

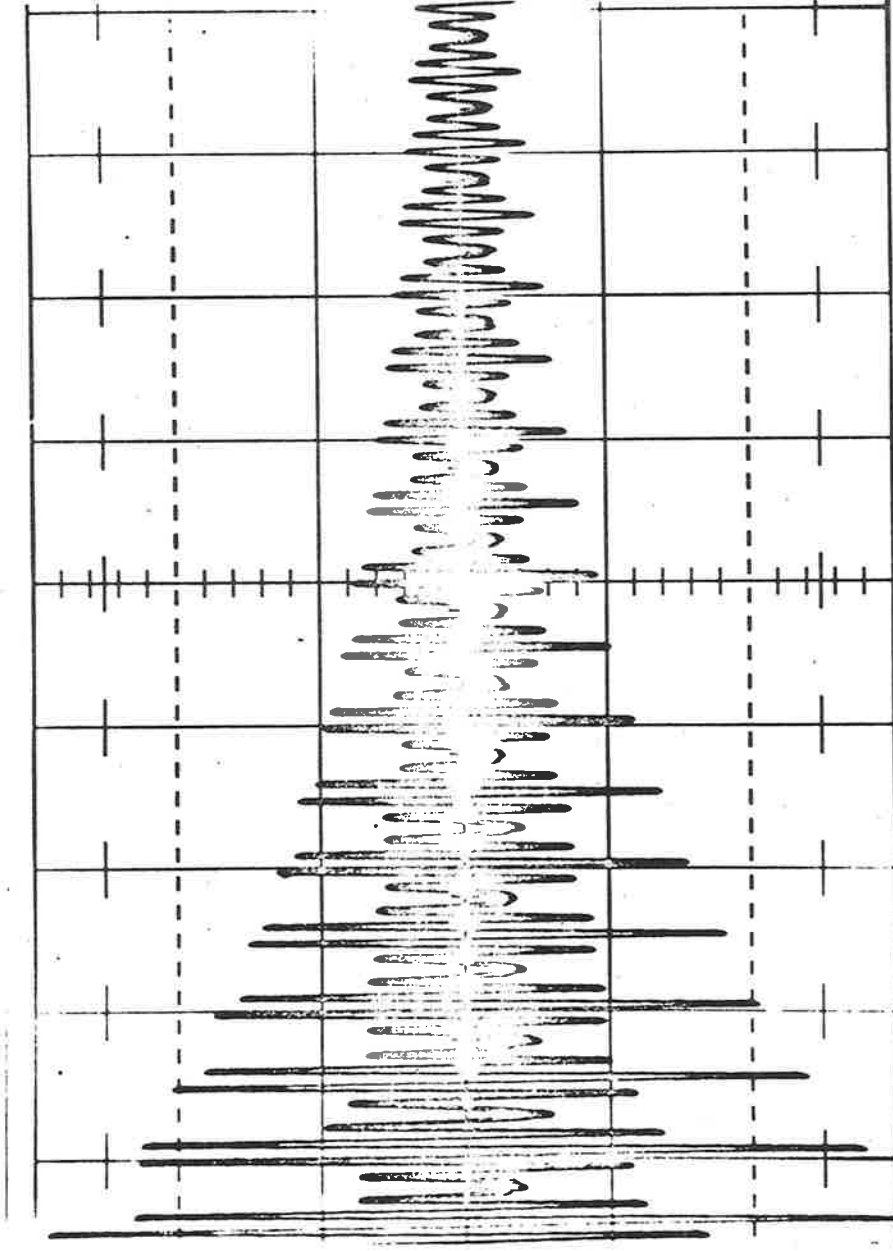


Fig 2.

DEPARTMENT OF ORGANIC CHEMISTRY  
THE UNIVERSITY

Address: BLOEMSIJNGEL 10, GRONINGEN (HOLLAND)  
Tel.: 05900-34841

HEAD: PROF. DR. H. WYNBERG

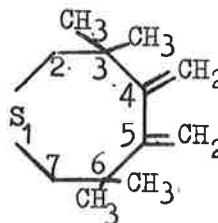
Groningen, June 15, 1966

Dr. B.L. Shapiro  
Dept. of Chemistry  
Illinois Institute of Technology  
Chicago, Ill. 60616  
U.S.A.

Dear Sir:

Title: Slow inversion rate of a sulphur containing 7-ring.

The A-60 proton magnetic resonance spectrum of 1-thia-4,5-dimethylene-3,3,6,6-tetramethylcycloheptane<sup>1)</sup> showed at room temperature (probe temperature about 37°C) the following absorptions: two singlets for the methyl groups, an AB multiplet for the 2 and 7 methylene groups and an AB multiplet for the exocyclic methylene groups. When the sample was warmed up, the two singlets collapsed at 92°C and the AB multiplet for the 2 and 7 methylene groups did the same at 112°C. Since this molecule gives two coalescence temperatures, it is very simple to calculate the flipping barrier. This turned out to be 8,3 kcal/mole.



We feel that it is possible with this system to test the various methods described in the literature for determination of rotation barrier energies, because here we have two temperature dependent absorptions; each can give a value for the energy. Subsequent work on this system and others will be done.

1) prepared by drs. Ae. de Groot and B. Evenhuis.

Yours truly,

S. van der Werf

## UNIVERSITY OF CALIFORNIA, IRVINE

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



SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY

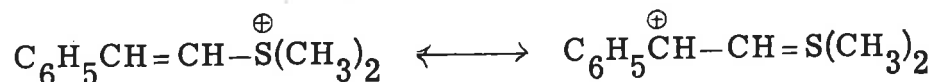
IRVINE, CALIFORNIA 92650

June 28, 1966

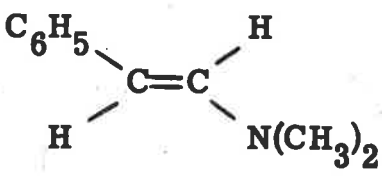
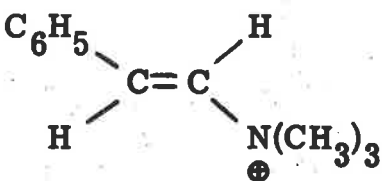
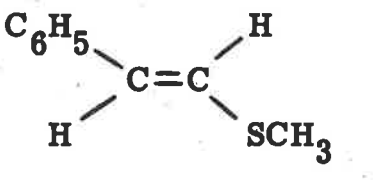
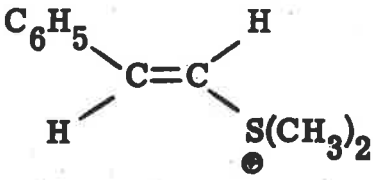
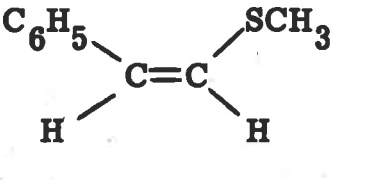
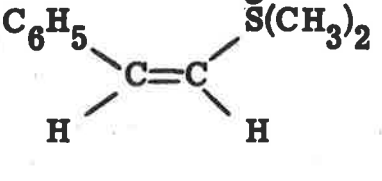
Dr. B. L. Shapiro  
 Department of Chemistry  
 Illinois Institute of Technology  
 Chicago, Illinois 60616

Dear Dr. Shapiro:

In a previous letter (No. 85 IITNMR) we described certain aspects of the n.m.r. spectra of cis and trans methyl styryl sulfides and dimethylstyrylsulfonium salts. The striking feature of these spectra is the larger chemical shift difference between the vinyl protons of the sulfonium salts relative to the sulfides. This is mostly associated with the low field resonance of the vinyl proton beta to the sulfonium substituent, and we attribute this partly to electronegativity effects and partly to d-orbital resonance in the sulfonium salts, which decreases the electron density at the  $\beta$ -carbon and causes a paramagnetic shift.

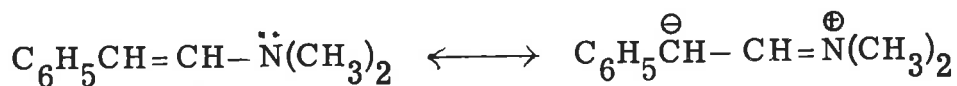


We have now observed the n.m.r. spectra of the analogous nitrogen compounds, trans-dimethylstyrylamine  $\text{C}_6\text{H}_5\text{CH}=\text{CHN}(\text{CH}_3)_2$ , and trans-trimethylstyrylammonium fluoborate  $\text{C}_6\text{H}_5\text{CH}=\text{CHN}(\text{CH}_3)_3^+\text{BF}_4^-$ . The pertinent chemical shift data are tabulated below along with the data for the sulfur compounds.

Compound	Solvent	Chemical Shift				$J_{HH}$ , cps
		$H_\alpha$ ppm	$H_\beta$ ppm	$(H_\beta - H_\alpha)$ cps	$CH_3$ ppm	
	$CDCl_3$	6.65	5.11	92.4	2.63	13.8
	$CH_3NO_2$	6.96 (7.22)	7.22 (6.96)	15	3.48	14.5
	neat	6.68	6.20	29	2.04	16.0
	$CH_2Cl_2$	6.95	7.65	42	3.02	15.0
	neat	5.98	6.30	19	2.01	11.0
	$CH_2Cl_2$	6.45	7.63	71	3.00	9.5



While we are not yet sure of the chemical shift assignment for the  $\alpha$  and  $\beta$  vinyl protons of the enammonium salt, it is quite apparent that the  $\beta$ -proton is some 1.8 - 2.1 ppm to lower fields than the  $\beta$ -vinyl proton of the enamine. This shift can hardly be attributed to d-orbital resonance in the salt but rather reflects the effect of p-orbital resonance in the enamine producing a diamagnetic shift of the  $\beta$ -vinyl proton.



The vinyl protons of the enamine are chemically shifted some 92 cps whereas the corresponding cis and trans sulfides are shifted only 19 and 29 cps. In contrast, the vinyl protons of the enammonium salt are chemically shifted only 15 cps whereas the corresponding cis and trans sulfonium salts are shifted 71 and 42 cps. Assuming electronegativity effects are not widely different between the sulfur and nitrogen analogs, the data indicate that 2p - 2p  $\pi$ -overlap in the enamine is substantially more effective than is 2p - 3p  $\pi$ -overlap in the sulfides, and that 2p - 3d  $\pi$ -overlap is effective only in the sulfonium salts.

One further point of interest concerns the observation of long-range coupling of N-methyl and S-methyl protons with the  $\beta$ -vinyl protons of the enamine, sulfides and sulfonium salts. This coupling is observable as a broadening of the  $\beta$ -vinyl resonance and, in certain cases, as a splitting of the methyl resonance. Thus, the N-methyl resonance of the enamine is a doublet,  $J = 0.4$  cps; the S-methyl resonance of the cis-sulfonium salt is a doublet,  $J = 0.2$  cps, which reduces to a singlet in the deuterium compound,  $\text{C}_6\text{H}_5\text{CD}=\text{CHS}(\text{CH}_3)_2^{\oplus} \text{BF}_4^{\ominus}$ . The methyl resonances of the enammonium salt, cis and trans sulfides and trans sulfonium salt are, however, not measurably split. The coupling in these cases must be less than 0.2 cps.

Sincerely yours,

*Marjorie C. Caserio*

Marjorie C. Caserio  
Assistant Professor of Chemistry

*Robert E. Pratt* <sup>MCC</sup>

Robert E. Pratt  
Post-doctoral Fellow

Dr. Lee's Professor of Chemistry:  
R. E. Richards F.R.S.  
Tel: Oxford 57757  
STD Code OOX2

PHYSICAL CHEMISTRY LABORATORY,  
SOUTH PARKS ROAD,  
OXFORD.

25th June 1966

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

Dear Barry,

We are continuing our work on the nuclear-electron Overhauser effect in solutions of free radicals. The results from proton resonances are almost always fully consistent with simple dipole-dipole coupling modulated by random diffusion in the solution. Good agreement with simple diffusion theory is obtained with a wide variety of compounds. In the special case of a substance which is exchanging with the radical by proton exchange, such as a phenol and its phenoxide, some of the proton resonances are actually enhanced instead of reversed, but this is a very rare phenomenon. At 3,300 gauss we can easily obtain reverse nuclear resonances with improvements of signal to noise of 100 to 150 times and at 12,500 gauss resonances can be inverted and increased in intensity by 10 to 50 times.

By contrast, fluorine resonances are often enhanced rather than reversed. We have been able to show that this arises in most cases from scalar coupling between the radical and the fluorine nuclei of the solvent modulated by the random molecular motion. We have tried fitting the results to simple diffusion theory and to various types of sticking model but none of them seems to be entirely satisfactory. It is evident that a more sophisticated model would be required to give accurate fit to all the experimental measurements. The practical result is that for fluorine resonances some can be strongly inverted, some strongly enhanced, and many give only small effects because of a balance between the dipolar and scalar coupling.

We have been able to show that in dilute solutions of radical, the "three spin" effect can be important, in which the electrons pump one set of nuclei into inverted population, and these in turn pump

- 2 -

another set of nuclei into an enhanced population. We have published some experimental results in Chemical Communications to show this effect, and have a paper in press in which the full theory is given.

We have been investigating the effect on  $^{13}\text{C}$  resonances and have found that we can observe these resonances in natural abundance at 3.5 Mc and 3,300 gauss in many compounds. Once again, some of the resonances are inverted (for example benzene, cyclohexane, methanol, methyl formate, methyl cyanide, methyl iodide) but in other cases the carbon -  $^{13}$  resonance is positively enhanced (for example chloroform, bromoform, methylene chloride, methylene bromide, methylene iodide). We are following up the question of the variation of the scalar coupling in these resonances and also trying to use them to help us to solve some chemical problems.

We are also investigating phosphorous resonances at 12,500 gauss. These are often strongly enhanced and we have been able to obtain some beautiful spectra.

We have been using a northern scientific C.A.T. on our spin echo apparatus with some success. In a Carr-Purcell sequence we advance the C.A.T. at each echo so that we store the peak of each echo in one channel, and use only the number of channels which are needed. This makes optimum use of the C.A.T. and works extremely well. We have also successfully used the micro-sampler that comes with this C.A.T. to accumulate free induction decays of quite short duration.

Yours sincerely,

Rex

Short Title - Dynamic Nuclear Polarisation.

## NORTHWESTERN UNIVERSITY

EVANSTON, ILLINOIS 60201

DEPARTMENT OF CHEMISTRY

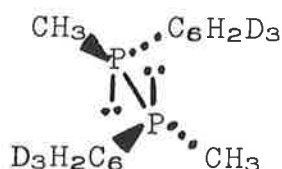
June 27, 1966

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

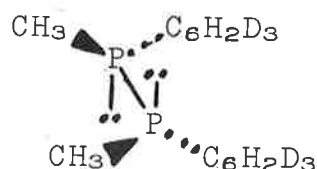
Dear Barry:

Configurational studies of nitrogenous compounds have occupied nmr spectroscopists for over ten years. The stability of phosphorus to inversion has prevented these methods from being applied to phosphorus compounds (in fact, optically active tertiary phosphines are known<sup>1</sup>). We have now observed that resonances of diphosphines exhibit reversible, temperature-dependent properties arising from phosphorus inversion. Apparently,  $p\pi-d\pi$  bonding between phosphorus atoms in diphosphines decreases the configurational stability with respect to monophosphines.<sup>2</sup>

1,2-Dimethyl-1,2-diphenyldiphosphine may exist in two diastereomeric forms, the meso (I) and the dl (II), each of which should produce separate and distinct resonances. Maier



I



II

has, indeed, reported that the  $^{31}\text{P}$  spectrum consists of two peaks.<sup>3</sup> To simplify analysis, we have synthesized the compound with deuterium in the ortho and para positions. The aromatic

Professor B. L. Shapiro

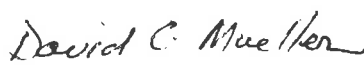
Page 2

June 27, 1966

resonances of the deuterated system consist at room temperature of two distinct peaks, which arise, respectively, from I and II (the methyl resonances are masked by solvent). The meso form (I) may be converted to the dl form (II) (and vice versa) by a single inversion about phosphorus. Between 130 and 180°, this process becomes fast on the nmr time scale, as judged by the broadening, coalescence, and sharpening of the meta resonances into a single peak. Analysis by the Gutowsky-Holm-Borčić method<sup>4</sup> gives an activation energy of 26+2 kcal/mole. The equilibrium constant at 32° (1.35) corresponds to a free-energy difference between diastereomers of 180 cal/mole. In a forthcoming Communication to the Journal of the American Chemical Society we will discuss the reasons for rejecting alternative hypotheses, such as equilibrating rotamers, temperature-dependent <sup>31</sup>P-H coupling constants, hindered rotation about the carbon-phosphorus bond, and dissociation-recombination.

Very truly yours,

  
Joseph B. Lambert

  
David C. Mueller

JBL/kc

1. L. Horner, et al., Tetrahedron Letters, No. 5, 161 (1961).
2. A. H. Cowley, Chem. Rev., 65, 617 (1965).
3. L. Maier, Ber., 94, 3043 (1961); J. Inorg. Nucl. Chem., 24, 275 (1962).
4. J. B. Lambert, W. L. Oliver, and J. D. Roberts, J. Am. Chem. Soc., 87, 5085 (1965).

Title: The Inversion of Diphosphines

Dr. H. Fritz  
c/o J.R. Geigy S.A.  
Basle 21 (Switzerland)

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

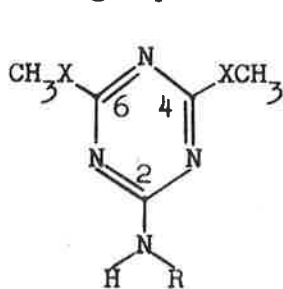
Chicago, Illinois 60616  
USA

June 30, 1966

Hindered rotation in trisubstituted (s)-triazines.

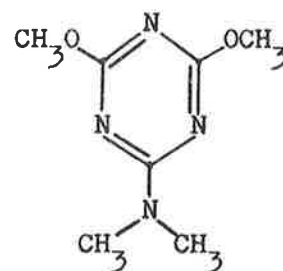
Dear Professor Shapiro,

We have examined the NMR spectra <sup>a)</sup> of several 2-monoalkylamino-4,6-OCH<sub>3</sub> (or SCH<sub>3</sub>) -(s)-triazines and found evidence of hindered rotation around the bond between carbon atom 2 and the nitrogen atom of the alkylaminogroup.



