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N - M - R Newsletter

No. 193

October, 1974

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Deadline Dates: No. 194: 4 November 1974
No. 195: 2 December 1974

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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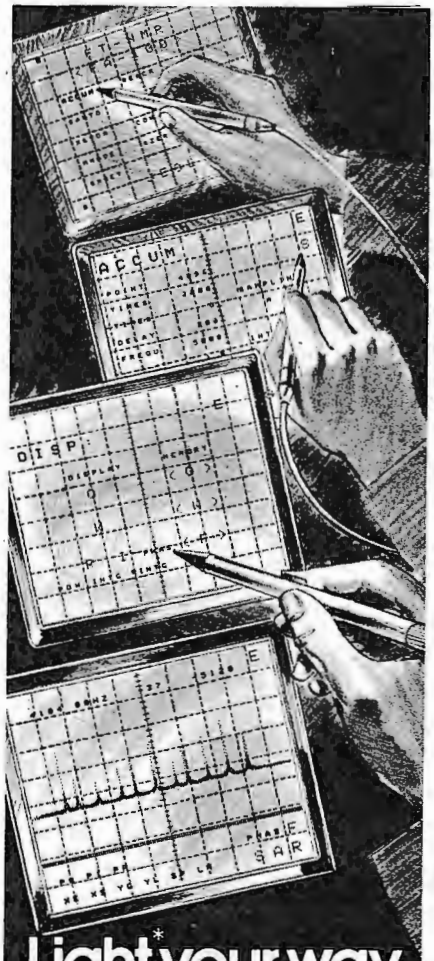

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Amsterdam, 27th August 1974.
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Dear Professor Shapiro,

^{13}C NMR of adsorbed species

We have recently been studying molecules adsorbed on surfaces by means of ^{13}C NMR. Confining ourselves to olefins adsorbed onto sodium-exchanged zeolites X and Y we found that the relaxation behaviour of olefins adsorbed on NaX zeolite differs widely from that of olefins adsorbed on NaY zeolite. Table I contains the T_1 values for 1-butene adsorbed on both types of zeolite. As far as the chemical shifts are concerned, the two systems showed hardly any difference (c.f. the results obtained by Michel[1]), suggesting that the very short spin-lattice relaxation times observed with the olefin-NaY zeolite system might be due to the presence of paramagnetic impurities. However, this conclusion is not in agreement with the nuclear Overhauser enhancement of about 3, measured by Michel[1]. These observations are being subject to a closer investigation.

As to the practical aspects of our measurements, performed on a XL-100/15 FT system, it turned out that the best results were obtained when an 8-mm NMR tube, filled with the solid, was placed in the centre of a 12-mm NMR tube, filled with a deuterated lock substance. The design of the 8-mm tube was similar to that of the Wilmad Taperlok sample tube (513A-7-SJH). Typically, line widths of 10 to 20 Hz were recorded. Spinning side bands were less than 5 %.

Please credit this contribution to Dr. R. Prins.

Yours sincerely,

A.D.H. Clague J.P.C.M. van Dongen I.E. Maxwell

1) D. Michel: Surface Science 42, 453 (1974)

Table I: T_1 values for 1-butene, adsorbed on NaX and NaY zeolite (loading 17 %w)

Type of zeolite	T_1 (ms) $H_2C=$	=CH-	$-CH_2-$	$-CH_3$
NaX*	154	230	350	600
NaY**	27	38	33	39

*accuracy ± 10 %

**accuracy ± 20 %

193-3 БЪЛГАРСКА
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Sofia. the 28 August 1974

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

Dear Professor Shapiro,

I was almost at a point to decide to communicate and the blue sheet has prompted me. Please credit this contribution to Dr S.Spassov's subscription.

TITLE: Integral Parameter Sensitivities and their use in the Total Line Shape Analysis.

The information that is necessary to reveal certain properties of a sample being investigated in a high resolution NMR experiment, is contained in the spectrum. The ultimate objective of the line shape analysis is to estimate the value of the exchange parameter, or the mean lifetime τ, among the other parameters presented in the exchange equation. This is commonly done by an iterative procedure.

