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Mailed:
March 29, 1963

Monthly Ecumenical Letters from Laboratories Of N-M-R

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DEADLINE FOR NEXT ISSUE
April 25, 1963

Una coleccion mensual de reportes privados y informales de los laboratorios de Resonancia Magnetica Nuclear.

La informacion aqui contenida es solamente para el uso del lector. Citarla no es permitido, excepto por arreglo directo con el autor del reporte, y el material citado debe ser referido como una "comunicacion privada".

NUCLEAR MAGNETIC RESONANCE SPECIALTIES
INCORPORATED

145 Greensburg Road New Kensington, Pa. Area Code 412 337-7727

March 22, 1963

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

Dear Barry:

Our engineering staff has recently been compelled to solve a rather common N.M.R. problem, namely, cooling our A-60 magnet system.

Other spectrometer owners may have, as we have, unfit raw water for cooling purposes. As an aid to other A-60 owners and prospective owners we will offer the following:

(1) Our engineering data is available for those interested in building their own system. (including complete parts list)

(2) We would consider supplying some of the parts that may be difficult to obtain in some areas.

(3) We would consider building the systems - ready for installation on special order.

I am hopeful this contribution to Mellonmr will keep me on your mailing list.

Sincerely,

Paul J. Yajko
Director

PJY/ams

The High Resolution Magnetic Double Resonance Spectra of F^{19} and Xe^{129} in XeF_4 and $XeOF_4$.

Thomas H. Brown, E. B. Whipple, and Peter H. Verdier

Union Carbide Research Institute
Tarrytown, New York

The F^{19} high resolution magnetic resonance spectra of XeF_4 and $XeOF_4$ have been measured in anhydrous hydrofluoric acid. Both show field-independent splittings due to Xe^{129}-F^{19} coupling and the absence of rapid chemical exchange. No Xe^{131}-F^{19} splitting is apparent in either case, and line intensity measurements require that this pattern be collapsed due presumably to the Xe^{131} quadrupole moment. Double resonance experiments demonstrate that the observed couplings are due to Xe^{129}, and show that four equivalent fluorine atoms are present in the molecules. They also serve to measure the Xe^{129} chemical shifts. XeF_4 has a Xe^{129}-F^{19} coupling constant of 3864 ± 10 cps and a F^{19} chemical shift, measured both at 56.44 and 15.00 Mc of 175.0 ± 1.5 ppm to lower field than the fluorine in the solvent HF. The Xe^{129} center frequency, 16.68703 ± 0.00003Mc, gives an estimated shift of -5817 ppm from atomic xenon. The oxyfluoride has a Xe^{129}-F^{19} coupling constant of 1163 ± 10 cps, an F^{19} shift of -291.6 ± 0.2 ppm from HF, and a xenon shift of -5540 ppm from atomic xenon.

4th OCEANS Abstracts

A limited number of copies of the Program and Abstracts of the recent 4th OCEANS (Omnibus Conference on the Experimental Aspects of Nuclear-magnetic-resonance Spectroscopy) held at Mellon Institute February 28 through March 2, 1963 inclusive are available on a first-come, first-served basis. It has been decided that the following price schedule will apply: until May 1, 1963 - $5.00, after May 1, 1963 - $2.00. These prices include postage. Checks, money orders, etc., should be made payable to "Treasurer, 4th OCEANS" and should accompany the order, which should be sent to the undersigned.

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

13 March 1963

KAMcL/DMT

Department of Scientific and Industrial Research
NATIONAL PHYSICAL LABORATORY
TEDDINGTON, Middlesex
Telegrams: Physics, Teddington Telephone: TEDdington Lock 3222, ext. 199

Please address any reply to
THE DIRECTOR
and quote:
Your reference:

BASIC PHYSICS DIVISION

11th March, 1963.

The absolute sign of the spin-spin coupling constant

Dear Dr. Shapiro,

I wish to report an experiment carried out by David Buckingham and myself to determine the absolute sign of the spin-spin coupling constant. The method used was that suggested by David in his recent paper (Trans.Far.Soc., 58, 2077, 1962). Its principle is to apply a large electric field to a sample whose N.M.R. spectrum is recorded simultaneously. This field causes the partial lining up of the electric dipoles in the sample (as in the Kerr effect) so that the nuclear magnetic dipole-dipole interaction is no longer averaged to zero but produces a splitting proportional to r^{-3} in the spectrum additional to that due to the isotropic spin-spin coupling. Depending upon the precise geometry of the experimental system and the molecule this extra splitting, which is proportional to the square of the electric field (E), either adds or subtracts from the spin-spin coupling. In our experiment the geometry was chosen to produce maximum effect, the electric field being applied parallel to the main magnetic field of the spectrometer.

We have studied two molecules so far: p-nitrotoluene at about 70°C and fluoroform at about -100°C. These molecules were chosen as having reasonably high dielectric constants and dipole moments whilst being easy to prepare in quite pure form so that the electric conductivity is low. In p-nitrotoluene the cross-ring coupling is small so that the ring proton spectrum approximates an AB system. With the line width available with our present apparatus (width at half-height ~ 3 cps) no other lines are seen. The observations were carried out on one half of the AB pattern and on applying the electric field a clear decrease was observed in the splitting between the peaks implying a positive J since the proton-proton direction is parallel to the electric dipole moment. Experiments with electric fields varying from ca. 8 Kv/cm to 25 Kv/cm confirmed that the splitting observed was proportional to E^2, in agreement with theory.

The result was finally checked by making a number of observations with fluoroform at one fixed field (ca. 10.5 Kv/cm). Without exception in some twenty traces a small increase (0.4 cps in 79.4 cps) in the peak splitting was noticed. Taken in conjunction with the molecular geometry, this confirms that the $H\backslash C/F$ coupling constant in this molecule is absolutely negative.

Further work is in progress to extend the range of these measurements.

It may be noted that these absolute signs are in agreement with the signs previously deduced from the assumption that the C^{13}-H coupling is positive.

Yours sincerely,

K.A. McLauchlan
Basic Physics Division

Dr. B.L. Shapiro,
Mellonmr,
Mellon Institute,
Pittsburgh, Pa.,
U.S.A.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
DEPARTMENT OF CHEMISTRY
CAMBRIDGE 39, MASSACHUSETTS

March 21, 1963

Dr. B. L. Shapiro
Mellon Institute
Pittsburgh, Pennsylvania

Dear Barry:

I hear through the Republican Underground that Keith McLauchlan has just written you about his elegant experiment to find the absolute sign of J. That makes this a good occasion for me to reinstate my subscription to MELLONMR by bringing your readers up to date on our own past and present activities in this direction.

For those who have not been following this work, a word of introduction is in order: It is common knowledge that the nuclear interactions of interest in magnetic resonance experiments in general depend on the orien - tation of the nuclear surroundings with respect to $\vec{H}_o$. Most of these interactions can be written in terms of second rank tensors. These include, for example,

a) the static dipolar interaction $\vec{I}_1 \cdot \left(\frac{r^2 - 3\vec{r}\vec{r}}{r^5}\right) \cdot \vec{I}_2$

b) the chemical shift $\vec{I} \cdot \overleftrightarrow{\sigma} \cdot \vec{H}_o$

c) the electron-coupled spin-spin interaction $\vec{I}_1 \cdot \overleftrightarrow{J} \cdot \vec{I}_2$

d) the quadrupole coupling $\overleftrightarrow{Q} : \overleftrightarrow{\nabla E}$

e) the spin-rotational interaction $\vec{I} \cdot \overleftrightarrow{C} \cdot \vec{J}$

f) the hyperfine coupling. $\vec{I} \cdot \overleftrightarrow{A} \cdot \vec{S}$, etc.