I a - f

	X	R
Ia	O	C <sub>2</sub> H <sub>5</sub>
b	O	CH(CH <sub>3</sub> ) <sub>2</sub>
c	O	C(CH <sub>3</sub> ) <sub>3</sub>
d	O	CH <sub>2</sub> CH <sub>2</sub> Cl
e	S	CH <sub>3</sub>
f	S	C <sub>2</sub> H <sub>5</sub>



II

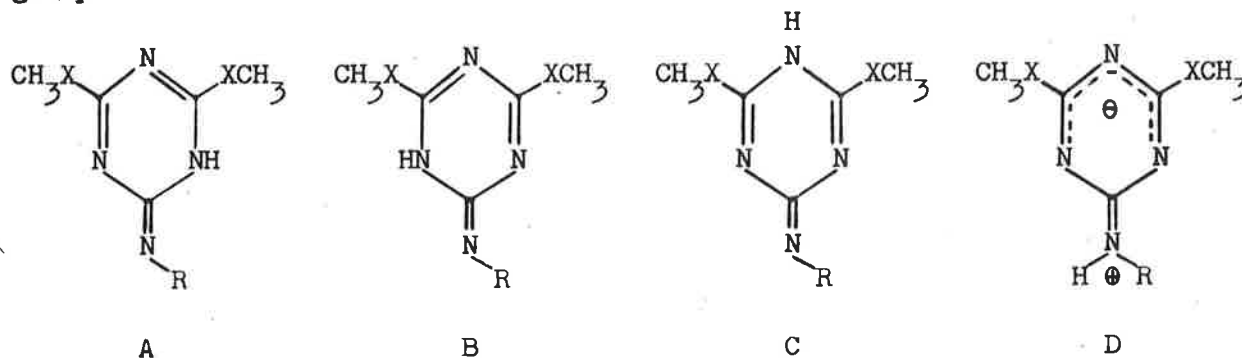
At normal temperature ( $\sim 30^{\circ}\text{C}$ ), the X-CH<sub>3</sub> resonances in I a - f are either broad or consist of two singlets with spacings of 2 - 4 cps (at 100 Mc). In contrast, II shows only one sharp line for the OCH<sub>3</sub> groups. All other CH signals in I a - f also have small linewidths. Results are given in the following table:

a) All spectra were measured on a Varian HA-100 spectrometer in CDCl<sub>3</sub> with TMS as internal standard. Concentrations were approximately 5 % (w/v).

Compound	$\tau(\text{XCH}_3)$ , ppm	$\Delta \nu$ ( $1/2$ ) cps
I a	6.07; 6.11	6
I b	6.09; 6.12	
I c	6.11	
I d	6.07; 6.09	
I e	7.55	4.5
I f	7.56	3
II	6.05	0.5

$\Delta \nu$  ( $1/2$ ) = linewidth at half signal height.

It can be excluded that the tautomers A, B or C are responsible for the different shielding of the  $\text{X-CH}_3$  groups, because all compounds I (except Ic) show vicinal coupling ( $J \sim 5 - 6$  cps) between NH and CH of the alkylamino groups.



It is reasonable to assume that strong contribution of the mesomeric form D to the ground state of the molecules causes this effect.

Quantitative temperature studies will be performed on these and related compounds and the results will be published elsewhere.

Yours sincerely,

*Hans Frit*

## UNIVERSITY OF CALIFORNIA, DAVIS

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



SANTA BARBARA • SANTA CRUZ

DEPARTMENT OF CHEMISTRY

DAVIS, CALIFORNIA 95616

July 1, 1966

C<sup>13</sup> Chemical Shift Difference Between  
Cis and Trans Diiodoethylene.

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

Dear Barry:

Savitsky and Namikawa (J. Phys. Chem., 67, 2754 (1963)) have previously reported the value 17.1 p.p.m. for the C<sup>13</sup> chemical shift difference between the cis and trans isomers of diiodoethylene. In view of the rather large solvent effects often found in C<sup>13</sup> magnetic resonance, for example the 7.3 p.p.m. difference between the C<sup>13</sup> shifts of neat CH<sub>3</sub>I and a dilute solution in cyclohexane (H. Spiesscke and W. G. Schneider, J. Chem. Phys., 35, 722 (1961)), and the fact the differential shielding had been determined using saturated solutions of the trans isomer (in CH<sub>2</sub>Cl<sub>2</sub>), we decided to explore the possibility that a significant portion of the difference might be due to some specific solute-solute interactions.

Accordingly, we prepared samples of C<sup>13</sup>-labelled trans-diiodoethylene and of C<sup>13</sup>-labelled mixtures of the isomers, and obtained their C<sup>13</sup> magnetic resonance spectra from solutions in which the total diiodoethylene present accounted for no more than five percent (in cyclooctane as the solvent). The spectra were obtained using rapid-passage, dispersion-mode conditions with proton spin-decoupling of each isomer in separate experiments. The resulting differential shielding is 17.3 p.p.m. for  $\sigma_{\text{trans}} - \sigma_{\text{cis}}$ , showing that the value obtained by Savitsky and Namikawa is due to intrinsic properties of the solute molecules, and not appreciably to intermolecular interactions.

With the C<sup>13</sup>-labelled compounds available from our syntheses we obtained the following additional n.m.r. parameters from proton spectra obtained on an A-60A (and from slow-passage, absorption mode C<sup>13</sup> spectra at 15.1 mc in the trans case):  $\sigma_{\text{trans}} - \sigma_{\text{cis}} = 43.4$  cps. and



Professor B. L. Shapiro

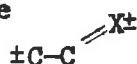
-2-

July 1, 1966

	<u>Trans</u>	<u>Cis</u>
$J_{CH}$	$201.0 \pm 0.1$ cps.	$193.2 \pm 0.2$ cps.
$J_{CCH}$	$-1.9 \pm 0.1$	$10.4 \pm 0.2$
$J_{HH}$	$14.3 \pm 0.2$	$6.2 \pm 0.2$
$J_{CC}$	$81.2 \pm 0.2$	$81.1 \pm 0.2$

The proton differential shielding was obtained using a 1:4 (cis to trans) mixture of isomers in cyclooctane in which the total diiodoethylene accounted for five percent of the material. The coupling constants were obtained using similar solutions at twenty-five percent, both of the mixture and of pure trans material. It was necessary to use time-averaging with a C-1024 in order to bring out the peaks containing information on  $J_{CC}$  for the cis-isomer.

We are now conducting some experiments to test the hypothesis of Savitsky and Namikawa that the differential shielding may be due to the relative contributions of structures of the type



in the ground-state of the two isomers. Using  $\text{CO}_2\text{CH}_3$  as X, we intend to vary the nature of X and the importance of the above structures, more or less continuously, by changing the hydrogen-bonding characteristics of the solvent. Samples of the esters with  $\text{C}^{13}$  placed at each carbon position are now being prepared so that dilute solutions may be employed, and so that the nature of X can be inferred from the carbonyl shielding. These experiments are being conducted by Mr. Paul Ellis.

Sincerely,



Gary E. Maciel  
Associate Professor  
of Chemistry

GEM:dh



UNITED STATES  
DEPARTMENT OF THE INTERIOR  
BUREAU OF MINES  
4800 FORBES AVENUE  
PITTSBURGH, PENNSYLVANIA 15213

Pittsburgh  
Coal Research Center

July 1, 1966

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

Dear Dr. Shapiro:

1967 Pittsburgh Conference

Although the 1967 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy is still several months away, Gerald Carlson (Mellon Institute) and his program committee have been busy organizing symposia. I have been chosen to organize the NMR meetings and, at the risk of being called prejudiced, I investigated the possibility of having a symposium devoted to carbon-13 nuclear magnetic resonance spectrometry.

Seven out of eight people invited to speak at the meeting have thus far accepted. A partial list of topics (not titles; subject to change) follows:

Instrumental Techniques for  $C^{13}$  NMR  
 $C^{13}$  NMR of Organometallic Compounds  
Conformational Analysis by  $C^{13}$  NMR  
 $C^{13}$  NMR of Organic Ions  
Solid State  $C^{13}$  NMR

The 1967 Conference will be held in Pittsburgh, March 5-10, 1967, and I am told that the Eighth Experimental NMR Conference will be held at Mellon Institute during the end of the preceding week.

I urge readers of I.I.T.N.M.R. to reserve these dates so they can attend these meetings. In addition I hope your readers will tell their chemist and physicist friends that the symposium was designed to interest the general scientific public as well as the  $C^{13}$  spectroscopist.

Sincerely yours,

H. L. Retcofsky  
Research Physicist  
Spectrometry  
(Program Committee,  
Pittsburgh Conference)

## CARNEGIE INSTITUTE OF TECHNOLOGY

SCHENLEY PARK

PITTSBURGH, PENNSYLVANIA 15213

July 9, 1966

DEPARTMENT OF CHEMISTRY

TELEPHONE: 621-2600  
AREA CODE 412

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

Dear Barry:

INDUCED PARAMAGNETIC RING CURRENTS

We have recently examined the quantum mechanical theory of induced ring currents in conjugated monocyclic polyenes, paying particular attention to the difference between  $4n$  and  $4n+2$  type systems. We find that the original London theory always predicts paramagnetic circulations for molecules with  $4n$  electrons. In such molecules, the direction of the current is opposite to that in benzene. However, these paramagnetic currents will be partly quenched by alternation of both ring and molecular non-planarity. For this type of molecule, the usual diamagnetic rules for corresponding proton nmr chemical shifts have to be reversed, those outside the ring being displaced to high field and those inside to low field. We have examined available experimental data on a number of annulenes and dehydroannulenes and found some evidence supporting these predictions. A full manuscript describing this work will appear shortly in the Journal of the American Chemical Society.

Yours sincerely,

*John*

John A. Pople  
Carnegie Institute of Technology  
and Mellon Institute

*Karl*

K. G. Untch  
Mellon Institute

JAP:kos

## INSTITUT DE CHIMIE DES SUBSTANCES NATURELLES

91 - GIF-SUR-YVETTE

TÉL. : 928. 46-76

July 2, 1966.

Another Secondary Deuterium Isotope Effect; Rabelais and the  
(No, not the Franciscans ! ) Nomenclature for Equivalence.

Dear Barry,

Together with Dr. S. Bory, we have measured the isotopic chemical shifts for the methyl groups of 2,2,6-trimethyl-6-deutero-cyclohexanone : + 0.2 (eq.Me-2), + 0.3 (ax.Me-2), and + 1.15 (eq.Me-6) (- 0.2 Hz, at 60 MHz), in carbon tetrachloride. In benzene solution, the corresponding shifts, also to high-field (plus sign) are respectively + 0.15, + 0.15, and + 1.1 ± 0.2 Hz. The sole 6-methyl group, geminal with the introduced deuterium atom, is affected substantially relative to the undeuterated molecule.

With reference to the Binsch (IITNMRN 87-32), and the Emsley, Fee-ney, Sutcliffe (IITNMRN 90-14), and after an exchange of ideas with Professor K. Mislow, who independently writes to you, I would like to submit the enclosed Table.

In it, the inverted A and E have their usual meanings of "whatever" and "there exists". The term "isochronous" had been very conveniently introduced by Abragam in his "Principles of Nuclear Magnetism", Oxford, 1961, p. 480; "anisochronous" is a neologism proposed by Binsch in a complementary way. The "isogamous" and "anisogamous" neologisms I am suggesting here refer to coupling constants, as the "isochronous" nomenclature describes the chemical shift situation, a coherent nomenclature has to include similar terms for both kinds of n.m.r. parameters. Furthermore, this vocabulary pleasantly implies the notion of nuclear polygamy, which could have been agreeable to our greatest national inventor of new words, and accounts for the above title. I have adopted "enantiomeric" and "diastereomeric", as defined by Mislow in his "Introduction to Stereochemistry", Benjamin, 1965, and the "enantiotopic", "diastereotopic" derivatives. Translation of all these words into French is adequate, pace Etienne; for example, Littré gives "topique" as an adjective with the meaning presently used by Mislow.

In my opinion, it is not enough for us n.m.r. spectroscopists to use more or less empirical and a posteriori descriptions in the spectral analysis of spin interaction systems, there ought to be an unified classification, in common with the organic chemists, and based only upon symmetry properties. All the words constructed with the "equivalence" root, should be banished, indiscriminate usage has worn out this term, "the word "structure" is an adequate comparison.

Sincerely yours,

  
Pierre Laszlo

Relationship of nuclei A and B	Chemical shift criterion	Coupling constants with nucleus X.	Relationships of X with A and B	$J_{AB}$ manifest in the spectrum
diastereotopic $H_A \neq H_B$	anisochronous $\nu_A \neq \nu_B$	anisogamous $J_{AX} \neq J_{BX} \quad \forall X$	diastereomeric	YES
enantiotopic $H_A = H_B$	isochronous $\nu_A \equiv \nu_B$	isogamous $J_{AX} \equiv J_{BX} \quad \forall X$	enantiomeric	NO (°)
enantiotopic $H_A = H_B$	isochronous $\nu_A \equiv \nu_B$	anisogamous $\exists X \rightarrow J_{AX} \neq J_{BX}$	diastereomeric	YES

(°) its detection is possible through recourse to either:

- a/ the  $^{13}\text{C}$  satellites: A and B become diastereotopic;
- b/ deuteration of A or B: "
- c/ use of a nematic solvent, where A and B are still isochronous, but now anisogamous.

For instance, the relationships between X and either A or B are enantiomeric for  $\text{C}_6\text{H}_6$  in an ordinary solvent, they become diastereomeric in a nematic solvent



TEXAS CHRISTIAN UNIVERSITY

Fort Worth, Texas 76129

Department of Chemistry

July 5, 1966

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

Dear Barry:

We have recently determined the n.m.r. spectra of several enamines of 1-azabicycloalkanes (Table I) and their corresponding iminium salts. The spectra of the former have permitted the assignment of structure to many unsymmetrical enamines and have led us to conclude that the position of the double bond is controlled by the same factors which are responsible for the relative stability of simple olefins and not those which are important for many enamines of cyclic ketones. The n.m.r. spectra of the iminium salts are quite characteristic and have proven useful in structure determinations.

A paper on this material has been accepted by J. Org. Chem. and a limited number of preprints are available.

Sincerely,

*Manfred G. Reinecke*

Manfred G. Reinecke  
Assistant Professor  
Department of Chemistry

<sup>a</sup> Unless otherwise noted all spectra were taken in CCl<sub>4</sub> solution with TMS as an internal standard on v.p.c. collected samples. Chemical shifts are expressed in  $\tau$ ,  $\delta$  in c.p.s. and doublets and triplets as  $d$  and  $t$  respectively. <sup>b</sup> Center of broad multiplet; <sup>c</sup> The relative area of these peaks suggests an approximately 2:1 mixture of the <sup>a</sup> to the <sup>b</sup> isomer. <sup>d</sup> In tetrachloroethylene, which has no appreciable effect on the chemical shift values; <sup>e</sup> The relative area of this peak (1:16) indicates that very little if any  $\Delta^{1(9)}$ -isomer is present; <sup>f</sup> Very distorted triplet with an apparent  $\delta$  of 7 c.p.s.; <sup>g</sup> Purified by vacuum distillation; <sup>h</sup> Phenyl protons.

TABLE I N.M.R. Spectra of Enamines of 1-Azabicycloalkanes<sup>a</sup>

Compound	H—C=C	CH <sub>2</sub> —N	C—CH <sub>3</sub>
(5)	5.92	7.14 <sub>t</sub> ( <u>J</u> =7)	
(6)	5.80	7.25 <sub>b</sub>	
(7)		7.23 <sub>t</sub> ( <u>J</u> =7)	8.50
(8a)	5.97 <sup>c</sup>	} 7.17 <sub>t</sub> ( <u>J</u> =7)	8.95 <sub>d</sub> ( <u>J</u> =7) <sup>c</sup>
(8b)			8.43
(9)	5.98 <sub>t</sub> ( <u>J</u> =4)	7.13 <sub>t</sub> ( <u>J</u> =7)	8.95
(10)	5.56	7.3 <sub>b</sub>	8.85
(11a)		} 7.3 <sub>b</sub>	8.44
(11b)	5.63 <sup>c</sup>		8.95 <sub>d</sub> ( <u>J</u> =7) <sup>c</sup>
(12)	5.89 <sup>e</sup>	7.15 <sub>b</sub>	9.1 <sub>b, f</sub>
(13)		7.2 <sub>b</sub>	2.95 <sub>b, h</sub>



# Imperial Chemical Industries Limited

HEAVY ORGANIC CHEMICALS DIVISION

Organic House, P.O. Box No. 2, Billingham, Co. Durham

Telephone: STOCKTON-ON-TEES 53601 Telex: 58523 ICIBILL, STOCKTON

Cables: ORGANIC, STOCKTON-ON-TEES, TELEX

Your Ref.

Our Ref. RES/JQ/JMR/AJW/MH.

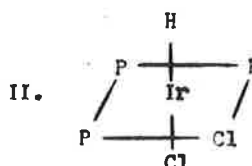
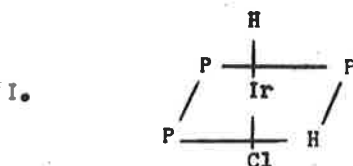
6th July, 1966.

Dear Professor Shapiro,

## IRIDIUM HYDRIDE SPECTRA ON THE VARIAN A-60A

We have been operating our new Varian A-60A spectrometer for some three months now, and are most impressed by the improved facilities compared with its parent A-60. These we have been using to good advantage in recording the spectra of saturated solutions of poorly soluble iridium hydride phosphine complexes over the region 0-2000 cps to high field of TMS, using the 5000 sec sweep.

The NMR spectra of complexes of the type  $\text{IrH}_2\text{ClP}_3$  (P = tertiary phosphine)\* are consistent with the configuration I, and the IrH part normally comprises two triplets for H, i.e.  $J_{\text{H}_1\text{P}_1} = J_{\text{H}_1\text{P}_2} \neq J_{\text{H}_1\text{P}_3}$ , and a quartet for H<sub>2</sub>, i.e.  $J_{\text{H}_2\text{P}_1} = J_{\text{H}_2\text{P}_2} = J_{\text{H}_2\text{P}_3}$ .



For complexes  $\text{IrHCl}_2\text{P}_3$  with configuration II, however, the Ir-H spectrum has been shown to vary with the phosphine ligand; in benzene solution the three coupling constants are equal for  $\text{Et}_3\text{P}$ , while with  $\text{Et}_2\text{PhP}$   $J_{\text{HP}_1} = J_{\text{HP}_2} \neq J_{\text{HP}_3}$ .

We have now found that this effect is due not only to the ligand but also to the solvent. The compound I, P =  $\text{Ph}_3\text{P}$ , has been studied in  $\text{CDCl}_3$ ,  $\text{CH}_2\text{Cl}_2$ ,  $\text{C}_6\text{D}_6$  and  $\text{C}_4\text{H}_8\text{O}$ . In deuteriochloroform, only, the coupling constant  $J_{\text{H}_2\text{P}_2}$  differs from  $J_{\text{H}_2\text{P}_1} = J_{\text{H}_2\text{P}_3}$ , the magnitudes being similar to those of the monohydride, II =  $\text{Et}_2\text{PhP}$ . The spectra of this complex in the other solvents all show the expected quartet; so also does the spectrum of the monohydride, II, P =  $\text{Ph}_3\text{P}$ , in deuteriochloroform.

