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Mailed: SEP 30 1960

Monthly
Ecumenical
Letters from
Laboratories
Of
N - M - R
No. 24

A monthly collection of informal private letters from laboratories of nmr.
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Modifications of Sample Spinning System

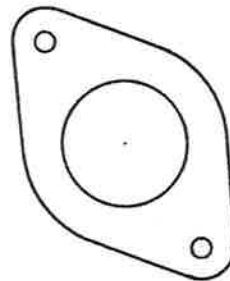
We have made two changes in our Varian probe assembly to ensure better alignment and smoother rotation of the NMR sample tubes.

First, if the air turbine mounting ring, Sketch #1, is modified so that its holding screws can be tightened while the sample tube is spinning in the turbine, the vibration due to the spinning can be minimized and the mount fixed in this position. The presence of the vibration can be noted by observing whether or not the detector level meter gives a higher and more erratic signal level when the sample is in motion as compared with the situation when the sample is stationary. The vibration can also be seen as base line noise on the scope and can sometimes be felt directly by the fingers. With this modification we find that when the probe is balanced with 20 db. of attenuation, the minimum observed on the detector level meter is essentially the same (about 20 μ a) whether the sample is rotating or not. Previously, an erratic reading of 50 μ a and higher was often observed when the sample was rotating.

Second, the bearing in the insert can be increased in length and its bottom configuration changed to reduce vibration of the spinning sample tube. The bearing that we use is shown in Sketch #2. This has proven very satisfactory, particularly when using the flat bottomed Wilmad tube.

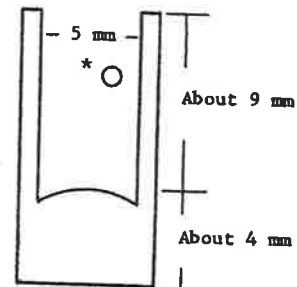
Robert B. Bradley
Laboratory of Physical Biology
National Institute of Arthritis
and Metabolic Diseases
National Institutes of Health

Sketch #1



Top View of Modified
Turbine Mounting Ring

Sketch #2



Side View of Nylon
Bearing for Spinning
Sample Tube

* Hole for removal

O.D. of bearing to fit insert

This bearing can also be made with an 8 mm base and a 5 mm top portion, if it is of value to use a small volume of solution.

HARVARD UNIVERSITY
DEPARTMENT OF CHEMISTRY

12 Oxford Street
Cambridge 38, Massachusetts, U.S.A.
3 September, 1960

Dr. B. L. Shapiro
Mellon Institute
4400 Fifth Ave.
Pittsburgh 13, Pa.

Dear Dr. Shapiro:

Many thanks for your letter of 3 August, and the back issues of MelloNMR enclosed. I have recently been doing some nitrogen (14)-proton double resonance studies. For a second radio frequency source I am using a very simple locked oscillator system that may be of interest to MelloNMR readers.

A block diagram of the system is shown in Figure 1. The output of a General Radio Type 1211 B Unit Oscillator is mixed with one of the 10Kc. harmonics of a General Radio Type 1213 D Unit Time/Frequency Calibrator. The audio frequency beat note from the calibrator is mixed in a phase detector with the audio frequency output of a Hewlett Packard 201CR oscillator. The phase detector is shown in Figure 2. The output of the phase detector is then returned to the grid of the 5763 tube in the Type 1211 B oscillator. The audio frequency of the Hewlett Packard oscillator is then monitored continuously with a H.P. 521 C electronic counter. The unit oscillator can be locked with the audio oscillator over a range of about 400 cycles/sec. The frequency stability of the whole system is about ± 0.2 cycles/sec. over about five minute periods, as measured with a H.P. 524 D electronic counter.

I would appreciate it very much if you could send me any additional back issues of MelloNMR from No. 11 through 21 that are still available.