In looking for the parts of these interactions which give rise to static spectral shifts and splittings, one finds for all of them a scalar part B_1 (sometimes zero) and a traceless second-rank tensor part $\overleftrightarrow{B}_2$. In a liquid, the effects of $\overleftrightarrow{B}_2$ are averaged to zero because of the spherical weighting of the average and only the scalar part (trace) is observed directly. Equivalently it can be said that the factor $P_2(\cos\theta) = \frac{1}{2}(3\cos^2\theta - 1)$ appearing in the secular part of the nuclear Hamiltonian has zero average over a spherical probability distribution. All of the electric field experiments in liquids arise from the recognition that a nonspherical average, such as might be obtained for polar molecules in an electric field; yields a nonzero result. For a molecule with dipole moment $\vec{\mu}$ acted upon by an (applied plus polarization) field $\vec{E}$ making an angle α with $\vec{E}_o$ in the laboratory, the result may be made quite definite. If the polar axis for the angle θ above makes an angle β with $\vec{\mu}$, then

$$\left\langle P_2(\cos\theta)\right\rangle = \frac{2}{5}\left(\frac{\mu E}{kT}\right)^2 P_2(\cos\alpha)\, P_2(\cos\beta) + O\left(\frac{\mu E}{kT}\right)^4.$$

Wherever $P_2(\cos\theta)$ appears in formulas for nuclear splittings or shifts in crystals, one can simply replace it by the above average to predict the residual splitting or shift in a polarized liquid.

Experiments based on this idea have recently been proposed in print by at least three sets of authors: M. W. P. Strandberg [Phys. Rev. 127, 1162, (1962)], A. D. Buckingham and E. G. Lovering [Trans. Farad. Soc. 58, 2077 (1962)], and A. M. Vasil'ev (JETP 43, 1526 (1962)], but they have been informally bandied about for at least four years. In 1959 one of my students (F. Mannis, now working with E. R. Andrew) and I proposed to look for quadrupole splittings of the I^{127} resonance in liquid CH_2I (chosen for large μ, low freezing point, and large e^2qQ). The transition $m \rightarrow m-1$ in the NMR spectrum is expected to shift by an amount

$$\Delta\nu = \frac{3e^2qQ(2m-1)}{4I(2I-1)h}\left\langle P_2(\cos\theta)\right\rangle$$

which for this case might well be more than a megacycle. Preliminary NMR experiments showed the impracticability of this experiment, arising from enormous quadrupolar relaxation broadening. We were not clever enough at the time (although we have now become wiser) to realize that the experiment

would probably succeed if we chose a substance with a rather small e^2qQ. This is because the splitting goes as e^2qQ whereas the relaxation rate, being proportional to the square of a matrix element, goes as $(e^2qQ)^2$. We are now pursuing this subject further.

From the way I have introduced this class of experiments, the rest seems obvious, but that is hindsight. It was not until about 2 years ago that I thought of looking for the anisotropy of chemical shifts under similar conditions. Saika and Slichter [JCP 22, 26 (1954] have predicted theoretically an anisotropy of the order of 2000 ppm in the F^{19} shift of covalently bound fluorine, and very large anisotropy should occur for many other nuclei as well (but, alas, not for protons). Mr. J. D. Macomber and I have been looking for some time for the expected electric field effect in CHF_3 and CH_3F, and have recently obtained the first tentative experimental confirmation of its existence. Experimental refinements now nearing completion are expected to make it accurately measurable.

At the same time we have been aware of the prospect of other electric field shifts and splittings such as those indicated in the early part of this letter and are investigating some of them as well. What had not entered my mind was the elegant possibility of determining the absolute sign of J from the dipolar splittings--the experiment that McLauchlan has been working on. I remember feeling pretty foolish when he and Buckingham told me about it in England last September.

Our tentative result on the F^{19} shift anisotropy in CHF_3 was obtained in a non-spinning cell which has a copper parallel-plate condenser immersed in the liquid, with $\vec{E} \parallel \vec{E}_o$. The trouble so far (apart from the necessity of separating the shift from the change in J-splitting arising from dipolar coupling) is in the rather large diamagnetic inhomogeneities of such a cell. Our best line widths are > 10 cps. We are completing construction of a 5mm o.d. spinning cell in which the electric field is produced in a thin annulus. The surfaces are coated with a 4000 Å film of Sb-doped SnO_2, which conducts well enough to act as a condenser plate but poorly enough to allow RF field penetration. As might be expected the debugging of such a cell and its thermostatting system have taken some time.

We will keep you informed of further developments as they occur.

Yours,

John

J. S. Waugh
Professor

JSW:smt

Physikalisch-Chemisches und Elektrochemisches
Institut der Technischen Hochschule München
Direktor: o. Prof. Dr. G. SCHEIBE

Dr. Herbert Zimmermann

München 2,
Arcisstraße 21
Telefon: 5592/351

26. 2. 1963

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

Sehr geehrter Herr Shapiro !

In der vergangenen Zeit haben wir uns mit Untersuchungen über den Protonenaustausch zwischen Karbonsäuren und Alkoholen beschäftigt. Man beobachtet bei solchen Systemen stets ein gemeinsames Hydroxyl-Protonensignal für Karbonsäure und Alkohol. Die chemische Verschiebung des Signals ist eine monotone Funktion des Molenbruchs. Daraus geht hervor, daß rascher Protonenaustausch stattfindet und daß die Protonen in diesen Systemen über größere Strecken wandern können. Es ist plausibel anzunehmen, daß die Protonenübertragung über Ionen erfolgt. Die Existenz von Ionen ist durch Leitfähigkeitsmessungen sichergestellt.[1] Die Leitfähigkeit nimmt mit steigender Temperatur zu. Die Systeme verhalten sich demnach wie Protonenhalbleiter. Die prototropen Reaktionen in Karbonsäure-Alkohol-Gemischen lassen sich an Hand eines Termschemas diskutieren(siehe Abbildung). Jeder Term hat eine doppelte Bedeutung. Besetzte Terme entsprechen Protonenzustände von Brönsted-Säuren. Leere Terme sind als Defektprotonen (DP) aufzufassen und entsprechen den korrespondierenden Basen. Das Termschema kann aus den freien Enthalpien (ΔG) entsprechender prototroper Reaktionen konstruiert werden. In der Abbildung sind neben den Termen die korrespondiesernden Säure-Base-Paare aufgeführt. Den Protonenübergängen zwischen verschiedenen Termen sind prototrope Reaktionen zuzuordnen:

(1) $2\ ROH \rightleftharpoons \overset{(-)}{RO} + \overset{(+)}{ROH_2}$

(2) $2\ RCOOH \rightleftharpoons \overset{(-)}{RCOO} + \overset{(+)}{RC(OH)_2}$

(3) $RCOOH + ROH \rightleftharpoons \overset{(+)}{ROH_2} + \overset{(-)}{RCOO}$

Die Protonenübergänge werden durch Zufuhr von Wärmeenergie ermöglicht.