We hope to continue this study, in the meantime it is submitted as a further subscription from the second author. The compounds were prepared by Dr. R.S. Coffey of these laboratories.

Yours sincerely,

*J. Quarby* J. M. Rowe A. J. Wilkinson

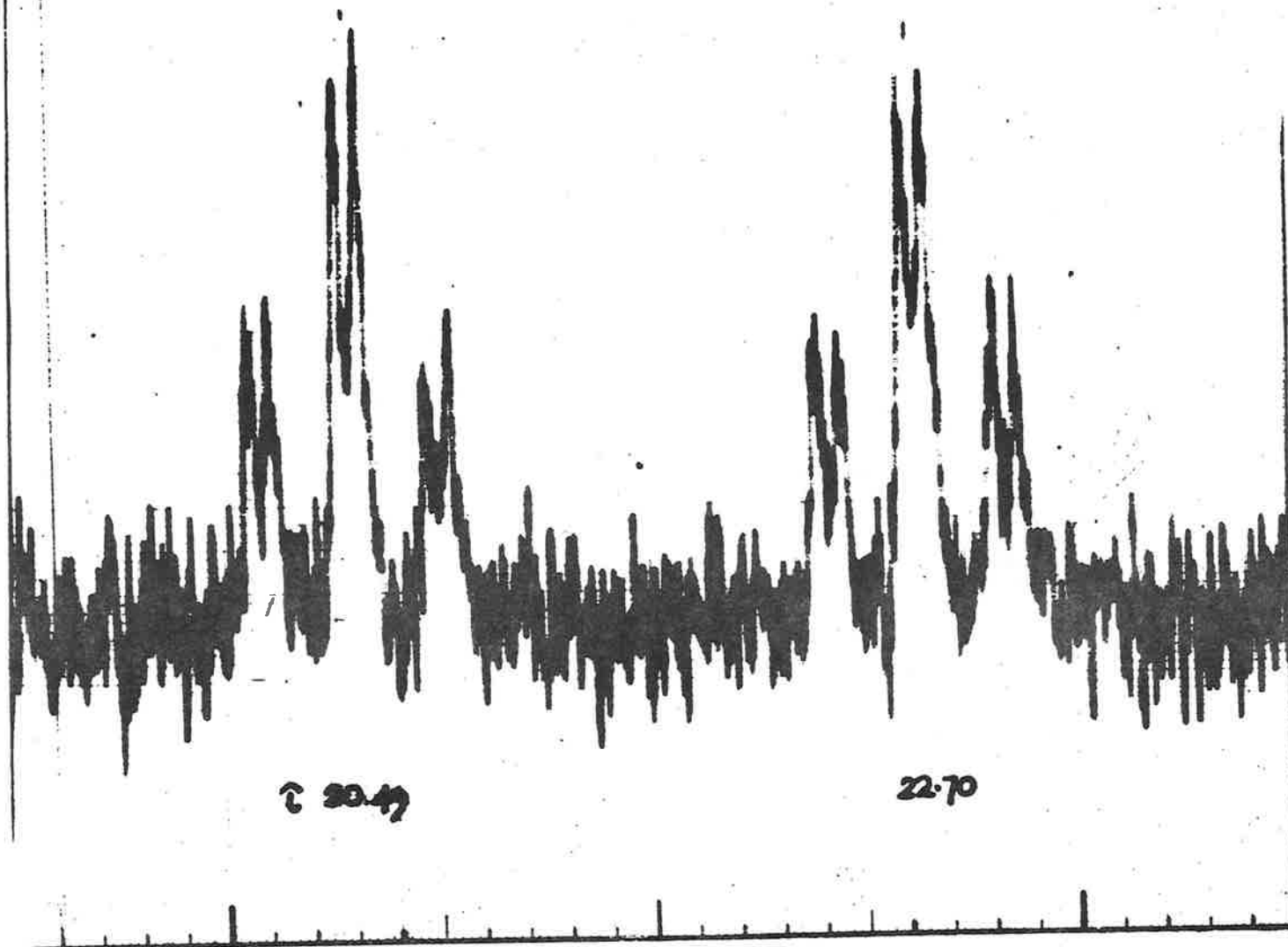
J. Quarby, J.M. Rowe, A.J. Wilkinson.

\* Chatt, Coffey & Shaw J.C.S., 1965, 7391

Taylor, Young & Wilkinson Inorg.Chem., 1966, 5, 20.



$$J_{H-P_1} = J_{H_1-P_2} = 20.5 ; J_{H_1-P_2} = 132.5 ; J_{H_1-H_2} = 5 \text{ c/s}$$



$$J_{H-P_1} = J_{H_1-P_2} = 17, J_{H_1-H_2} = 4.5$$



# The Principles of **NUCLEAR MAGNETIC RESONANCE**

## **A WORKSHOP**



**Sponsored by the  
UNIVERSITY OF HOUSTON  
30 Aug. - 1 Sept., 1966**

## **NUCLEAR MAGNETIC RESONANCE WORKSHOP**

**August 30, 31 - September 1, 1966**

A gathering of scientists interested in high-resolution Nuclear Magnetic Resonance is being sponsored by the University of Houston. This discussion will center largely on elementary principles, but small seminar groups will be encouraged to consider NMR problems at all levels of complexity. Special sections, such as use of NMR in college curricula, and use of NMR in the small industrial laboratory will be scheduled if the demand is sufficient. Therefore the workshop should be of value to most persons now using NMR, as well as to those contemplating the use of NMR.

### **ENROLLMENT**

All sessions will meet in the Lamar Fleming Building on the University of Houston campus. The total number of participants will be limited, since this should make both the laboratory sessions and the informal discussions of greater value to those attending. Enrollments will be accepted in the order in which they are received.

### **FEES**

A charge of \$75, payable to the University of Houston NMR Workshop, will be made to cover laboratory fees, workshop materials, lunches for three days, and a banquet (Wednesday, 31 August). Graduate students and undergraduates may enroll for a fee of \$20.

### **HOUSING AND MEALS**

Several motels and hotels are located within a convenient distance of the campus. A list will be sent to each applicant on receipt of the registration form, or the Workshop will make reservations for you. Noon meals will be served in the University of Houston dining halls, and this cost is included in the registration fee.

## Program

### TUESDAY, 30 AUGUST

Introduction to Nuclear Magnetic Resonance (3 lectures)  
Nugent Chamberlain, Esso Research and Engineering

### WEDNESDAY, 31 AUGUST

Determination of Structure by Nuclear Magnetic Resonance  
Nugent Chamberlain, Esso Research and Engineering

Applications of NMR  
Ray Ettinger, Varian Associates

Interpretation of Spectra  
William B. Smith, Texas Christian University

### THURSDAY, 1 SEPTEMBER

Relationship of NMR Parameters to Structure

William B. Smith, Texas Christian University

The NMR Spectrometer  
Ray Ettinger, Varian Associates

## LABORATORY AND DISCUSSION GROUPS

Two sessions each day will be devoted to groups of ten participants. The laboratory sections will be permitted to observe operation of and to operate the Varian A-60 and HA-100 spectrometers, and the C-1024 computer of average transients. Some sessions will be devoted to the interpretation of NMR spectra.

## REGISTRATION

The Workshop will acknowledge receipt of applications, and notify persons of acceptance. Complete Workshop materials will be available to participants in the Chemistry Office of the University of Houston after August 28. Registration forms and checks should be mailed to: Dr. M. R. Willcott, Chemistry Department, University of Houston, Houston, Texas 77004.

## REGISTRATION FORM

Name \_\_\_\_\_

Address \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

If you have had any experience with NMR, describe briefly:

\_\_\_\_\_

If you wish the Workshop to make motel reservations for you, complete the following:

Number of persons \_\_\_\_\_ Arrival date \_\_\_\_\_ Departure date \_\_\_\_\_

Make check payable to: University of Houston NMR Workshop

Mail check to: Dr. M. R. Willcott  
Department of Chemistry  
University of Houston  
Houston, Texas 77004

# Ford Motor Company

ENGINEERING AND RESEARCH STAFF

20000 ROTUNDA DRIVE  
P. O. BOX 2053  
DEARBORN, MICHIGAN 48121

July 13, 1966

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

Dear <sup>a</sup>Berry:

Sorry to be tardy in my subscription.

As part of a general talk that I will give at the Colloque Ampere in Ljubljana, I wrote down the expression for the Fermi part of the spin-spin coupling constant between two nuclei A and B as a power series in the Hartree-Fock (dummy) correlation parameter,  $\lambda$ :

$$J = J_0 + \lambda J_1 + \dots$$

where

$$J_0 = (4\pi\gamma_A/3)2 \sum_{\mu} \left[ \langle \mu(1) | \delta(r_{1A}) \mu_B(1) \rangle - \langle \mu(1) | \mu_B(1) \rangle \langle \bar{\mu}(2) | \delta(r_{2A}) | \bar{\mu}(2) \rangle \right. \\ \left. - \sum_{\nu \neq \mu} \langle \nu(1) | \mu_B(1) \rangle \langle \nu(2) | \delta(r_{2A}) | \mu(2) \rangle \right],$$

a sum of contributions from individual orbitals in the H-F (or other separable-potential)  $\psi_0$ :

$$\psi_0 = \alpha(1)\bar{\alpha}(2)\beta(3)\bar{\beta}(4) \dots,$$

and where the perturbed orbitals are solutions to the one-electron equations

$$[h_o(1) - \epsilon_{\mu}] \mu_B(1) = -(16\pi\beta\hbar\gamma_B/3) [\delta(r_{1B}) - c_{\mu}] \mu(1).$$

Professor B. L. Shapiro

Page 2

July 13, 1966

The w.f. to first order in  $I_{B,z}$  which enters into  $J_0$  can be written as the sum of "triplets"

$$\psi'_{001} = \sum_{\mu} G^{\mu} \equiv \frac{1}{2} \sum_{\mu} \alpha(1)\bar{\alpha}(2)\dots[\mu_B(m)\mu(n)-\mu(m)\mu_B(n)]\Sigma_3(mn) \dots$$

where  $\Sigma_3(mn)$  is the triplet spin function of  $M_s = 0$ , but note that the  $\mu_B$  are not eigenfunctions of  $h_0$  but a complicated sum of such eigenfunctions.

If  $\mu_B$  is expanded in a finite set of functions as

$$\mu_B = \sum c_n |n\rangle$$

then it can be obtained from the sequence of linear equations

$$\sum_n \langle m | h_0 - \epsilon_{\mu} | n \rangle c_n = -\langle m | (16\pi\beta\hbar\gamma_B/3) [\delta(r_B) - c_{\mu}] | \mu \rangle$$

but unlike all such other expressions--i.e. for chemical shift, electric polarizability, etc.--there is a singular function in the right hand integral which can only be removed if one of the approximate functions  $|n\rangle$  contains a multiple of  $r_B^{-1}$ . This leads to a divergence in the r.h.s. which must be identically cancelled by a term in the l.h.s., but which in turn leads to a new divergence which in turn must be cancelled identically by a third term. This process of cancelling of divergences is finite so that one can then proceed with the remaining linear equations, dropping off the last few so as not to overdefine the problem. A conclusion from this type of argument is that the Least Combination of Atomic Orbitals method for such calculations is highly suspect since the divergence does not appear! Professor R. M. Pitzer at Caltech has recently told me that he is beginning calculations for J's which will include  $r_B^{-1}$  times exponentials in his basis set.

As a point of minor interest, we have demonstrated generally that couplings between nuclei which are permutationally equivalent--the F's of  $CF_3H$ , but not of  $KF_5$ --do not affect the spectrum, even though their part of the Hamiltonian does not commute with the remainder, as is necessary for the proof of Gutowsky, McCall and Slichter in liquids. This permutationally equivalent nuclei play a role in liquid crystal spectra similar to that of "magnetically" equivalent nuclei in liquids, i.e. they are magnetically equivalent. In the  $AX_2$  and  $AX_3$  examples in Buckingham and Pople's paper the  $J_{xx}$  terms are explicitly seen not to affect the spectrum.

Yours sincerely,

Jeremy I. Musher  
Mathematical and Theoretical Sciences  
(Summer Visitor)

JIM:pjt

Title: On Spin-Spin Coupling Constants.

## THE UNIVERSITY OF ROCHESTER

COLLEGE OF ARTS AND SCIENCE

RIVER CAMPUS STATION

ROCHESTER, NEW YORK 14627

DEPARTMENT OF CHEMISTRY

July 13, 1966

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

Dear Barry:

RA-1 Spectrum Accumulator

We have now been using a JEOL RA-1 Spectrum Accumulator connected to our A-60 for some months and thought that readers of IITNMRN might be interested in our experiences with it.

In general, these have been quite satisfactory. Apart from some easily cured trouble arising from pickup of transients when the RA-1 was first installed and some erratic behavior from a relay, which we replaced, we have had no trouble with the hardware. A hazard with a stored program computer such as the RA-1 is that the program can be damaged if the operator makes a mistake. There have been occasions when the RA-1 has refused to work for no obvious reason, but we have found that the frequency of such stoppages appears to vary inversely with the experience of the operator, so we suspect that the RA-1 is not the culprit. Such difficulties are easily remedied by reading the program tape in again.

So far we have made one simple and useful addition to the connection unit. This is a circuit which automatically attenuates the signal to a pre-set level while the RA-1 is searching for the trigger and restores it to the original level as soon as the RA-1 starts accumulating. There is a spare relay connection in the unit available for the attenuator circuitry. We find that by using a fairly high concentration of TMS and attenuating the peak to a convenient level for triggering we can use a relatively noise-free trigger, thereby reducing broadening of the spectrum on long runs. By this means we have maintained good ringing on sharp peaks on overnight runs.

Professor B. L. Shapiro

July 13, 1966

We have been planning to build more versatile sweep offset circuitry, following a suggestion by Roy King, but so far have found this unnecessary. The present system, in which the RA-1 switches the A-60 sweep offset in while accumulating and out while searching for the TMS trigger, has proved quite adequate (though it does involve sweeping from high field to low). We have accumulated expanded portions of spectra, using sweep offset and a TMS trigger, without having any trouble from line broadening.

The RA-1 is also being used to calculate e.s.r. spectra, which are calculated in the form of simulated spectra or as stick diagrams, and drawn out on the A-60 recorder.

The greatest drawback to the routine use of the RA-1 in conjunction with the A-60 is the present lack of an adequate instruction manual. We have been writing our own as we go, supplementing the instructions supplied by JEOL with what we have learned from their engineer and from trial and error. When the instructions are available anyone who can use the A-60 can quickly learn to use the RA-1 as well. Our RA-1 is now being used on a routine basis by an undergraduate assistant.

Yours sincerely,



L. D. Colebrook

## CALIFORNIA STATE COLLEGE



## AT LOS ANGELES

Department of Chemistry

5151 State College Drive, Los Angeles, California 9003

(San Bernardino and Long Beach Freeways Interchange)

Telephone 225-1631 (Area Code 213)

July 14, 1966

Dear Barry:

I hope this note meets the July 15 deadline. As you will recall, in a previous newsletter contribution, we mentioned that in our studies of complexes between several salts and dimethylformamide, separate resonance signals were observed for bulk and complexed amide. We indicated at that time that temperature studies were in progress to determine rate data for the exchange of solvent molecules between the bulk and complexed environments. In this letter we wish to present the results of a few of our temperature studies.

In Figure 1 are plots of  $\ln \Delta$  versus  $1/T$  for solutions of a variety of salts in DMF. The quantity  $\Delta$  is essentially the difference in line width of the complex amide signal (aldehyde proton) at  $T_0$ , a temperature at which exchange is very slow, and  $T$ , some higher temperature. Since  $k$ , the rate constant for the exchange is proportional to  $\Delta$ , activation enthalpies were obtained from these plots. The values obtained are 28, 19, 17, 18, and 11 Kcal/mole for the  $TiCl_4$ ,  $AlCl_3$ ,  $GaCl_3$ ,  $BeCl_2$  (both solutions) and  $SbCl_5$  solutions respectively. Our studies also yield coordination numbers for the complexes along with rate constants for the exchange process at each temperature. We have submitted a paper on these studies and will gladly supply preprints to those interested.

