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Monthly
Ecumenical
Letters from
Laboratories
Of
N - M - R
No. 20

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DIVISION OF APPLIED CHEMISTRY

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May 10, 1960.

Dr. A.A. Bothner-By,
Assistant Director of Research,
Mellon Institute,
4400 Fifth Avenue,
Pittsburgh 13, Pennsylvania,
U.S.A.

Dear Aksel:

I'm sorry to be remiss in my contributions to M.E.L.L.O.N.M.R. However, our new instrument has just gone on the air. Therefore all I can contribute at the present time is a rather negative result.

There are many cases of quadrupole broadening of protons attached to nitrogen. Protons attached to chlorine have always given a single sharp line with no evidence of quadrupole broadening or spin coupling. Presumably this is due to rapid relaxation of the chlorine nuclear spin by interaction of its quadrupole with an unsymmetric electric field gradient. I don't know of any examples of broadening of a fluorine resonance line due to coupling with a nucleus having a quadrupole moment.

Perchloryl fluoride, ClO_2F , has three oxygens and a fluorine tetrahedrally arranged about the chlorine. It does not have an observable dipole moment so the electric field about the chlorine should be reasonably symmetric. Therefore one might hope to observe spin coupling with the chlorine, or broadening of the fluorine resonance signal.

The gas was condensed into a sample tube and sealed under vacuum. Upon warming to room temperature a mobile liquid is obtained, with the pressure in the tube being about ten atmospheres. No signal could be found from this sample so presumably this is a case of (a) Quadrupole broadening of a fluorine resonance

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signal via spin coupling, (b) Quadrupole broadening caused by chlorine. The next thing is to take the wide line spectrum upon the liquid and see what happens.

A note in the last issue of M.E.L.L.O.N.M.R. about thermocouple protection of the magnet prompts this mention of our arrangement. The thermocouples from each magnet coil are joined in series and attached to a small indicating meter relay, "Simplytrol", which is put in the same circuit as the pressure switch. This serves as an inexpensive protection against overheating in either or both coils, and also gives an indication of the actual internal temperature.

Yours truly,

Syd Brownstein

S. Brownstein.

SB/mfc



U. S. NAVAL ORDNANCE TEST STATION
CHINA LAKE, CALIFORNIA

IN REPLY REFER TO:
5058/DWM:c1
29 April 1960

5058/DWM:c1
29 April 1960

11ND-NOIS-2700/4 (12-68)

Dr. Aksel A. Bothner-By
Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Penna.

Dear Dr. Bothner-By:

Enclosed are the latest results on the NMR spectra of divinylmercury, tetravinyltin and trivinylaluminum etherate. We feel that the present set of spectral parameters is good to ± 0.2 cps.

The proton spectra of divinylmercury and tetravinyltin exhibit nice satellite spectra from coupling of all three vinyl protons with magnetic isotopes Hg^{199} , Sn^{117} and Sn^{119} (all with spin $I = 1/2$). Normal abundances of these are 16.9, 7.7 and 8.7%, respectively. The metal-proton spin-coupling constant has the useful effect of expanding the pseudochemical-shift values in the satellite spectra. Since the electro-positive metal atoms tended to bring about collapse of the main vinyl spectra, it was found useful here to obtain approximate δ - and J -values by first-order treatment of satellite lines and then to refine these before attempting to compute the main spectra.

Computation of the ABC-type spectra was accomplished using the IBM-709 SPIN program written by Holder, Abell and von Holdt of the Lawrence Radiation Laboratory, Livermore, California. SPIN is a complete NMR spectrum program which will handle a 32×32 Hamiltonian matrix, hence spin systems of up to five spin $1/2$ nuclei.

The spectra were run at 40 mc. using vacuum-distilled liquids. The tetravinyltin spectrum was measured with cyclohexane as an external standard in Willmad concentric tubes. Standardization of the other spectra has not yet been done.

Divinylmercury

The Hg^{199} satellites were treated separately as ABC-type spectra. The fact that high- and low-field sets are unmixed indicates that all Hg -H coupling constants are of the same sign. (The work of Narasimhan and Rogers on diethylmercury shows the $Hg-CH_2$ and $Hg-CH_3$ J -values are of opposite sign). From the parameters obtained for the satellite spectra the values for the main spectrum were determined and found to give good agreement with observed frequencies and intensities. Results are summarized in the final table.

