

## No. 251

### August, 1979

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All Newsletter Correspondence, Etc., Should Be Addressed To:

Dr. Bernard L. Shapiro Department of Chemistry Texas A&M University College Station, TX 77843 U.S.A.

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(i)

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PHONE: (403) 432-5460

June 21, 1979

Professor B.L. Shapiro Department of Chemistry Texas A & M University College Station, Texas 77843

Dear Barry:

### <sup>19</sup>F Laser CIDNP Enhancements

Recently, Kaptein and coworkers<sup>1</sup> have proposed the use of a laser induced CIDNP experiment to probe the surface accessibility of tyrosyl residues in proteins. We have been involved in the biosynthetic incorporation of fluorotyrosyl residues into proteins such as alkaline phosphatase<sup>2</sup> and the M13 gene 8 protein<sup>3</sup>, and the use of different experiments such as the solvent isotope shift to probe their accessibility. In this letter, we would like to show you some of our first 254 MHz <sup>19</sup>F NMR laser CIDNP experiments on fluorotyrosine. Figure 1 shows the spectrum in the light and the dark, with the enhancement being ca. 40x. Note the change of sign from the <sup>1</sup>H CIDNP on TYR.

<sup>1</sup>Kaptein et al., Nature 274, **293** (1978).

<sup>2</sup>Hull and Sykes, Biochem. <u>15</u>, 1535 (1976).

<sup>3</sup>Hagen et al., Biochem. <u>17</u>, 3860, <u>18</u>, 0000 (1979).

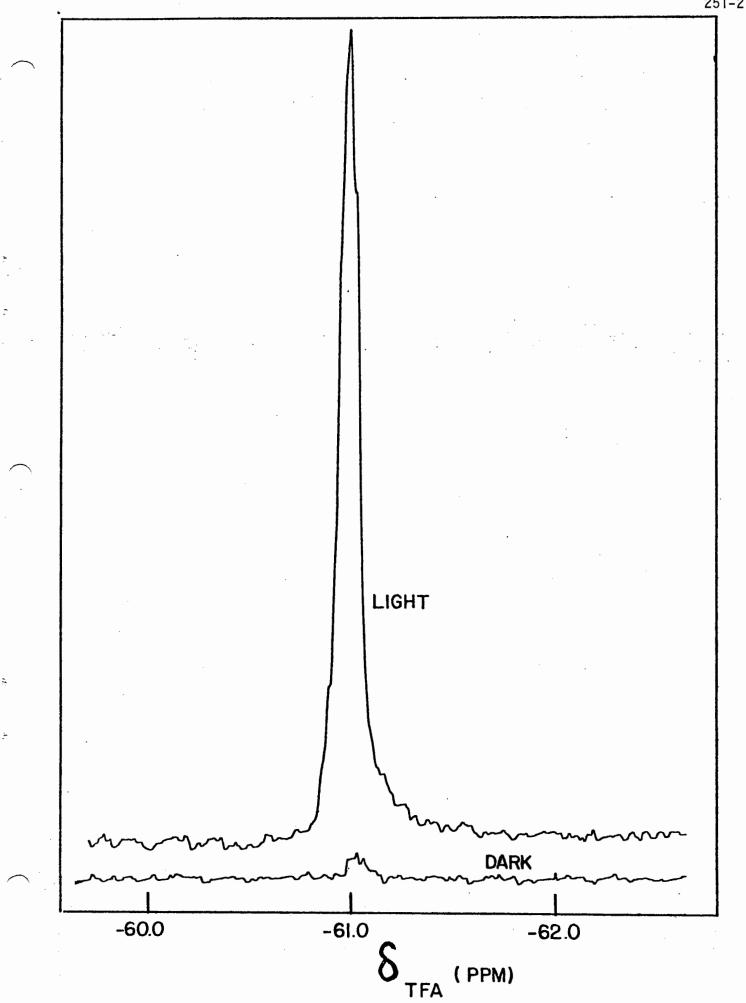
Best regards,

John H. Baldo

Brian D. Sykes

/vb

P.S. We would like to thank Bob Swindlehurst for the loan of the laser.



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#### DEPARTMENT OF PHYSICS INSTITUTE OF CYBERNETICS ACADEMY OF SCIENCES OF THE ESTONIAN SSR

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Nº \_\_\_

#### ИНСТИТУТ КИБЕРНЕТИКИ АН ЭСТОНСКОЙ ССР СЕКТОР ФИЗИКИ

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\_\_\_\_\_<u>June 1, \_\_\_\_\_\_197\_9</u>

Professor Bernard L. Shapiro Department of Chemistry Texas A & M University College Station, Texas 77843 U S A

Dear Professor Shapiro,

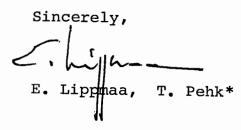
One more Estonian silicon and carbon chemical shift standard for solids (and liquids)

In a recent issue (Newsletter 233-9) the combinatorics of the cubic  $Q_8M_8$ , or Si<sub>16</sub>O<sub>20</sub>(CH<sub>3</sub>)<sub>24</sub>, was presented by Haigh to demonstrate a large number of possible isotopic isomers of this compound, which we used as a secondary reference in our first low-field high resolution <sup>29</sup>Si NMR experiments. This compound is fine for setting up magic angle spinning cross polarization experiments, but it is not especially suited as a reference compound. It has quite a complicated <sup>29</sup>Si spectrum due to deformation of the cube in the solid phase (see Figure).

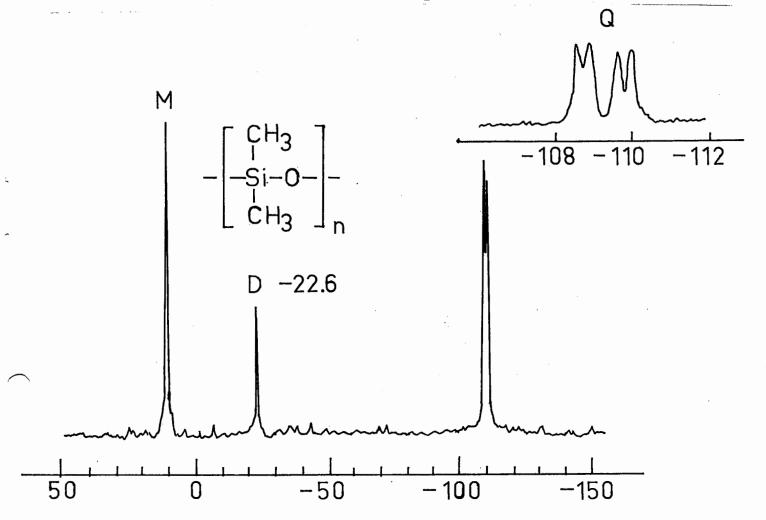
The lack of an inner reference in solid state high resolution NMR can be a real problem if close chemical shifts in different samples are to be compared. We propose methyl silicone rubber,  $D_n$  or  $[-Si(Me)_2O-]_n$ , for this purpose. It separates easily from any powder and most liquids, and can be used as a piece of tubing around the inner perimeter of the sample spinner. It has the following advantages:

- 1. It gives narrow <sup>13</sup>C and <sup>29</sup>Si NMR lines even without high-power decoupling.
- 2. The resonances are close to those of TMS ( $\delta^{13}C = 1.5$  ppm and  $\delta^{29}Si = -22.6$  ppm at 30°C).