The influence of a given parameter on the line shape is different. The role that the lifetime plays in shaping the exchange spectrum is known to be of special importance. It is a rather well known fact, that not all of the parameters could be equally well iterated at a given value of the lifetime τ. The process of iteration is even seriously disturbed, if all of the parameters are tried to be refined. This note is intended to supply information concerning the choice of the parameters to be varied in the course of iteration, at any particular lifetime. This is done on the basis of sensitivities.

The sensitivity of a function G with respect to a certain parameter p is understood to be G's partial derivative[1]:

$$s = \partial G/\partial p \qquad (1)$$

In the case of exchange between two sites, the equation is:

$$G = f(\delta, T_2, P, \tau, \nu) \qquad (2)$$

and δ is the peak separation at slow exchange; T_2 is the relaxation parameter; P is population and ν is the scanning frequency.

From Eqns.(1) and (2) follows that s is a function of the parameters

and of the frequency ν. The presence of ν in the expression for s is a complication, since it would be not an easy task to decide at which particular frequency two or more sensitivities should be compared. Since the information is contained in the spectrum as a whole, an easy way to avoid the above mentioned obstacle, is to integrate the sensitivity function s over the scanning frequency ν. The sensitivity should always be taken with positive sign. The figure thus obtained is called integral parameter sensitivity (IPS):

$$S = \int_a^b |s| \, d\nu \quad \text{or} \quad S = \int_a^b (s)^2 \, d\nu \qquad (3)$$

The IPS expresses the overall influence of a given parameter on the shape of the exchange function-the larger the value of S, the greater the parameter's role in forming the exchange spectrum.

On the figure, the IPS towards the following parameters: 1-δ, 2'-T_{2A}, 2''-T_{2B}, 3-p_A, 4-τ, are plotted. τ is taken as an independent variable. For convenience logarithmic scale for both horizontal and vertical axes is adopted.

It is seen from the figure that, for example, in the region of very fast exchange, the shape of the function is much less sensitive to changes in δ, T_{2A}, T_{2B} and p_A, than to the variation of τ. It is therefore useless to vary these parameters in this particular region of the exchange parameter τ -it simply results in wastage of computer time and may even lead to a divergent process of calculation.