Sincerely,

Anthony Fratiello

*Anthony Fratiello*

Don Paul Miller

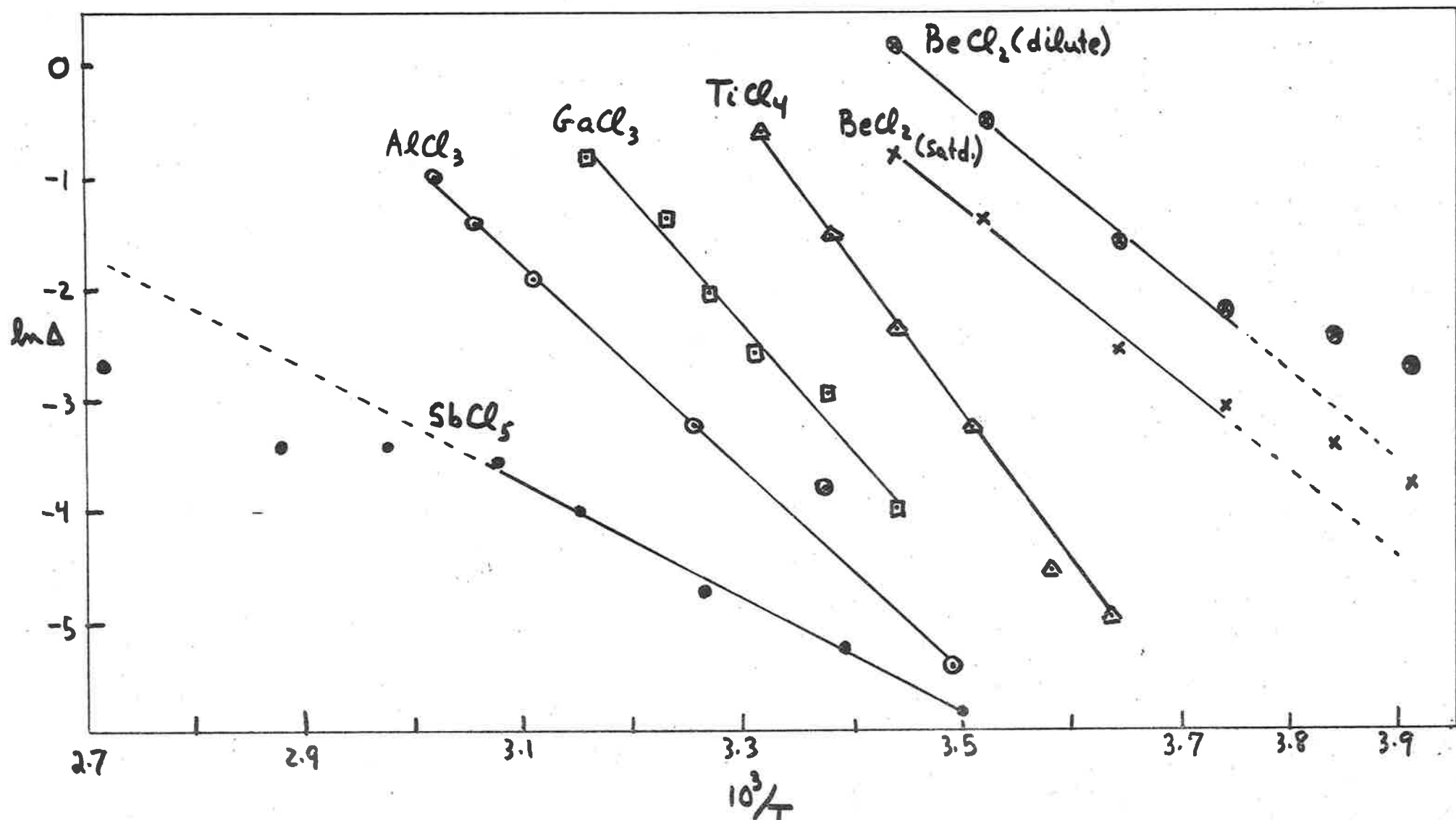
*Don Paul Miller*

Ronald Schuster

*Ronald Schuster*

FMS/ml





"Kinetic Theory of Nuclear Spin Relaxation in Gases"  
R. G. Gordon  
J. Chem. Phys. 44, 228 (1966)

"NMR Spin-Lattice Relaxation in Solid Solutions of C<sub>6</sub>H<sub>6</sub> and C<sub>6</sub>D<sub>6</sub>: Evidence for Concerted Motions by Adjacent Molecules"  
J. E. Anderson  
J. Chem. Phys. 43, 3575 (1965)

"Use of Spin Echoes in a Pulsed Magnetic-Field Gradient to Study Anisotropic, Restricted Diffusion and Flow"  
E. O. Stejskal  
J. Chem. Phys. 43, 3597 (1965)

"Four-Proton System in Barium Bromide Dihydrate"  
J. W. McGrath  
J. Chem. Phys. 43, 3746 (1965)

"NMR Studies of Some Liquid Crystal Systems"  
J. C. Rowell, W. D. Phillips, L. R. Melby and M. Panar  
J. Chem. Phys. 43, 3442 (1965)

"Proton Magnetic Resonance Shifts in Aqueous Solutions of Paramagnetic Metal Ions"  
Z. Luz and R. G. Shulman  
J. Chem. Phys. 43, 3750 (1965)

"Electron Transfer Rates for Unsubstituted and Dimethyl-substituted Tris(1,10-phenanthroline) Iron(II) and -(III) Ions from NMR Studies"  
D. W. Larsen and A. C. Wahl  
J. Chem. Phys. 43, 3765 (1965)

"Buxus Alkaloids. Part VI. The Constitutions of Cyclo-microbuxine, Cyclomicrobuxinine, and Alkaloid-L"  
T. Nakano and M. Hasegawa  
J. Chem. Soc. (Lond.) 6688, (1965)

"The Preparation of Some 4,6-Disubstituted Pyrimidine-5-carboxylic Acids"  
M. D. Mehta, D. Miller, and E. F. Mooney  
J. Chem. Soc. (Lond.) 6695 (1965)

"Applications of Proton Resonance Spectroscopy to Structural Problems. Part XXIII. Determination of Hammett  $\sigma$ -Constants for Heterocycles"  
A. R. Katritzky and F. J. Swinbourne  
J. Chem. Soc. (Lond.) 6707 (1965)

"Metal- $\beta$ -Diketone Complexes. Part I. Metal-Carbon Bonding in Some Platinum Acetylacetonates"  
J. Lewis, R. F. Long, and C. Oldham  
J. Chem. Soc. (Lond.) 6740 (1965)

"Organometallic Reactions. Part III. Trimerisation of Isocyanates by Organotin Alkoxides and Oxides"  
A. J. Bloodworth and A. G. Davies  
J. Chem. Soc. (Lond.) 6858 (1965)

"Organophosphorus Chemistry. Part III. The Reactions of Phospha-alkenes with Nucleophiles and as Intermediates in Polymer Synthesis"  
H. Goldwhite, R. N. Haszeldine and D. G. Rowsell  
J. Chem. Soc. (Lond.) 6875 (1965)

"Organophosphorus Chemistry. Part IV. Phospha-Alkenes as Intermediates in the Reaction of 1,1,2,2-Tetrafluoro-ethylphosphine with Dimethylamine or Aniline"  
M. Green, R. N. Haszeldine, B. R. Iles, and D. G. Rowsell  
J. Chem. Soc. (Lond.) 6879 (1965)

"Deuterium and Tritium Exchange Reactions of Phenols and the Synthesis of Labelled 3,4-Dihydroxyphenylalanines"  
G. W. Kirby and L. Ogunkoya  
J. Chem. Soc. (Lond.) 6914 (1965)

"Tetranortriterpenoids. Part I. [Bicyclononanolid. Part I.] The Constitution of Swietenine"  
J. D. Connolly, R. Henderson, R. McGrindle, K. H. Overton, and N. S. Bhacca  
J. Chem. Soc. (Lond.) 6935 (1965)

"Cyclitols. Part XXI. Benzyl Ethers of Myoinositol. Aromatisation of a Tosyl Derivative of Myoinositol"  
S. J. Angyal and M. E. Tate  
J. Chem. Soc. (Lond.) 6949 (1965)

"The Synthesis of an Analogue of Griseofulvin"  
R. F. Curtis, C. H. Hassall, and D. W. Jones  
J. Chem. Soc. (Lond.) 6960 (1965)

"Photo-oxygenation of 3-Acetoxyergosta-3,5-7,22-tetraene and Related Compounds"  
P. Bladon and T. Sleight  
J. Chem. Soc. (Lond.) 6991 (1965)

"The Reaction of Dipyrromethene Salts with Methyl Ketones and with Dicyanomethane"  
P. Bamfield, A. W. Johnson, and J. Leng  
J. Chem. Soc. (Lond.) 7001 (1965)

"Studies on Sesquiterpenoids. Part XI. Structure and Stereochemistry of Xanthumin, a Stereoisomer of Xanthinin"  
H. Minato and Isao Horibe  
J. Chem. Soc. (Lond.) 7009 (1965)

"The Dimroth Rearrangement. Part V. The Mechanism of the Rearrangement of 1-Alkyl-1,2-dihydro-2-iminopyrimidines in Aqueous Solution"  
D. D. Perrin and I. H. Pitman  
J. Chem. Soc. (Lond.) 7071 (1965)

"The Production of Free Radicals in the Reaction of Grignard Reagents with Organic Halides in the Presence of Cobaltous Chloride"  
D. I. Davies, D. H. Hey, and M. Tiecco  
J. Chem. Soc. (Lond.) 7062 (1965)

"Mycotoxins. Part II. The Constitution of Ochratoxins A, B, and C, Metabolites of Aspergillus ochraceus Wilh."  
K. J. van der Merwe, P. S. Steyn and L. Fourie  
J. Chem. Soc. (Lond.) 7083 (1965)

"Preparations and Reactions of 1,3-Dithia-2-silacyclopentane Derivatives"  
E. W. Abel, D. A. Armitage, and R. P. Bush  
J. Chem. Soc. (Lond.) 7098 (1965)

"The Removal of Toluene-p-sulphonyl Groups from Sulphonamides. Part II. Phenylglyoxal Anil Dimers"  
T. Bryce, G. R. Proctor, and M. A. Rehman  
J. Chem. Soc. (Lond.) 7105 (1965)

"Naturally-occurring Thiophens. Bithienyl Derivatives from Tagetes minuta L."  
R. E. Atkinson, R. F. Curtis, and G. T. Phillips  
J. Chem. Soc. (Lond.) 7109 (1965)

"Phosphorus-Nitrogen Compounds. Part XVII. The <sup>1</sup>H and <sup>31</sup>P Nuclear Magnetic Resonance Spectra of Dimethylamino-derivatives of Hexachlorocyclotriphosphazatriene"  
R. Keat, S. K. Ray, and R. A. Shaw  
J. Chem. Soc. (Lond.) 7193 (1965)

"Perfluoroalkyl Derivatives of Nitrogen. Part XIX. Synthesis, Pyrolysis, and Nuclear Magnetic Resonance Evidence for Restricted Inversion at the Nitrogen Atom of Trishepta-fluoro-n-propylhydroxylamine"  
R. E. Banks, M. G. Barlow, R. N. Haszeldine, and M. K. McCreath  
J. Chem. Soc. (Lond.) 7203 (1965)

"The Chemistry of Fungi. Part L. Rosenonolactone"  
G. A. Ellestad, B. Green A. Harris, W. B. Whalley and H. Smith  
J. Chem. Soc. (Lond.) 7246 (1965)

- "Über den Verlauf der Bergmann-Sternschen Azlactonsynthese; 2-Alkyliden-Pseudooxazolone-(5)"  
W. Steglich und R. Hurnaus  
Tetrahedron Letters 383 (1966)
- "Photochemische Reaktionen von Aromaten mit Dienen: Cycloaddukte von Benzol und einigen seiner Derivate an Isopren und von Benzol an Butadien"  
G. Koltzenburg und K. Kraft  
Tetrahedron Letters 389 (1966)
- "1,3-Cycloadditionen von Azomethin-Yliden aus Aziridin-Carbonestern"  
R. Huisgen, W. Scheer, G. Szeimies, und H. Huber  
Tetrahedron Letters 397 (1966)
- "Zur 1,3-Dipolaren Cycloaddition Aromatischer Nitriloxide an Azodicarbonsäureester"  
R. Huisgen, H. Blaschke und E. Brunn  
Tetrahedron Letters 405 (1966)
- " $\Delta^3$ -Ox Diazoline-(1,3,4) Bei der Oxydation von Benzoyl-hydrazonen mit Bleitetraacetat"  
R. W. Hoffmann und H. J. Luthardt  
Tetrahedron Letters 411 (1966)
- "Synthesis of Hexaoxadiamantanes"  
O. Vogl and B. C. Anderson, and D. M. Simons  
Tetrahedron Letters 415 (1966)
- "Procyanidino-(-)-Epicatechin, ein Zweiarmlig Verknüpftes, Kondensiertes Proanthocyanidin aus Aesculus Hippocastanum"  
W. Mayer, L. Goll, E. Moritz von Arndt und A. Mannschreck  
Tetrahedron Letters 429 (1966)
- "The Structure of the Principal Alkaloid from Valeriana Officinalis(L.)"  
K. Torssell und K. Wahlberg  
Tetrahedron Letters 445 (1966)
- "2-Benzylamino-4-Benzylimino-2-Pentene. Evidence for Another Non-Classical Aromatic System Involving  $\pi$ -Electronic Interaction Through the Hydrogen Bond"  
L. C. Dorman  
Tetrahedron Letters 459 (1966)
- "Prostaglandins I - Total Synthesis of 9 $\beta$ ,15 $\xi$ -Dihydroxyprost-13-Enoic Acid"  
J. F. Bagli, T. Bogri and R. Deghenghi, and K. Wiesner  
Tetrahedron Letters 465 (1966)
- "Specific Deuteration  $\beta$  to A Keto Group Reduction of  $\alpha$   $\beta$ -Ethylenic Ketones with Lithium and Deuterated Propylamine"  
M. Fetizon and J. Gore  
Tetrahedron Letters 471 (1966)
- "Dimers of Perchloro-(3,4-Dimethylenecyclobutene) I, Perchloro-(3,4,7,8-Tetramethylene-Tricyclo[4.2.0.0<sup>2,5</sup>]<sub>1</sub>Octane)"  
K. Mano, K. Kusuda, A. Fujino and T. Sakan  
Tetrahedron Letters 489 (1966)
- "Hindered Rotation in 1,2-Dineopentyltetramethylbenzene"  
D. T. Dix, G. Fraenkel, H. A. Karnes and M. S. Newman  
Tetrahedron Letters 517 (1966)
- "The Synthesis and Valence Isomerization of 1,2-Diphenylcyclobutene"  
M. A. Battiste and M. E. Burns  
Tetrahedron Letters 523 (1966)
- "A Facile Phenol Synthesis. An Improved Route to 6-Methoxy-2-Tetralone"  
R. L. Kidwell and S. D. Darling  
Tetrahedron Letters 531 (1966)
- "A New Pyrrolizidine Aminoalcohol in Alkaloids from Crotalaria Species"  
C. K. Atal and K. K. Kapur, and C. C. J. Culvenor and L. W. Smith  
Tetrahedron Letters 537 (1966)
- "The Full Structures of Three Chromomycins, A<sub>2</sub>, A<sub>3</sub>, and A<sub>4</sub>"  
M. Miyamoto, Y. Kawamatsu, K. Kawashima, M. Shinohara, and K. Nakanishi  
Tetrahedron Letters 545 (1966)
- "The Structure of Nimbinic Acid"  
C. R. Narayanan and R. V. Pachapurkar  
Tetrahedron Letters 553 (1966)
- "Couplages a Distance Entre Protons Dioxanniques"  
J. Delmau and J. Duplan  
Tetrahedron Letters 559 (1966)
- "The Configuration and Formation of Percival Dianhydro-osazone"  
H. El Khadem and M. M. A. Abdel Rahman  
Tetrahedron Letters 579 (1966)
- "The Preparation of 1-O-Tosyl-( $\rightarrow$ )-Inositol from Quebrachitol"  
S. D. Gero  
Tetrahedron Letters 591 (1966)
- "The Structural Studies of Formycin and Formycin B"  
G. Koyama, K. Maeda and H. Umezawa, and Y. Iitaka  
Tetrahedron Letters 597 (1966)
- "Lithiation of N, N-Dimethyl- $\beta$ -Phenylethylamine"  
N. S. Narasimhan and A. C. Ranade  
Tetrahedron Letters 603 (1966)
- "The Structure of Leucotylic Acid, A New Triterpenic Acid from a Lichen"  
I. Yosioka, T. Nakanishi and E. Tsuda  
Tetrahedron Letters 607 (1966)
- "Pyxinic Acid, A Novel Lichen Triterpene with 3- $\beta$ -Hydroxyl Function."  
I. Yosioka, A. Matsuda and I. Kitagawa  
Tetrahedron Letters 613 (1966)
- "Mass Spectra of Caucalol Diacetate and Related Compounds"  
S. Sasaki, Y. Itagaki, H. Moriyama, K. Nakanishi  
Tetrahedron Letters 623 (1966)
- "A New Synthesis of Nebularine and 7-D-Ribofuranosylpurine (Synthesis in Nucleoside Antibiotics, Part I)"  
T. Hashizume and H. Iwamura  
Tetrahedron Letters 643 (1966)
- "Neu 1,3-Dipolare Cycloadditionen der Nitrilylide und eine Tautomerie bei Imidchlorlden"  
R. Huisgen und R. Raab  
Tetrahedron Letters 649 (1966)
- "Über das V lenzismere System 9,10-Dihydro-9,10-Athano-Naphthalin - 1,6-Athano-Cyclodecapentaen"  
E. Vogel, W. Maier und J. Eimer  
Tetrahedron Letters 655 (1966)
- "Glycozoline, A Carbazole Derivative from Glycosmis Pentaphylla (Retz) DC."  
D. P. Chakraborty  
Tetrahedron Letters 661 (1966)

"The Addition of Dimethylketen to some Heterocyclic Bases"  
R. N. Pratt, S. A. Procter, and G. A. Taylor  
Chem. Commun. 574 (1965)

"N.m.r. Solvent Shifts and the Conformations of  $\alpha\beta$ -Unsaturated Ketones"  
C. J. Timmons  
Chem. Commun. 576 (1965)

"The Photochemical Rearrangement of Benzylenol Ethers of  $\Delta^4$ -3-Oxo-steroids"  
J. T. Pinhey and K. Schaffner  
Chem. Commun. 579 (1965)

"A Novel Phosphorus(V)  $\rightarrow$  Phosphorus(III) Rearrangement: A Possible Example of Phenylphosphinidene as a Reaction Intermediate"  
M. J. Gallagher and I. D. Jenkins  
Chem. Commun. 587 (1965)

"The Structure of Kitol"  
B. V. Burger and C. F. Garbers  
Chem. Commun. 588 (1965)

"Conformations of the Bicyclo[3,2,2]non-2-ene Skeleton"  
C. W. Jefford, S. Mahajan and K. C. Ramey  
Chem. Commun. 616 (1965)

"Addition Reactions of Cyclopentadienylidene: Synthesis of Spiro[2,4]heptadienes"  
R. A. Moss  
Chem. Commun. 622 (1965)

"Tetrafluorotyrosine: Synthesis and Properties"  
R. Filler and H. H. Kang  
Chem. Commun. 626 (1965)

"Synthesis of 1,2-Anhydro-D-glucitol"  
E. Haslam and T. Radford  
Chem. Commun. 631 (1965)

"West African Timbers. Part XIV. The Structure of Turraeanthin, a Triterpene Monacetate from Turraeanthus africanus"  
C. W. L. Bevan, D. E. U. Ekong, T. G. Halsall, and P. Toft  
Chem. Commun. 636 (1965)

"The Bacterial Pigment from Pseudomonas lemmonieri; Structure of a Degradation Product"  
G. Ferguson, D. R. Pollard, J. Monteath Robertson, and D. M. Hawley, G. O. P. Doherty, N. B. Haynes, D. W. Mathieson, and W. B. Whalley  
Chem. Commun. 640 (1965)

"The Nenitzescu Indol Synthesis: Formation of Isomeric Indoles"  
G. R. Allen, Jr., C. Pidacks and M. J. Weiss  
Chem. Ind. (London) 2096 (1965)

"Elucidation of the Structures of the Sapogenins of Polygala Senega by a Direct Correlation with Medicagenic Acid"  
Y. Shimizu and S. W. Pelletier  
Chem. Ind. (London) 2098 (1965)

"Isomerisation of Diterpene Alkaloids Containing and Oxazolidine Ring"  
S. W. Pelletier, K. W. Gopinath and K. Kawazu  
Chem. Ind. (Lond.) 28, (1966)

"Di-tert.-Butyl Trioxide"  
N. A. Milas  
Chem. Ind. (London) 66 (1966)

"The Nenitzescu Indole Synthesis: A Reaction Mechanism"  
G. R. Allen, Jr. and M. J. Weiss  
Chem. Ind. (London) 117 (1966)