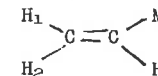
Tetravinyltin

Two sets of satellites arising from Sn^{117} and Sn^{119} spin-coupling were observed as closely spaced doublets, since the gyromagnetic ratios of these two nuclei differ by only 4.6%. The Sn^{119} spectra were analyzed as separate ABC-type systems, assigning line groups by assuming the relationship $J_{cis}^{trans} > J_{gem}$ for the vinyl protons. When the agreement between computed and observed frequencies for the satellites was within experimental error, the corresponding parameters for the main spectrum were calculated. These also resulted in an acceptable fit to the observed spectrum.

Trivinylaluminum etherate

The liquid studied is a one-to-one addition product of diethyl ether with freshly prepared trivinylaluminum, since the pure vinyl compound polymerizes rapidly when isolated. Coupling constants from the previously studied materials and "guessed" chemical shifts were used as initial approximations for the spectrum computations. Subsequent trial- and-error refinement of parameters brought the computed frequencies into agreement with the observed to ± 0.2 cps. This case convinced us of the need for a good least-squares refinement program which is now being written.

Summary of NMR Spectral Parameters



Chemical Shifts, cps (relative to H_1) at 40 mc.

	H_1	H_2	H_3
Divinylmercury	0	-22.0	-53.6
Tetravinyltin	0	-17.9	-25.1
Trivinylaluminum etherate	0	-12.7	-22.1

Proton-Proton J -Values, cps

	J_{12}	J_{23}	J_{13}
Divinylmercury	3.5	13.1	21.0
Tetravinyltin	3.7	13.2	20.3
Trivinylaluminum etherate	6.1	14.8	20.9