- 3. The chemical shift anisotropies of both nuclei are very low. No sidebands are created upon sample spinning and the magic angle setting is not critical.
- 4. The temperature dependence of carbon <sup>13</sup>C chemical shift in methyl silicone rubber is very low, only 0.001 ppm/°K, which is about an order of magnitude less than in TMS.
- 5. It might be a good <sup>1</sup>H reference too. We have not tried yet.
- 6. It can be used both with the conventional FT (or just CW) and the high power solid state FT NMR spectrometers, such as the Bruker Physik CXP 200/300.



\* Presently Dept. of Chemistry, The Florida State University (Laboratory of Prof. G.C.Levy).





UNIVERSITÉ DE DROIT, D'ÉCONOMIE ET DE SCIENCES D'AIX-MARSEILLE FACULTÉ DES SCIENCES ET TECHNIQUES DE SAINT-JÉROME

Laboratoire des Organométalliques J. C. MAIRE, Professeur.

2.07.1979

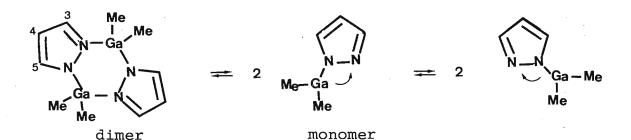
JCM/YB/192-79

Prof.B.L.SHAPIRO Texas A and M University College of Science College Station, TEXAS 77843

#### Object : 1-Dimethylgallyl pyrazole dimer-monomer equilibrium.

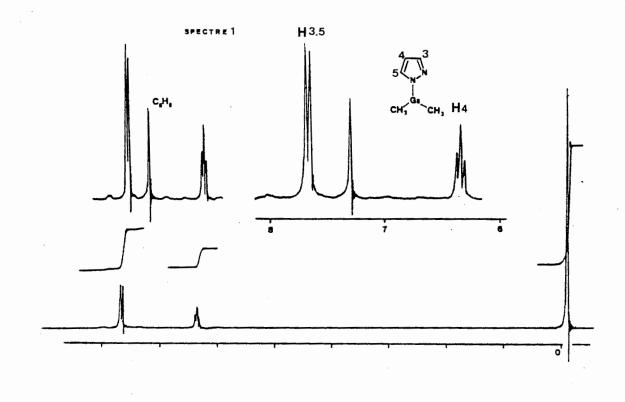
The reaction between trimethylgallium and pyrazole gives 1-dimethylgallyl pyrazole, which, in the solid state, is a dimer (see below : D.F.Rendle, A.Storr, and J.Trotter, Canad.J.Chem., 1975, <u>53</u>, 2930).

The <sup>1</sup>H n.m.r. spectrum, recorded in carbon tetrachloride, (figure 1) shows the equivalence of protons  $H_3$  and  $H_5$ . Proton  $H_4$  gives a triplet and protons  $H_3$  and  $H_5$ , a doublet. Thus, in this solvent, the dimeric structure of the title compound remains. When hexadeuterioacetone is used as solvent, all the signals are spitted into two (figure 2), corresponding to an equilibrium mixture of dimer and monomer. For the monomeric form, the intensity of the signals shows that protons  $H_3$  and  $H_5$ have the same chemical shift, consequently we are in presence of a metallotropic phenomena with the dimethylgallium exchanging between the two pyrazolyl nitrogens.



We have reccorded the spectrum of 1-dimethylgallyl pyrazole in different solvents ; the results are gathered in table 1. A more complete description of this work, including <sup>13</sup>C n.m.r. study, is being prepared for publication. Yours Sincerely. D.BOYER JC.MAIRE R.GASSEND TELGUERO

rue Henri Poincaré - 13397 MARSEILLE Cedex 4 tél.: 98-09-01 (570).



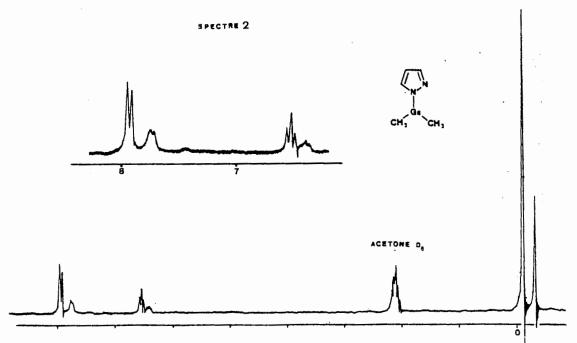


Table.	1
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SOLVANT	s Has ppm	5H4 ppm	5 H6 ppm	JH3H₄ Hz
c ci,	d 7e70	t 8 <sub>1</sub> 40	s -0,10	. 2
C.D.	d 7,55	t 6,22	s - 0,22	2
C D CI,	d 7,75	1 6,45	• _ D,02	2
CD,0D	d 7,90	t 6,60	5 - 0.10	2
ເຊີເຊັ	d 7,93	1 6.53	• - 0,07	2
0	d 7.75	1 6,43	\$ -0,30	2
CH, CN	d 7,55	1 6,27	= - 0,25	2
	d 7,48	t 5,22	s - 0,45	2
DMSO D6	de 8,10	te 5,50	10-0.03	[
DIOXANE	d 7,95	1 6,65	s - 0,00	i i
	7,70		5 - 0,2 5	·

Dr. Alois Steigel Institut für Organische Chemie der Universität Düsseldorf Direktor: Professor Dr.L.Birkofer

4 Düsseldorf, den July 5, 1979 Universitätsstraße 1 Fernsprecher 311-2298/99

Professor B. L. Shapiro Department of Chemistry Texas A and M University College Station. Texas 77843

### $^{13}$ C NMR Study of Diethylmalonic Hydrazide and its Methyl Derivatives

Dear Professor Shapiro,

The tendency of cyclic hydrazides to assume the unsymmetric tautomeric lactam-lactim form has been suspected for a long time and was termed "Dikationeffekt" by  $Arndt^{1}$ . However, reliable information on the degree of such a structural preference is still very limited. In recent studies<sup>2</sup> on N-phenyl- and N-triazolyl-dialkylmalonic hydrazides, contradictory results have been obtained. While UV measurements indicated the presence of the lactam-lactim structure, IR and <sup>1</sup>H-NMR data were interpreted in favor of the dilactam structure.