"(+)-N-Methyl-corydine, a new Quaternary Alkaloid from Fagara nigrescens Fries"  
A. M. Kuck  
Chem. Ind. (London) 118 (1966)

"Chemistry of Dibenzo(g,p)Chrysene"  
R. E. Erickson and D. Schnipp  
Chem. Ind. (London) 161 (1966)

"Spectromètre pour résonance paramagnétique électronique de grande sensibilité permettant l'observation simultanée de l'absorption et de la dispersion"  
M. Decailliot et J. Uebersfeld  
Compt. Rend. 262, Ser. B, 141 (1966)

"Réaction de carbènes de du réactif de Simmons-Smith sur quelques composés alléniques"  
Y. Vo-Quang, L. Vo-Quang, G. Emptoz et P. Savignat  
Compt. Rend. 262, Ser. C, 220 (1966)

"Nuclear Magnetic Resonance (Part II)"  
D. J. E. Ingram  
Contemp. Phys. 7, 103 (1965)

"Proton Nuclear Magnetic Resonance Studies of Ethylenediaminetetraacetic Acid and its Metal Complexes"  
R. J. Kula  
Dissertation Abstr. 26, 3009 (1965)

"A Study of the Reactions of Some Non-Metallic Binary Fluorides"  
Brother G. C. Demitras  
Dissertation Abstr. 26, 3024 (1965)

"The Chemistry and Transition Metal Complexes of 2,8,9-Trioxa-1-Phospha-Adamantane"  
T. J. Huttemann, Jr.  
Dissertation Abstr. 26, 3025 (1965)

"1,2,2-Paracyclophanes and Para-Xylylenes"  
H. S. J. Chow  
Dissertation Abstr. 26, 3034 (1965)

"An Investigation for Alkaloids in Charpentiera Obovata Gaud"  
T. R. Pattabhiraman  
Dissertation Abstr. 26, 3044 (1965)

"Studies in Nuclear Magnetic Double Resonance"  
J. M. Anderson  
Dissertation Abstr. 26, 3053 (1965)

"Nuclear Magnetic Resonance Studies of Calcium Boroaluminates Glasses and Nuclear Magnetic Resonance Studies of Diffusion in Glassy and Crystalline Lithium Borates"  
S. G. Bishop  
Dissertation Abstr. 26, 3435 (1965)

"The Analysis and Results of Nuclear Magnetic Resonance Investigations of Vitreous and Crystalline Borates"  
S. Greenblatt  
Dissertation Abstr. 26, 3439 (1965)

"O-Esters of Trinitromethane in the Reaction of 1,3-Dipolar Cycloaddition"  
V. A. Tartakovskii, I. E. Chlenov, G. V. Lagodzinskaya, and S. S. Novikov  
Doklady- Chem. Sect. (English Transl.) 161, 257 (1965)

"Ring Enlargement in the Photolysis of Phenyl Azide"  
W. von E. Doering and R. A. Odum  
Tetrahedron 22, 81 (1966)

"Photo-addition Reactions of Dimethyl Maleate and Dimethyl  
Acetylene Dicarboxylate with Norbornene"  
M. Hara, Y. Odaira and S. Tsutsumi  
Tetrahedron 22, 95 (1966)

"Studien in der Caranreihe—II. Über die Pyrolyse der  
Acetate von (+) (1R:4R:6S)-Caran-trans-4-ol und (+)  
(1R:4S:6S)-Caran-cis-4-ol; (+) (1R:6S)- $\Delta^4$ -Caren und  
(+) (1R:4R)-trans- $\Delta^2,8$ -p-Menthadien"  
K. Gollnick und G. Schade  
Tetrahedron 22, 123 (1966)

"Studien in der Caranreihe—III. Zur Darstellung des (+)  
(1R:6S)- $\Delta^4(10)$ -Caren I " $\beta$ -Caren";"  
K. Gollnick und G. Schade  
Tetrahedron 22, 133 (1966)

"Studien in der Caranreihe—IV. Über die Umlagerung von  
(—) (1R:4S:6S)- $\Delta^2$ -Caren-trans-4-OL in (—) (5S)-1-Methyl-  
5-( $\alpha$ -Hydroxyisopropyl)-Cyclohexa-1,3-Dien"  
K. Gollnick, G. Schade und S. Schroeter  
Tetrahedron 22, 139 (1966)

"Carbonyl  $C^{13}$  Chemical Shifts in Substituted Benzaldehydes"  
A. Mathias  
Tetrahedron 22, 217 (1966)

"Fulvenes and Thermochromic Ethylenes—XXXVI. NMR. Studies  
in the Series of 2,3,6,7-Dibenzheptafulvene and 2,3,6,7-  
Dibenzotropone"  
M. Rabinovitz, I. Agranat and E. D. Bergmann  
Tetrahedron 22, 225 (1966)

"Axial and Equatorial Thiols—II. 3 $\beta$ - and 3 $\alpha$ -Thiol Deriva-  
tives of 5 $\beta$ -Pregnan-20-one and 5 $\beta$ -Androstan-17-One"  
D. A. Swann and J. H. Turnbull  
Tetrahedron 22, 231 (1966)

"Determination of the Conformations of Epimeric 2,3-Diphenyl-  
3-Amino-1-Propanols. Simplified ABCX NMR Spectra"  
G. Fodor, R. E. Reavill, J. Stefanovsky, B. Kurtev, and  
H. J. Bernstein  
Tetrahedron 22, 235 (1966)

"The NMR Spectra of Taxinine and its Derivatives"  
M. C. Woods, K. Nakanishi, and N. S. Bhacca  
Tetrahedron 22, 243 (1966)

"Vitamin A Analogues—I. Synthesis of 4-Thia-Ionone"  
J. L. Baas, A. Davies-Fidder and H. O. Huisman  
Tetrahedron 22, 259 (1966)

"Vitamin A Analogues—II. Synthesis of 4-Thia-Vitamin A"  
J. L. Baas, A. Davies-Fidder, F. R. Visser and H. O. Huisman  
Tetrahedron 22, 265 (1966)

"Vitamin A Analogues—III. Determination of the Stereo-  
chemical Configuration of Some Polyenes of the 4-Thia-  
Vitamin A Series by NMR Spectrometry"  
P. K. Korver, C. Kruk, P. J. van der Haak, J. L. Baas, and  
H. O. Huisman  
Tetrahedron 22, 277 (1966)

"Vitamin A Analogues—IV. Attempted Synthesis of 2,4,4-  
Trimethyltetrahydrothiopyrone-3"  
J. L. Baas, A. Davies-Fidder and H. O. Huisman  
Tetrahedron 22, 285 (1966)

"Vitamin A Analogues—V. Synthesis of 9-, 13-, and 9,13-  
Desmethyl Homologues of Vitamin A"  
P. J. van den Tempel and H. O. Huisman  
Tetrahedron 22, 293 (1966)

"Contribution to the Chemistry of Cacalol"  
P. Joseph-Nathan, J. J. Morales and J. Romo  
Tetrahedron 22, 301 (1966)

"The Absolute Configuration of Thujane"  
G. Ohloff and G. Uhde, A. F. Thomas, and E. Sz. Kovats  
Tetrahedron 22, 309 (1966)

"The Structural Elucidation of Sitsirikine, Dihydrositsirikine  
and Isositsirikine. Three New Alkaloids from Vinca Rosea  
Linn"  
J. P. Kutney and R. T. Brown  
Tetrahedron 22, 321 (1966)

"Studies in the Chemistry of the Eremophilane Sesquiterpenes"  
L. H. Zalkow, A. M. Shaligram and Shih-En Hu, and C. Djerassi  
Tetrahedron 22, 337 (1966)

"Triterpenoids—I. The Structure of Isoaescigenin. Some  
Derivatives of Aescigenin"  
J. B. Thomson  
Tetrahedron 22, 351 (1966)

"Modified Steroid Hormones—XLII. Partial Synthesis of 17 $\alpha$ -  
Acetoxy-6-Acetoxyethylpregna-4,6-Diene-3,20-Dione and its  
2 $\alpha$ -Acetoxy Derivative, Two Acetylated Metabolites of  
Megestrol Acetate"  
G. Cooley, M. T. Davies, B. Ellis and V. Petrow  
Tetrahedron 22, 365 (1966)

"Modified Steroid Hormones—XLIII. The Stereochemistry of the  
Epimeric 17 $\alpha$ -Acetoxy-6-Hydroxy-6-Hydroxymethylpregn-4-Ene-  
3,20-Diones"  
D. Burn, G. Cooley, M. T. Davies, B. Ellis, V. Petrow and  
J. P. Yardley  
Tetrahedron 22, 369 (1966)

"Modified Steroid Hormones—XLIV. The Preparation and Stereo-  
chemical Analysis of Steroidal-2-Acetoxy-4,6-Dien-3-Ketones"  
F. K. Butcher, G. Cooley, M. T. Davies and V. Petrow  
Tetrahedron 22, 377 (1966)

"Untersuchung der indirekten Kernspinkopplung zwischen  
Protonen und Kohlenstoff-13 über mehrere Bindungen"  
E. Sackmann und H. Dreeskamp  
Spectrochim. Acta 21, 2005 (1965)

"The Conformation of Westphalen's Biol Diacetate"  
C. R. Narayanan and K. N. Iyer  
Tetrahedron Letters 285 (1966)

"Stereochemistry of the Michael Addition. An Interesting  
Solvent Effect"  
R. A. Abramovitch and D. L. Struble  
Tetrahedron Letters 289 (1966)

"The Structure of  $\beta$ -Sinensal"  
R. A. Flath, R. E. Lundin, and R. Teranishi  
Tetrahedron Letters 295 (1966)

"A Facile Ring Cleavage of 3-Aminothietane Dioxides"  
L. A. Paquette and M. Rosen  
Tetrahedron Letters 311 (1966)

"The Structure of  $\alpha$ - and  $\beta$ -Bourbonene, Sesquiterpenic Hydr-  
carbons from the Essential Oil Geranium Bourbon"  
J. Krepsinsky, Z. Samek and F. Sorm  
Tetrahedron Letters 359 (1966)

"The Structure of Ostreogrycin A"  
G. R. Delpierre, F. W. Eastwood, G. E. Gream, D. G. I.  
Kingston, P. S. Sarin, L. Todd and D. H. Williams  
Tetrahedron Letters 369 (1966)

"The Reaction of Oxymercurials with Active Methylene Compounds and the Demercuration of the Resulting Mercurials with Acid and Base"  
K. Ichikawa, O. Itoh, T. Kawamura, M. Fujiwara, and T. Ueno  
J. Org. Chem. 31, 447 (1966)

"Stereochemistry of the Bimolecular Metal Reduction of Benzaniline"  
J. J. Eisch, D. D. Kaska, and C. J. Peterson  
J. Org. Chem. 31, 453 (1966)

"Mechanism of Ozonation Reactions. II. Aldehydes"  
R. E. Erickson, D. Bakalik, C. Richards, M. Scanlon, and G. Huddleston  
J. Org. Chem. 31, 461 (1966)

"Reactions of Phosphorus Compounds. VII. A General Chain-Extension Reaction"  
E. E. Schweizer, L. D. Smucker, and R. J. Votral  
J. Org. Chem. 31, 467 (1966)

"The Constitution of Crude O,O-Dimethyl- and O,O-Diethyl-dithiophosphoric Acids Prepared from Phosphorus Pentasulfide"  
A. E. Lippman  
J. Org. Chem. 31, 471 (1966)

"Unsymmetrically Substituted  $\alpha$ -Diketones in the Oxyphosphorane Condensation.  $P^{31}$  and  $H^1$  Nuclear Magnetic Resonance Spectra"  
F. Ramirez, A. V. Patwardhan, and C. P. Smith  
J. Org. Chem. 31, 474 (1966)

"A Convenient Synthesis of Ester p-Tosylhydrazones and Studies of the Thermal Decomposition of Some of Their Sodium Salts"  
R. M. McDonald and R. A. Krueger  
J. Org. Chem. 31, 488 (1966)

"Solvolysis of  $\beta$ -Cyclopropylethyl p-Bromobenzenesulfonate"  
R. R. Sauer and R. W. Ubersax  
J. Org. Chem. 31, 495 (1966)

"Catalpa Glycosides. III. The Structure of Catalposide"  
J. M. Bobbitt, D. W. Spiggle, S. Mahboob, H. Schmid, and W. von Philipsborn  
J. Org. Chem. 31, 500 (1966)

"Photochemistry of Carbohydrate Derivatives. Photolysis of D-Galactose Diethyl Dithioacetal"  
D. Horton and J. S. Jewell  
J. Org. Chem. 31, 509 (1966)

"The Structure and Total Synthesis of Takatonine"  
S. Kubota, T. Masui, E. Fujita, and S. M. Kupchan  
J. Org. Chem. 31, 516 (1966)

"Stereochemistry of Enolization of 17-Keto Steroids"  
J. Fishman  
J. Org. Chem. 31, 520 (1966)

"The Oxidation of Steroidal Amines to Nitro Steroids"  
C. H. Robinson, L. Milewich, and P. Hofer  
J. Org. Chem. 31, 524 (1966)

"New Enynolic Acids from Acanthosyris. Structures and Chemistry"  
R. G. Powell, C. R. Smith, Jr., C. A. Glass and I. A. Wolff  
J. Org. Chem. 31, 528 (1966)

"Conformational Analysis. V. 2-Chloro- and 2-Bromotetrahydropyran"  
G. E. Both and R. J. Ouellette  
J. Org. Chem. 31, 544 (1966)

"Conformational Analysis. VI. 1-Vinylcyclohexanol"  
R. J. Ouellette, K. Liptak, and G. E. Both  
J. Org. Chem. 31, 546 (1966)

"Thiazine Derivatives. III. The Synthesis of Some 2-Substituted 5,6-Dihydro-1,3,4-thiazines and Tetrahydro-1,3-thiazines Related to Cepham"  
A. I. Meyers and J. M. Greene  
J. Org. Chem. 31, 556 (1966)

"The Structure of Couper's Compounds. Chemical Studies and  $P^{31}$  Nuclear Magnetic Resonance Spectra on Couper's Compound and Related Structures"  
A. G. Pinkus and P. G. Waldrep  
J. Org. Chem. 31, 575 (1966)

"Bridged Polycyclic Compounds. XXXII. The Proton Magnetic Resonance Spectra of Some Dibenzobicyclo[2.2.2]octadienes"  
S. J. Cristol, T. W. Russell, J. R. Mohrig and D. E. Plorde  
J. Org. Chem. 31, 581 (1966)

"Bridged Polycyclic Compounds. XXXIII. Thermal Rearrangement of Benzonorbornadiene"  
S. J. Cristol and R. Caple  
J. Org. Chem. 31, 585 (1966)

"2-Methyl-5,6-dihydro-1,4-dithiin and 2-Methyl-5,7-dihydro-5H-dithiepin from 2-Chloroallylthioalkanethiols"  
A. T. Bottini and E. F. Bottner  
J. Org. Chem. 31, 586 (1966)

"Conformational Analysis. VII. The Conformational Preference of the Nitro and Carboethoxyl Groups"  
R. J. Ouellette and G. E. Booth  
J. Org. Chem. 31, 587 (1966)

"1,3- and 3,4-Cycloaddition Reactions of 1,1-Diphenyl-1,3-butadiene with Tetracyanoethylene"  
J. J. Eisch and G. R. Husk  
J. Org. Chem. 31, 589 (1966)

"The Reaction of  $\Delta^2$ -Cyclohexenone with  $\beta$ -Methylallylmagnesium Chloride and Isobutylmagnesium Chloride. The Effect of Added Cuprous Chloride"  
L. Mandell and J. M. Brodmann  
J. Org. Chem. 31, 591 (1966)

"Ozonolysis of 1,2,3,4,7,7-Hexachlorobicyclo[2.2.1]hepta-2,5-diene"  
R. C. Slagel  
J. Org. Chem. 31, 593 (1966)

"Organic Disulfides and Related Substances. XV. Attempted Syntheses of Mercapto Disulfides"  
L. Field and H. K. Kim  
J. Org. Chem. 31, 597 (1966)

"The Isomerization and Disproportionation of 8-t-Butylchroman and Related t-Butyl phenyl Ethers"  
I. J. Borowitz and G. J. Williams  
J. Org. Chem. 31, 603 (1966)

"Chemistry of Carbon Diselenide. II. Alkylation of Substituted Selenoureas"  
J. S. Warner and T. F. Page, Jr.  
J. Org. Chem. 31, 606 (1966)

"Buxus Alkaloids. X. The Isolation and Constitution of Cyclovirobuxeine-B"  
S. M. Kupchan and G. Ohta  
J. Org. Chem. 31, 608 (1966)

"The Free-Radical Reaction of Ferrocene with Maleic Anhydride"  
R. E. Bozak  
J. Org. Chem. 31, 610 (1966)

"Proton Magnetic Resonance Spectra of Tetracyclines"  
M. S. von Wittenau and R. K. Blackwood  
J. Org. Chem. 31, 613 (1966)

"A Convenient Preparation of  $\gamma$ -Keto Acids"  
A. Takeda, K. Takahashi, S. Torii, and T. Moriwake  
J. Org. Chem. 31, 616 (1966)

"Decomposition of Ethyl Diazoacetate by a  $\pi$ -Allylic Palladium Chloride Complex"  
R. K. Armstrong  
J. Org. Chem. 31, 618 (1966)

"Trimethylsilyl Derivatives of Salicyclic Acid"  
E. G. Chozy, Jr., and M. B. Neuwirth  
J. Org. Chem. 31, 632 (1966)

"Desaimantation d'un Systeme de Spins Nucleaires Dilues  
Relaxation Spin-Reseau dans le Silicium Tres Pur"  
B. Sapoval et D. Lepine  
J. Phys. Chem. Solids 27, 115 (1966)

"Relaxation Nucleaire de  $^{29}\text{Si}$  A Basse Temperature par  
Impuretes Paramagnetiques Localisees"  
D. Jerome et J. M. Winter  
J. Phys. Chem. Solids 27, 129 (1966)

"A Nuclear Magnetic Resonance Study of Decomposition in  
Neutron-Irradiated LiF"  
C. D. Knutson, H. O. Hooper and P. J. Bray  
J. Phys. Chem. Solids 27, 147 (1966)

"A Note on the Analysis of the Line Shape of Nuclear Magnetic  
Resonance Dispersion; Relation Between Spin-Lattice  
Relaxation Time and Modulation Frequency"  
H. Nagasawa  
J. Phys. Soc. Japan 20, 1808 (1965)

"Nuclear Magnetic Resonance of  $\text{Co}^{59}$  in Ferromagnetic Cobalt  
Compounds  $\text{Co}_3\text{B}$  and  $\text{Co}_2\text{B}$ "  
T. Shinohara and H. Watanabe  
J. Phys. Soc. Japan 20, 2020 (1965)