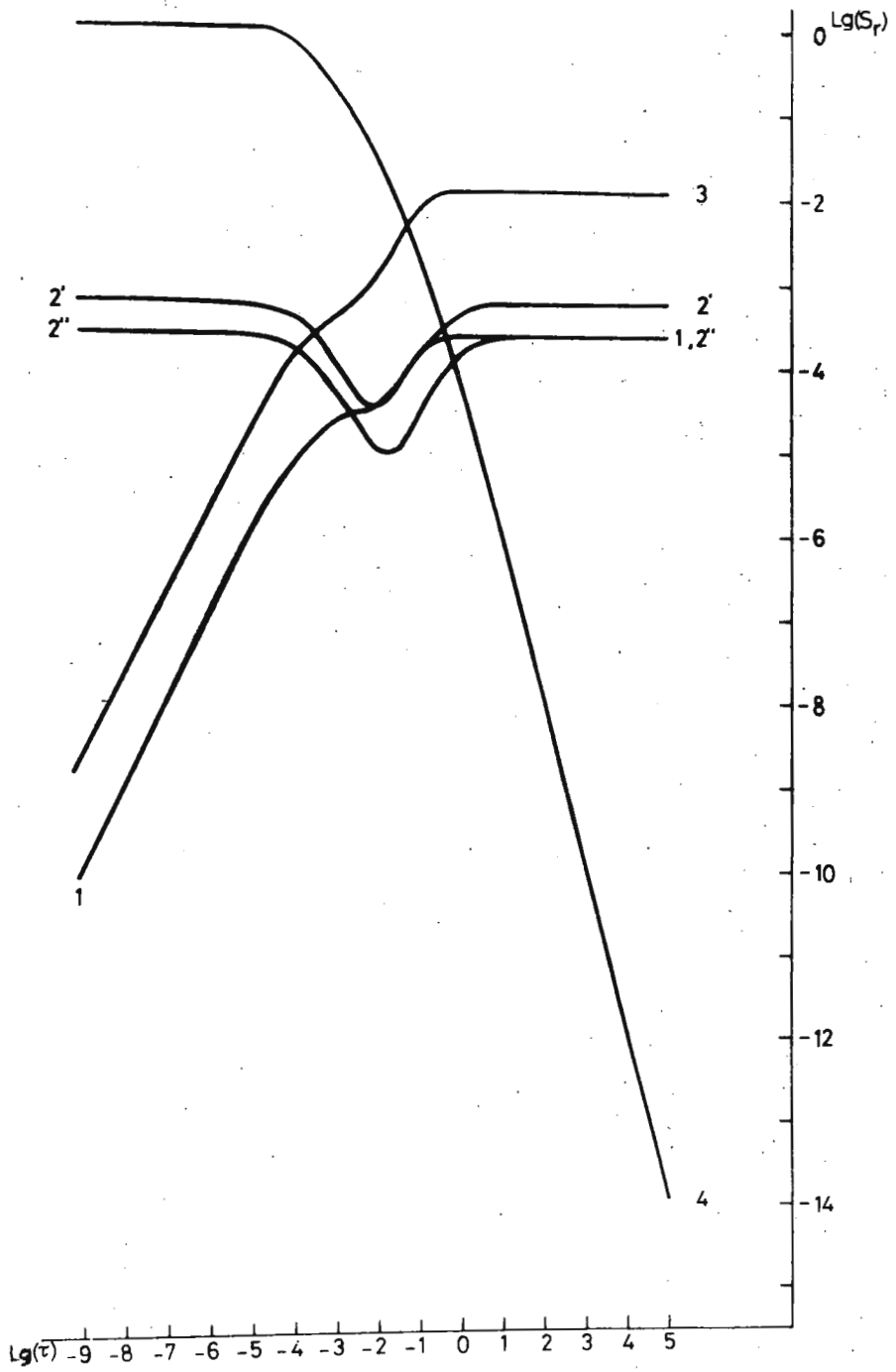
A more detailed description of this approach, along with results of its application, are to be published shortly.

Sincerely yours

Valentin Dimitrov

Valentin Dimitrov

1. V.S.Dimitrov, Org.Magnetic Resonance, 6(1974) 16



δ = 20 Hz; $T_{2A} = T_{2B}$ = 0.6 sec; p_A = 0.7;

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193-6

AIR MAIL

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

uw ref. | onze ref. MA/4585/eds | toestel 2762 | datum 2nd September 1974

Dear Professor Shapiro,

Concentration dependency of an epoxy proton signal.

In 1972 we measured a 220 MHz spectrum of cis-9,10-epoxy stearic acid methylester and the epoxy signal was found at δ 2.69 (CCl_4, internal TMS). Recently another sample of this compound was measured, but by chance the concentration was much higher and δ 2.71 was found. This unexpected difference proved to be reproducible, and the accompanying table shows the chemical shift as a function of relative concentration in CCl_4 (at 29°C). The δ-values for the terminal CH_3 and for the OCH_3 group is also given.

Conc.	CH_3	OCH_3	epoxy-H
0.67 M	0.885	3.595	2.713
0.33 M	0.887	3.599	2.704
0.17 M	0.886	3.600	2.697
0.08 M	0.887	3.601	2.693
0.04 M	0.887	3.603	2.691

The methyl group is constant within 0.002, the methoxy group exhibits a small downfield shift (0.008), but the shift of the epoxy protons is 0.022 and is to higher field.
An attempt to find out whether this relatively large effect is due to intermolecular interactions between the epoxy group and the carbomethoxy group or between epoxy groups failed.
1,2-epoxy dodecene has been measured at concentrations ranging from 0.046 to 1.10 M. The epoxy signals remained constant within 0.004 ppm.
An identical experiment with mixtures of 1,2-epoxy dodecene and methyl stearate revealed that the δ 2.72 and δ 2.28 epoxy signals remained constant within 0.004 and 0.001 ppm respectively, but that the signal at 2.559 shifts to 2.567 after dilution with a factor of 25.
This shift is in a direction opposite to the "dilution shift" in the epoxy stearate.