Metal-Proton J -Values, cps

	J_1	J_2	J_3
Divinylmercury	162.7	301.1	132.4
Tetravinyltin	90.7	182.9	99.4

Sincerely,

Donald W. Moore

Donald W. Moore
Physical Chemistry Branch

Fig. 1 40 mc. NMR spectrum of Divinylmercury showing Hg ¹⁹⁹ satellites

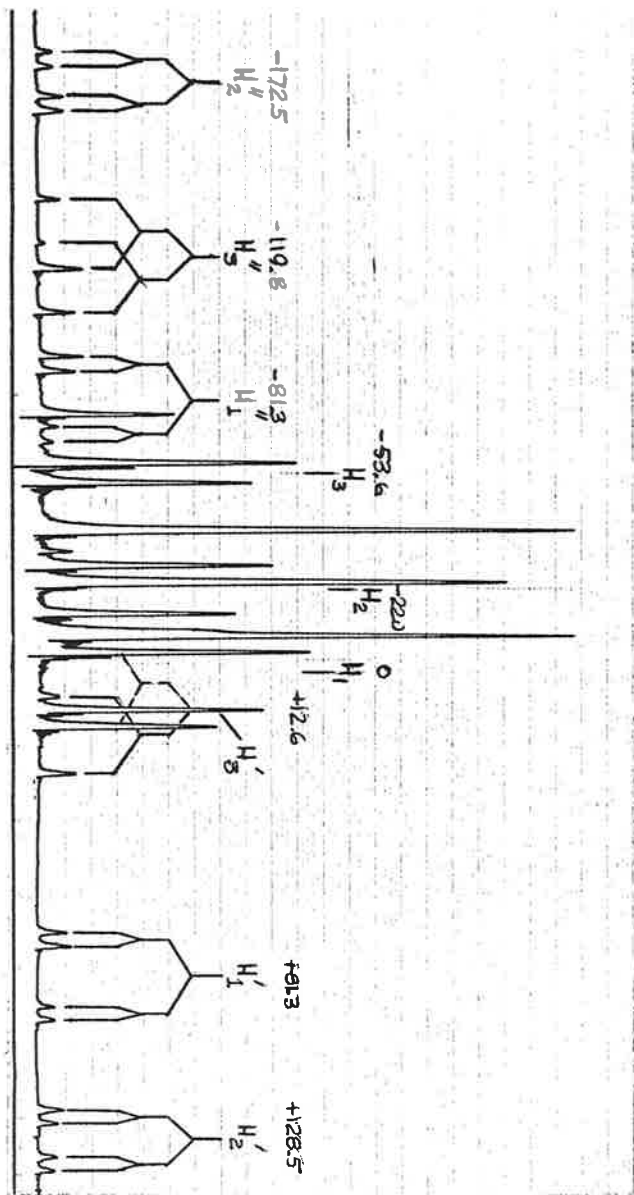


Fig 2 Tetravinyltin
40 mc

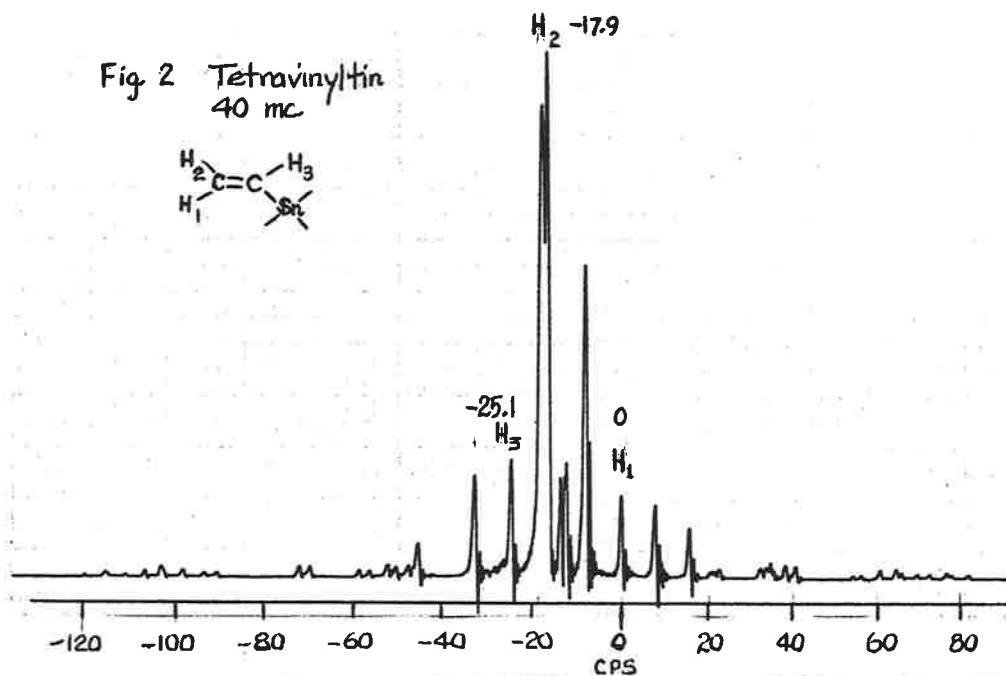
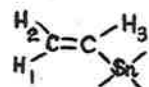
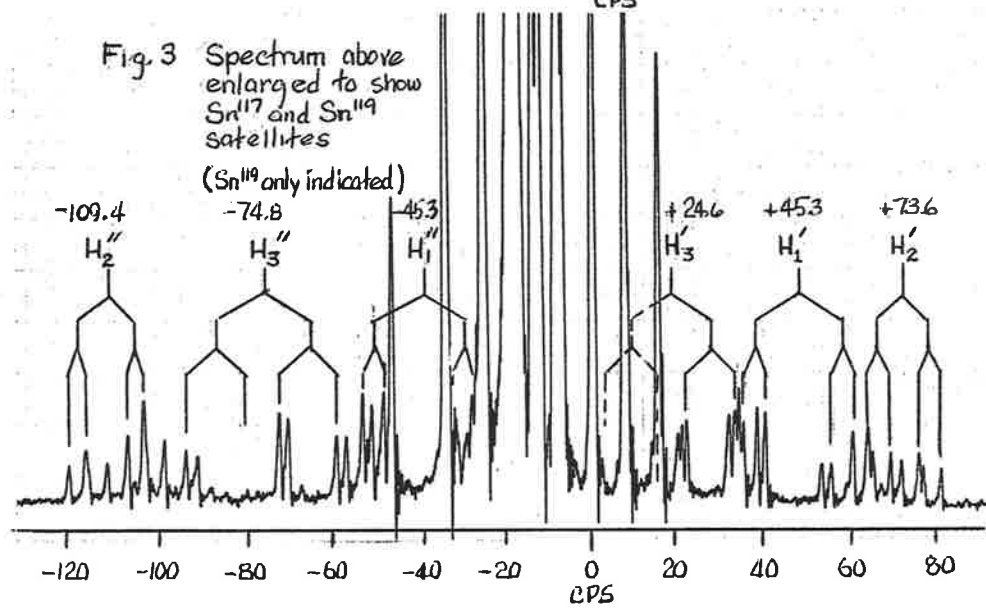