"Polymers of Fluorocarbon Ethers and Sulfides. I. Tri-  
fluoromethyl Trifluorovinyl Ether and Sulfide"  
W. S. Durrell, E. C. Stump, Jr., G. Westmoreland and C. D.  
Padgett  
J. Polymer Sci. A3, 4065 (1965)

"Fluorothiocarbonyl Compounds. V. Polymerization of Thio-  
carbonyl Compounds"  
W. J. Middleton, H. W. Jacobson, R. E. Putnam, H. C. Walter,  
D. G. Pye, and W. H. Sharkey  
J. Polymer Sci. A3, 4115 (1965)

"Preparation and Characterization of Some Star- and Comb-  
Type Branched Polystyrenes"  
T. Altares, Jr., D. P. Wyman, V. R. Allen, and Meyersen  
J. Polymer Sci. A3, 4131 (1965)

"Self-locking Nuclear Resonance Magnetic Field Strength  
Meter for 11-65 Mc/s"  
H. R. Oppliger, P. Schmid and Hs. H. Günthard  
J. Sci. Instr. 42, 865 (1965)

"A Frequency Modulated Nuclear Resonance Search Oscillator"  
D. T. Edmonds  
J. Sci. Instr. 43, 63 (1966)

"A Study of the Wool-Water System by Means of Pulsed Nuclear-  
Magnetic-Resonance Techniques"  
L. J. Lynch and K. H. Marsden  
J. Textile Inst. Trans. 57, T1 (1966)

"Nuclear Spin-Lattice Relaxation and Molecular Association  
in Acetic Acid"  
B. N. Bhar  
Nuovo Cimento 40B, 416 (1965)

"Distortional Narrowing of NMR Rotary Saturation Lines"  
I. R. McDonald  
Physics Letters 19, 653 (1966)

"Spin Echoes from Broad Resonance Lines with High Turning  
Angles"  
W. B. Mims  
Phys. Rev. 141, 499 (1966)

"Nuclear Moments of  $\text{Sc}^{43}$  and  $\text{Sc}^{47}$ "  
R. G. Cornwell, W. Happer, Jr. and J. D. McCullen  
Phys. Rev. 141, 1106 (1966)

"Nuclear Moments of  $\text{Pm}^{147}$ "  
J. Reader  
Phys. Rev. 141, 1123 (1966)

"Fine Structure in Nuclear Resonance"  
R. A. Ferrell and W. M. MacDonald  
Phys. Rev. Letters 16, 187 (1966)

"Synthesis of Poly(arylene sulphones) by Polycondensation of  
Arylsulphonyl Chlorides under Friedel-Crafts Conditions"  
M. E. A. Cudby, R. G. Feasey, B. E. Jennings, M. E. B. Jones  
and J. B. Rose  
Polymer 6, 589 (1965)

"Reactivity Ratios for the Copolymerization of Acrylates and  
Methacrylates by Nuclear Magnetic Resonance Spectroscopy"  
N. Grassie, B. J. D. Torrance, J. D. Fortune and J. D.  
Gemmell  
Polymer 6, 653 (1965)

"The Physical Properties of Phospholipids. I. Solid State  
and Mesomorphic Properties of Some 2,3-Diacyl-DL-Phos-  
phatidylethanolamines"  
D. Chapman, P. Byrne and G. G. Shipley  
Proc. Roy. Soc. (London) Ser. A 290, 115 (1966)

"Chemia Cyjanku Karbonylu. XIII. Nowe Dane O Przebiegu  
Reakcji Cyjanku Karbonylu Ze Związkami Nienasyconymi.  
O Reakcji Cyjanku Karbonylu Z CIS I TRANS  $\alpha$ - $\beta$ -Dwumety-  
lostyrenami"  
O. Achmatowicz i K. Belniak  
Roczniki Chemii 39, 1685 (1965)

"Acylation of Streptomycin Series (1). Preparation and  
Characterization of Crystalline N-monoacetyldihydro-  
desoxystreptomycin Monosulfate"  
M. Katayama, T. Sato and H. Ikeda  
Sci. Papers Inst. Phys. Chem. Res. (Tokyo) 59, 153 (1965)

"Theory of Nuclear Magnetic Resonance in Ferrometals"  
L. L. Buishvili, N. P. Giorgadze, and G. A. Kharadze  
Soviet Physics—Solid State (English Transl.) 7, 2258 (1966)

"Pyrylium Salts Formed by Diacylation of Olefins—XVI. A  
Low-Melting Hydrate of 2,6-DI-n-Propyl-4-Methylpyrylium  
Perchlorate"  
A. T. Balaban, E. Romas and C. Rentia  
Tetrahedron 22, 1 (1966)

"Fluorocyclopentanes—IV. The Fluorination of Cyclopentane  
with Cobalt Trifluoride"  
A. Bergomi, J. Burdon, T. M. Hodgins, R. Stephens and J. C.  
Tatlow  
Tetrahedron 22, 43 (1966)

"Nitropyrroles—I. The Preparation and Properties of 2- and  
3-Nitropyrrole"  
K. J. Morgan and D. P. Morrey  
Tetrahedron 22, 57 (1966)

"Hydrogen Isotope Exchange in the Cyclopentadienyl Rings of Cobalticinium Compounds"  
D. N. Kursanov, V. N. Setkina, N. K. Baranetskaya, G. G. Dvoryantseva, and R. B. Materikova  
Doklady-Chem. Sect. (English Transl.) 161, 336 (1965)

"Solfanilammedi fluorurate. - Nota II. Trifluorometil-pirimidino-solfanilammedi"  
M. G. Biressi, M. Carissimi e F. Ravenna  
Gazz. Chim. Ital. 95, 1293 (1965)

"Steroidi conteneti funzioni azotate. - Nota II. Sintesi di 17-azido e 17-amino-5 $\alpha$ -pregnani e loro derivati"  
G. Nathansohn, G. Winters e A. Vigevani  
Gazz. Chim. Ital. 95, 1338 (1965)

"Sulla 1,2-difenil-3,5-dichetopirazolidina. - Nota VIII. Tautomeria e spettri di risonanza magnetica nucleare di alcuni derivati"  
R. Mondelli e L. Merlini  
Gazz. Chim. Ital. 95, 1371 (1965)

"Phenylphosphonitriles. II. Friedel-Crafts Reactions of 2,4,6-Trichloro-2,4,6-triphenyltriphosphonitrile with Benzene"  
B. Grushkin, M. Gali Sanchez, M. V. Ernest, J. L. McClanahan, G. E. Ashby, and R. G. Rice  
Inorg. Chem. 4, 1538 (1965)

"The Stereochemistry of Some Cobalt(III) Complexes with Ethylenediamine-N,N'-diacetic Acid and Some N-Substituted Analogs"  
J. I. Legg, and D. W. Cooke  
Inorg. Chem. 4, 1576 (1965)

" $\beta$ -Diketone Complexes of Cobalt(III). I. Synthesis and Spectroscopy of Bis(acetylacetonato)diaminecobalt(III) Salts"  
R. D. Archer and B. P. Cotsoradis  
Inorg. Chem. 4, 1584 (1965)

"The Fluorosulfuric Acid Solvent System. II. Solutions of Antimony Pentafluoride, Antimony Tetrafluoride Mono-fluorosulfate, and Antimony Pentafluoride-sulfur Trioxide Mixtures"  
R. C. Thompson, J. Barr, R. J. Gillespie, J. B. Milne, and R. A. Rothenbury  
Inorg. Chem. 4, 1641 (1965)

"The Preparation and Properties of Some Tetracoordinate Boron Compounds. The Pseudo-Metal Ion Concept"  
L. E. Toporcer, R. E. Dessy, and S. I. E. Green  
Inorg. Chem. 4, 1649 (1965)

"Polycyclic Group V Ligands. II. 2,6,7-Trioxa-1,4-diphosphabicyclo[2,2,2]octane and Derivatives"  
K. J. Coskran and J. G. Verkade  
Inorg. Chem. 4, 1655 (1965)

"Identification of Organosilanes by Nuclear Magnetic Resonance Spectroscopy"  
J. F. Hampton, C. W. Laceyfield, and J. F. Hyde  
Inorg. Chem. 4, 1659 (1965)

"Organosulfur Derivatives of the Metal Carbonyls. VIII. New Aspects of the Chemistry of Methylthio Derivatives of Iron Carbonyl"  
R. B. King and M. B. Bisnette  
Inorg. Chem. 4, 1663 (1965)

"Chloride Alkoxides of Pentavalent Tungsten"  
O. J. Klejnot  
Inorg. Chem. 4, 1668 (1965)

"Comments on the Reaction of Amines with 1,2-Dicarboclovododecaborane(12)"  
M. F. Hawthorne, P. A. Wegner, and R. C. Stafford  
Inorg. Chem. 4, 1675 (1965)

"The Reaction of Peroxydisulfuryl Difluoride with Anhydrides of Certain Perhalocarboxylic Acids to Form Perhaloacyl Fluorosulfates"  
Inorg. Chem. 5, 169 (1966)

"Phenylphosphonitriles. III. Structural Identification of Geometric Isomers of Tetrameric Phenylphosphonitrilic Chloride"  
B. Grushkin, A. J. Berlin, J. L. McClanahan, and R. G. Rice  
Inorg. Chem. 5, 172 (1966)

"The Descriptive Chemistry of Phosphorus Trioxide"  
J. G. Riess and J. R. Van Wazer  
Inorg. Chem. 5, 178 (1966)

"Studies of Ligand Exchange Reactions between Zirconium and Hafnium Chelates by Nuclear Magnetic Resonance"  
A. C. Adams and E. M. Larsen  
Inorg. Chem. 5, 228 (1966)

"Nuclear Magnetic Resonance Studies of Ligand Exchange for Some Group IVb  $\beta$ -Diketones"  
T. J. Pinnavaia and R. C. Fay  
Inorg. Chem. 5, 233 (1966)

"Proton Magnetic Resonance Study of  $\beta$ -Diketones and Its Application to Thermodynamic Formation Constants of Various  $\beta$ -Diketone Metal Chelates"  
R. L. Lintvedt and H. F. Holtzclaw, Jr.  
Inorg. Chem. 5, 239 (1966)

"Hydrogen Bonding to Metal  $\beta$ -Ketoenolates"  
T. S. Davis and J. P. Fackler, Jr.  
Inorg. Chem. 5, 242 (1966)

"Infrared and Proton Magnetic Resonance Spectra of Imidazole,  $\alpha$ -Alanine, and L-Histidine Complexes in Deuterium Oxide Solution"  
R. H. Carlson and T. L. Brown  
Inorg. Chem. 5, 268 (1966)

"Organonitrogen Derivatives of Metal Carbonyls. I. Reactions between Metal Carbonyl Anions and Haloalkyl Amines"  
R. B. King and M. B. Bisnette  
Inorg. Chem. 5, 293 (1966)

"Organonitrogen Derivatives of Metal Carbonyls. II. Arylazo Derivatives of Molybdenum"  
R. B. King and M. B. Bisnette  
Inorg. Chem. 5, 300 (1966)

"Organonitrogen Derivatives of Metal Carbonyls. III. Reactions between Metal Carbonyl Anions and Chloromethyl Isocyanate"  
R. B. King and M. B. Bisnette  
Inorg. Chem. 5, 306 (1966)

"Ennefluoro-2-(fluorosulfato)butane and Trifluoroacetyl Fluorosulfate"  
J. J. Delfino and J. M. Shreeve  
Inorg. Chem. 5, 308 (1966)

"Polycyclic Borazines"  
J. L. Boone and G. W. Willcockson  
Inorg. Chem. 5, 311 (1966)

"Interaction of Decaborane and Aqueous Ammonia"  
E. L. Muetterties and F. Klanberg  
Inorg. Chem. 5, 315 (1966)

"Reaction Product from Rhodium Trichloride and Acrylonitrile"  
K. C. Dewhirst  
Inorg. Chem. 5, 319 (1966)

"Preparation and Properties of the Monoborane Adduct of N,N,N'-Tetramethylethylenediamine"  
A. R. Gatti and T. Wartik  
Inorg. Chem. 5, 329 (1966)



"The Chemistry of the Euphorbiaceae. XIV. The Flavones from a Beyeria SP."  
R. M. Dawson, C. A. Henrick, P. R. Jefferies, and E. J. Middleton  
Australian J. Chem. 18, 1871 (1965)

"Nuclear Magnetic Resonance Spectra of Polycyclic Aromatic Compounds. II. Substituted Benz[a]Anthracenes"  
T. J. Batterham, L. Tsai and H. Ziffer  
Australian J. Chem. 18, 1958 (1965)

"Derivatives of Orthoacids. III. Sulphonamide Derivatives"  
G. Crank and F. W. Eastwood  
Australian J. Chem. 18, 1966 (1965)

"Convenient Preparation of a Crystalline Derivative of Meso-Tartraldehyde"  
S. J. Angyal and S. D. Gero  
Australian J. Chem. 18, 1973 (1965)

"Chemistry of Pyrrolic Compounds. II. Some Aspects of the Chemistry of Dipyrrolic Ketones"  
P. S. Clezy and A. W. Nichol  
Australian J. Chem. 18, 1977 (1965)

"The Alkaloids of Atherosperma Moschatum Labill. II. Non-Phenolic Alkaloids"  
I. R. C. Bick and G. K. Douglas  
Australian J. Chem. 18, 1997 (1965)

"Insect Venoms, Attractants, and Repellents. VII. A Stereospecific Synthesis of Synthesis of Iridodial"  
S. A. Achmad and G. W. K. Cavill  
Australian J. Chem. 18, 1989 (1965)

"The Chemistry of the Euphorbiaceae. XV. Further Diterpenes from Ricinocarpus Stylosus: 1a,19-Dihydroxy-16a-(—)-Kauran-17-OIC Acid"  
C. A. Henrick and P. R. Jefferies  
Australian J. Chem. 18, 2005 (1965)

"Chemical Studies of the Proteaceae. I. Grevillea Robusta A. Cunn. And Orites Excelsa R. Br."  
E. Ritchie, W. C. Taylor, and S. T. K. Vautin  
Australian J. Chem. 18, 2015 (1965)

"The Constituents of Melicope Broadbentiana F. M. Bail. The Structures of Melibentin, Melicopol, and Methylmelicopol"  
E. Ritchie, W. C. Taylor and S. T. K. Vautin  
Australian J. Chem. 18, 2021 (1965)

"Oxidation of 3-Carene with Mexoric Acetate"  
Z. G. Isaeva, B. A. Arbuzov, V. V. Ratner, and I. P. Povodyreva  
Bull. Acad. Sci. USSR, Div. Chem. Sci. (English Transl.) 450 (1965)

"Decomposition of Unsymmetrical Geminate Amines"  
R. G. Kostyanovskii and O. A. Pan'shin  
Bull. Acad. Sci. USSR, Div. Chem. Sci. (English Transl.) 549 (1965)

"N, N'-Methylene- and Benzylidenebisethylenimines"  
R. G. Kostyanovskii and O. A. Pan'shin  
Bull. Acad. Sci. USSR, Div. Chem. Sci. (English Transl.) 553 (1965)

"Photochemical Chlorination of Chloro(3,3,3-Trifluoropropyl)silanes and the Synthesis of Chloro(3,3,3-Trifluoropropenyl)silanes"  
M. A. Kadina and V. A. Ponomarenko  
Bull. Acad. Sci. USSR Div. Chem. Sci. (English Transl.) 633 (1965)

"N-Alkoxyethylethylenimines"  
R. G. Kostyanovskii and O. A. Pan'shin  
Bull. Acad. Sci. USSR Div. Chem. Sci. (English Transl.) 721 (1965)

"Synthesis and Transformations of Tribromogermene"  
V. F. Mironov and T. K. Gar  
Bull. Acad. Sci. USSR Div. Chem. Sci. (English Transl.) 740 (1965)

"The Interaction of Cumulene Systems with Organometallic Complexes III. Iron Carbonyl Complexes of Hexapentaene and of Tetra- and Dimethylbutatriene"  
A. Nakamura  
Bull. Chem. Soc. Japan 38, 1868 (1965)

"Studies of Hydrogenolysis. XLIX. The Selective Hydrogenolysis of  $\alpha$ ,  $\beta$ -Epoxyketones"  
S. Mitsui, Y. Senda, T. Shimodaira and H. Ichikawa  
Bull. Chem. Soc. Japan 38, 1897 (1965)

"Some Acetylene Complexes of Vanadium"  
R. Tsumura and N. Hagihara  
Bull. Chem. Soc. Japan 38, 1901 (1965)

"Applications de la Spectrographie de Résonance Magnétique Nucléaire (R.M.N.) dans le Domaine des Dérivés Polycycliques à Caractère Aromatique. X"  
R. H. Martin, R. Flammang et M. Arboui  
Bull. Soc. Chim. Belges 74, 418 (1965)

"On the N.M.R. Spectra of Methyltintrichloride Solution"  
E. V. Van den Berghe and G. P. Van der Kelen  
Bull. Soc. Chim. Belges 74, 479 (1965)

"Occurrence in Seed Oils of Some Fatty Acids with Conjugated Unsaturation"  
C. Y. Hopkins and M. J. Chisholm  
Can. J. Chem. 43, 3160 (1965)

"A Linear Correlation of Proton Chemical Shifts in Some Alkyl Chlorides and Hydrocarbons with the Occupation Number of the Hydrogen Orbitals"  
P. Hruska, G. Kotowycz and T. Schaeffer  
Can. J. Chem. 43, 3188 (1965)

"Desert Plant Constituents. II. Ocotillol: An Intermediate in the Oxidation of Hydroxy Isooctenyl Side Chains"  
E. W. Warnhoff and C. M. M. Halls  
Can. J. Chem. 43, 3311 (1965)

"Reactions of Nitro Sugars. II. The Conversion of Methyl 6-Deoxy-6-Nitro-Hexopyranosides into Deoxynitroinositols by Alkaline Glycoside Cleavage"  
H. H. Baer and W. Rank  
Can. J. Chem. 43, 3330 (1965)

"Nuclear Magnetic Resonance and Infrared Studies of Acylated Imidazolidinones and Imidazolidinethiones"  
R. Greenhalgh and M. A. Weinberger  
Can. J. Chem. 43, 3340 (1965)

"Syntheses with 5-Dibromomethyl- and 5-Formylpyrromethenes"  
A. Markovac and S. F. MacDonald  
Can. J. Chem. 43, 3364 (1965)

"Addition of Fluoroacetylenes to Group V Hydrides"  
W. R. Cullen, D. S. Dawson and G. E. Styan  
Can. J. Chem. 43, 3392 (1965)

"Demjanov Rearrangement of 9-Xanthenemethylamine"  
S. N. Alam and D. B. MacLean  
Can. J. Chem. 43, 3433 (1965)