Whatever the explanation may be, anyone who is working with epoxy fatty acid esters must be aware of this concentration effect when spectra, measured at different concentrations are compared.

Yours sincerely,

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September 3, 1974

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

Dear Professor Shapiro,

I have received your reminder, so I must apologise for the delay and I send my contribution, the title of which is

"Temperature Dependence of the Proton Magnetic Resonance Spectra of Halogeno Bis (NN' - diethyl-dithiocarbamato) Iron (III)"

The proton magnetic resonance spectra between $+60^{\circ}C$ to $-60^{\circ}C$ on a series of intermediate spin ($S = 3/2$) halogeno bis (diethyldithiocarbamato) iron (III) have been recorded. The pseudo contact shift (PCS) at various temperatures has been estimated from the experimentally measured paramagnetic anisotropies. The results show the marked effect of T^{-2} term in the Iodo derivative and it is rationalised in terms of the large ground state zero field splitting of the ferric ion. The temperature variation of the contact shift (CS) shows deviations from the curie-law behaviour.

The temperature variation PMR study has revealed a conformational change in these molecules, which has been ascribed to a hindered rotation about S_2C-N bond. The thermodynamic parameters describing this kinetic process have been calculated.

Sincerely yours

M.M. Dhingra

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№ 56

12th September 1974

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

^{29}Si NMR and d-Orbital Participation

Dear Prof. Shapiro,

Recently we have been studying silicon-29 chemical shifts with particular emphasis on the influence of distant substituent groups. In the first papers in this field[1,2] the ^{29}Si shifts in compounds of the series $Me_{4-n}SiX_n$ (X = OR, F, Cl) were discussed in terms of the $(p-d)_\pi$-interaction between silicon and the electronegative group X. However, in several later studies[3,4,5] good correlation with experiment has been obtained in calculations assuming only a negligible role of silicon d-orbitals in the Si-X bonds. A clear contradiction exists between these two approaches and we want to present results from our laboratory (obtained in collaboration with Dr. J.Schraml in Prague) that tend to show that while the gross tendencies of ^{29}Si shifts may be rationalized without $(p-d)_\pi$-interaction, this concept is useful for the interpretation of shift differences between some homologous series of compounds.

Table ($\delta_{^{29}Si}$ ppm from TMS, downfield shifts positive)

	Compound MY	$\delta_{^{29}Si}$		$\Delta\delta_{^{29}Si} = \delta_{^{29}Si}(MY) - \delta_{^{29}Si}(MH)$	
		Y=Cl	$Y=NH_2$	Y=Cl	$Y=NH_2$
	Me_3SiCH_2Y	+2.5	+0.5	+2.5	+0.5
I	$(EtO)Me_2SiCH_2Y$	+8.9(6)	+13.2	-4.6(6)	-0.3
	$(EtO)_2MeSiCH_2Y$	-17.2(6)	-9.9	-11.1(6)	-3.8
	$(EtO)_3SiCH_2Y$	-59.7(6)	-50.2	-15.2(6)	-5.7
II	$Me_3SiOCH_2CH_2Y$	+18.3	+16.9	+4.8	+3.4
	$Me_2Si(OCH_2CH_2Y)_2$	-1.6	-3.2	+4.5	+2.9
	$MeSi(OCH_2CH_2Y)_3$		-41.5		+3.0

All substituent effects $\Delta\delta_{^{29}Si}$ are negative in the first section of the Table and positive in the second - e.g. electron-withdrawing groups Y increase the silicon shielding in compounds of the type $-O-\overset{|}{Si}-\ldots-Y$ and decrease it in the $-\overset{|}{Si}-O-\ldots-Y$ type compounds. Such a dependence of shielding on the position of atoms in a chain is typical of unsaturated compounds. Indeed, in compounds of the type $A=B-\ldots-Y$ the electron-withdrawing substituents Y decrease the shielding of A and increase (when others, such as nonbonded interactions are negligible!) the shielding of B. The first case is well known and universal, and good examples of the latter case are provided by the ^{13}C shifts of ketones, ^{31}P shifts in compounds of the type $O=P\lesseqgtr$, ^{14}N shifts in nitro compounds and isonitriles.