Eine energielose Protonenwanderung ($\Delta G = 0$) entlang der Ortskoordinate des gleichen Terms ist möglich durch Protonenübertragung zwischen den korrespondierenden Säure-Base-Paaren, z.B.

$$\overset{(+)}{ROH_2} + ROH \rightleftharpoons ROH + \overset{(+)}{ROH_2}$$

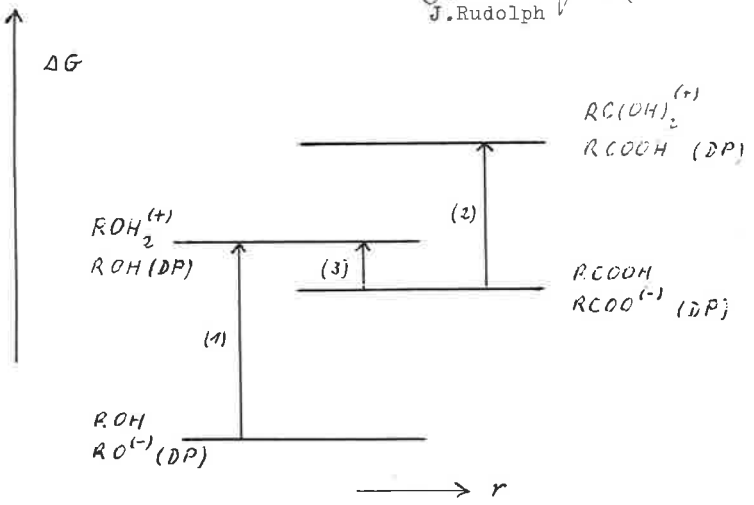
Mit diesen Vorstellungen in Einklang stehen Untersuchungen der Konzentrationsabhängigkeit der Leitfähigkeit. Während reine Alkohole und reine Säuren nur sehr geringe Leitfähigkeit zeigen, wird die Leitfähigkeit bei Säure-Alkohol-Gemischen erhöht. Reaktion (3) ist in diesen Systemen dominierend. Es werden mehr Ionen durch thermische Anregung gebildet als durch Reaktion (1) und (2) und die Leitfähigkeit ist erhöht.

Abschließend möchte ich die Gelegenheit benützen, Ihnen für die Übersendung der Mellon-Letters herzlich zu danken.

Mit freundlichen Grüßen

H. Zimmermann

J. Rudolph



1) S.S.Drutman, J.allg.Chem. (UDSSR) 25, 30 (1955)

COMMONWEALTH OF AUSTRALIA

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANIZATION

DIVISION OF COAL RESEARCH

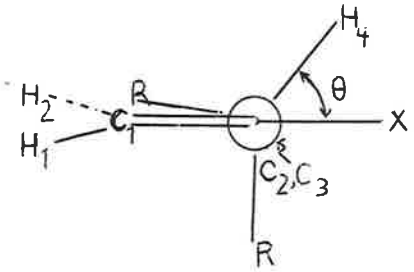
TELEGRAMS: FUELRES, SYDNEY
TELEPHONE: WW 0261
The Laboratory is situated in Delhi Road, North Ryde.

P.O. BOX 175,
CHATSWOOD, N.S.W.

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

Dear Dr. Shapiro ,

We have recently established[1,2] the steric dependence of allylic coupling (both cisoid 2:4 and transoid 1:4) on the magnitude of the angle θ , by examination of the NMR spectra of a number of compounds (mainly 6-substituted Δ^4-cholestene-3-ones) of reasonably well defined stereochemistry.



The main finding is that the coupling constant is at a numerical maximum (no sign implied , but presumed negative[3]) when θ is 90° and that it vanishes when θ is either 0 or 180°. This is in line with conformational studies[4,5,6] on acyclic derivatives and in agreement with theory[6,7]. To confirm our findings , I have been searching for examples of allylic and homoallylic coupling in the literature, especially that concerned with structural determinations on natural products. I have succeeded in finding some dozens of cases , but must have inevitably missed a great number.

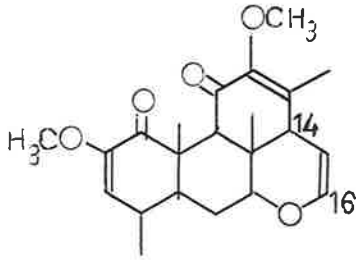
I would like to appeal to your readers to communicate to me any data of this nature: published coupling constants, spectra, odd unpublished data, samples of compounds with likely structures or just comments. To illustrate the sort of examples I have in mind , consider the structure of anhydroneoquassin (I)[8] where a large (3.1 c/s) allylic coupling was reported between H_{16} and H_{14}. Although the stereochemistry of quassins is not yet fully defined , θ must be at least 75° and most likely 90°. Similarly , with catalposide (II)[9] , the reported allylic coupling between H_3 and H_5 may help in elucidating the conformation.

I am pessimistic about being able to fit the J v. θ relation to any function[6] - we have only a 0-3 c/s range as against 0 - 12 c/s for the Karplus rule and also, the angles become a bit doubtful—but it should prove useful , nevertheless, because the usual choice is between stereochemistries with widely differing θ values.

Yours sincerely

(S. Sternhell)

1. Collins , Hobbs and Sternhell - Tetrahedron letters (in print)
2. Pinhey and Sternhell - Tetrahedron letters (in print)
3. Bishop - Annual Reports, 58 60 (1961)
4. Whipple , Goldstein and McClure - J.Am.Chem.Soc. 82 3811 (1960)
5. Bothner-By, Naar-Colin and Gunther - ibid. 84 2748 (1962)
6. Whipple - J.Chem.Phys., 35 1039 (1961)
7. Karplus - J.Chem.Phys. , 33 , 1842 (1960)
8. Carman and Ward - Aust.J.Chem. , 15 , 807 (1962)
9. Bobbitt, Spiggle, Mahboob, von Philisborn and Schmid , Tetrahedron letters 321 (1962)



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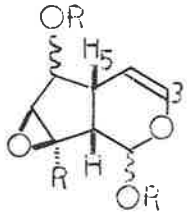


II

Dr. Hubert Schmidbaur
INSTITUT FÜR ANORGANISCHE CHEMIE
DER UNIVERSITÄT MARBURG
DIREKTOR: PROF. DR. MAX SCHMIDT

3550 MARBURG, 2. 3. 1963
Gutenbergstraße 18
Fernruf 731, Nebenstelle 3514
bei Durchwahl 733614

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

Sehr geehrter Herr Doktor Shapiro,

In unserem hiesigen Institut wird derzeit ein NMR-laboratorium installiert, das schon in kurzer Zeit in Betrieb genommen werden soll. Um dieses Laboratorium auch hinsichtlich der Literatur über NMR-spektroskopie komplett einzurichten, wären wir Ihnen sehr dankbar, wenn Sie uns in Zukunft regelmässig ein Exemplar Ihrer Mellon-NMR-letters übersenden könnten. Wir haben dankenswerterweise schon bisher im Institut für Anorganische Chemie der Universität München im dortigen NMR-laboratorium Zugang zu den Mellon letters gehabt und konnten dort auch seit längerer Zeit unsere Messungen ausführen. Aus unseren bisherigen Ergebnissen der Untersuchungen an Organoheterosiloxanen darf ich Ihnen noch kurz einige Beispiele anführen.