Fig. 3 Spectrum above
enlarged to show
Sn¹¹⁷ and Sn¹¹⁹
satellites
(Sn¹¹⁹ only indicated)



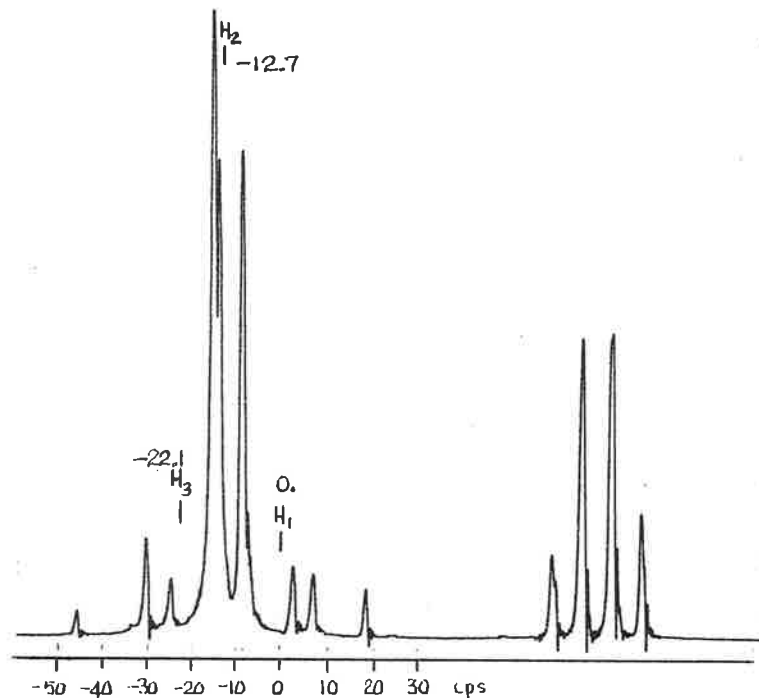


Fig. 4 40 mc. NMR spectrum of Trivinylaluminum etherate with CH₂ portion of ether spectrum

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
DEPARTMENT OF CHEMISTRY
CAMBRIDGE 39, MASSACHUSETTS

May 18, 1960

Dr. Aksel A. Bothner-By,
Mellon Institute,
Pittsburgh, Penna.

Dear Aksel:

Some time ago you sent us a copy of your IBM 704 program for the calculation of high resolution spectra. In its original form it is rather difficult to use in analyzing complex spin systems because of the form of its output: namely, a table of numerical line frequencies and intensities. In order to use the output for a five spin case (210 lines) it is absolutely essential that a plot be laboriously constructed. Even then, it is often hard to compare the result with experiment when there are many nearly overlapping lines in the theoretical spectrum, since it is hard to know to what extent their intensities should be added.

Accordingly some changes have now been made. The lines resulting from your program are first ordered according to their frequencies. All of the lines, or those lying in any chosen range of frequency, are then displayed on the 704's cathode ray tube. In this display, each line is replaced by a Lorentz curve whose characteristic width is chosen in advance, according to the experimental resolution. The tails of all component lines are added where they overlap. The end result is a calculated spectrum which can be compared directly with experiment. The pattern can be photographed by flipping a switch, or cards containing new chemical shifts and coupling constants can be fed in immediately. M. P. Barnett is writing a program which will permit immediate modification of the parameters by punching in the new ones on a Flexewriter keyboard.

Copies of the complete program are available to anyone who has access to a 704 with 8192 words of core storage and a cathode ray tube display. The display part of the program does not work for more than five spin-1/2 nuclei.

Yours sincerely,

C. S. Johnson, Jr.

C. S. Johnson, Jr.

J. S. Waugh
J. S. Waugh

OH CHEMICAL SHIFTS IN CHELATE STRUCTURES

J. H. Chaudet

Research Laboratories, Tennessee Eastman Company, Division of Eastman Kodak Company, Kingsport, Tennessee

The chemical shift values at 40 mc. for the OH proton in several chelates were compared with the effectiveness of the compounds in stabilizing cellulose acetate butyrate against degradation by ultraviolet radiation. A direct relation between the chemical shift values and stabilizing effectiveness was found. The results are summarized in the following table.