So, on the basis of ^{29}Si chemical shifts, the behavior of the $Si-O$ bond is qualitatively similar to the polarization of a multiple bond. The same conclusion regarding the $Si-F$ bond may be drawn from the data presented by Ernst, Spialter et al.[4]

for *meta*- and *para*-substituted phenyltrifluorosilanes, where structures of the type $F^{\delta+} = SiF_2 - \ldots - X^{\delta-}$ with d-orbital participation in the ground state may play an important role. In general, the $(p\text{-}d)_\pi$-interaction in Si - X bonds seems to be the most reasonable explanation for diamagnetic ^{29}Si shifts under the influence of distant electronegative substituents Y.

Sincerely,

Endel Lippmaa, Märt Mägi, Tõnis Pehk

1. P.C.Lauterbur in Determination of Organic Structures by Physical Methods, Ed. by F.C.Nachod, W.D.Phillips, vol.2, chap.7, 1962, Academic Press, N.Y. and London.
2. B.K.Hunter, L.W.Reeves, Canad.J.Chem., 46, 1399 (1968).
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4. C.R.Ernst, L.Spialter, G.R.Buell, D.L.White, J.Organometal.Chem., 59, C13 (1973).
5. R.Radeglia, G.Engelhardt, J.Organometal.Chem., 67, C45 (1974).
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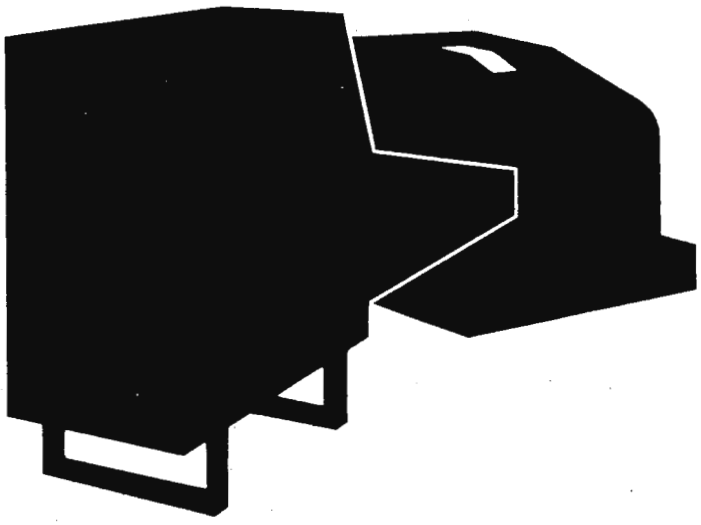

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IN REPLY REFER TO:
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18 September 1974

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

Title: ^{13}C Line-Width Variation in Polymer Side-Chains; Postdoctoral Associateship

Dear Barry:

In an earlier letter (Nov. '73) I mentioned the observation of a large variation in ^{13}C nmr line-widths for n-octyl side-chains bound to a linear aluminoxy soap backbone. The material under study was a high molecular weight product which forms stable gel solutions in hydrocarbons at relatively low concentrations, ~ 5% or so, and the purpose of the nmr study was to gain some idea of the gel structure. We have now put our conclusions on a more solid ground with the observation of closely similar ^{13}C spectra for benzene solutions of poly-(1-decene) - essentially polyethylene with n-octyl groups on the odd carbons.

Both spectra exhibit an increase in line-width starting with 1.3 Hz at C-5 and continuing back along the alkyl chain to 5 Hz at the point of attachment to the backbone. In addition, the poly-olefin spectrum shows further broadening of the two lines (5.3 and 6.5 Hz) arising from the main polyethylene chain.

Clearly, these observations provide a nice demonstration of the expected dependence of T_1 on the correlation times for segmental motions in the critical region where these times approach the nuclear precession period, in this case around 4×10^{-8} sec.