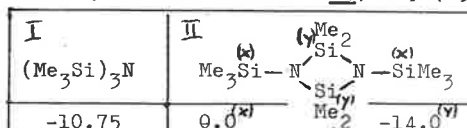
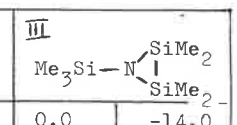
(1) (2)	(1) δ_{CH_3}	δ_{CH_2}	(2) J_{HCCH}	J/Δ	$J_{HC^{207}Pb}$	$J_{HCC^{207}Pb}$	$J_{HC^{119}_{117}Sn}$
$Me_3SiOSiMe_3$	-3.7	-3.7	-	-	-	-	-
$Me_3SiOGeMe_3$	0	-21.0	-	-	-	-	-
$Me_3SiOSnMe_3$	0	-21.0	-	-	-	-	57.2 54.3
$Me_3SiOPbMe_3$	+2.8	-73.0	-	-	69.5	-	-
$Me_3SiOSiEt_3$	0	-56.5	-31.3	8.0	0.32	-	-	-
$Me_3SiOGeEt_3$	0	-63.0	-50.7	8.0	0.65	-	-	-
$Me_3SiOSnEt_3$	+2.0	-74.0	-59.8	8.0	0.56	-	-	-
$Me_3SiOPbEt_3$	+5.0	-97.5	-10.5	8.0	0.59	42.0	169.0	-
$Me_3SiOSiEt_3$	0	-56.5	-31.3	8.0	0.32	-	-	-
$Me_3GeOSiEt_3$	-25.5	-59.0	-31.3	8.0	0.29	-	-	-
$Me_3SnOSiEt_3$	-24.0	-57.2	-27.5	8.0	0.27	-	-	57.2 54.4
$Me_3PbOSiEt_3$	-71.0	-54.5	-22.0	8.0	0.25	69.0	-	-

Tabelle 1: Chemische Verschiebungen und Kopplungskonstanten der PMR-spektren einiger Hexaalkylheterosiloxane.(Me=Methyl, Et=Äthyl, (1) für das linke, (2) für das rechte Element der Formeln).

Dr. H. Schmidbaur
INSTITUT FÜR ANORGANISCHE CHEMIE
DER UNIVERSITÄT MARBURG

Wir interpretieren die angegebenen Daten unter Berücksichtigung der konkurrierenden Faktoren Hybridisierung, Elektronegativität und $d_\pi p_\pi$-bindungsstabilisierung so, dass die O-X-bindung für X=Si,Ge,Sn,Pb zunehmend an polarem Charkter gewinnt und $d_\pi p_\pi$-bindungsanteile verliert, während gleichzeitig die π-bindungsanteile der angegliederten O-Si-bindung laufend zunehmen und bei den Plumbo-siloxanen den stärksten "Silanolat-charakter" erreichen. Die H-C-(C)-X-kopplungskonstanten beweisen dabei im Vergleich mit den analogen Werten bei den reinen Tetraalkylverbindungen die beachtliche Umhybridisierung des Restes R_3X von sp^3 in Richtung auf sp^2.

Im Zusammenhang mit Untersuchungen an verwandten Silazan-verbindungen haben wir auch die NMR-spektren einiger neuartiger Silazan-ringsysteme, die kürzlich von W. Fink, Zürich, dargestellt wurden(Angew. Chem. 73, 736 (1961), Helv. chim. Acta, im Druck), vermessen. Diese Spektren bestätigen nicht nur die unten formulierte Struktur dieser kleinen anorganischen Ringe, sondern lassen auch aus den Chemischen Verschiebungen deutlich die hohe $d_\pi p_\pi$-bindungsstabilisierung dieser Systeme erkennen, die die erstaunliche Stabilität dieser Verbindungen begründet. Zum Vergleich haben wir den Wert des Tris-Trimethylsilyl-amins beigefügt. Über ein zu II isostrukturelles Siloxalan-vierringsystem haben wir bereits berichtet. (J. Amer.chem.Soc.84,1069 (1962)).

Formel:	I $(Me_3Si)_3N$	II 	III 
Chem. Versch.:	-10.75	0.0(x) -14.0(y)	0.0 | -14.0
Intensitäten :	-	(x)6 : 4(y)	3 : 4

Alle Werte hier und in Tabelle 1 in (c/s) bei 60 Mc, alle Substanzen in CCl_4-lösung mit Konzentrationen von 5±1 %, gemessen gegen TMS als internem Standard vergleichbarer Konzentration, bei 23°C.
Die Ergebnisse werden an anderer Stelle ausführlicher diskutiert.
Wir danken Herrn Doz. Dr. H. P. Fritz für die Gelegenheit zu unseren Messungen auch an dieser Stelle sehr herzlich.

Mit freundlichen Grüssen
H. Schmidbaur
(H. Schmidbaur)

TEL. NO. 31751
JRSs/ET

School of Chemistry
THE UNIVERSITY
LEEDS 2

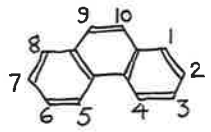
12th March, 1965

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

Dear Dr. Shapiro,

Our apologies for the lateness of this contribution.

Mr. K.D. Bartle and I have recently begun to study some simple and substituted polynuclear aromatic hydrocarbons, with particular reference to their detection and analysis in complex mixtures. One example is phenanthrene, the spectrum of which has yet to be completely assigned: we have observed some proton-shift effects in substituted phenanthrenes which may be of interest to HelloNMR readers. Figure 1 shows the low-field (aromatic) part of the NMR spectrum of phenanthrene and four substituted derivatives in approximately 10% solutions in CCl_4 recorded on the Varian A-60 spectrometer at the Bradford Institute of Technology. The lower field group of at least five lines in phenanthrene (fig.1a) (Jonathan, Gordon, and Dailey, J. Chem. Phys., 1962, 36, 2443) has been previously assigned to the 4,5 protons (Bernstein, Schneider, and Pople, Proc. Roy. Soc., 1956, A236, 515) and we have confirmed that in 4-methyl-phenanthrene the area ratio of the low to high-field groups in Fig.1b is 1:3 compared with 1:4 in phenanthrene. The introduction of the methyl group produces an extra low-field shift of the 5-proton of about 0.25 ppm. We have also observed what is probably a high-field shift of the 4-proton when a methyl (and ethyl) group is substituted at position 3 (fig.1c). The group of lines to the low-field side of the broad singlet at 496 c/s occurs at almost the same position and with almost the same separations as the low-field group in phenanthrene, and is therefore assigned to proton 5. The broad singlet is presumably proton 4: the adjacent methyl group not only produces a high-field shift of about 0.2 ppm but also removes the fine structure.



Substituents at the 9 and 10 positions also have interesting effects. Deliberate introduction of asymmetry, as in 9-methyl-10-ethyl phenanthrene, appears to split the 4,5 protons into two rather similar groups of lines (fig.1d): a similar splitting appears to occur in 9-methylphenanthrene, but here (fig.1e) one of the groups appears to overlap the remainder of the aromatic protons. However, the area ratios of the three groups of lines in fig.1d are about 1:1:2 (moving from low to high field), which suggests that one of the two low-field multiplets (at 475 c/s) is assignable to the 1,8 protons, that have been "moved out" of the high-field group by steric effects of the substituents.