In order to examine the suitability of  ${}^{13}C$  NMR in solving this structural problem, we synthesized the methyl derivatives 2-5 of diethylmalonic hydrazide 1. The sizeable shift differences between the

R R <sup>1</sup>		$\mathbf{X}_{_{\mathrm{Et}}}^{^{\mathrm{Et}}}$	R		$\sum_{i=1}^{2} \sum_{i=1}^{2} \sum_{j=1}^{2} \sum_{j=1}^{2} \sum_{j=1}^{2} \sum_{i=1}^{2} \sum_{j=1}^{2} \sum_{j=1}^{2} \sum_{j=1}^{2} \sum_{j=1}^{2} \sum_{j=1}^{2} \sum_{i=1}^{2} \sum_{j=1}^{2} \sum_{j$	:	R N		F		
	R	$R^1$		R	$R^1$		R	$\delta(^{13}\rm{CO})^{3)}$		R	$\delta(^{13}CO)^{3})$
$\frac{1a}{2a}$	$^{\rm H}_{\rm CH_3}_{\rm CH_3}$	н н Сн <sub>3</sub>	$\frac{\frac{1b}{2b}}{\frac{4}{5}}$	н СН <sub>3</sub> н СН <sub>3</sub>	H H CH <sub>3</sub> CH <sub>3</sub>	$\frac{6}{7}$	н Сн <sub>3</sub>	179.4 174.3	8 9	н Сн <sub>3</sub>	180.8 178

C-3 and C-5 resonances in the fixed lactam-lactim compounds  $\underline{4}$  and  $\underline{5}$  amounting to 9.3 and 6.6 ppm, respectively, seemed to provide a good basis for the purpose. However, since the blocking CH<sub>3</sub> groups are in  $\beta$ -position to these resonances, a knowledge of the  $\beta$ -effects in the lactam and lactim functions is required.

Upfield shifts of 5.1 and 2.8 ppm have been observed<sup>3)</sup> in the pyrrolidinone 7 and the pyrrolinone 9 and were attributed to the blockage of hydrogen bonding by methylation. Double bonds in  $\alpha\beta$ - or  $\beta\gamma$ -position reduce the magnitude of the upfield shift<sup>3)</sup>. As crude lactim models, carbonic acids might be taken into consideration.

The hypothetical  $\beta$ -effects of compounds 2-4 and of the lactim site in compound 5 given in the Table were determined by assuming that

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both compounds  $\underline{1}$  and  $\underline{2}$  either are present in the dilactam form  $\underline{a}$  or in the lactam-lactim form b.

Table. <sup>13</sup>C Shift Effects of  $\beta$  Methyl Groups on C-3 and C-5 in Methyl Derivatives of Diethylmalonic Hydrazide Assuming Structural Equality with the Demethyl Compounds.

Solvent	Methyl Derivative		Desmethyl Compound		Structural	Site of	Shift
Solvent	No. $\delta(^{13}C)$		No.	$\delta(^{13}{\rm C})$	Туре	Methylation	Difference
CDC13	5	173.5	4	176.8	Lactam-Lactim	Lactam	-3.3
[d <sub>6</sub> ]dmso	5	172,6	<u>4</u>	175.3	Lactam-Lactim	Lactam	-2.7
[D <sub>6</sub> ]DMSO	<u>2b</u>	170.6	$\frac{1b}{or}$	173.9 <sup>a)</sup> 173.3 <sup>b)</sup>	Lactam-Lactim	Lactam	-3.3 or -2.7
CDC13	<u>5</u>	166.9	$\underline{2b}$	174.8	Lactam-Lactim	Lactim	-7.9
[d <sub>6</sub> ]dmso	5	166.0	<u>2b</u>	172.7	Lactam-Lactim	Lactim	-6.7
[D <sub>6</sub> ]DMSO	<u>4</u>	165.9	$\frac{1b}{or}$	172.7 <sup>c)</sup> 173.3 <sup>d)</sup>	Lactam-Lactim	Lactim	-6.8 or -7.4
$CDC1_3$	3	173.5	<u>2a</u>	174.8	Dilactam	Lactam	-1.3
[d <sub>6</sub> ]dmso	3	172.3	<u>2a</u>	172.7	Dilactam	Lactam	-0.4
[D <sub>6</sub> ]DMSO	<u>2a</u>	170.6	<u>1a</u>	173,3	Dilactam	Lactam	-2.7

- a) Calculated by comparison of  $\delta(C-3)$  of 2 (172.7 ppm) with the average shift of 1 (173.3 ppm).
- b) Calculated from  $\delta(C-5)$  of 4 (175.3 ppm) by subtraction of the  $\epsilon$ -effect of the lactim methyl group in 5 on C-5 (2.0 ppm).
- c) Use of  $\delta(C-3)$  of compound 2.
- d) Corresponds to the lactam shift calculated in b), since identical to the observed shift of compound 1.

Evidently, the  $\beta$ -shifts obtained by assuming the presence of the tautomeric lactam-lactim forms <u>1b</u> and <u>2b</u> are internally consistent and favorably compare with the lactam effects of compounds <u>5</u> and <u>9</u> and with the  $\beta$ -effects of methyl carboxylates (methyl acetate: -7.4 ppm). On the other hand, the assumption of the presence of the dilactam forms <u>1a</u> and <u>2a</u> seems to give less satisfactory results. To interpret these hypothetical values one would have to invoke a +2.3 ppm sterical shift effect in compound <u>3</u> and a reduction of the upfield shift by 2.4 ppm relative to the methylpyrrolidinone 7.

Yours sincerely,

Plans Hespel Alois Steigel

- (1) F.Arndt, Angew. Chem. <u>61</u>, 397 (1949).
- (2) M. Woodruff and J. B. Polya, Aust. J. Chem. <u>29</u>, 2491 (1976); <u>30</u>, 421 (1977): 31, 1855 (1978).
- (3) G. Fronza, R. Mondelli, E.W. Randall, and G.P. Gardini, J. Chem. Soc., Perkin Trans. 2, 1746 (1977).

Prof. Dr. D. Leibfritz Universität Bremen Fachbereich Chemie/Biologie

NW 2 Leobenerstraße 2800 Bremen 33 Telefon (04 21) 218-2818/2841

July 6, 1979/t

Professor B.L. Shapiro Dept. of Chemistry Texas A & M - University College Station, Texas 77843 USA

Dear Dr. Shapiro,

#### Differentiation of Aromatic and Aliphatic Carbons

Sometimes it may be difficult to assign the unsaturated carbons in a molecule, if both olefinic and aromatic carbons are present. The off-resonance spectrum or a fully coupled spectrum may fail, if many lines cover a narrow range.

In this case one can add an equimolar amount or a slight excess of N-chloro-sulfonylisocyanat (NCSA) to the sample in a chloroform solution. The reaction can be done in the NMR sample tube, while it is cooled under a rinsing water-tab. The sample can be measured without a purification, unless the olefin undergoes a cationic polymerization. Then one removes the slight polymeric by-product via filtering on a very short silica gel column.