Yours very truly,

John D. Baldeschwieler

John D. Baldeschwieler

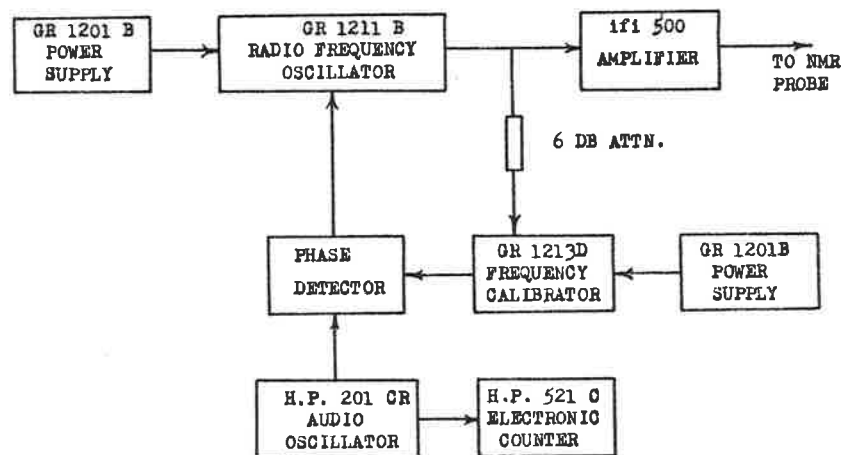


FIGURE 1

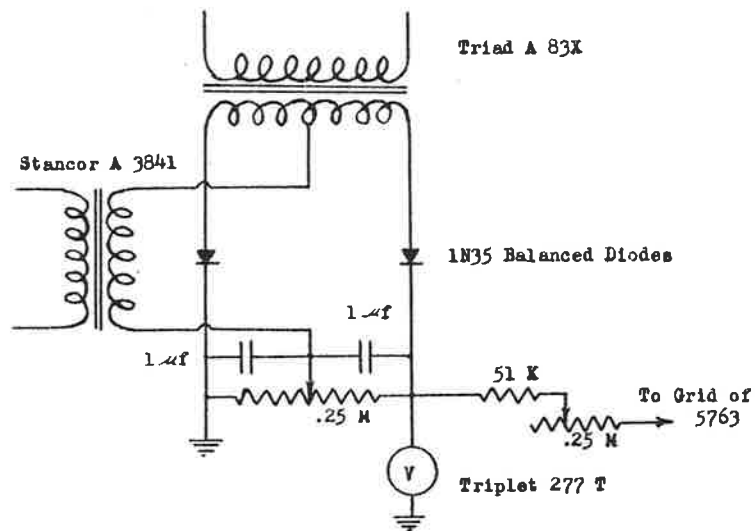


FIGURE 2

CARNEGIE INSTITUTE OF TECHNOLOGY

SCHENLEY PARK

PITTSBURGH 13, PENNSYLVANIA

CHEMICAL AND PETROLEUM
RESEARCH LABORATORY

August 2, 1960

Dr. Aksel A. Bothner-By
Assistant Director of Research
Mellon Institute for Industrial Research
4400 Fifth Avenue
Pittsburgh 13, Pennsylvania

Dear Dr. Bothner-By:

You indicated that you would be happy to accept some items of general interest from the Chemical and Petroleum Research Laboratory. Nuclear magnetic resonance spectroscopy is one of the five categories of spectroscopy with which the American Petroleum Institute Research Project 44 and the Manufacturing Chemists Association Research Project are concerned.

As of about April 1, 1960, we contacted practically every laboratory operating some type of commercial nuclear magnetic resonance spectrometer. Many other laboratories have been reached through the distribution of information by the API and the MCA. The many replies which have been received have been most gratifying. About 30 laboratories have expressed considerable interest in contributing spectra, but of course, many are not able to contribute immediately but hope to do so "toward the end of the year" or "as soon as the bugs have been worked out".

Regarding the recent communications on standardization of spectra by Drs. Shoolery, Johnson, and Anderson of Varian Associates and by Dr. Chamberlain of Humble Oil and Refining Company, as reported in M. E. L. L. O. N. - M. - R. - Numbers 19 and 20 respectively, we feel that a certain amount of flexibility in these matters is justifiable. The use of multiple scales, as proposed by Dr. Chamberlain, might be a good thing for the interim, pending complete agreement on all points. Contributions of spectra are encouraged from all investigators, regardless of personal or party likes and dislikes. The revision of spectra, from one form to a more suitable form at a later date, will be a routine operation for both the API and the MCA Projects, just as it has been for the other four categories of spectral data.

Nugent Chamberlain has kindly offered to provide the API Research Project 44 with the spectra of the paraffins and cycloparaffins of the API Standard and Research Hydrocarbons. These spectra will be run at a frequency of 60 megacycles. In addition, Nugent will soon be contributing a sizeable lot of spectra recorded at 40 megacycles. The latter spectra will be issued in one complete lot.

Dr. Aksel A. Bothner-By

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August 2, 1960

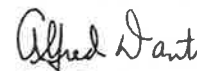
Dr. William Ritchey of the Standard Oil Company of Ohio has kindly offered to provide the API Research Project 44 with the spectra of all the aromatic compounds in the series of Research Hydrocarbons.

Dr. Bothner-By is carrying out investigations on certain compounds of the olefin series and some low-boiling paraffins.

Investigators will be considered for the remaining families of compounds.

The interest and assistance of all the recipients of M. E. L. L. O. N. - M. - R. - in the work of the API and MCA Research Projects is very much appreciated.