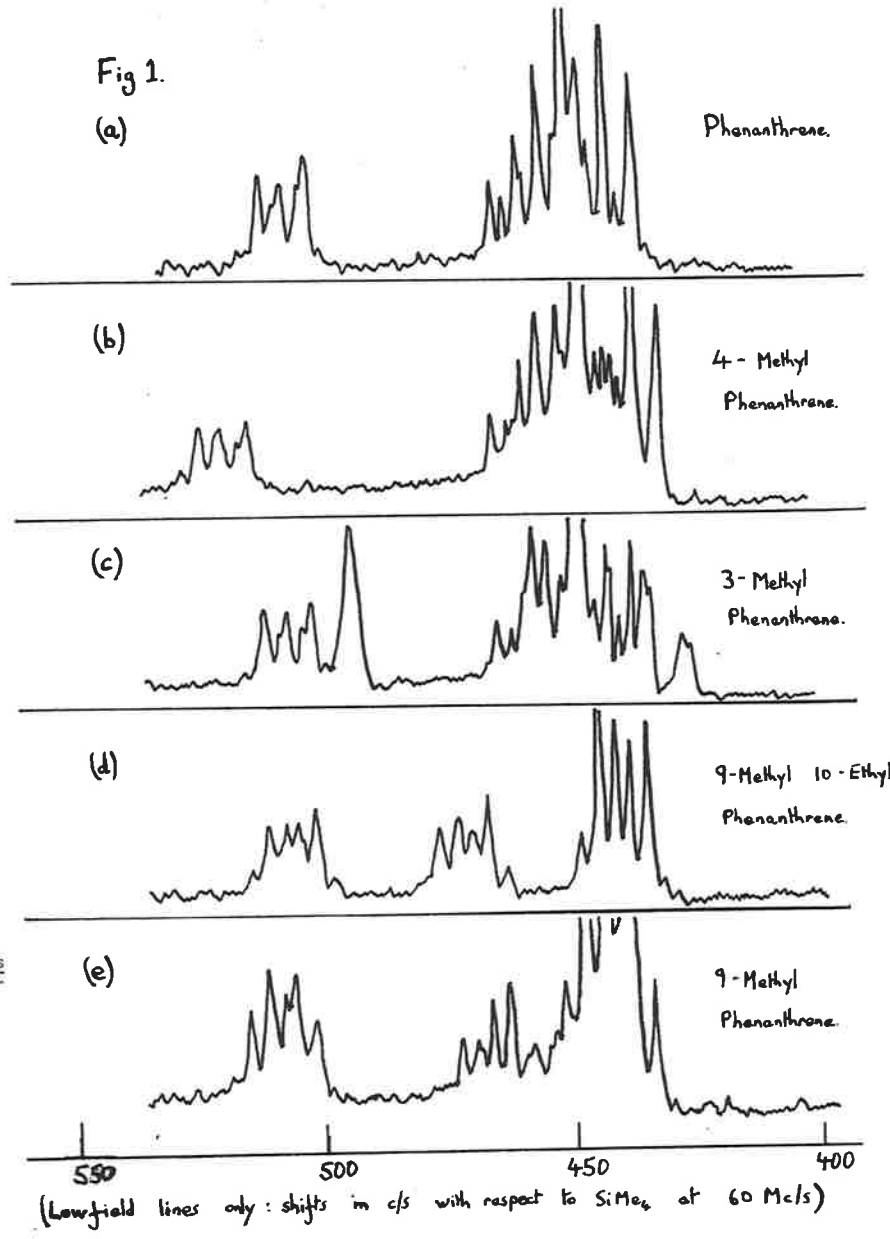
We hope by this kind of study to assist in the complete assignment of the phenanthrene spectrum.

Yours sincerely,

K.D. Bartle
(K.D. Bartle)

J.A.S. Smith
(J.A.S. Smith)

Fig 1.



(Low field lines only : shifts in c/s with respect to $SiMe_4$ at 60 Mc/s)

MONSANTO CHEMICAL COMPANY

800 NORTH LINDBERGH BOULEVARD
ST. LOUIS 66, MISSOURI

March 14, 1963

Dr. Robin K. Harris
Mellon Institute
4400 Fifth Avenue
Pittsburgh, Pennsylvania

Dear Dr. Harris:

I promised you I would send you a list of some of the simple phosphorus compounds on which we need P^{31} chemical shift data and, where pertinent, coupling constants. All of these compounds would be very useful in filling out our picture of P^{31} chemical shifts. In addition, it would be particularly valuable to obtain data on any mixed compounds involving a fluorine atom bonded directly to a phosphorus having an unshared pair of electrons or having a sulfur in the isolated (nonbridging) position. We have no good data on the cross terms in our equation for chemical shifts of mixed compounds for fluorine on a $:P\overset{\cdot}{-}$ or $SP\overset{\cdot}{-}$. Similarly, we would like to have any available data on compounds in which there is one or more hydrogens directly bonded to an $SP\overset{\cdot}{-}$ moiety.

If you know of any person or groups that might have the requested information, it would be greatly appreciated if you would pass these articles along. By the way, would it be possible to print this in Mellon's Monthly Ecumenical Letters put out by Dr. Bothner-by? I would greatly appreciate your finding this out for me.

Very sincerely,

John R. Van Wazer
Senior Scientist

Enclosure
cof

THE 37 MOST-WANTED N.M.R. CHEMICALS

$CH_3P(OCH_3)_2$

$CH_3P(OC_2H_5)_2$

$CH_3P(O)(OtC_4H_9)_2$

$CH_3P(S)(OCH_3)_2$

$C_2H_5P(OCH_3)_2$

$C_2H_5P(OC_2H_5)_2$

$C_2H_5P(O)(OCH_3)_2$

$C_2H_5P(S)(OCH_3)_2$

$C_2H_5P(S)(OC_2H_5)_2$

$(CH_3)_2CHP(O)Cl_2$

$(CH_3)_2CHP(O)(OC_2H_5)_2$

$C_6H_5P(OtC_4H_9)_2$

$C_6H_5P(S)(OCH_3)_2$

$C_6H_5P(S)(OC_2H_5)_2$

$C_6H_5P(S)(OC_6H_5)_2$

$C_6H_5P(O)(O\text{-iso-}C_3H_7)_2$

$C_6H_5P(O)(O\text{-t-}C_4H_9)_2$

$C_6H_5P(S)(O\text{-t-}C_4H_9)_2$

$(CH_3)_2P(OC_2H_5)$

$(CH_3)_2P(S)(OC_2H_5)$

$(CH_3)_2P(SC_2H_5)_2$

$(CH_3)_2P(O)(SC_2H_5)_2$

$(CH_3)_2P(S)(SC_2H_5)_2$

$(CH_3)_3PO$

$(CH_3O)_2P(O)Cl$

$(C_2H_5O)P(S)Cl_2$

$(C_2H_5O)P(S)(SC_2H_5)_2$

$[(CH_3)_2CH\text{-}S]_3PS$

$t\text{-}C_4H_9\text{-}S\text{-}PCl_2$

$(tC_4H_9O)_2P(S)H$

$(C_6H_5)_2P(S)H$

$[(iC_3H_7)_2N]PCl_2$

$[(t\text{-}C_4H_9)_2N]PCl_2$

$(C_2H_5O)_2P(SC_2H_5)$

$[(C_2H_5)_2N]P(O)Cl_2$

$(CH_3O)_2P[OC(CH_3)_3]$

$(C_2H_5O)_2P(O)[OC(CH_3)_2C\ N]$

TELEPHONE
34 0484 LMJ:BE

University of Melbourne
Department of Chemistry
Parkville, N.2, Victoria

15th March, 1963.