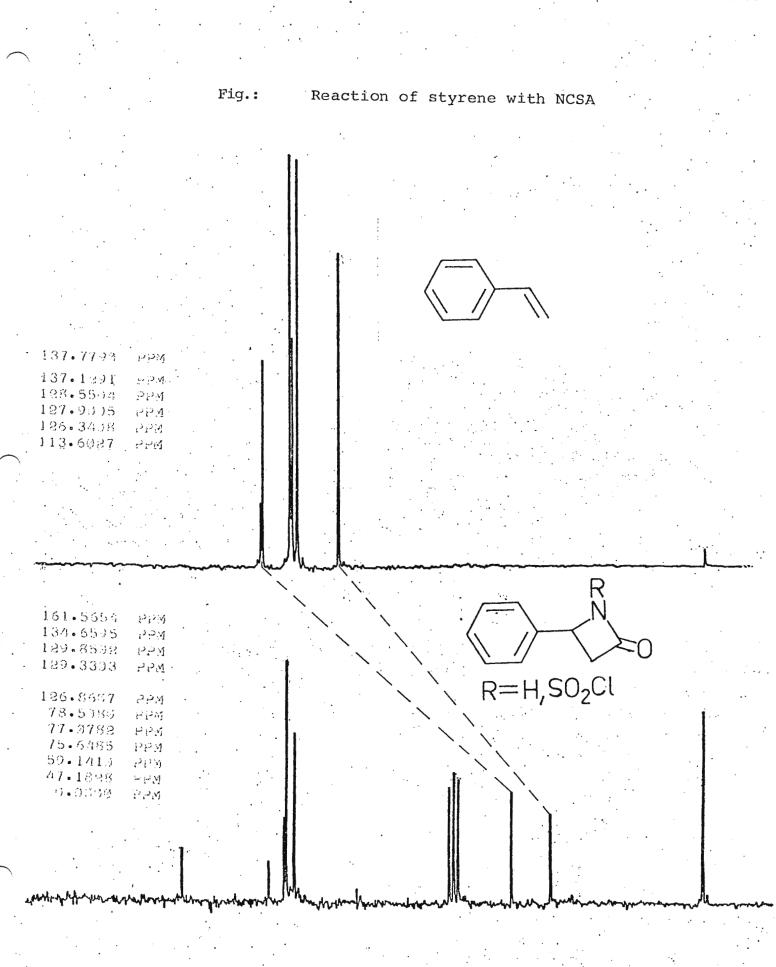
Conjugated double bonds usually do not react twice in a 2+2- cyclo addition, but only once and then rearrange<sup>1)</sup>. Nevertheless, the olefinic carbons change their chemical shift considerably.

The NCSA is commercially available (f.e. Aldrich). An application is shown in the figure.

Yours sincerely,

1. Leibfrig E. Haupt

"H.Bestian, Pure and Appl. Chem. 27, 611 (1971)



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#### STANFORD UNIVERSITY STANFORD, CALIFORNIA 94305

#### STANFORD MAGNETIC RESONANCE LABORATORY

(415) 497-4062 (415) 497-6153

July 9, 1979

Professor Bernard L. Shapiro Department of Chemistry Texas A&M University College of Science College Station, TX 77843

Waistline Broadening Using High Field Nuclear Magnetic Resonance

Dear Dr. Shapiro:

It has come to my attention through direct association with those organisms whose nocturnal habits and harried manners identify them as NMR spectroscopists, that there is a marked similarity in the basic morphology of these creatures. This manifests itself in the middle portion of the torso where a large swelling takes place. This has been identified by Oink and Glutton (1) as the Spare Tire Effect (STE). Since the author noticed a similar change to her own physique upon direct association with these subjects, it was decided that an investigation was in order, since the possibility existed that the phenomenon may have been contagious.

#### EXPERIMENTAL

Several subjects were observed during scheduled feeding times to determine the quantity and type of nourishment consumed. Contrary to the expected result, it was discovered that, with few exceptions, subjects ate relatively small amounts of food with a normal or below normal percentage falling into the junk food category (2). This line of investigation proving inconclusive, the author next attempted to observe the environment in which these creatures dwell, hoping to derive from this source some further insight into the problem. This line of investigation proved more effective in that indirect evidence was gathered which was more in line with the expected result. Table I summarizes this data: Professor Shapiro July 9, 1979

Change Noted	Time	Weight of Operator
Six-Pack of Beer Disap- pears from Lab Refrigerator	2200-0800	210
2" layer of peanut shells around NMR console	2000-0800	190
Trash can overflowing with Frito-Lay bags	2200-0800	230
Chocolate colored smears on oscilloscope screen	1800-0800	250
		<u> </u>

TABLE I

All of the above changes occurred between the times indicated. A direct observation was not possible because standard technique dictates that no investigator in his right mind works during these hours (3). The operator weight shown is that of the spectroscopist assigned to the instrument during the indicated times. This indirect evidence provided the impetus to further the investigation by interviewing the subjects involved during the data gathering phase shown in Table I. The subjects are numbered consecutively, number 1 being the first listed. The results of this phase of the investigation are shown in Table II. The responses are in answer to the question, "What did you do last night?"

TABLE II

Subject Number	Response
1	Ran spectra, counted spiders on wall.
2	Ran spectra, kicked computer
3	Ran spectra, ran spectra, ran spec <sub>t</sub>
4	Burp!

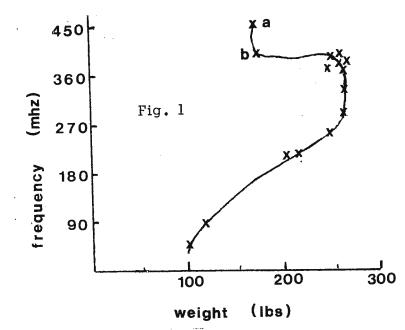
Upon further interrogation, all subjects insisted that, to the best of their knowledge, nothing had been consumed other than a great deal of energy. None of the interviewees noticed the changes taking place during their stay in the lab, though #2 stated that he slipped on a cylindrical object which crushed under his weight, forcing him to kick the computer.

Professor Shapiro July 9, 1979

These direct lines of research exhausted, the author decided to get an historical overview of the situation by speaking with what Cohn and Jardetzky (4) have characterized as "old-timers" (OT). All of the subjects interviewed stated that, even though the state-of-the-art in instrument technology has vastly changed, one item in particular has gotten noticably larger (besides spectroscopists) and this is the <u>MAGNET</u>. There seemed a direct correlation between weight of spectroscopists and the size of the magnets with which each worked (this being readily observable while interviewing the OT's).

#### RESULTS

Upon further observation of OT's, it was ascertained that when fields were low, so was their weight. To double-check the correlation, other subjects were analyzed and the results are shown in Fig. 1.



The subjects represented by points a and b were 6'5" and had only been in the field two years. The higher field at 450 MHz has also been available for a short period of time so the larger weight gain associated with it should be expected in the near future.

#### DISCUSSION

The results of this study have far-reaching effects on all those associated with nuclear magnetic resonance. For the sake of brevity, a few of the major ones have been summarized below: Professor Shapiro July 9, 1979

1. Spectroscopists can gorge themselves with impunity.

2. Spectroscopists should get hazardous duty pay (this applies to administrators also, see ref. 3).

3. Spectroscopists should be supplied a clothing allowance over and above their regular salaries, since magnets are getting increasingly larger, it follows that they will also.

In conclusion, NMR need no longer be only an acronym for nuclear magnetic resonance, but can now also represent "NO MORE RAZZING" by skinny family and friends, as scientific evidence now exists which supports the conclusion that its job, not jaws.