Very truly yours,



Alfred Danti

In Charge, Spectral Data

American Petroleum Institute Research Project 44

Manufacturing Chemists' Association Research Project

AD:hm

THE UNIVERSITY OF BIRMINGHAM



Department of Chemistry,
EDGBASTON,
BIRMINGHAM. 15.
22nd. August 1960

TELEPHONE
BILLY OAK 1201

Dr. A.A. Bothner-By,
Editor, M.E.L.L.O.N.M.R.,
Mellon Institute,
4400, Fifth Avenue,
Pittsburgh 13, PA., U.S.A.

Dear Dr. Bothner-By,

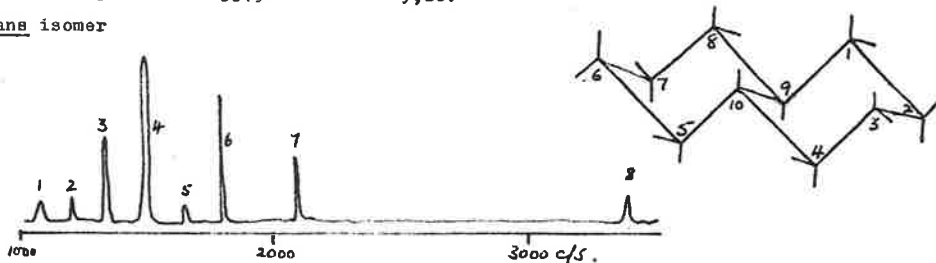
We hope that this account of the fluorine spectra of the two stereoisomers of perfluorodecalin will be of some interest to readers of M.E.L.L.O.N.M.R. The spectra, measured at 30.107 Mc/s. with trifluoroacetic acid as external reference, indicate that the cis isomer is undergoing rapid interchange between its two conformations with consequent loss of axial/equatorial identity of the fluorine nuclei and simplification of the spectrum compared with that of the trans isomer which must have a fixed configurations:

cis isomer.



Peak	Position	Assignment
1	1363 c/s.	2,3,6,7 pairs (?)
2	1647 "	1,4,5,8 "
3	3375 "	9,10.

trans isomer



	Peak	Position	(2) Separation	Assignment
I	1	1079 c/s.		
	3	1356 "	277 c/s.	1,4,5,8 a
	6	1821 "		
	7	2102 "	281 "	1,4,5,8 e
II	2	1219 "		
	4	1508 "	289 "	2,3,6,7
	(6)	(1821) "	(313) "	a and e
	8	3387 "		9,10.
	5	1673 "		impurity (<u>cis</u> isomer)

Group I. The assignment of this AB quadruplet is made on the basis that peaks 1,3 are broader, due to unresolved coupling, than 6,7 which would arise if coupling across an intervening C-C bond is greater between nuclei trans to each other than for those which are gauche

e.g. 1a is trans to 2a and 9, gauche to 2e, whilst 1e is gauche to 2a, 2e and 9.

Group II. This is also a symmetrical pattern. The small high field component was resolved at the appropriate position to low field of the larger peak 6 in a run at 37.635 Mc/s. carried out for us by Mr. B.A. Evans of Mullard Research Laboratories. The group would appear to be another AB quadruplet in which the two centre peaks are merged (the peak is some 50 c/s. wide at half height) because of the small chemical shift between the axial and equatorial nuclei. Relative intensity measurements are insufficiently accurate to make a precise evaluation of the shift but it could be 50-60 c/s. (2 p.p.m.).

Thus there emerge two points of similarity with the spectra of fluorinated cyclohexanes reported by Feeney and Sutcliffe in M.E.L.L.O.N.M.R.21 :

- (a) The coupling constant between gem fluorine nuclei is about 280 c/s.
- (b) The chemical shift between axial and equatorial nuclei is appreciable only when these are located near other substituents in the ring.

Incidentally, we are at present examining the spectra of highly fluorinated cyclopentanes and find much the same effects except that the geminal coupling constant is slightly smaller at about 265 c/s.

With thanks for continuing to send the Newsletter and apologies for the delay in making a contribution,

Yours sincerely,

J. Homer.
J. Homer
L.F. Thomas.
L.F. Thomas.

Buenos Aires, september 5, 1960.

Dr. Aksei Bothner-By
 Mellon Institute
 1400 Fifth Avenue
 Pittsburgh 13, Pa.
 U.S.A.