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

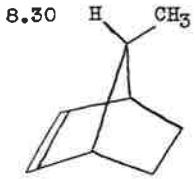
Dear Barry,

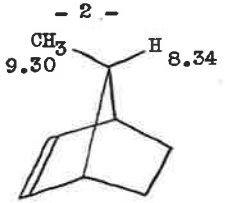
Now that the initial pandemonium of my move to Melbourne has subsided I find I have time to pay my subscription to M.E.L.L.O.N.M.R. which, incidently, seems to be going from strength to strength.

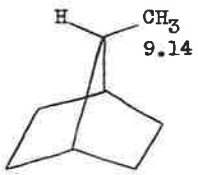
The recent paper by Pople (J. Chem. Phys., 1962, 37, 60) in which he predicts the principle susceptibilities for such systems as C=O and C=C has no doubt stimulated a number of people to reconsider the possibility of determining these quantities from the long range shielding contributions of such bonds to the chemical shifts of neighbouring protons in suitable model compounds. My own feeling is that this objective is still worthwhile but that a survey of many such systems is needed in order to adequately test the point dipole approximation. Accordingly, I would like to present some data which we have obtained in the hope that other readers will do likewise. If sufficient data becomes available it will then be worthwhile getting down to the task of analysing the results.

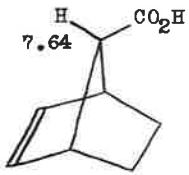
Our work in this field has been confined to the bicycloheptene and bicyclo-octene systems in which the geometry is well defined and conformational effects are minimal. The only conformational problem concerns freely rotating methyl groups which we feel can be adequately treated by the Narashiman and Roger's model (J. Chem. Phys., 1959, 31, 1302).

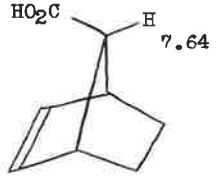
Our survey includes the following examples (τ-values):

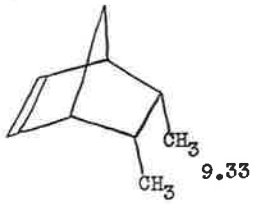


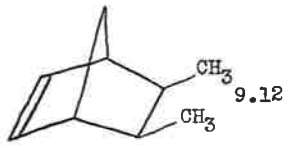


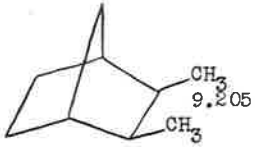


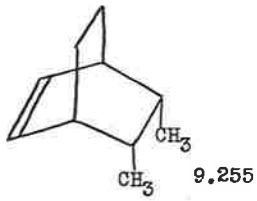


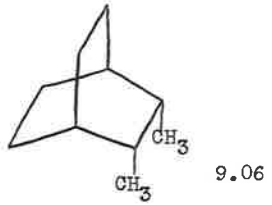












Consideration of these results indicates that the calculations will have to include terms arising from C-C as well as C=C anisotropies, and this is why much more data is required before one can hope to test the invariance of bond anisotropies and the accuracy of the point dipole approximation.

I hope all goes well with you and that it will not be too long before we have another opportunity of sharing a bottle of Chateau Lafitte. Give my best regards to your colleagues.

With best wishes,

Yours sincerely,

Lloyd.

LLOYD M. JACKMAN.

M onthly
E cumenical
L etters from
L aboratories
O f
N - M - R

A monthly collection of informal private letters from laboratories of NMR.
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CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE

INSTITUT DE CHIMIE DES SUBSTANCES NATURELLES
GIF-SUR-YVETTE (S.-&-O.)

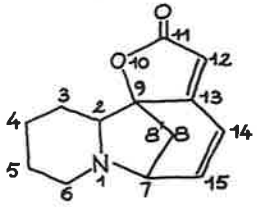
TEL. : 928 46-76

Dr. Aksel A. BOTHNER-BY
Mellon Institute
4400 fifth Avenue
PITTSBURGH, 13 Pennsylvania

Dear Dr. Bothner-By,

We apologize for not sending our contribution sooner and we hope that you will be merciful and you will find the present work interesting enough to be included in MELLONMR Letters.

We found the double irradiation method to be a very powerful tool in the elucidation of the two complex NMR spectra of phyllochrysine (P) and securinine (S), two isomer alkaloids corresponding to the plane formula (fig. 1) (1, 2).

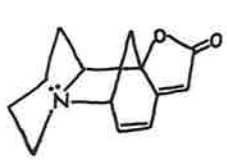


(fig. 1)

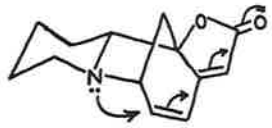
We send the whole interpretation of the two spectra run in $CDCl_3$ with both A60 (Gif) and HR-60 (Zürich) ; we also send the figures of double irradiation : the arrow represents the location of the proton (-s) which is irradiated by the strong r.f. field, the distance Δ in c/s gives the place of the decoupled proton (-s) and its appearance.

P corresponds to the relative configuration (fig. 2) while S corresponds to the relative configuration (fig. 3).

Two absolute configurations (enantiomers) belong to each relative configuration, and virosecurinine which proved to be the enantiomer of securinine (2) has also been isolated (3). The main difference between S and P consists in the difference of configuration at C_2. In fact, proton 2 in the NMR spectra of S and P appears at two different fields : δ 3,67ppm for P, and δ 2,02ppm for S. The magnetic influence of the conjugated C=O to two double bonds and that of the oxygen of the lactone ring are not easy to evaluate and we could not predict the relative position of proton 2 in the two spectra.

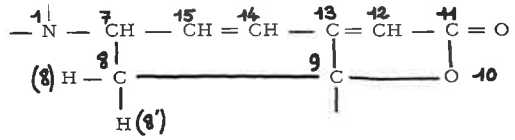


(fig. 2)



(fig. 3)

The double irradiation method allowed us to settle the following sequence :



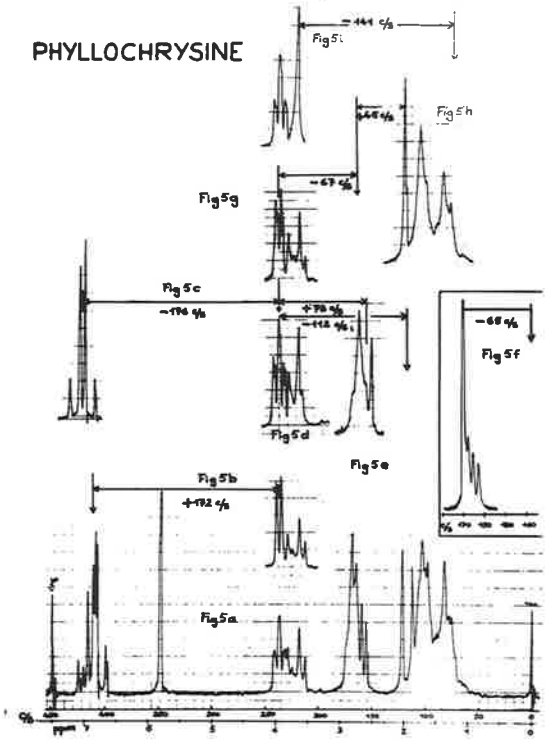
(fig. 4)

for the two alkaloids.