#### REFERENCES

- Oink, O. and Glutton, I.M.A., "A Study of Obesity on Malibu Beach Using UV Techniques." J. Fat Soc. <u>3</u>, 21-30 (1975).
- McDonald, R., "An Annotated List of Junk Food." Grease Lett. <u>27</u>, 9-56 (1971).
- 3. Bureaucrat, B.S., "How to be an Effective Administrator," U.S. Government Publication #5,260, pp. 1,255-50,976 (1950).
- 4. Cohn, M. and Jardetzky, O., personal communication (1978).

Sincerely yours,

Petunia

Petunia Pig Administrative Services Coordinator



BRUKER announces a new landmark in <u>low-cost</u> high-resolution superconducting NMR spectrometers with

# <sup>1</sup>H frequency of 250 MHz

.....



ER Instruments, Inc.

MANNING PARK BILLERICA, MASSACHUSETTS 01821 (617) 667-9580

July 12, 1979

Dr. B. Shapiro Texas A & M University Department of Chemistry College Station, TX 77843

Dear Barry,

I would like to take this opportunity to draw your readers' attention to a number of openings in the area of NMR and ESR applications/marketing, where we would like to expand our staff by filling the following positions:

1. Product Manager, High Power Pulse NMR

For this position, we seek a Ph.D in Physical Chemistry with a strong background in instrumentation, experience in pulsed NMR spectrometry, and its applications in physics as well as chemistry.

2. High-Resolution High-Field NMR Applications Chemist (Ph.D)

The ideal individual has several years of experience in high-field NMR and its applications in chemistry and bio-logy, as well as a flair for instrumentation both in terms of hard- and software.

3. EPR Postdoctoral Position

For this position, we anticipate a physical chemist familiar with biochemical/biophysical applications of EPR and a good understanding of instrumentation.

4. NMR Field Engineers

The ideal candidates have several years of experience with radio frequency technology and digital electronics, as well as some understanding of cryogenics.

Our Applications Laboratory at Billerica presently accommodates the following instrumentation:

- \* WH-360 Widebore
- \* WM-250
- \* WP-200 Widebore
- \* WP-80 DS

All Systems Are Fully Broadbanded

251-17

By fall of this year, we intend to add the following equipment:

\* WH-400

\* CXP-200

\* ER-200

Applicants are kindly requested to submit their resume to the undersigned.

Sincerely,

belie

Dr. F. W. Wehrli Executive Vice President Bruker Instruments, Inc.

Dr. B.L. Shapiro

July 10, 1979

6. Block averaging of transformed data will allow further averaging without memory overflow and will thus allow reduction in noise, but will make no difference in the dynamic range, which is ADC limited. The averaging of blocks of transformed data will not average out the round-off error noise of the Fourier transform in small wordlength computers.

Thus, my conclusions are unchanged from earlier notes on the subject except to say that there seems to be an awful lot of snow swirling around in certain commercial publications on the subject. I hope this provides a suitable entrenching tool.

1. J.W. Cooper, J. Magn. Resonance, 22, 345 (1976).

2. J.W. Cooper, L.S. McKay and G.B. Pawle, ibid., 28, 405 (1977).

3. J.P. Marchal, J. Brondeau and D. Canet, ibid., 33, 469 (1979).

4. R. Kaiser, TAMUNMR 249, 36.

Sincerely,

yin los

James W. Cooper Assistant Professor of Chemistry

JWC/lpl



#### TUFTS UNIVERSITY

Department of Chemistry

July 10, 1979

Dr. B.L. Shapiro TAMUNMR Newsletter Department of Chemistry Texas A&M University College Station, TX 77843

"More (or less) on Dynamic Range"

Dear Barry:

After your Dynamic Range Memorial Issue (TAMUNMR 249) and your blue-green note, I felt I might well summarize the results of dynamic range theories developed by various workers. (1-4)

1. We define dynamic range as the ability to measure small <u>peaks</u> in the presence of large peaks. The ability to measure a noisy baseline is irrelevant, although a small amount of noise will allow slightly smaller signals to be eventually averaged into the spectrum.

2. The maximum theoretical dynamic range that one can measure is 2d+1, where d is the number of bits in the ADC. The smallest peak that can be measured is that which has an intensity of about 1/2 bit, as-suming that the large peak fills the ADC. Signal averaging allows the observation of this additional 1/2 bit, but does not allow further improvement in dynamic range because the ADC never measures smaller peaks.

3. As Professor Kaiser has noted, the Fourier transform leaves the rms S/N unchanged and thus, the dynamic range unchanged (Parseval's theorem). (Thus Parseval's search for the large dynamic range Holy Grail is unwarranted).

4. Dynamic ranges larger than that apparently predicted by this 2<sup>d+1</sup> theory are, however, routinely observed, when the large peak has a substantially greater linewidth than the small peak. This occurs because the large peak thus dies out faster in the fid, leaving the remainder of the fid to register the smaller peak unhindered by the large peak. Some data have been tabulated for this case (2).

5. Computer word lengths only limit the dynamic range of FT data when the round-off error (noise) of the Fourier transform becomes significant. This occurs only when a short wordlength (16 or perhaps 20-bit) computer is used in a single-precision integer mode transform. A longer wordlength only allows a greater dynamic range in that the Fourier transform no longer limits the native dynamic range of the computer's ADC and the spectrometer's amplifiers.

#### 251-19

#### FREIE UNIVERSITÄT BERLIN

Fachbereich Chemie (FB 21) Institut für Organische Chemie (WE 02)

Dr.K.Roth

Freie Universität Berlin, FB 21. WE 02, Takustraße 3, 1000 Berlin 33

Professor B.L.Shapiro Dep.of Chemistry Texas A&M University College Station, Texas

#### A Simple Resolution Enhancement Technique: Delayed Fourier Transform.

FU

In general the shape of the magnetic field is a complex mixture of several field gradients which leads to a non-exponential FID and after FT to a non-Lorentzian line. One way of separating the broad from the sharp components exists in the subdivision of the FID into a first part which contains more information about the broad part and into a second one with predominant information about the sharp component of the peak. Therefore by omitting a number of data points from the beginning of a normal FID line broadenings due to field inhomogeneities can be partially compensated.

This principle of the so-called <u>Delayed Fourier Transformation</u> (DFT) was first described by Seiter et al (1) who developed this technique for the separation of the broad and sharp components in bilayers. Very recently we have investigated the application of the DFT technique to nmr spectra with line width in the order of 0.1 Hz.

A typical result for a <sup>1</sup>H routine spectrum (fig.1) shows the expected discrepancy from the theoretical Lorentz form. After cutting-off the first third of the FID the lineshape becomes nearly Lorentzian and the line width decreases by a factor 2.

Any delay  $\mathcal{T}$  results in a linear phase shift of  $360 \cdot \mathcal{C} \cdot \Delta \mathcal{V}$ . For larger spectral width this phase shift cannot be compensated by the use of the conventional software without baseline distortions and one has to subdivide the spectrum into several portions of signals. Then  $\mathcal{T}$  is restricted to a value which results in a phase shift within the plotted range which can be compensated by the usual phase correction routine.