Dear Dr. Bothner-By:

Some time ago I wrote you about the possibility of the assignment of relative signs of some of the J-couplings in spectra of the ABX_3 type observing the behaviour of the X_3 group of lines at different spectrometer frequencies. This method has been successfully used in the analysis of the spectra of some olefines, of the ABX_3 as well as of the ABX_2 type. (to appear shortly in the J.Chem.Phys. and Journal de Physique et le Rad;)

To make the method clear we include some graphs which show the results of calculations made for the X_3 and X_2 group of lines for several sets of parameters with values similar to the actually found in experimental spectra. The figures show the difference between spectra in which J_{AX} and J_{BX} are of the same or of different sign. Unfortunately we have been unable to make the exact ABC_2 and ABC_3 treatments, but it should be noted that the perturbed ABX_2 treatment by F.S. Mortimer (J. of Mol. Spec. 3, 335, (1959), gives quite similar results except for low spectrometer frequencies where the frequencies of the transitions diverge. The X approximation works very well in the ABX_3 case due to the big chemical shift of the methyl groups. In the ABX_2 systems it still accouns well for the frequencies but not so well for intensities.

Below are listed some of our results.

Yours sincerely,

L. G. de Kowalewski
 Lora G. de Kowalewski

V. J. Kowalewski
 V. J. Kowalewski

	J_{AB}	J_{AX}	J_{BX} (cps)
cis crotonic acid	11.4	± 7.5	∓ 1.9
trans crotonic acid	14.9	± 6.7	∓ 1.7
methyl ester of the trans crotonic acid	15.4	± 7.0	∓ 1.6
3 - propenyl bencene	14.8	± 7.0	∓ 1.0
cynnamic alcohol	15.0	± 4.7	∓ 0.5
anetol	15.4	± 6.68	∓ 1.72

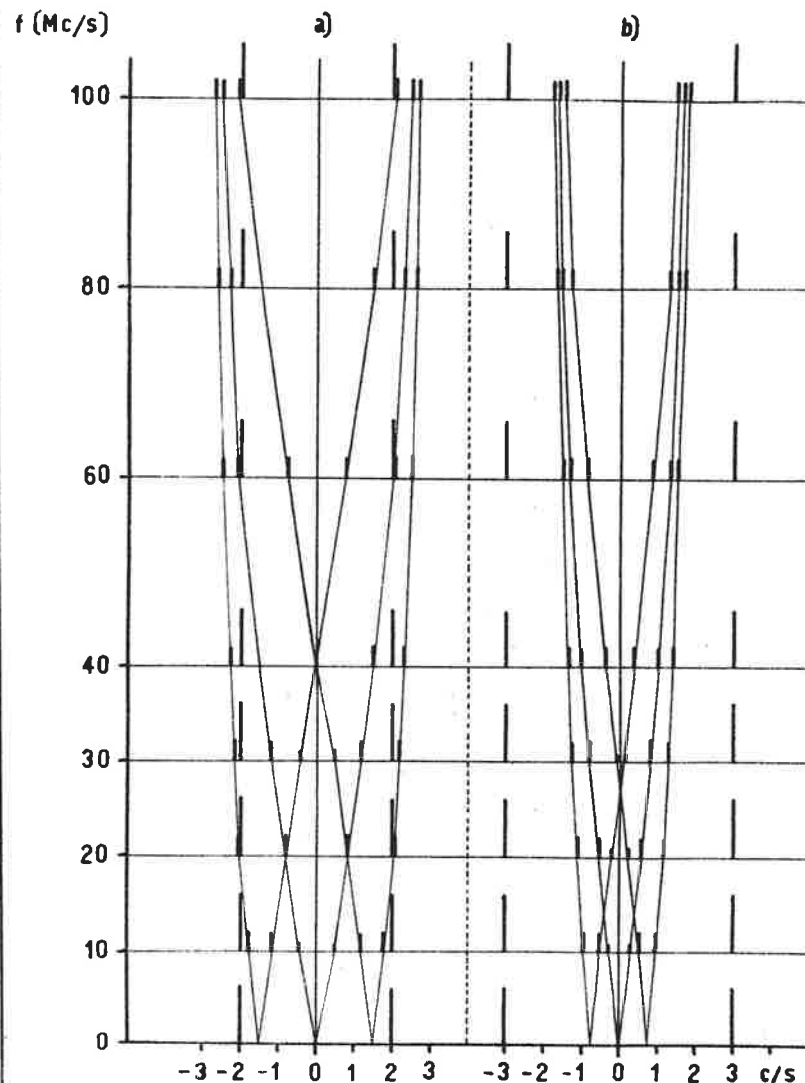


Fig. 2(a) X_3 group of lines of an ABX_3 spectrum for different spectrometer frequencies. $|J_{AB}| = 10$ cps; $|J_{AX}| = 5.0$ cps; $|J_{BX}| = 1.0$ cps; $\delta_{AB} = 0.15$ ppm; J_{AX} and J_{BX} with different signs.