PHYLLOCHRYSINE (fig. 5)

1) - By irradiation of proton 7 (3.90ppm), with Δ =+176c/s (±), the protons 14 and 15 appear as a pure AB system :

$$J_{14,15} = 9.3\text{c/s} \; ; \; \left|\delta(H_{14}) - \delta(H_{15})\right| = 0.18\text{ppm}$$



2) - By irradiation of proton 8 (2.64ppm), with Δ =-67c/s, proton 7 appears as a quadruplet corresponding to the X part of the following ABX system : - CH_{14} = CH_{15} - CH_7-

$J_{15,7} + J_{14,7} = 6.7$c/s and since the difference of chemical shifts between protons 15 and 7, 14 and 7 respectively, are much greater than the corresponding coupling constants, we are allowed to write (by a single measure on the spectrum) : $J_{15,7} = 4.5$c/s and

$$J_{14,7} = 1.1\text{c/s}.$$

The position of ethylenic protons of P at lower field than those of S is due in the latter compound to a more effective participation of the electronic doublet of nitrogen to the conjugated system (fig. 3) ; this fact is supported by the yellow colour and U.V. spectra of P and S.

(±) - the + sign means that the decoupled proton is at a higher field location respect to that of the irradiated proton.

3) - By irradiation of proton 7 (3.90ppm), with Δ =+73c/s, proton 8 appears at 2.64ppm as a doublet, J =9,2c/s, which belongs to the half part of an AB system (the two protons on the carbon 8: H_8 and $H_{8'}$) This means that protons 8 and 8' are coupled together with J = 9,2 c/s ; that proton 8 is coupled with proton 7 and that proton 8' has no coupling with proton 7. Actually, by irradiation of proton 8 (2.64ppm), with Δ =+45c/s, proton 8' appears at 1.90ppm as a singulet. The simultaneous irradiation of protons 15 and 14, with Δ =+172c/s, makes proton 7 appear as a doublet, corresponding to the coupling between protons 7 and 8, $J_{8,7}$ = 4,5c/s. However, by irradiation of the proton 8' (1.90ppm) with Δ =-112c/s, proton 7 is only very slightly pertubed, which means that $J_{7,8'}$ is very near zero.

Since protons 8 and 8' and 7 do not show couplings other than 8, 7 and 8,8' we are allowed to say that C 9 does not bear any proton ; the condition of J =0 in the Karplus curve cannot be satisfied for two protons (on the same carbon) at the same time.

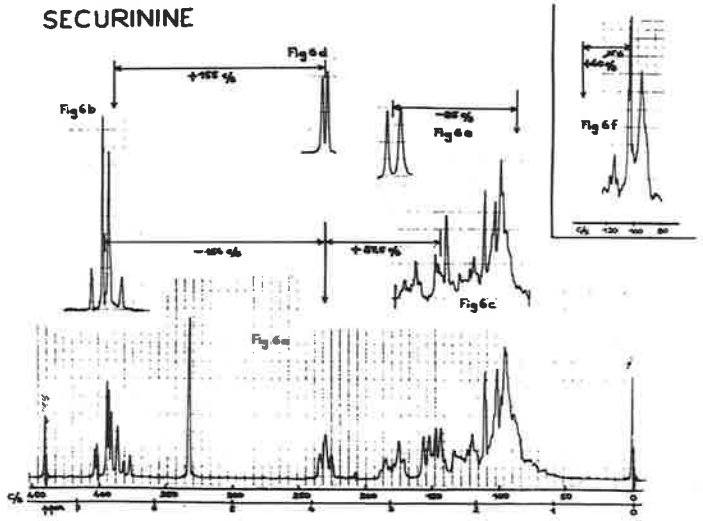
SECURININE (fig. 6)

Identical operations allowed to find the same sequence (fig. 4) for S.

The double irradiation method proved also to be very useful for evaluating the chemical shift of hidden protons.

For instance, proton 2 (3.67ppm) appears as a singlet by irradiation 141c/s at the upper fields ; the chemical shift of the two protons on C 3 is around 1,32ppm.

The two protons on C 6 appear at 2,88ppm under a single peak when irradiation is carried out 65c/s at the upper fields ; then, the chemical shift of the two protons on C 5 is around 1,80ppm.



SECURININE ($CDCl_3$)

δ ppm	Intensity	Appearance	
Between 6.75 - 6.30	2	Octet : AB part of an ABX $J_{14,15} = 9,3$ $\delta(H_{14}) - \delta(H_{15}) = 0,18$	H_{14} and H_{15}
5.54	1	singlet	H_{12}
3.84	1	rough triplet $J_{7,15} = 4.6$ $J_{7,14} = 1.2$ $J_{7,8} = 4.5$	H_7
2.99	1	trebled doublet $J_{6e,6a} = 10$ $J_{6e,5e} = 3.6 = J_{6e,5a}$	H_6 équatorial
near 2.50	2 = 1 + 1	quadruplet $J_{8,8'} = 9.5$ $J_{8,7} = 4.3$	H_8
		hidden peak	H_6 axial
2.02	1	quadruplet $J_{2,3a} = 9.5$ $J_{2,3e} = 4$	H_2 axial
1,77	1	doublet $J_{8,8'} = 9.5$	$H_{8'}$
between 1.80-1.0	6		$2H_3$, $2H_4$, $2H_5$

Chemical shifts δ are given in ppm referred to TMS (internal reference). The coupling constants J are given in c/s. a = axial position ; e = equatorial position.

REFERENCES :

1 - S. SAITO, K. KODERA, N. SUGIMOTO, Z. HORII and Y. TAMURA, Chemistry and Industry, 1962, 1652.

2 - J. PARELLO, A. MELERA and R. GOUTAREL, Bull. Soc. Chim. Fr., under press.

3 - T. NAKANO, T.H. YANG and S. TERAO, Chemistry and Industry, 1962, 1651.

PHYLLOCHRYSINE ($CDCl_3$)

δ ppm	Intensity	Appearance	
between 7.00 - 6.54	2	Octet : AB part of an ABX $J_{14,15} = 9.3$ $\delta(H_{14}) - \delta(H_{15}) = 0,18$	H_{14} and H_{15}
5.74	1	singlet	H_{12}
3.90	1	rough triplet $J_{7,15} = 4.5$ $J_{7,14} = 1.1$ $J_{7,8} = 4.5$	H_7
3.67	1	quadruplet $J_{2,3a} = 11$ $J_{2,3e} = 4.8$	H_2 axial
2.64	1	quadruplet $J_{8,8'} = 9.2$ $J_{8,7} = 4.5$	H_8
2.88	2	single peak	H_6 (axial and equatorial)
1.90	1	doublet $J_{8',8} = 9.2$	$H_{8'}$
between 1.84-1.0	6		$2H_3$, $2H_4$, $2H_5$

Yours sincerely,

Joseph PARELLO
Institut de Chimie des Substances Naturelles
Gif s/Yvette
(S. et O)

Attilio MELERA
Varian Research Laboratory
43, Klausstrasse, Zürich 8

Scale Expansion for the A-60

It is frequently the case that an A-60 will deliver resolution or fine structure of such quality that the slowest available sweep rates are inadequate for either seeing this fine structure in detail (because of ringing distortion) or making precise measurements of it. To get around this problem, there are at least two possibilities; one of these is to slow the sweep down by using a slower speed motor. This approach has been described by T. J. Flautt of Procter & Gamble (see MELLONMR 51, 19 (1963)). Another approach, which we have used with convenience and success is to expand the scale so that it is possible to make the full-scale scan width any arbitrary number of cycles, and in particular, to get any desired size of sweep less than the 50 cps full-scale which is the normal minimum. The following description and diagrams follow a basic suggestion from Varian Associaties, who we thank for the ideas.