## BERLIN

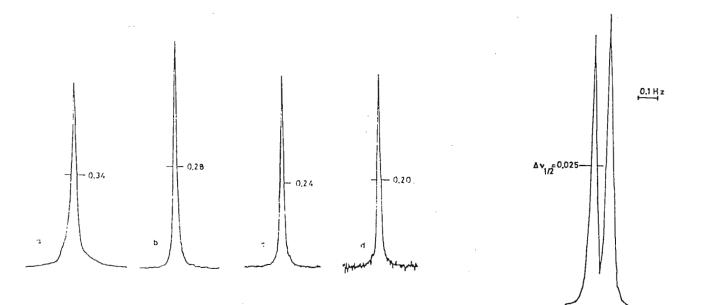
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July 19, 1979

The outstanding feauture of the DFT technique lies in the fact that all other resolution enhancement techniques can be applied subsequently and result in a further gain of resolution. In fig.2 the spectrum of ODCB is given after cutting-off 1,245 data points of the 32 K FID which corresponds to a delay of over 2 sec and after treating this FID with the conventional Convolution difference technique. The spectrum shows a beautiful shape and a line width of 0.025 Hz!

(1) C.H.A.Seiter et al, JACS <u>94</u>,2535(1972)

Sincerely Yours,



<sup>1</sup>H spectra of  $CHCL_3/CDCl_3$  mixture (1/10) at 270 MHz, aquisition time 20.48 s, delay (no. of data points / time in s) a(0/0) b(200/2) c(400/4) d(600/6).

fig.2 Small portion of <sup>1</sup>Hspectrum of ODCB(270 MHz) after a delay of 1,245 points and Conv.Diff. witha line broadening of .015 Hz.

#### 251-21

#### Université de Nancy I

D. CANET Maître de Conférences

> Professor B.L. SHAPIRO Department of Chemistry Texas A & M University

COLLEGE STATION, Texas 77843 U.S.A.

<u>Suggested title</u> : Isotropic phases of cationic surfactants as studied by proton NMR

#### Nancy, July 20, 1979

#### Dear Professor Shapiro

Thank you for your two reminders. I wish to report a study of the different phases formed by the systems n-alkylammonium chloride/D<sub>2</sub>O. At first, our purpose was to determine by NMR the concentrations at which appear these phases. Although somewhat disregarded for surfactants, proton NMR fairly easily provides the required information, as far as isotropic phases are concerned. The joined figure shows a plot of T1 and linewidth versus amphiphile concentration for the resonance corresponding to almost all methylenes in the aliphatic chain. The sharp decrease of T1 indicates the formation of globular micelles whereas the abrupt increase of linewidth, at higher concentrations, is attributed to the formation of rod shaped micelles. This procedure is rather convenient since it just takes one day to get all those data.

<sup>2</sup>H NMR of deuterium oxide allows the characterization of anisotropic phases.

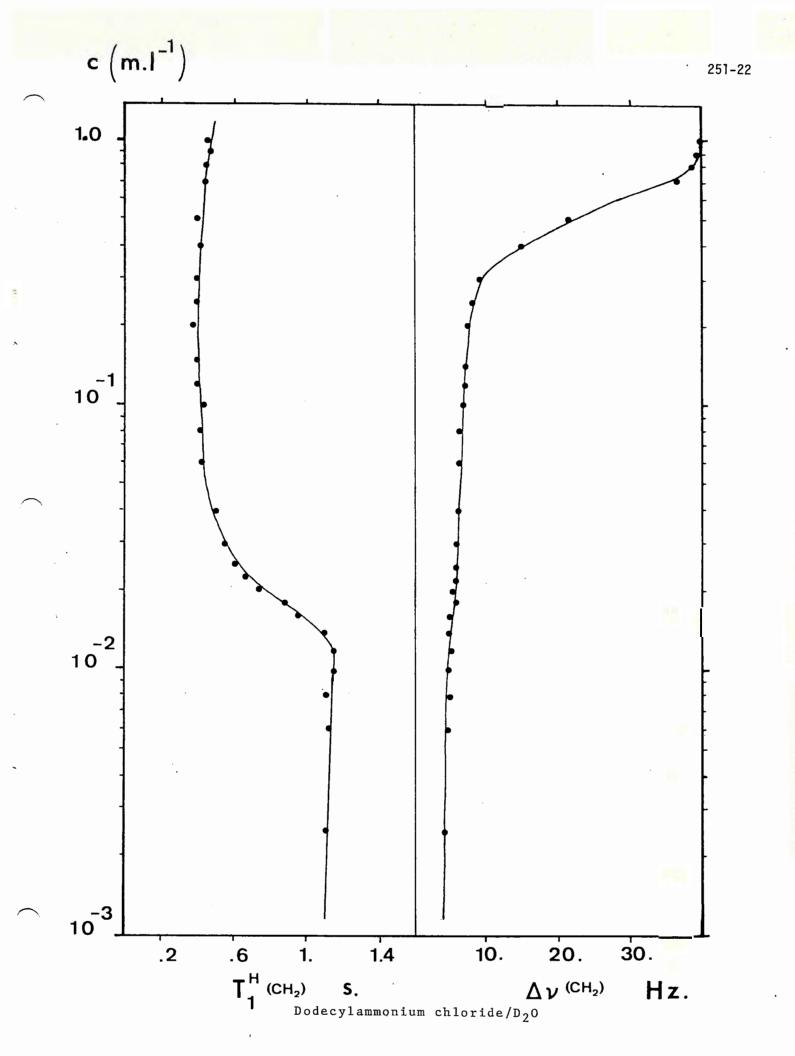
A parallel study of carbon-13 relaxation of these different aggregates is in progress. It shows that the local mobility along the aliphatic chain is the same regardless the considered phases, except for the terminal methyl.

Details concerning these experiments will hopefully be published soon.

Yours sincerely

CANET

Encl.



UNIVERSITY of PENNSYLVANIA

#### PHILADELPHIA 19104

#### Department of Chemistry

July 24, 1979

Dr. Bernard L. Shapiro Texas A&M University NMR Newsletter Department of Chemistry Texas A&M University College Station, Texas 77843

#### THE FOUR CHAMBERED ROTOR

Dear Dr. Shapiro:

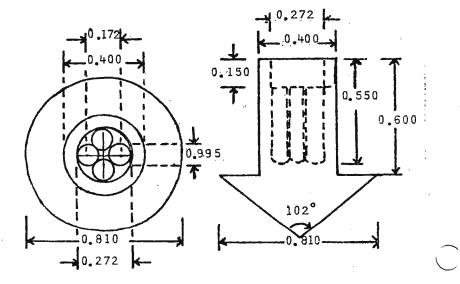
We have recently developed a rotor suitable for magic angle spinning of liquid or gel samples. Conventional rotor designs of the Andrew or bullet variety tend to have unstable spinning performance when the sample chamber has liquid in it. By drilling four holes equally spaced about the center of a solid Andrew rotor small separate sample chambers are made. This four chambered rotor spins reliably solid, liquid, and gel materials. Presumably it works by minimizing vortex formation and keeping a vortex out of the center of the spinning axis. The rotor is illustrated in the Figure.