Fig. 2(b) Same as 2(a) but with J_{AX} and J_{BX} of the same sign.

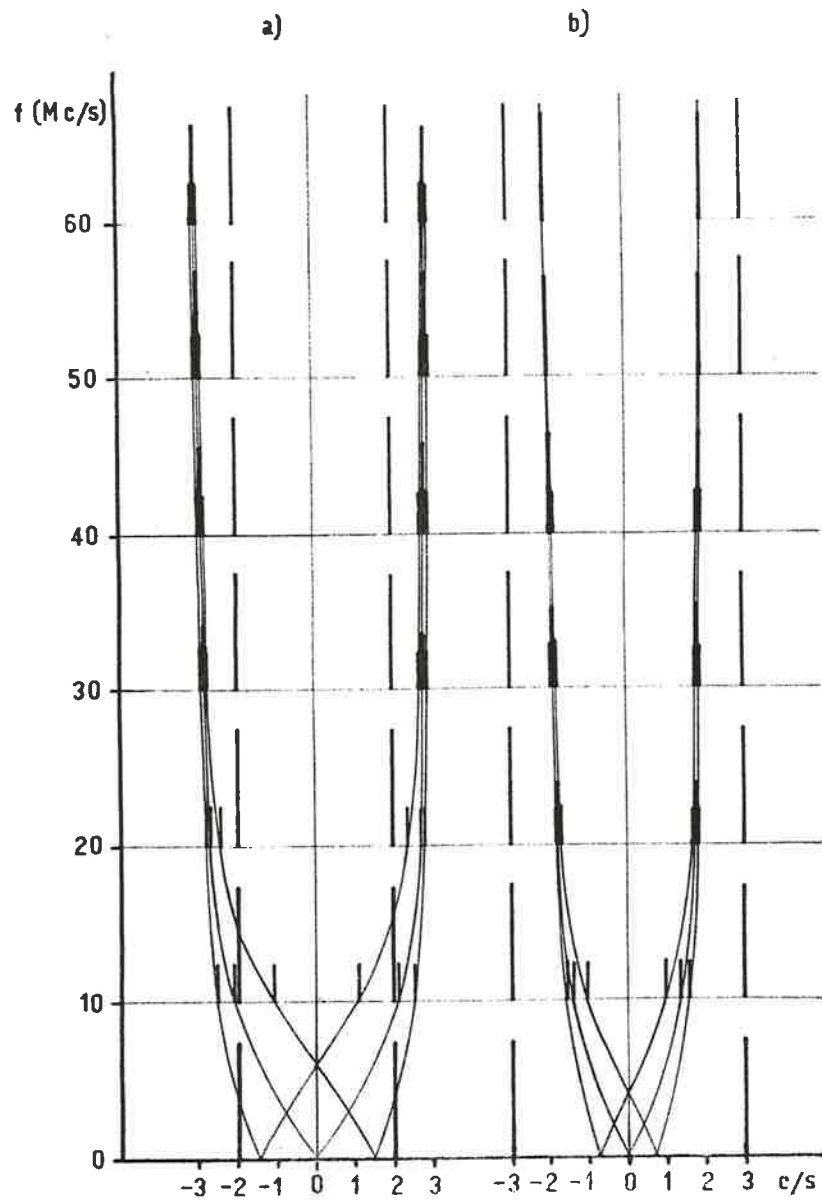


Fig. 1(a) X_3 group of lines of an ABX_3 spectrum for different spectrometer frequencies. $|J_{AB}| = 10$ cps; $|J_{AX}| = 5.0$ cps; $|J_{BX}| = 1.0$ cps; $\delta_{AB} = 1.0$ ppm; J_{AX} and J_{BX} with different signs.
 Fig. 1(b) Same as 1(a) but with J_{AX} and J_{BX} of the same sign.