<u>Chelate</u>	<u>OH Chemical Shift, Cps.</u>	<u>Stabilizing Effectiveness, Hr.</u>
None	---	200
2, 6-Dihydroxybenzophenone	-160	600
Phenyl salicylate	-220	1000
2, 2'-Dihydroxybenzophenone	-220	1000
2, 4-Dihydroxybenzophenone	-280	2400
2-Hydroxy-4, 4'-dimethoxybenzophenone	-310	>2600

NMR spectra were recorded on methylene chloride or acetone solutions of the chelates (concn., approx. 10% by wt.). Water was used as the external standard for determining the OH peak positions. Mixtures of the chelates with 2, 6-dihydroxybenzophenone were used in determining the relative OH chemical shift values.

Exposures to ultraviolet radiation were made in an Atlas Twin-Arc Weather-Ometer that had been modified by the addition of ten 20-watt Westinghouse fluorescent sun lamps to remedy the short-ultraviolet deficiency of the enclosed carbon arcs. Samples were pre-

pared by milling the compounds, in 1% concentration, with the plastic. The effectiveness of a compound as a stabilizer was measured in terms of the hours of exposure required to cause a 25% loss in flexural strength.

The above data show that the more negative the chemical shift value of the additive, the greater is its stabilizing effectiveness. It is suggested that the OH chemical shift position in these compounds is a measure of hydrogen bond strength. The stabilization effectiveness of the chelate-type structure has long been associated with hydrogen bonding.

The OH chemical shift position for the chelates is not concentration dependent, as is the OH resonance in systems that contain intermolecular hydrogen bonds. The chelate OH proton exchanges readily when a mineral acid is added to the solution.

Some dihydroxybenzophenones which contain two chelate structures give two OH resonances. 2, 2'-Dihydroxy-4-methoxybenzophenone is an example, the two OH peaks occurring at -207 and -268 cps. Symmetrical dichelate structures, such as 2, 6-dihydroxybenzophenone, have a single OH resonance.

Further investigations of these chelates are being made.



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Baytown, Texas

May 19, 1960

Dr. A. A. Bothner-by
Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Pa.

Dear Aksel:

In MELLONMR No. 13 Jim Shoolery, LeRoy Johnson, and Wes Anderson proposed a scheme for the presentation of NMR spectra. This scheme provides data which would be usable directly by the operator when tetramethylsilane is the internal reference compound and peak displacements are measured by the audio-image method. The proposal has considerable merit under these conditions, and therefore deserves consideration. It is not, however, well suited to some other important operating conditions and procedures. I should like to discuss some of these other situations and propose modifications of Jim's proposal to make the scheme more widely applicable at this time.

Although tetramethylsilane has proved to be a good internal reference with wide application, it has not proved to be universally applicable. It does not appear to be suitable in aqueous solutions or at elevated temperatures or for the study of many silicon compounds. It will still be necessary to use other internal reference materials for many studies. If each spectrum is marked with only a single set of scales, it will either be necessary to shift the scale zero and possibly its direction for each reference compound, or else to give up the convenience of recording the audio calibration oscillator readings directly.

When a proton-stabilized or similar high-stability instrument and an X-Y recorder are available, it is possible to achieve quite satisfactory accuracy for the measurement of chemical shifts by using a standardized horizontal scale which is simply shifted slightly from spectrum to spectrum to correspond to the solvent shifts indicated by the reference material.

Dr. A. A. Bothner-by - 2

May 19, 1960

In this case operating convenience does not dictate a particular location for the scale zero. It's just as easy to set the tetramethylsilane (or other) peak on 10 or on 7 as it is to set it on zero. Under these conditions one never employs the laborious audio-image method for measuring peak separations. This speeds up the work considerably and permits the study of many more samples in a given time. We find this system to be precise to about ± 3 cps at 60 mc and we expect the precision to be improved still further in the future. It will still be necessary to use the audio-image method for determining peak separations with precisions greater than 1 cps for the foreseeable future, but this can generally be confined to the production of permanent record spectra. It is not often required for analytical work.

When the operator is thus divorced from the audio oscillator, he finds the parts-per-million scale to be more desirable than the frequency scale because it is frequency independent and permits the direct comparison of the chemical shifts of spectra run at all frequencies. Spectroscopists who operate in this manner do not think in terms of cycles per second but in terms of parts per million, and the frequency scale has little utility in the study of chemical shifts. It does, of course, have utility in the measurement of coupling constants. The operating convenience, accuracy and general utility of the highly stabilized spectrometer are so great that it seems quite reasonable that high-stability systems will be placed into use as fast as developments and economics will permit. We should, therefore, adopt a "standard" scale which will be satisfactory for these future operating practices as well as for the current ones.