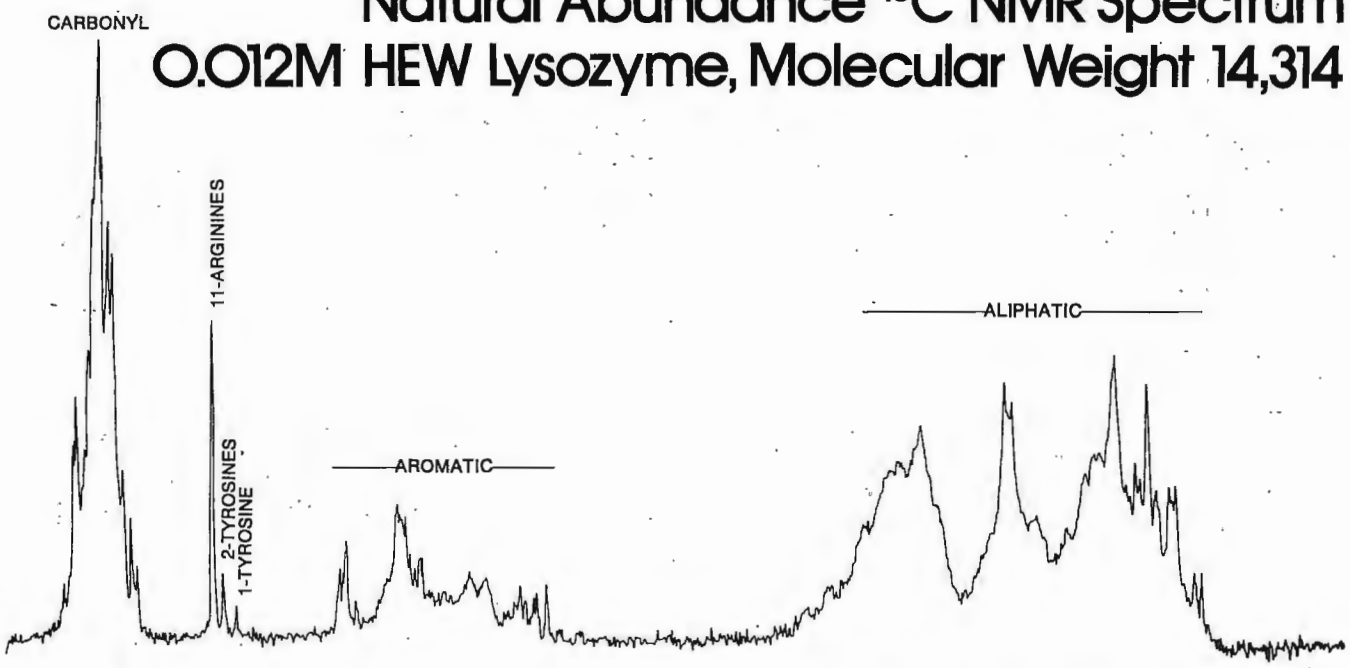
Applicants for our 1975 Postdoctoral Research Associateship are being sought. The program here is administered by the National Research Council, with appointees holding temporary civil service positions for one or two years. (I am sorry that we can only accept U. S. citizens.) We would be pleased to receive research proposals from graduates interested in either theoretical or experimental aspects of nitrogen-15 nmr, the study of molecular motions in polymer systems, or any other research-worthy subject. A brochure detailing facilities in the Michelson Lab and the NRC-NWC postdoctoral program will be sent on request.

Sincerely,

Don

DONALD W. MOORE

Natural Abundance ^{13}C NMR Spectrum
0.012M HEW Lysozyme, Molecular Weight 14,314



Proton-decoupled natural abundance ^{13}C spectrum of hen egg white lysozyme in 0.15M NaCl in 9:1 H_2O/D_2O, pH 4.0, 45°C. Recorded with a TT-14 system[1] at 15.08 MHz using a 20 mm sample tube; 37,107 90° pulses; 4096 time-domain points; and 1.165 Hz digital line broadening. This spectrum demonstrates that in a small protein, single carbon resonances such as the one assigned above to C^{ζ} in a single tyrosine residue, can be observed after only five hours of signal averaging with use of the 20 mm sample technology developed at Indiana University by Dr. Adam Allerhand.[2,3]

1. Sample run at the University of Chicago, courtesy of Dr. Philip Keim, Pritzker School of Medicine.
2. A. Allerhand, R.F. Childers, E. Oldfield, J. Magn. Resonance, *11*, 272 (1973).
3. E. Oldfield and A. Allerhand, Proc. Nat. Acad. Sci. USA, *70*, 3531 (1973).