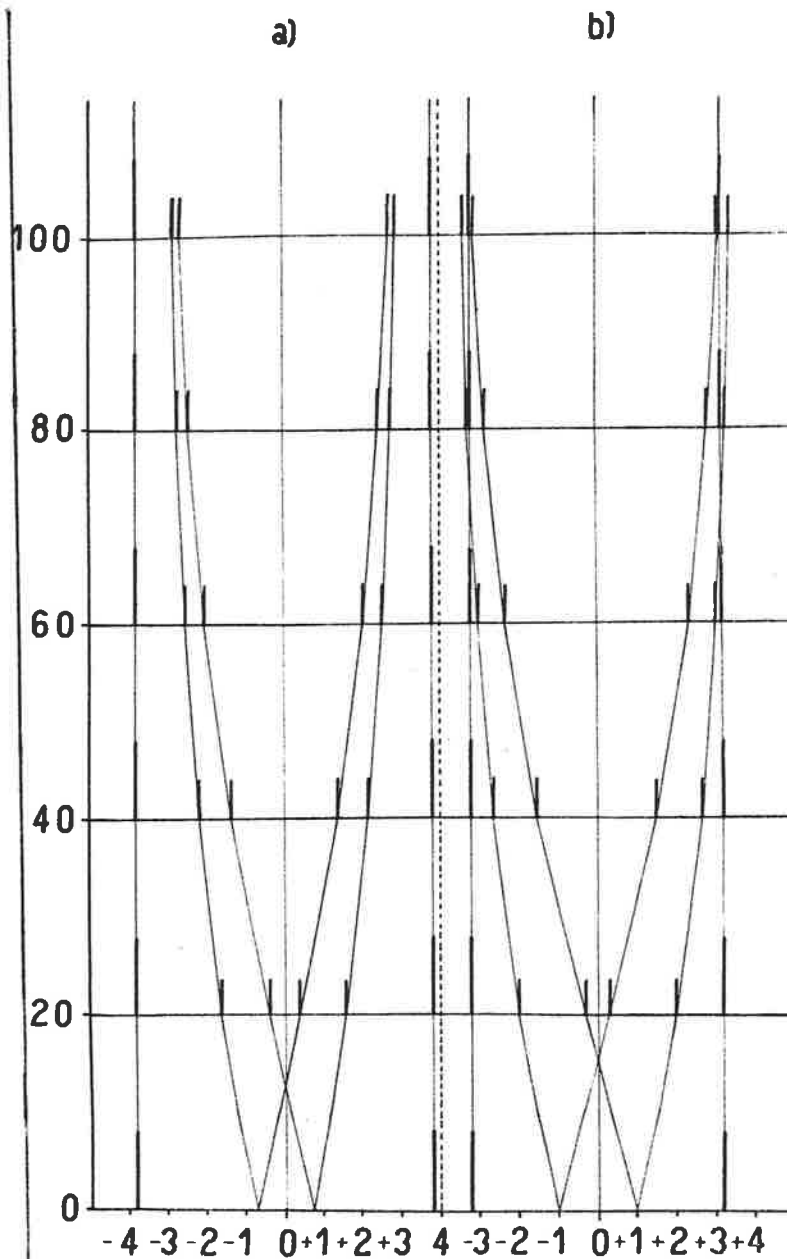


Fig. 3(a) X_2 group of lines of an ABX_2 spectrum for different spectrometer frequencies. $|J_{AB}| = 14.0$ cps; $|J_{AX}| = 7.0$ cps; $|J_{BX}| = 0.6$ cps. $\delta_{AB} = 0.25$ ppm; J_{AX} and J_{BX} of the same sign.
 Fig. 3(b) Same as 3(a) but with J_{AX} and J_{BX} of different signs.

The Standard Oil Company

(An Ohio Corporation)

Chemical and Physical Research Division

4440 Warrensville Center Road

Cleveland 28, Ohio

L. C. HUGHES
PLANTATION MANAGER

August 25, 1960

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

Dear Barry:

Here is the information on our automatic timer that you requested on your last trip here to Cleveland.

The automatic timer is used to turn on our slow sweep unit and recorder when we are running spectra. We have it adjusted so that we can sweep from well below the aromatic region to well past tetramethylsilane, and it will then shut off the recorder and sweep unit simultaneously.

The main advantage in having such a unit is that the operator is no longer "tied down" to the NMR console while running a sample. It is nice to know that the unit will stop automatically when the spectra is done, and not have to worry about kicking out the Super Stabilizer in case you should be away from the console for any length of time.

There are many ways that such a unit might be built, and enclosed is a schematic diagram of the way in which we constructed our unit. Two thermal delay relay tubes were used even though one could do the job if it were really pushed hard.

There is a difference in delay time in a thermal relay when operated from a cold start as opposed to operating it from a hot start, i.e., immediately after having been used. This timing difference can be as high as 50%. To minimize this, a smaller timed value was selected to precede the main tube. In this way, the deviation in time is limited to the smaller value only and is therefore not objectionable.

Dr. B. L. Shapiro

-2-

August 25, 1960

Relay A should be a 4 pole normally open 6 VAC relay. Two sets of contacts are placed in parallel with the up field increase sweep lever. The other set of contacts go to the motor drive on the recorder. We do not switch the servo motor on and off as we have not found it necessary to do so.

One other item of interest pertains to the "Varian Field Homogeneity Control" unit. The vertical Y control on this unit has a tendency to be overly sensitive. Should you wish to decrease this sensitivity, Varian recommends that you change resistors, (R-205 and R-206) from 820 ohms to 2200 ohms. We use a dpdt switch mounted on the control panel to select the proper resistance values. This enables us to increase or decrease the vertical Y sensitivity quickly whenever we wish to do so.