There is a loss in filling factor with this rotor design, with the example shown having a sample volume of about .2ml versus about .4ml for the same size rotor with a single open sample chamber. To date, we have been unable to spin fluted versions of the four chambered rotors stably, so rotation rates for liquids have been limited to about 2.5 kHz versus 4.5 kHz for the fluted version with a solid sample.

Graduate students T.A. Cross, J.A. DiVerdi, and M.H. Frey have been working on the development of magic angle spinning.

Sincerely,

Stanley J. Opella Assistant Professor of Chemistry



### UNIVERSITY of PENNSYLVANIA

#### PHILADELPHIA 19104

#### Department of Chemistry

July 24, 1979

Dr. Bernard L. Shapiro Texas A&M University NMR Newsletter Department of Chemistry Texas A&M University College Station, Texas 77843

NMR Technicians

Dear Dr. Shapiro:

The Department of Chemistry at the University of Pennsylvania has two technical NMR positions available. Interested candidates should write directly to S.J. Opella.

- 1) Applications oriented. Primary responsibilities are to help students and postdoctoral fellows obtain analytical <sup>13</sup>C and <sup>1</sup>H NMR spectra, instruct users on spectrometer operations, and help maintain commercial spectrometers.
- Electronics oriented. Participate in construction and modification of NMR spectrometers with both digital and radiofrequency electronics.

The University of Pennsylvania is well equipped for NMR analysis and research. Technical staff will have opportunities to participate in active research programs on organic, biological, and physical problems. The University of Pennsylvania is an Affirmative Action/Equal Opportunity Employer.

Sincerely,

auley Stanley J. OpeNa

Assistant Professor of Chemistry

### Southern Research Institute



2000 NINTH AVENUE SOUTH BIRMINGHAM, ALABAMA 35205 TELEPHONE 205-323-6592

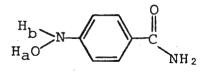
July 17, 1979

Professor B. L. Shapiro Department of Chemistry Texas A & M University College Station, Texas 77843

Title: Vicinal Spin-Spin Coupling Between OH and NH Protons

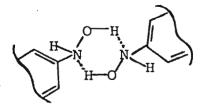
Dear Barry:

We have recently observed spin-spin coupling between the protons on the hydroxylamino group in p-(hydroxylamino)benzamide. In DMSO-d<sub>6</sub>



solution (27 mg/0.4 ml),  $J_{ab}$ =1.8 Hz. This coupling was confirmed by spin-decoupling. So far as we know, there has been no previous report of spin-spin coupling between OH and NH protons.

We do not know whether this observation was made possible by slow exchange caused by interaction with the solvent, or whether there may be intermolecular hydrogen bonding such as:



Dimerization by means of hydrogen bonding between hydroxylamino and carboxamide groups is, of course, also possible. The amide protons are nonequivalent, but have chemical shifts of about 7.0 and 7.7 ppm which appear to be very close to these of unsubstituted benzamide,

("The Aldrich Library of NMR Spectra", Vol. VII, p. 73.)

If others have observed similar couplings, we'd be interested in hearing from them.

Martha Thorpe Senior Chemist Molecular Spectroscopy Section

MCT/cpm

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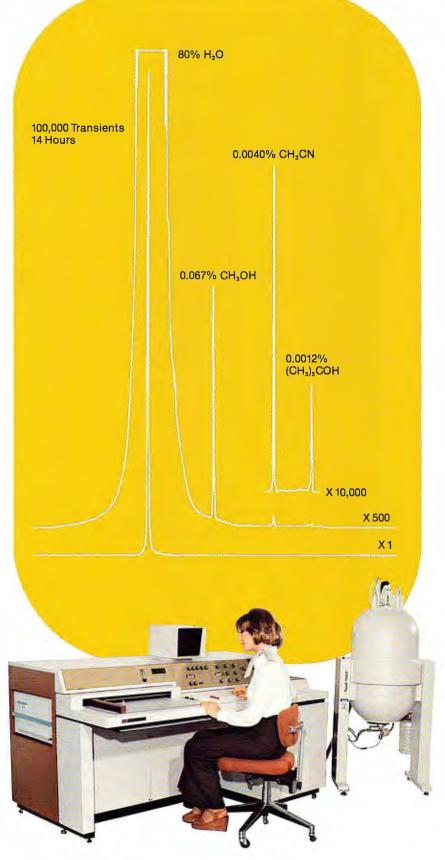
Then, there's the matter of the XL-200's broadband accessory which, with only a single probe, enables you to observe a host of nuclei (including <sup>13</sup>C) between 20 and 81 MHz. And there's the remarkable low-loss dewar system, which operates over three months on only 25 liters of liquid helium.

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Cable address: Technology

Professor B. L. Shapiro Department of Chemistry Texas A&M university College Station, Texas 77843 USA

#### HYDROCARBON CHAIN CONFORMATION IN LYOTROPIC LIQUID CRYSTALS

Dear Professor Shapiro,

We are continuing our deuterium NMR studies of the ternary system water-sodium octanoate-decanol using deuterated octanoate and decanol. We are interested in the effects of composition and phase structure on hydrocarbon chain conformation and mobility in the different liquid crystalline phases that are formed in this system. Figure 1 shows a typical spectrum from the lamellar phase containing perdeuterated octanoate. The spectrum shows five resolved quadrupole splittings. Generally it is found that the average orientation of the initial chain segments is similar for decanol and octanoate and that the amount of trans conformations in the hydrocarbon chain of the octanoate ion increases with increasing decanol content.

Ulf Henriksson

Sincerely Yours Tomas Klasop

5 kHz

Fig. 1. Fourier transform of <sup>2</sup>H quadrupole echo from a lamellar phase containing sodium octanoate- $d_{15}$ . Mole ratio octanoate:decanol:water = 1 : 1.04 : 9.30. Temperature 20°C.



#### DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE PUBLIC HEALTH SERVICE NATIONAL INSTITUTES OF HEALTH BETHESDA, MARYLAND 20014

July 5, 1979

Dear Barry:

#### Temperature Calibration Curves at 220 MHz

Chemical shifts in methanol and ethylene glycol have long been used for measurement of temperature in proton NMR. Reliable calibration curves of  $\delta$  vs. T were established at 60 MHz,<sup>1</sup> and most people use these with appropriate scaling to other frequencies. Since we were concerned about possible magnification of errors at high frequency, we decided to make a careful check on the relations at 220 MHz.

Dave Raiford, who spent last summer with us, and Cherie Fisk built an adaptor for our HR-220 probe to permit temperature measurement with a thermocouple while the sample is spinning. Details of the measurements will be published in *Anal. Chem.*,<sup>2</sup> but the conclusion is that Van Geet's 60 MHz results, scaled up to 220 MHz, are in excellent agreement with our new data. A comparison for the methanol results is shown below; the ethylene glycol agreement is even better.

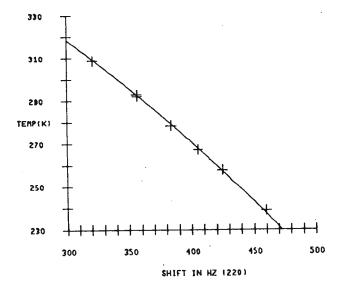
Sincerely,

Tel

Edwin D. Becker

<sup>1</sup>A. L. Van Geet, Anal. Chem. <u>42</u>, 679 (1970); *ibid.*, <u>40</u>, 2227 (1968).