HIGH SENSITIVITY CARBON-13 FT-NMR
with a TT-14 System

The TT-14 FT NMR System combines a state-of-the-art solid state console, Nicolet NMR-80 computer, 20 mm sample probe developed by Dr. Adam Allerhand, and a time-proved Varian 14 kilogauss magnet into the highest sensitivity carbon-13 performance available today. Features of this system are:

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- multipulse capability, including automatic T_1 measurements;
- disk memory unit for program and data storage;
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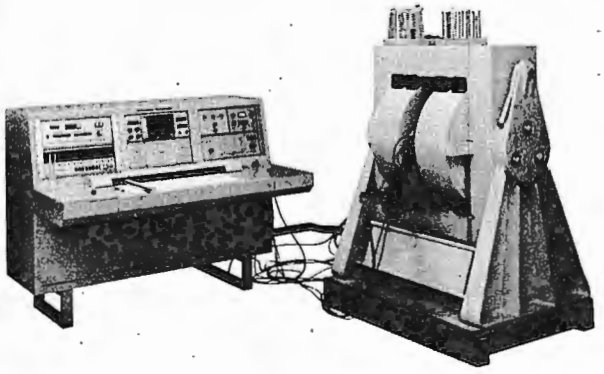

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Telephone 01-928 7900 ext 649

Dr Bernard L Shapiro
Dept. of Chemistry
Texas A and M University
College Station
Texas 77843 U.S.A.

Our reference

Date October 4, 1974

FT-NMR OF CHICKEN MEAT

Dear Professor Shapiro

We are about to acquire a PFT-100 spectrometer for a variety of analytical situations concerned with this laboratory's role in environmental and consumer protection and other regulatory processes.

As an example of some of the FT work that we have done recently, note the attached chicken NMR spectra. In the literature, there are many ^{13}C spectra of enzymes and other chemically defined protein samples; however meat protein in toto can also yield useful spectra after removal of fat and then denaturation. In Figure 1, chicken protein has a ^{13}C spectrum, albeit a little noisy here, that shows distinct correlations with the ^{13}C spectrum of the constituent amino acids (Figure 2). The chicken meat was freeze-dried, defatted and ground to a partially fibrous powder. When "reconstituted" in boiling deuterated 6M GuCl, a very thick gel was produced that had the normal aroma of cooked chicken. The intensities of the carbonyl absorptions are enhanced in the protein spectrum as one would expect from shorter relaxation times in the polyamide state.

In Figure 2, the constituent amino acids are revealed except for a few which are degraded in hot acid hydrolysis (6M HCl at 120°C for 16 hours). For those engaged in amino acid analysis of samples $\geq$1 gram, ^{13}C FT NMR may provide a more specific, convenient and faster alternative to the chromatographic-based procedures used at present. Specificity is much greater as each amino acid has several lines in the ^{13}C spectrum. Several software routines have recently appeared for processing the data of conventional analyses and include corrections for extinction coefficients; these likely can be adapted for on-line analysis in the NMR computer.

In Figure 3, we present another aspect of chicken NMR as purchased in the local shops. The ^{31}P FT spectrum is of raw meat sampled with a cork-borer and the chemical shift is consistent with PO_4^{3-}. From other available data, we believe that the bulk of the signal arises from degraded polyphosphate salts. These substances are added to many frozen and canned meats and to convenience foods for moisture retention and palatibility. This particular spectrum was taken with a ^{13}C probe at 25.1 MHz at reduced magnetic field with block averaging and computer lock on HMPA.