Both have been in operation for several months and have been completely satisfactory. In the event that further information is desired, please do not hesitate to contact us.

Sincerely,

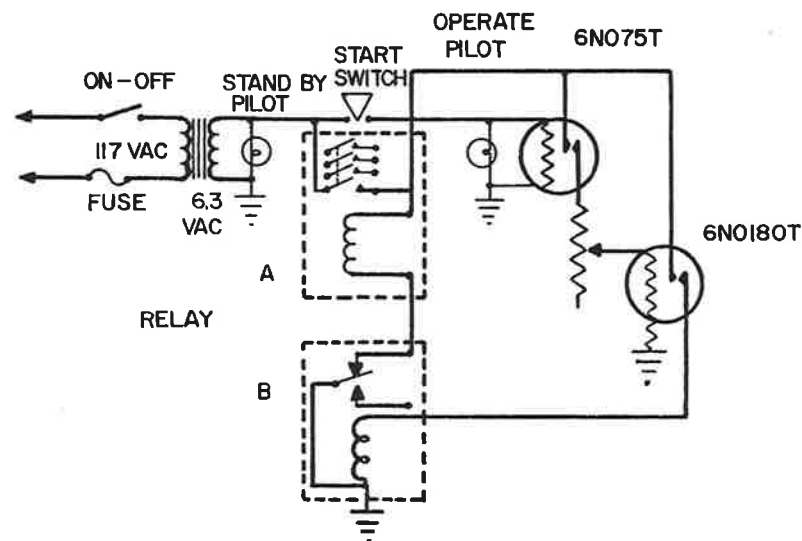
H. Grossman

H. Grossman

W. M. Ritchey

W. M. Ritchey

NMR TIMER



RELAYS SHOWN NOT ENERGIZED

Contribution from the Department of Chemistry,
The University of Florida

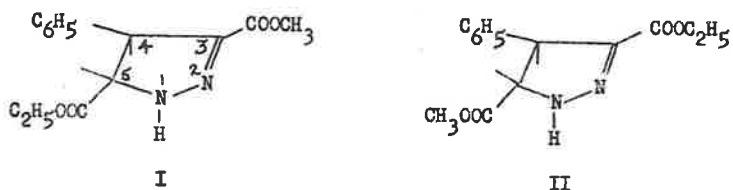
Application of Nuclear Magnetic Resonance Spectrometry

to Determination of Pyrazoline Structures

By Wallace S. Brey, Jr., and W. M. Jones

In the latter part of the nineteenth century, Buchner and his co-workers (E. Buchner and H. Dessauer, *Ber.*, 26, 258 (1893); E. Buchner and C. von der Heide, *ibid.*, 35, 31 (1902)) made the interesting observation that the reactions of methyl diazoacetate with ethyl cinnamate and ethyl diazoacetate with methyl cinnamate led to two different isomeric 2-pyrazolines, melting at 107° (I) and 76° (II), respectively.

Geometrical configurations have recently been assigned



(W. M. Jones, *J. Am. Chem. Soc.*, 82, 3136 (1960)) as a result of an examination of the decomposition products of these two materials and the application of the rule that "the geometrical configuration of the primary cyclopropane resulting from the decomposition of a 2-pyrazoline is determined by the relative thermodynamic stabilities of the intermediate 1-pyrazolines."

There still remained, however, uncertainty in the distinction between the two isomers I and II. The assignment shown has been established by comparing the proton magnetic resonance spectra of these two compounds with four model compounds, containing various substituents in the 3 and 5 positions.

NMR spectra were determined for the mixed esters and the model compounds in saturated solutions in carbon tetrachloride. A Varian 4300-2 high-resolution spectrometer, operating at 56.4 megacycles, was employed. Shifts were measured with respect to benzene as an internal reference, using sidebands applied by a

calibrated audio oscillator. The amount of benzene added to the solutions was adjusted to give a peak of the same size as that of the phenyl group of the sample.

In Table I are presented the chemical shifts of the hydrogen atoms in the six compounds, expressed in parts per million displacement of the resonance upfield from the reference.