<sup>2</sup>D. S. Raiford, C. L. Fisk and E. D. Becker, Anal. Chem. in press.



Methanol calibration:

Curve-Van Geet;<sup>1</sup>

Data points-Raiford, et al.2

Carr Laboratory, Department of Chemistry MOUNT HOLYOKE COLLEGE South Hadley, Massachusetts 01075



Telephone 413 538-2214

June 6, 1979

Professor B.L. Shapiro Texas A&M University Department of Chemistry College Station, Texas 77843

Dear Barry,

Re: Postdoctoral Position. 12 <sup>9</sup>Xe Chemical Shifts.

<u>Xenon</u> may not have top priority among nuclei you want to study (it doesn't appear on the Varian, JEOL, or Bruker ready reference lists), but we have found it not too difficult to observe.  $^{12.9}$ Xe (s = 1/2, 26.2% natural abundance) has sufficient solubility in many solvents at atmospheric pressure to be observed in a reasonable period of time on our JEOL FX90Q. It shows a very large solvent shift (95 ppm) from methanol to chloroform centered about water at 24.789637 MHz. The relaxation time, as might be expected, is quite long (> 100 secs). We are using the large solvent dependent shift as a probe of a number of inhomogeneous systems.

<u>Postdoctoral Position</u>. I have a postdoctoral position open immediately. Main research areas are involved with conformational analysis of molecules of biological interest using multinuclear nmr:  $^{31}$ P,  $^{13}$ C,  $^{15}$ N,  $^{11}$ B,  $^{129}$ Xe,  $^{113}$ Cd, etc. on our new JEOL FX90Q. Studies include insulin, steroids, cyclic peptides, cell-metabolism and conformational analysis of small molecules using shift reagents. Salary \$12,000/yr. A combined postdoctoral/instructorship appointment is possible. This might be particularly useful to persons preparing for a teaching career. Send resume and letters of reference.

Sincerely yours,

Then Williamon

Kenneth L. Williamson

KLW/sh

#### Standard Oil Company (Indiana)

Standard Oil Research Center Post Office Box 400 Naperville, Illinois 60540 312-420-5111

July 11, 1979

Professor Bernard L. Shapiro Department of Chemistry Texas A & M University College Station, Texas 77843

Subject: NMR Equipment Available

Dear Barry:

We have recently ordered a superconducting nmr system. In order to make room in our laboratory, we are planning to retire our HX-90 and TT-14 spectrometers. We would like to take this opportunity to notify your readers that we have this equipment for sale.

The HX-90 has a deuterium lock, broadband <sup>1</sup>H decoupler and observe capabilities for <sup>1</sup>H, <sup>31</sup>C, <sup>19</sup>F and <sup>31</sup>P. The TT-14 has a widegap HA-60 magnet which might be of interest for magic angle spinning studies. The console is a WP-60 concole equipped for FT <sup>13</sup>C only with internal/ external <sup>2</sup>H lock and broadband <sup>1</sup>H decoupling. The computers for both systems will be retained by our laboratory for use with other spectrometers.

Anyone interested in these spectrometers may call me at 312- 420-5217 for more details. The HX-90 is available immediately while the TT-14 will be available by the end of the year.

Sincerely,

G. J. Ray Mail Station F-9

GJR/jh



DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

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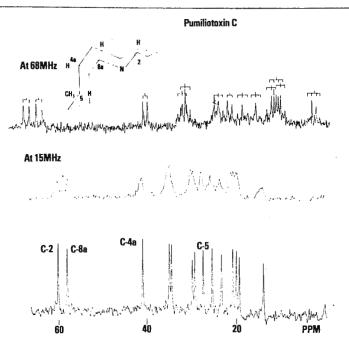
NATIONAL HEART, LUNG, AND BLOOD INSTITUTE

Prof. B. L. Shapiro Department of Chemistry Texas A&M University College Station, Texas 77843

Dear Barry,

Off resonance C-13 spectra at low and high frequencies; Position Open

The carbon-13 spectra of methine carbons in alicyclic systems often fail to produce clean-cut doublets under off-resonance conditions. The standard way to distinguish them from triplets is to assume that peaks in the off-resonance spectrum occurring at the same frequency as those of noise-decoupled spectra arise from triplets or singlets, and do not represent methine carbons. The off-resonance spectrum for Pumioliotoxin C at 15 MHz shown here contains instances of the failure of this rule. C-2, 4a and 5 produce broad peaks which look a good deal more like the center of triplets than doublets. Only C-8a produces a well-formed doublet. The origin of the effect is evidently the close coupling of the protons, for 8a is also the only carbon bearing no proton involved in an axial-axial interaction. Faced by such effects, it is a great aid to be able to obtain a spectrum at 68 MHz on the NIH hybrid supercon system, which produces discrete doublets for all four methines.



#### July 26, 1979

#### Prof. B. L. Shapiro

Position open:

Jonathan Costa has a position open in the National Institute of Mental Health for a Visiting Scientist or Postdoctoral Fellow interested in investigating biological problems by NMR. The work will probably be performed largely on the Heart Institute's Nicolet 360, due to be delivered in December. Interested persons should write Dr. Costa, at the Clinical Neuropharmacology Branch of the National Institute of Mental Health, Building 10, Rm. 3D48, NIH, Bethesda, MD 20205.

Yours very truly,

Bol

R. J. Highet Laboratory of Chemistry

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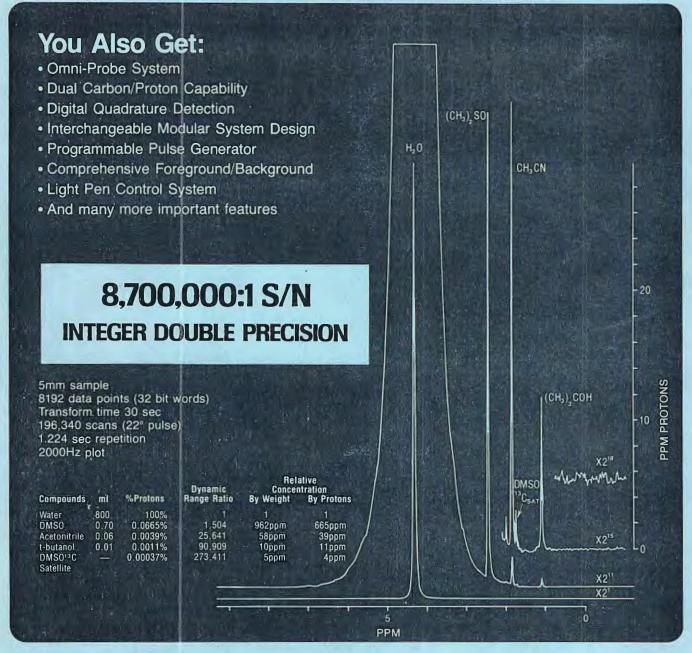
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