"Substitutions en Serie Naphtalénique. II. Dérivés du Diméthoxy-2,7 Naphtalène"  
J.-C. Richer et Y. Pépin  
Can. J. Chem. 43, 3443 (1965)

"Compounds of Silicon. Part V. Octaisopropylsilsesquioxane, its Bromination, and <sup>1</sup>H Nuclear Magnetic Resonance Spectroscopy"

J. H. Bishop, J. M. E. Goldschmidt, R. A. Shaw, and A. R. Katritzky  
J. Chem. Soc. (Lond.) 7325 (1965)

"Compounds Related to The Steroid Hormones. Part XV. Proton Magnetic Resonance Spectra of 11-Oxo-steroids"

G. F. H. Green, J. E. Page, and S. E. Staniforth  
J. Chem. Soc. (Lond.) 7328 (1965)

"Polyfluorocycloalkenes. Part III. Reactions with Alcohols under Ionic Conditions to give Ethers"

A. B. Clayton, J. Roylance, D. R. Sayers, R. Stephens, and J. C. Tatlow  
J. Chem. Soc. (Lond.) 7358 (1965)

"Polyfluorocycloalkenes. Part V. Fluorination of Alkoxy-polyfluorocycloalkenes by Cobaltic Fluoride"

A. B. Clayton, R. Stephens and J. C. Tatlow  
J. Chem. Soc. (Lond.) 7370 (1965)

"Hydrido-complexes of Iridium(III) Stabilised by Tertiary Phosphines and Arsines"

J. Chatt, R. S. Coffey, and B. L. Shaw  
J. Chem. Soc. (Lond.) 7391 (1965)

"Alkaloid Biosynthesis. Part IX. The Ipecacuanha Alkaloids"

A. R. Battersby, R. Binks, W. Lawrie, G. V. Parry and B. R. Webster  
J. Chem. Soc. (Lond.) 7459 (1965)

"Ring- $\alpha$  Expansion and Fine Structure of 7,12-Dihydro-1-hydroxypleiadene-7,12-dione and Related Compounds. Trans-annular Reactions of 7-, 8-, and 9-Membered Rings Fused to Planar Systems"

M. E. C. Biffin, L. Crombie, and J. A. Elvidge  
J. Chem. Soc. (Lond.) 7500 (1965)

"Pentafluoroethyl Derivatives of Selenium"

N. Welcman and H. Regev  
J. Chem. Soc. (Lond.) 7511 (1965)

"Constituents of *Withania somnifera* Dun. Part IV. The Structure of Withaferin A"

D. Lavie, E. Glotter, and Y. Shvo  
J. Chem. Soc. (Lond.) 7517 (1965)

"New Observations on the Condensations of Malonic Acid with Naphthalene-1,8-diamine and *o*-Phenylenediamine"

R. F. Smith and M. M. Holmer  
J. Chem. Soc. (Lond.) 7531 (1965)

"Studies on Oxime Hydrochloride. Part III, Long Range Anisotropy Effect of Hydroxyimino Groups of Dioximes"

H. Saito and K. Mukada  
J. Mol. Spectr. 18, 355 (1965)

"1,3-Elimination-Rearrangement of 2-Acetoxy-6-bromocyclohexanones"

F. G. Bordwell and K. M. Wellman  
J. Org. Chem. 31, 351 (1966)

"The Synthesis of 2-Nitrocyclohexanone and the Reaction of Acetyl Nitrate with Cyclohexene"

A. A. Griswold and P. S. Starcher  
J. Org. Chem. 31, 357 (1966)

"Reactions of 14-Hydroxy-14-azadispiro[5.1.5.2]pentadec-9-ene-7,15-dione and Related Compounds. I. The 3,11-Dimethyl Derivative"

D. V. Nightingale and D. N. Heintz  
J. Org. Chem. 31, 361 (1966)

"Structure of the Isomers of 1,4-Dinitro-2,3-butanediol"

F. I. Carroll  
J. Org. Chem. 31, 366 (1966)

"Preparation of the 4-Iodo-1,2,3,4-tetramethylcyclobutenyl Cation. Ultraviolet and Nuclear Magnetic Resonance Spectra of Cyclobutenyl Cations in Methylene Chloride, Sulfuric Acid, and Liquid Sulfur Dioxide"

E. H. Gold and T. J. Katz  
J. Org. Chem. 31, 372 (1966)

"The Uranediol Rearrangement"

H. Hirschmann, F. E. Hirschmann, and A. P. Zala  
J. Org. Chem. 31, 375 (1966)

"Reactions with Base of 2-Propargylthioethanol and the 2-(2-Haloallylthio)ethanols"

A. T. Bottini and E. F. Böttner  
J. Org. Chem. 31, 385 (1966)

"Base-Induced Cyclization Reactions of Propargyloxopropanol and the 3-(2-Haloallyloxy)propanols"

A. T. Bottini and E. F. Böttner  
J. Org. Chem. 31, 389 (1966)

"1,3-Bis(fluoroxy)perfluoropropane and other Oxygen-Containing Compounds by Direct Fluorination"

J. H. Prager  
J. Org. Chem. 31, 392 (1966)

"Alkali Sensitivity of a Dinitro Compound in the Cyclopropane Series"

W. G. Brown and F. H. Greenberg  
J. Org. Chem. 31, 394 (1966)

"cis- and trans-1,3-Diphenylpropene"

E. K. Raunio and W. A. Bonner  
J. Org. Chem. 31, 396 (1966)

"Cyclopropylpyridines. Synthesis and Electronic Interaction"

A. P. Gray and H. Kraus  
J. Org. Chem. 31, 399 (1966)

"The Vapor Phase Rearrangement of Thioncarbonates and Thion-carbamates"

H. Kwart and E. R. Evans  
J. Org. Chem. 31, 410 (1966)

"The Thio-Claisen Rearrangement. The Mechanism of Thermal Rearrangement of Allyl Aryl Sulfides"

H. Kwart and E. Robert Evans  
J. Org. Chem. 31, 413 (1966)

"Observations Regarding the Mechanism of Olefin Epoxidation with Per Acids"

H. Kwart and D. M. Hoffman  
J. Org. Chem. 31, 419 (1966)

"Cycloheximide Transformations. I. Kinetics and Mechanisms in Aqueous Acid"

E. R. Garrett and Robert E. Notari  
J. Org. Chem. 31, 425 (1966)

"Stereochemical Course of the Robinson Annulation Reactions. cis-9-Hydroxy-10-methyldecalin-2,5-dione"

T. A. Spencer, H. S. Neel, D. C. Ward  
J. Org. Chem. 31, 434 (1966)

"Additivity of Chemical Shifts in the Decalin Ring System. Determination of Configuration of cis-9-Hydroxy-10-methyl-decalin-2,5-dione"

K. L. Williamson, L. R. Sloan, T. Howell, and T. A. Spencer  
J. Org. Chem. 31, 436 (1966)

"Hydrogen Chemical Shifts of Aliphatic Compounds in Carbon Tetrachloride, Trifluoroacetic Acid, and Trifluoroacetic Acid-Sulfuric Acid"

P. E. Peterson  
J. Org. Chem. 31, 439 (1966)

- "New Heteroaromatic Systems Derived from o-Formylthiophene-boronic Acids"  
S. Gronowitz and A. Bugge  
Acta Chem. Scand. 19, 1271 (1965)
- "Reduction of Conjugated Cyclopropyl Ketones with Lithium in Liquid Ammonia"  
T. Norin  
Acta Chem. Scand. 19, 1289 (1965)
- "Homologous Aliphatic C<sub>30</sub> — C<sub>45</sub> Terpenols in Birch Wood"  
B. O. Lindgren  
Acta Chem. Scand. 19, 1317 (1965)
- "NMR Studies and the Absolute Configuration of Solanum Alkaloids (Spiroaminoketal Alkaloids)"  
P. M. Boll and W. von Philipsborn  
Acta Chem. Scand. 19, 1365 (1965)
- "Nucleophilic Aromatic Substitution with Ethylenediamine"  
B. Lamm  
Acta Chem. Scand. 19, 1492 (1965)
- "Primary Hydrogen Isotope Effects in the Bromination of 2-Bromo-1,3,5-trimethoxybenzene and 2,4-Dibromo-1,3,5-trimethoxybenzene. Proximity Effects of Bromine"  
E. Helgstrand  
Acta Chem. Scand. 19, 1583 (1965)
- "Studies on Orchidaceae Alkaloids III. The Alkaloids in Dendrobium primulinum Lindl. and Dendrobium chrysanthum Wall"  
B. Lüning and K. Leander  
Acta Chem. Scand. 19, 1607 (1965)
- "The Addition of Grignard Reagents to Pyridazines. IV. t-Butylmagnesium Chloride and 3-Methoxy-6-phenylpyridazine"  
I. Crossland and L. Kjaergaard Rasmussen  
Acta Chem. Scand. 19, 1652 (1965)
- "Siphulin, a Chromenone Lichen Acid"  
T. Bruun  
Acta Chem. Scand. 19, 1677 (1965)
- "The Reaction of 5-Bromo- and 2-Bromopyrimidine with Organolithium Compounds"  
S. Gronowitz and J. Røse  
Acta Chem. Scand. 19, 1741 (1965)
- "Constituents of Umbelliferous Plants. VII. Coumarins from the Fruits of Peucedanum palustre L. The Structure of Two New Coumarins"  
B. E. Nielsen and J. Lemmich  
Acta Chem. Scand. 19, 1810 (1965)
- "Oxidative Conversion of Homovanillic Acid to a Fluorescent Compound"  
H. Corrodi and B. Werdinius  
Acta Chem. Scand. 19, 1854 (1965)
- "The Chemistry of the Order Araucariales. 3. Structure and Configuration of Araucarolone and Some Related Compounds from Agathis australis"  
C. R. Enzell and B. R. Thomas  
Acta Chem. Scand. 19, 1875 (1965)
- "3-Methyl-3-butenylglucosinolate, a New Isothiocyanate-Producing Thioglucoside"  
A. Kjaer and W. Wagnières  
Acta Chem. Scand. 19, 1989 (1965)
- "Proton Nuclear Magnetic Resonance Studies of Several Polyalcohols, Hydroxy Acids, and Derivatives of D-Gluconic Acid"  
D. T. Sawyer and J. R. Brannan  
Anal. Chem. 38, 192 (1966)
- "Isolation and Identification of C<sub>13</sub> to C<sub>17</sub> Alkyl-naphthalenes, Alkylbiphenyls, and Alkylidibenzofurans from the 275° to 305° C. Dinuclear Aromatic Fraction of Petroleum"  
F. Fu-Hsi Yew and B. J. Mair  
Anal. Chem. 38, 231 (1966)
- "Time-Averaged Proton Magnetic Resonance Analysis of Micro Samples from Open-Tube Gas Chromatographs"  
R. E. Lundin, R. H. Elsen, R. A. Flath, N. Henderson, T. R. Mon, and R. Teranishi  
Anal. Chem. 38, 291 (1966)
- "NMR-spektroskopische Untersuchungen zur Kinetik des Benzoloxyl-Oxepin-Gleichgewichtes"  
H. Gunther  
Angew. Chem. 77, 1083 (1965)
- "Die Autoxydation des 4,6-Di-tert.-butyl-resorcins"  
H. Musso und D. Maaben  
Ann. Chem. 689, 93 (1965)
- "Die Struktur der Ketipinsäure und ihrer Derivate"  
R.-D. Stachel  
Ann. Chem. 689, 118 (1965)
- "Synthese von Desoxyinosadiaminen"  
M. Nakajima, A. Hasegawa und T. Kurokawa  
Ann. Chem. 689, 229 (1965)
- "Synthese des 2-Desoxy-streptamins und des (±)-Hyosamins"  
M. Nakajima, A. Hasegawa und N. Kurihara  
Ann. Chem. 689, 235 (1965)
- "Synthese des Actinamins"  
M. Nakajima, N. Kurihara, A. Hasegawa und T. Kurokawa  
Ann. Chem. 689, 243 (1965)
- "Kernmagnetische Relaxation von Mehr-Spin-Systemen"  
D. Fenzke  
Ann. Physik 16, 281 (1965)
- "Protonenrelaxation in paramagnetischen Lösungen unter Berücksichtigung der Nullfeldaufspaltung"  
Von U. Lindner  
Ann. Physik 16, 319 (1965)
- "Proton Magnetic Resonance Spectra of Some  $\beta$ -Methylstyrenes,  $\beta,\beta$ -Dimethylstyrenes, and  $\alpha$ -Methylstilbenes; and a Discussion of Long-range Shielding and Coupling Effects in These and Related Systems"  
H. Rottendorf, S. Sternhell, and J. R. Wilmshurst  
Australian J. Chem. 18, 1759 (1965)
- "The Nuclear Magnetic Resonance Spectra of the Codeine Isomers and Their Derivatives"  
T. J. Batterham, K. H. Bell, and U. Weiss  
Australian J. Chem. 18, 1799 (1965)
- "A Proton Magnetic Resonance Study of Molecular Overcrowding in N-Alkyl-o-Nitroanilines"  
I. D. Rae  
Australian J. Chem. 18, 1807 (1965)
- "Aromatic Nitrogen Bridgehead Compounds. I. The Dipyrrolo [1,2-c:2',1'-e]imidazolium and Related Cations"  
I. C. Calder and W. H. F. Sasse  
Australian J. Chem. 18, 1819 (1965)
- "The Structure of Trichlorocodide"  
T. J. Batterham and U. Weiss  
Australian J. Chem. 18, 1863 (1965)

"Polyacetylenverbindungen, LXXXIX. Über ein neues Thiophen-furan-Derivat"

F. Bohlmann und C. Arndt  
Chem. Ber. 99, 135 (1966)

"Polyacetylenverbindungen, XC. Synthese des Thioäthers aus *Anthemis carpatica* Willd"

F. Bohlmann und A. Seyberlich  
Chem. Ber. 99, 138 (1966)

"Polyacetylenverbindungen, XCI. Struktur und Synthese eines  $C_{18}$ -Ketoacetats aus *Cosmos sulphureus* Cav."

F. Bohlmann, K.-M. Kleine und H. Bornoski  
Chem. Ber. 99, 142 (1966)

"Reaktionen von Diazoessigsäure-Äthylester mit Acetalen und Orthocarbonsäure-trialkylestern"

A. Schönberg und K. Praefcke  
Chem. Ber. 99, 196 (1966)

"Über das Bis-biphenyl-dichlor-diacetyl"

A. Schönberg und K. Praefcke  
Chem. Ber. 99, 205 (1966)

"Nickel(0)-dialkylcyanamid-carbonyl"

H. Bock und H. tom Dieck  
Chem. Ber. 99, 213 (1966)

"Über Aminomethylierungsprodukte von Methantricarbonsäureestern und ihre weiteren Umsetzungen"

H. Böhme und L. Häfner  
Chem. Ber. 99, 281 (1966)

"Cyansäureester als Dipolarophile bei 1,3-Cycloadditionen"

D. Martin und A. Weise  
Chem. Ber. 99, 317 (1966)

"Notiz über die Bildung von N-[Pyridyl-(4)],-pyridon-(4) aus 4-Allyloxy-pyridin-N-oxid"

B. S. Thyagarajan, K. K. Balasubramanian und R. Bhima Rao  
Chem. Ber. 99, 368 (1966)

"Cyclisierungen in der Pyridinreihe"

E. Winterfeldt  
Chem. Ber. 98, 3537 (1965)

"Photoreaktion von Cyclohexadien-(1,4)-dicarbonsäure-(1,2)-anhydrid mit Butin-(2)"

R. Askani  
Chem. Ber. 98, 3618 (1965)

"Reaktionen von  $\beta$ - und  $\gamma$ -Keto-carbonsäureestern mit Äthylendiaminen"

H. Baganz und S. Rabe  
Chem. Ber. 98, 3652 (1965)

"Cyclopentadienon-Äthylacetal"

E. Vogel und E.-G. Wyes  
Chem. Ber. 98, 3680 (1965)

"Über die Umsetzung von Bromdiacetyl sowie alicyclischen 1-Chlor-dionen-(2,3) mit primären und sekundären Aminen zu Reduktionen und einige Eigenschaften der Produkte"

H. Simon, G. Heubach, W. Bitterlich und H. Gleinig  
Chem. Ber. 98, 3692 (1965)

"Umsetzungen des Dimethyl-oxo-sulfoniummethylids mit Olefinen, Aromaten und Alkylierungsmitteln"

H. König, H. Metzger und K. Seelert  
Chem. Ber. 98, 3712 (1965)

"Reaktionen des Dimethyl-oxo-sulfoniummethylids mit Azomethinen, Azinen, Hydrazonen und Nitrilen"

H. König, H. Metzger und K. Seelert  
Chem. Ber. 98, 3724 (1965)

"Über neue, stabile Schwefel-Ylide"

H. König und H. Metzger  
Chem. Ber. 98, 3733 (1965)

"NMR- und IR-Spektren von Oxoniumsalzen"

F. Klages, J. E. Gordon und H. A. Jung  
Chem. Ber. 98, 3748 (1965)

"Synthese und Eigenschaften eines bicyclischen Oxoniumsalzes"

F. Klages und H. A. Jung  
Chem. Ber. 98, 3757 (1965)

" $\alpha$ -Rhodomycinon,  $\beta$ -Rhodomycinon, 10-Desoxy- $\gamma$ -rhodomycinon"

H. Brockmann, J. Niemeyer, H. Brockmann, Jr. und H. Budzikiewicz  
Chem. Ber. 98, 3785 (1965)

"Tetramethyl-, Dewar-phthalsäure"-dimethylester"

R. Criegee und F. Zanker  
Chem. Ber. 98, 3838 (1965)

"Vierringsynthesen durch photosensibilisierte Cycloaddition von Maleinsäureanhydrid an halogenierte Olefine"

R. Steinmetz, W. Hartmann und G. O. Schenck  
Chem. Ber. 98, 3854 (1965)

"Eine neue Synthese von arylsubstituierten 3-Aza-pyrylium-Salzen und ihre Umsetzung mit CH-aciden Verbindungen"

R. R. Schmidt  
Chem. ber. 98, 3892 (1965)

"Zur Anlagerung organischer Azide an winkelgespannte Doppelbindungen"

R. Huisgen, L. Möbius, G. Miller, H. Stangl, G. Szeimies, und J. M. Vernon  
Chem. Ber. 98, 3992 (1965)

"Einige Beobachtungen zur Addition organischer Azide an CC-Dreifachbindungen"

R. Huisgen, R. Knorr, L. Möbius, und G. Szeimies  
Chem. Ber. 98, 4014 (1965)

"Zerfallsreaktionen des Diazoessigsäure-allylesters"

W. Kirmse und H. Dietrich  
Chem. Ber. 98, 4027 (1965)

"A Stable Methylzirconium Compound"

J. R. Surtees  
Chem. Commun. 567 (1965)

"The Reaction of Tetraphenylallene with the Alkali Metals"