Table I

NMR Chemical Shifts of Substituted Pyrazolines

Values are in parts per million;
positive shifts are to higher magnetic field

Com- pound	Substituent group		H in OCH ₃	H in CH ₂ of C ₂ H ₅	H in CH ₃ of C ₂ H ₅	H in C ₆ H ₅	H on ring carbons
	3	5					
I	COOCH ₃	COOC ₂ H ₅	3.60	3.03	5.96	0.043	2.75 3.03
II	COOC ₂ H ₅	COOCH ₃	3.56	3.19	6.08	0.055	2.75 2.93
III	COOCH ₃	H	3.63	-	-	0.108	(a)
IV	COOC ₂ H ₅	H	-	3.14	6.06	0.078	(a)
V	COOCH ₃	COOCH ₃	3.49 3.60	-	-	0.041	2.71 3.01
VI	COOC ₂ H ₅	COOC ₂ H ₅	-	3.01 3.14	5.98 6.08	0.046	2.71 2.94

(a) Splitting by H in position 5 prevented analysis.

The resonance of the hydrogen atoms in an OCH₃ group is a single sharp peak, while each ethyl group shows the usual 4 and 3 multiplets with coupling constants of 7.1 - 7.2 cycles. The two hydrogen atoms attached to the ring carbons show a characteristic non-equivalence quartet, which was analyzed in the usual manner to give the chemical shifts shown. In many of the spectra, this quartet overlapped that of the methylene hydrogens of the ethyl group, but the differences in relative spacings within the two types of quartet permitted unambiguous assignments to be made.

The assignments of structures I and II, as indicated in the table, to the high and low melting isomers, respectively, may be confirmed by examination of the chemical shifts of the H atoms in alkoxy groups. The carbomethoxy group in the 3 position of III, a known compound, has a value of 3.63. In compound V, there are carbomethoxy groups in both 3 and 5 positions; the respective chemical shifts may be assigned as 3.60 and 3.49. The value of 3.60 appears also in I, corresponding to a carbomethoxy group in the 3 position; in II, the lower value of 3.56 for the same substituent group is consistent with the location of this group in the 5 position. Even more striking is the distinction between the chemical shifts of the carboethoxy hydrogens in the two positions. In IV, the methylene protons appear at 3.14, a value also found for one of the two groups in compound VI, and comparable to the value 3.19 for compound II. In contrast, the other methylene hydrogens in the diethyl ester, VI, have a chemical shift at 3.01, which corresponds to the value of 3.03 in compound I, indicating that the ethyl group in the latter is in the 5 position. In parallel fashion, the resonances for the methyl hydrogens in the ethyl esters appear at 6.06-6.08 for the 3 position and 5.96-5.98 for the 5 position.

There are two possible factors that can explain the result that the resonances for a carboalkoxy group always appear at higher field when the group is in the 3 position than when it is in the 5 position. The first of these is based upon the conjugation of the carbonyl group of the position 3 substituent with the ring double bond, which would be expected to reduce the conjugation of the unshared electrons on the ether oxygen of the ester unit with the carbonyl group and therefore increase the electron density on the ether oxygen and in turn on the alkyl group. It is not expected, however, that this effect would be transmitted to the extent it is to the methyl group in the ethyl radical. The second factor, which would apply particularly to this group, is related to the fact that in the trans configuration of these compounds, the alkyl group of the ester in position 3 has a higher probability than does the group in position 5 of being located in front of the face of the phenyl ring. It is known that the ring current effect in the aromatic ring shifts resonances of groups to higher field when the groups are in this sort of location with respect to the ring.

Chemical shifts of the hydrogens attached to carbons in the pyrazoline ring display an interesting pattern. The resonance at higher field appears at 3.01-3.03 when there is a carbomethoxy group in position 3 but at 2.93-2.94 when there is a carboethoxy group in this position. The resonance at lower field appears at 2.71 when positions 3 and 5 have the same

substituent group but at 2.75 for the mixed esters. This indicates that there is some change in the precise ring geometry related to the presence of the alkyl groups in the esters.

Finally, the position of the phenyl resonance is of interest. In compounds III and IV, the phenyl group has the greatest freedom of rotation about its bond to the ring, and the chemical shift has the highest positive value. The other four compounds listed in Table I have about the same rotational freedom for the phenyl, slightly less than that in III and IV, and the chemical shifts of the phenyl protons fall in a relatively narrow range, somewhat below III and IV. In addition to the results reported in Table I, we have obtained spectra of solutions of oils isolated from the mother liquors resulting from filtration of I and II from their crude reaction mixtures. Evidence has been presented (W. M. Jones, J. Am. Chem. Soc., 82, 3136 (1960)) that these mother liquors are rich in the cis-4-phenyl-3,5-dicarboalkoxy-2-pyrazolines. In these spectra can be seen bands characteristic of either compound I or II, as well as additional bands which are believed to be characteristic of the cis isomers, since the phenyl peaks fall at lower fields, one chemical shift being negative. In the cis isomer, the phenyl group is wedged between the two substituents. The shift of the resonance of the phenyl group to lower magnetic field is therefore attributed to steric effects, very probably a direct "repulsive unshielding" of the phenyl protons.

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