Spectral scales which are standardized in direction, length, and units, are highly desirable for a collection of reference spectra which are to be searched during the course of identifying unknown materials. We find the standardized spectra save considerable time in recognizing and identifying functional groups and molecular configurations. To reduce the rate of obsolescence, the standard chosen should be applicable to as many foreseeable situations as possible.

One solution to both the operating convenience problem and the desirability of having completely standardized scales is the presentation of multiple scales on the spectrum. The frequency scale could, as the Varian group proposes, start at the internal reference peak and run in either direction as dictated by the operating convenience of the audio-image method. There could then be two parts-per-million scales, one of which corresponds directly to the frequency scale. The other would be the "standard" scale in which positive numbers would increase from left to right with the zero being somewhere near the center or left end of the scale like the API or "tau" scales. The frequency scale and its corresponding parts-per-million scale would change in zero position and sometimes in direction with changes in internal reference material, but the "standard" scale would not change, and would be applicable to all internal reference compounds and operating conditions.

Dr. A. A. Bothner-by - 3

May 19, 1960

The MELLONMR 13 letter proposes a scan length of 8.33 ppm with tetramethylsilane at the upper end of the scale. According to our data, such a scan would exclude a few of the resonances observed in high molecular weight condensed-ring aromatic molecules and would exclude aldehydic hydrogen resonances. Inclusion of all the aromatic resonances requires a scan length of about 9 ppm below TMS, while inclusion of the aldehydic hydrogen resonances requires a scan length of 10 ppm below TMS. I think the aldehyde group is sufficiently common in organic chemistry to warrant its inclusion in the "standard scan", thus dictating a scan length of 10 ppm from TMS. Actually, of course, one must allow a little room on each side of the end resonances for full presentation of their skirts and for convenient termination of the scales. This requirement extends the total scale length to about 10.5 ppm. The length of 12.5 ppm specified for the API spectra was based, as I remember, on operating convenience as dictated by the characteristics of the Varian time-base recorder rather than by a real need for the presentation of 12.5 ppm of spectrum. With an X-Y recorder, aspect ratio and scan length are independent, so that a reconsideration of this specification can be made when X-Y recorders achieve widespread use.

Very truly yours,

N. F. Chamberlain

N. F. Chamberlain

NFC:ms

WASHINGTON UNIVERSITY
SAINT LOUIS

DEPARTMENT OF PHYSICS

May 23, 1960

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

Dear Dr. Bothner-By:

The description of the resolution and sensitivity of a high resolution spectrometer is a relatively straightforward matter, but I am unaware of any sort of general yardstick that might be applied to the description of such a spectrometer's instability. Consequently, I would like to offer a possible way, for which I do not necessarily claim originality and completeness, that might be useful in comparing various spectrometers.

One can identify a root mean square error per second that is essentially independent of the sweep rate and which describes the instability of a spectrometer, at least in first approximation. Running the quartet in acetaldehyde several times at a number of different sweep rates and then measuring the separation between adjacent lines yielded the following information:

R	\bar{t}	σ	σ'
.064 cps/s	44.6s	$\pm .660$ cps	$\pm .015$ cps/s
.16	17.8	.238	.013
.24	11.9	.164	.014
.40	7.13	.0835	.012

where R = sweep rate, \bar{t} = the average time between successive peaks, σ = the observed rms error arising in the measurement of the spacing between two successive peaks, and $\sigma' = \sigma/\bar{t}$.

At 56.4 mc/s this amounts to an instability of $\pm 2.5/10^9$. The instrument is a Varian V-4302 spectrometer replete with flux stabilizer. Blotter paper has been placed over the pole cap covers and stabilizer coils. One inch of styrofoam covers the yoke. The water is held to $\pm .01^\circ\text{C}$ for periods of at least an hour at a time, and the air is held to roughly $\pm 0.3^\circ\text{C}$.

WASHINGTON  UNIVERSITY
SAINT LOUIS

DEPARTMENT OF PHYSICS

Dr. Aksel A. Bothner-By
May 23, 1960
Page 2.

The quantity seems to have value in judging the suitability of various methods for determining the chemical shift as well as in making line width and area measurements. Furthermore it offers a means of objectively deciding what stabilization measures are necessary through comparison of this quantity for spectrometers in different laboratories.

Yours sincerely,
Thomas R. Hughes
Thomas R. Hughes

TRH/ab

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* * * * *

DEADLINE!!!

For Issue No. 21 is
Monday, June 27.

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