P. Dowd  
Chem. Commun. 568 (1965)

"The Adducts of Dimethylketen and 9-Methylacridine"

S. A. Procter and G. A. Taylor  
Chem. Commun. 569 (1965)

"A Photochemical Rearrangement involving Aminated Quinones"

D. W. Cameron and R. G. F. Giles  
Chem. Commun. 573 (1965)

"Apparatus for Measuring Nuclear Relaxation Time"  
R. K. Mazitov and V. I. Ionov  
Instr. Exp. Tech. (USSR) (English Trans.) 616, (1965)

"Nuclear Magnetic Resonance Study of Proton Exchange Involving Ion Pairs of Ammonium Salts in *t*-Butyl Alcohol"  
M. Cociver  
J. Am. Chem. Soc. 88, 672 (1966)

"Nuclear Magnetic Resonance Study of Salt Effects on the Proton Exchange of Ammonium Salts in *t*-Butyl Alcohol"  
M. Cocivera  
J. Am. Chem. Soc. 88, 677 (1966)

"Electronic Character of Metal-Anion Bonds"  
G. W. Parshall  
J. Am. Chem. Soc. 88, 704 (1966)

"Organometallic Chemistry of the Transition Metals. XIII. A  $\pi$ -Benzyl Derivative of Molybdenum with a Temperature-Dependent Proton Nuclear Magnetic Resonance Spectrum"  
R. B. King and A. Fronzaglia  
J. Am. Chem. Soc. 88, 709 (1966)

"Proton Magnetic Resonance Spectroscopy. Configurational Stability of Neohexyl(3,3-dimethylbutyl) Organometallic Compounds"  
M. Witanowski and J. D. Roberts  
J. Am. Chem. Soc. 88, 737 (1966)

"Nuclear Magnetic Resonance Spectroscopy. Analysis of the Proton Spectrum of 7,7-Dicyanonorcaradiene"  
C. Ganter and J. D. Roberts  
J. Am. Chem. Soc. 88, 741 (1966)

"Nitrogen-15 Magnetic Resonance Spectroscopy. VII. Chemical Shifts of *para*-Substituted Nitrobenzenes"  
D. T. Clark and J. D. Roberts  
J. Am. Chem. Soc. 88, 745 (1966)

"Proximity Effects. XLIV. Stereospecific Synthesis and Solvolysis of *cis*- and *trans*-5-Phenylcyclooctyl and *cis*- and *trans*-5-Phenylcyclooctyl-1,2,2,8,8- $d_5$  Tosylates"  
A. C. Cope and R. B. Kinnel  
J. Am. Chem. Soc. 88, 752 (1966)

"The Stereochemical Course of a Robinson-Schöpf Biogenetic-Type Reaction. The Conformation of Certain Tricyclic Tropane Congeners"  
L. A. Paquette and J. W. Heimaster  
J. Am. Chem. Soc. 88, 763 (1966)

"The Stereochemistry of  $\Delta^2$ -Thiolane Formation from Episulfides"  
J. R. Lowell, Jr., and G. K. Helmkamp  
J. Am. Chem. Soc. 88, 768 (1966)

"On the Mechanism of Iodination. The Addition of Iodine to 9,10-Dihydro-9,10-ethenoanthracene"  
D. D. Tanner and B. G. Brownlee  
J. Am. Chem. Soc. 88, 771 (1966)

"Reactions Proceeding by the [3.2.1] Bicyclic Path"  
M. S. Newman and C. Courduvelis  
J. Am. Chem. Soc. 88, 781 (1966)

"The Acid-Catalyzed Isomerization of  $\alpha$ - and *cis*- and *trans*- $\gamma$ -Methylallyl Alcohols"  
W. G. Young and J. S. Franklin  
J. Am. Chem. Soc. 88, 785 (1966)

"The Structure of the Cactus Sterol Macdougallin (14 $\alpha$ -Methyl- $\Delta^8$ -cholestene-3 $\beta$ ,6 $\alpha$ -diol). A Novel Link in Sterol Biogenesis"  
J. C. Knight, D. I. Wilkinson, and C. Djerassi  
J. Am. Chem. Soc. 88, 790 (1966)

"The Mitomycin Antibiotics. Synthetic Studies. XII. Indoloquinone Analogs with Variations at Positions 5 and 6"  
W. A. Remers and M. J. Weiss  
J. Am. Chem. Soc. 88, 804 (1966)

"Isolation, Structure, and Radiochemical Synthesis of 3,6-Dimethyl-4-hydroxy-2-pyrone"  
T. E. Acker, P. E. Brenneisen, and S. W. Tanenbaum  
J. Am. Chem. Soc. 88, 834 (1966)

"Sulfur Dioxide Insertion. II. S-Sulfinatopentacarbonylmanganese(I) Complexes"  
F. A. Hartman, and A. Wojcicki  
J. Am. Chem. Soc. 88, 844 (1966)

"Bicyclo[2.1.0]pent-2-ene"  
J. I. Brauman, L. E. Ellis, E. E. van Tamelen  
J. Am. Chem. Soc. 88, 846 (1966)

"Comments on the Factors Controlling Product Stereospecificity in the Reduction of Carbonyl Compounds with Alcohol Dehydrogenase and Diphosphopyridine Nucleotide"  
G. J. Karabatsos, J. S. Fleming, N. Hsi, and R. H. Abeles  
J. Am. Chem. Soc. 88, 849 (1966)

"A Novel Amination Reaction of Carboxylic Acid Derivatives with Tetrakis(dimethylamino)titanium"  
H. Weingarten, and W. A. White  
J. Am. Chem. Soc. 88, 850 (1966)

"Pyracycloquinone"  
B. M. Trost  
J. Am. Chem. Soc. 88, 853 (1966)

"The [1,1]Ferrocenophane System"  
W. E. Watts  
J. Am. Chem. Soc. 88, 855 (1966)

"Steroids. CCXCII. Synthetic Studies on Insect Hormones. II. The Synthesis of Ecdysone"  
J. B. Siddall, A. D. Cross, J. H. Fried  
J. Am. Chem. Soc. 88, 862 (1966)

"Methanolysis of 7-Chloronorbornadiene under Alkaline Conditions. Evidence for the Formation of a Labile Tricyclic Intermediate"  
H. Tanida, T. Tsuji, T. Irie  
J. Am. Chem. Soc. 88, 864 (1966)

"A New and Convenient Synthesis of Glyoxals, Glyoxalate Esters, and  $\alpha$ -Diketones"  
N. Kornblum, and H. W. Frazier  
J. Am. Chem. Soc. 88, 865 (1966)

"Prostaglandins in Human Seminal Plasma"  
M. Hamberg and B. Samuelsson  
J. Biol. Chem. 241, 257 (1966)

"Magnetic Susceptibility Anisotropy Studies on Osmocene: Correlation of  $\pi$ -Electron Ring Currents with Chemical Shifts and Bonding in Metallocenes"  
L. N. Mulay and V. Withstandley  
J. Chem. Phys. 43, 4522 (1965)

"Analysis of Carr-Purcell Spin-Echo NMR Experiments on Multiple-Spin Systems. I. The Effect of Homonuclear Coupling"  
A. Allerdaad  
J. Chem. Phys. 44, 1 (1966)

"Sign of  $J_{HF}$  in  $CH_2Cl_2$ "  
E. L. Mackor and C. MacLean  
J. Chem. Phys. 44, 64 (1966)

- "Configuration and Conformation of 2,3-Dichloro-1,4-Dioxane, M.P. 52°"  
R. R. Fraser and C. Reyes-Zamora  
Can. J. Chem. 43, 3445 (1965)
- "Reactions of Ethyldiazoacetate with Cyclopentadiene Synthesis of the Epimeric Ethyl Bicyclo[3.1.0]Hex-2-ene-6-Carboxylates"  
J. Warkentin, E. Singleton, and J. F. Edgar  
Can. J. Chem. 43, 3456 (1965)
- "Delphinium Alkaloids. The Structures of the Alkaloids of Delphinium Cardinale Hook and the Alkaloid Delatine"  
M. H. Benn  
Can. J. Chem. 44, 1 (1966)
- "Investigation of the Structure of 1,5-Methylene-Quinolinizinium Ion"  
O. E. Edwards, C. Fodor, and L. Marion  
Can. J. Chem. 44, 13 (1966)
- "Alkaloids of the Seeds of Aspidosperma Fendleri Woodson"  
R. H. Burnell and J. D. Medina  
Can. J. Chem. 44, 28 (1966)
- "Nuclear Magnetic Resonance Studies. VIII. Determination of the Thermodynamic Parameters Governing Associations of Substituted Benzaldehydes in Toluene Solution"  
R. E. Klinck and J. B. Stothers  
Can. J. Chem. 44, 37 (1966)
- "Nuclear Magnetic Resonance Studies. IX. The Chemical Shift of the Formyl Proton in Aliphatic Aldehydes"  
R. E. Klinck and J. B. Stothers  
Can. J. Chem. 44, 45 (1966)
- "Polyoxyphenols of Western Red Cedar (Thuja Plicata Donn) III. Structure of Plicatic Acid"  
J. A. F. Gardner, E. P. Swan, S. A. Sutherland, and H. McLean  
Can. J. Chem. 44, 52 (1966)
- "The Occurrence of 2,3-Dibromobenzyl Alcohol 4,5-Disulfate, Dipotassium Salt, in Polysiphonia Lanosa"  
J. H. Hodgkin, J. S. Craigie, and A. G. McInnes  
Can. J. Chem. 44, 74 (1966)
- "Selective Nucleophilic Substitution and Preferential Epoxide Formation"  
A. J. Dick and J. K. N. Jones  
Can. J. Chem. 44, 79 (1966)
- "Structures of Chlorobium Chlorophylls (660)"  
A. S. Holt, J. W. Purdie, and J. W. F. Wasley  
Can. J. Chem. 44, 88 (1966)
- "Synthesis and Nuclear Magnetic Resonance Spectrum of 3',4',6'-Tri-O-Acetyl-2-(Methylthio)-D-Glucopyrano[2',1':4,5]-2-Thiazoline"  
T. Ito  
Can. J. Chem. 44, 94 (1966)
- "Reactions of Amides and Related Compounds. III. N.M.R. Investigation of the Protonation of N,N-Dimethylnitrosoamine, N,N-Diethylnitrosoamine, and N-Nitrosopiperidine"  
S. J. Kuhn and J. S. McIntyre  
Can. J. Chem. 44, 105 (1966)
- "Organophosphorus-Uranyl Complexes Studied by Nuclear Magnetic Resonance Spectroscopy"  
J. L. Burdett and L. L. Burger  
Can. J. Chem. 44, 111 (1966)
- "Proton Magnetic Resonance Line Width as a Function of Polymer Tacticity"  
S. Brownstein and D. M. Wiles  
Can. J. Chem. 44, 153 (1966)
- "Reactions of Organotin Compounds. VI. The Reactions of Tin Hydrides with Perfluorovinyl Tin Compounds"  
A. D. Beveridge, H. C. Clark, and J. T. Kwon  
Can. J. Chem. 44, 179 (1966)
- "The Proton Magnetic Resonance Spectra and Tautomeric Equilibria of Aldoses in Deuterium Oxide"  
R. U. Lemieux and J. D. Stevens  
Can. J. Chem. 44, 249 (1966)
- "The Oxidation of Phenols. II. The Oxidation of 2,4-Di-*t*-Butylphenol with Peroxy R Radicals"  
E. C. Horswill and K. U. Ingold  
Can. J. Chem. 44, 269 (1966)
- "Stereochemical Studies. V. Rearrangements Accompanying Attempts to Epimerize 4-Oxa-3-Oxo-5 $\alpha$ -Cholestane and 4-Oxa-3-Oxo-5 $\beta$ -Cholestane by Brewster and Kugera's Method"  
W. H. Lunn and J. T. Edward  
Can. J. Chem. 44, 279 (1966)
- "Sulfonate Esters of  $\alpha$ -Chloroaldoximes, Aldoximes, and Amidoximes Via "Sulfene" Addition"  
W. E. Truce and A. R. Naik  
Can. J. Chem. 44, 297 (1966)
- "The Preparation of 5-Alkylthio, Branched Tryptamines"  
J. K. Horner, J. I. DeGraw, and W. A. Skinner  
Can. J. Chem. 44, 307 (1966)
- "A General Method for Selective N-Methylation of Substituted Tryptamines"  
J. K. Horner and W. A. Skinner  
Can. J. Chem. 44, 315 (1966)
- "The Nuclear Magnetic Resonance Spectrum of Vinylene Carbonate"  
K. A. McLaughlan and T. Schaeffer  
Can. J. Chem. 44, 321 (1966)
- "Kinetic Hydrogen Isotope Effects in Aromatic Bromodeprotonation"  
B. T. Baliga and A. N. Bourns  
Can. J. Chem. 44, 379 (1966)
- "Organic Sulfur Mechanisms. III. A Brief Investigation into the Reaction of Benzonitrile Oxide with Sulfenes"  
J. F. King and T. Durst  
Can. J. Chem. 44, 409 (1966)
- "Concerning the Reaction Between Maleic Anhydride and Indene"  
N. S. Isaacs  
Can. J. Chem. 44, 415 (1966)
- "Nuclear Spin Relaxation in Gaseous H<sub>2</sub> at Low Densities"  
W. N. Hardy  
Can. J. Phys. 44, 265 (1966)
- "Eine einfache Synthese der Norcaryophyllensäuren"  
H.-D. Scharf und F. Korte  
Chem. Ber. 98, 3672 (1965)
- "Synthesen von 1,3-Dienen über die Photoaddition von Olefinen an Halogenolefine, II. 3-[Indenyl-(2)]-cumarine"  
C. H. Krauch und W. Metzner  
Chem. Ber. 99, 88 (1966)
- "Synthesen von Heterocyclen, 78. Die Gewinnung von  $\beta$ -Lactamen durch Umsetzung Schiffischer Basen mit Cyanessigsäurehalogeniden"  
E. Ziegler und T. Wimmer  
Chem. Ber. 99, 130 (1966)

"Morellic, Isomorellic and Gambogic Acids"  
C. G. Karanjgaonkar, P. Madhavan Nair and K. Venkataraman  
Tetrahedron Letters 687 (1966)

"The Acylation of Enolate Anion by Acid Halides and Dimethylketene"  
K. Yoshida and Y. Yamashita  
Tetrahedron Letters 693 (1966)

"The Structure of Saikogenins A, B, C and D, Triterpenoid Alcohols of Bupleurium Falcatum L."  
T. Kubota, F. Tonami, and H. Hinoh  
Tetrahedron Letters 701 (1966)

"Nuclear Magnetic Resonance Spectra of N-Nitrosourethans"  
R. A. Moss  
Tetrahedron Letters 711 (1966)

"Konformative Beweglichkeit Flexibler Ringsysteme. Untersuchungen Mit Hilfe der Protonenresonanzspektroskopie"  
S. Kabuß, A. Luttringhaus und H. Friebolin, H. G. Schmid, and R. Mecke  
Tetrahedron Letters 719 (1966)

"O-Methylation of  $\beta$ -Dicarbonyl Compounds"  
R. Chong and P. S. Clezy  
Tetrahedron Letters 741 (1966)

"Zur Konjugation in Makrocyclischen Bindungssystemen III. Synthese und Eigenschaften von Tribenzocyclododekahexaen"  
H. A. Staab, F. Graf und B. Junge  
Tetrahedron Letters 743 (1966)

"Zur Konjugation in Makrocyclischen Bindungssystemen IV. Synthese und Eigenschaften von 1:2,5:6,9:10-Tribenzocyclododeke-1,5,9-Trien-3,7,11-Triin"  
H. A. Staab und F. Graf  
Tetrahedron Letters 751 (1966)

"Über die Pyrolyse von Aliphatischen Enolacetaten (1)"  
H. G. Hauthal, H. Schmidt  
Tetrahedron Letters 759 (1966)

"The Calculation of Directly Bonded  $^{13}\text{C}$ -H and  $^{13}\text{C}$ - $^{13}\text{C}$  Coupling Constants using Delocalized Molecular Orbital Theory"  
F. B. van Duijneveldt, V. M. S. Gil and J. N. Murrell  
Theoret. Chim. Acta 4, 85 (1966)

"Nuclear Magnetic Resonance Studies of Heterocyclic Fluorine Compounds. Part 1.—Oxazetidines and Oxazines"  
J. Lee and K. G. Orrell  
Trans. Faraday Soc. 61, 2342 (1965)

"Bis- $\pi$ -cyclopentadienyl-titan-mercaptide"  
H. Köpf und M. Schmidt  
Z. Anorg. Allgem. Chem. 340, 139 (1965)

"Zur Reaktion von N-Carboäthoxy-azepinen mit Metallhexacarbonylen und Eisenpentacarbonyl"  
Von E. O. Fischer und H. Mühle  
Z. Anorg. Allgem. Chem. 341, 137 (1965)

"Zur Kenntnis des Methyl difluorophosphins"  
F. Seel, K. Rudolph und R. Budenz  
Z. Anorg. Allgem. Chem. 341, 196 (1965)

"Hochauflöste Protonenresonanzspektren mit direkter magnetischer Dipol-Dipol-Wechselwirkung, Teil III"  
G. Englert und A. Saupe  
Z. Naturforsch. 20a, 1401 (1965)

"Kernmagnetische Relaxation und Korrelation im System Wasser - Sauerstoff"  
R. Hausser und F. Noack  
Z. Naturforsch. 20a, 1668 (1965)

"Die Medienabhängigkeit der Protonenresonanz von Tetramethylsilan, Benzol und Chloroform: Suszeptibilitätseffekt und Wasserstoffbrücken-Verchiebung"  
H. J. Friedrich  
Z. Naturforsch. 20b, 1021 (1965)

"NMR-spektroskopische Untersuchungen an Benzylisochinolin-Derivaten"  
L. Pohl und W. Wiegreb  
Z. Naturforsch. 20b, 1032 (1965)

" $^{31}\text{P}$ -Kernresonanzdaten der Alkali-diphenylphosphide"  
E. Fluck und K. Issleib  
Z. Naturforsch. 20b, 1123 (1965)

"Theorie der Multipolrelaxation. II Bewegungsgleichungen bei beliebigem Spin"  
H. Schwegler  
Z. Physik 189, 163 (1966)

"Theorie der Multipolrelaxation. III. Behandlung von Experimenten der reinen Quadrupol-Resonanz"  
H. Schwegler  
Z. Physik 189, 176 (1966)

"Advances in Magnetic Resonance"  
J. S. Waugh, ed.  
Academic Press, N. Y. 1965

"Nuclear Magnetic Resonance in a Flowing Liquid"  
A. I. Zhernovio, and G. D. Latyshev  
Consultant Bureau, N. Y. 1965