The scale expander (see attached Figure 1) places a variable resistor (R-1) in series with a resistor in the D.C. sweep circuit of the probe when the switch (S-1) is in the open position. In the closed position, the A-60 functions normally (whatever that is!).

For R-1, we use a Borg 10-turn potentiometer, Model 205, with a total resistance of 20,000 ohms and a linearity of 0.1%. The switch, S-1 (chosen to have a very low contact resistence) is a JBT, type #ST52K, DPST; the two "on" terminals are soldered together as are the two "off" terminals.

The potentiometer is fitted with a Duo-Dial (Beckmann Type RB) and the scale expander is calibrated by means of the usual sideband techniques. (The total cost of the pot, switch and dial is less than $25.00.)

We found that in our particular case, a dial setting of 516 gave a sweep width of 12.5 cps full-scale. On our instrument, this turned out to be about the limit of acceptable sweep linearity and still provides a four-fold sweep expansion. The calibration is checked periodically, but seems to hold indefinitely.

If the ultimate use of such scale expansion is desired, it will, of course, still be desirable to use the 500-second sweep time. In general, but particularly in this event, it will be found that for most cases a considerable amount of extra RF attenuation will be required to avoid saturation effects. The simple (if inelegant) method we use is to insert a 30 db fixed attenuator as indicated on the attached Figure 2, which also includes the details of this attenuator. This extra attenuation is always used when the scale expansion is being employed, and the RF dial setting is usually kept above .07 mG to keep the rest of the electronics happy. With this scheme and the otherwise optimum RF setting, it is sometimes impossible to achieve quite perfect phasing using the tuning range of the Detector Phase control on the front of the spectrometer console. There is surely some simple resolution to this minor problem, and suggestions will be gratefully received.

Finally, we have mounted the pot, dial and switch on a small plate affixed to the outside of the right-hand end of the console for convenience and to keep it somewhat out of the way of incorrigible switch- and dial-twiddlers. We have used this scale expansion system routinely for well over a year and are quite happy with it.

B. L. Shapiro
J. A. Lorenc
J. G. Rickert

Mellon Institute
Pittsburgh, Pennsylvania

18 March 1963

FIG. 1

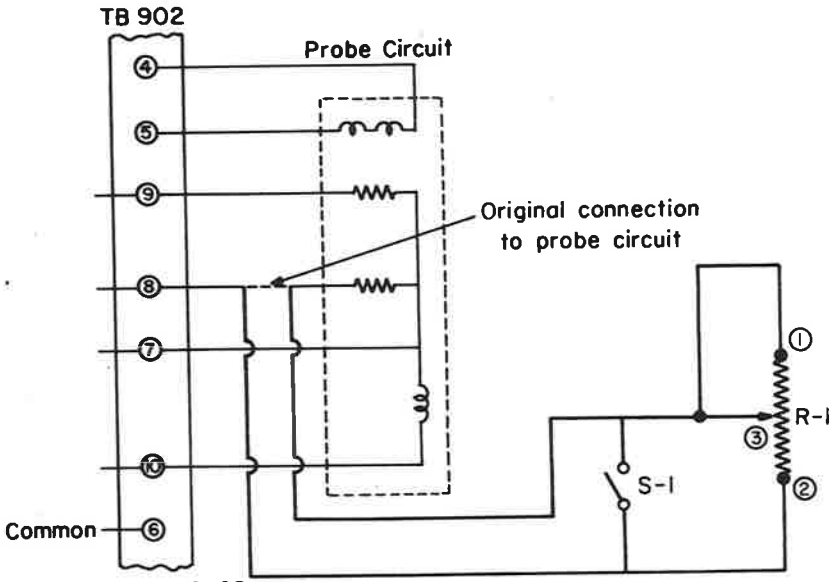
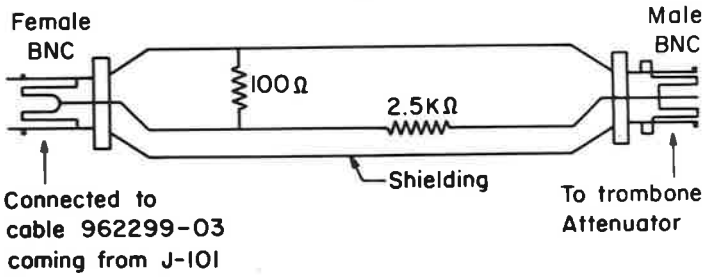


FIG. 4-62
RECORDER SCHEMATIC — SCALE EXPANDER

FIG. 2



30db ATTENUATOR

ADVANCE NOTICE

NMR Spectroscopy Institute

CANISIUS COLLEGE • BUFFALO, NEW YORK

JUNE 24-~~29~~ 28, 1963

ELEMENTARY SESSION

Lecturers:

DR. RALPH GOLIKE
E. I. du Pont de Nemours Co., Buffalo

MR. LEROY JOHNSON
Varian Associates, Palo Alto, California

DR. HERMAN A. SZYMANSKI
Canisius College, Buffalo

GUEST LECTURERS
DR. KENNETH BARTZ HUMBLE OIL

The elementary sessions are designed for those who require a beginning course in NMR spectroscopy including an introduction to basic theory and heavy emphasis on interpretation of NMR spectra, laboratory techniques, industrial applications of NMR, etc. No background in NMR spectroscopy will be required of participants and simple laboratory exercises will be included in the course.

Because of the laboratory exercises the group will be limited in size to 30 participants.

Monday

A. Elementary theory of NMR and NMR spectra
B. The simple NMR instrument
C. Laboratory work sessions

Tuesday

A. Interpretation of NMR spectra
B. Quantitative Analysis
C. Laboratory and work sessions

Wednesday

A. Solvent effects, conformational analysis
B. Special instrumentation techniques
C. Laboratory and work sessions

Thursday

A. Applications of NMR spectroscopy to industrial problems
B. Interpretation of NMR spectra
C. Laboratory and work sessions

Friday

A. Applications of NMR spectroscopy
B. Panel Discussion

ADVANCED SESSION

will HANDLE INTERPRETATION SESSIONS FOR THE GROUP

Lecturers:

DR. DAVID J. WILSON,
University of Rochester

DR. LAWRENCE COLEBROOK,
University of Rochester

The advanced sessions are designed for those having a good mathematical background but very little background in magnetic studies. No laboratory exercises are planned for this group. The topics covered will be concerned with high resolution NMR.

Monday, Tuesday and Wednesday

A. Mathematical background
B. Basic principles of magnetic resonance

Thursday and Friday

A. Spin decoupling techniques
B. Solvent effects
C. Conformational Analysis
D. Special techniques

Symposium ends at noon.

The Fee for both sessions will be $100
College Teachers 50

For further information write:

DR. H. A. SZYMANSKI
CANISIUS COLLEGE, BUFFALO, N. Y.

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