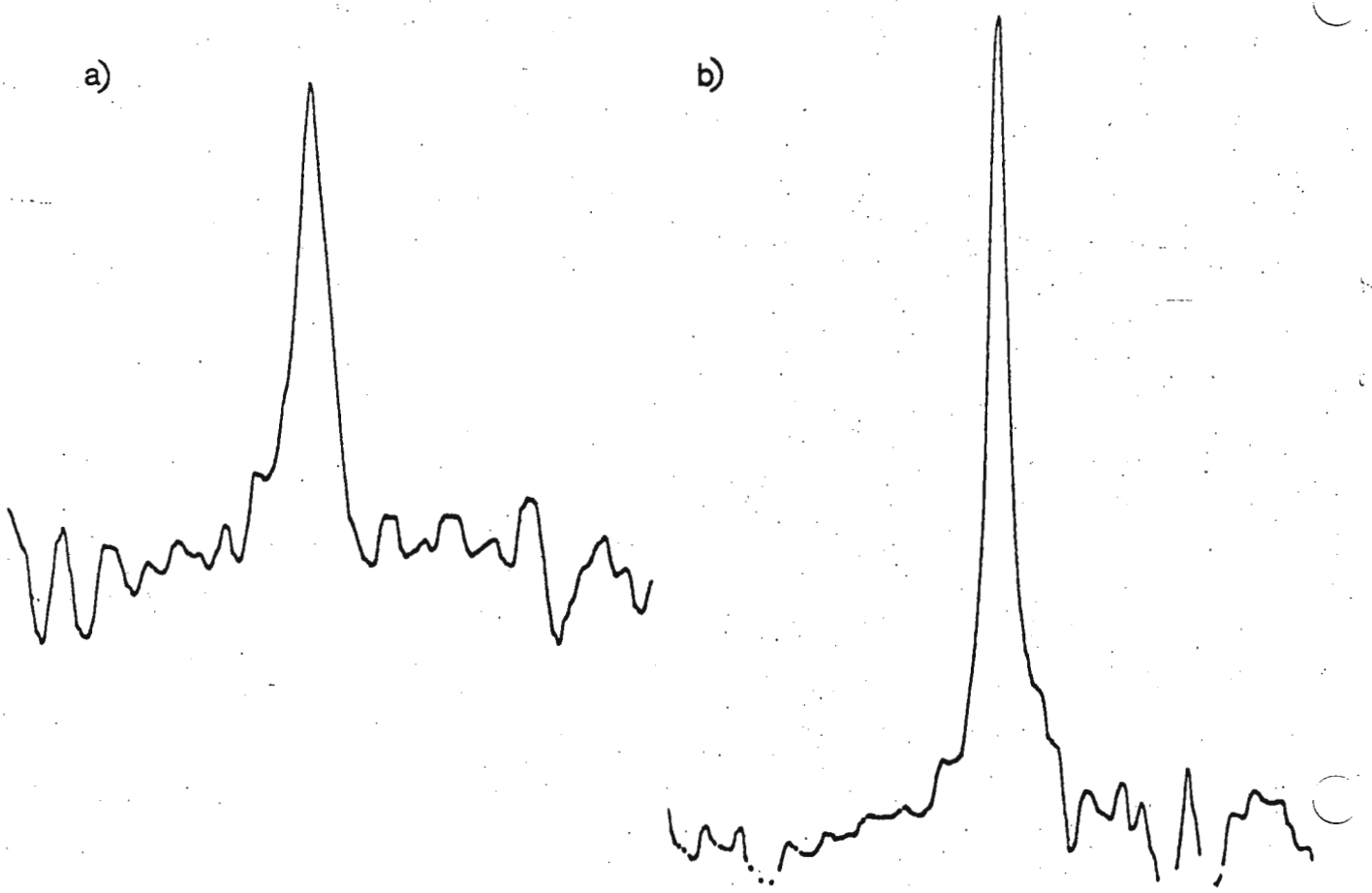


Fig. 3 ^{31}P NMR spectra of chicken meat, a) leg, b)breast. (Accumulation time 90 sec.)

We acknowledge the co-operation of P Beynon (JEOL) and P Baker (PCMU) in obtaining these spectra and the Government Chemist for authorisation to release these details. Please credit this opening communication to I K O'Neill.

I K O'Neill M A Pringuer H Prosser C Richards.

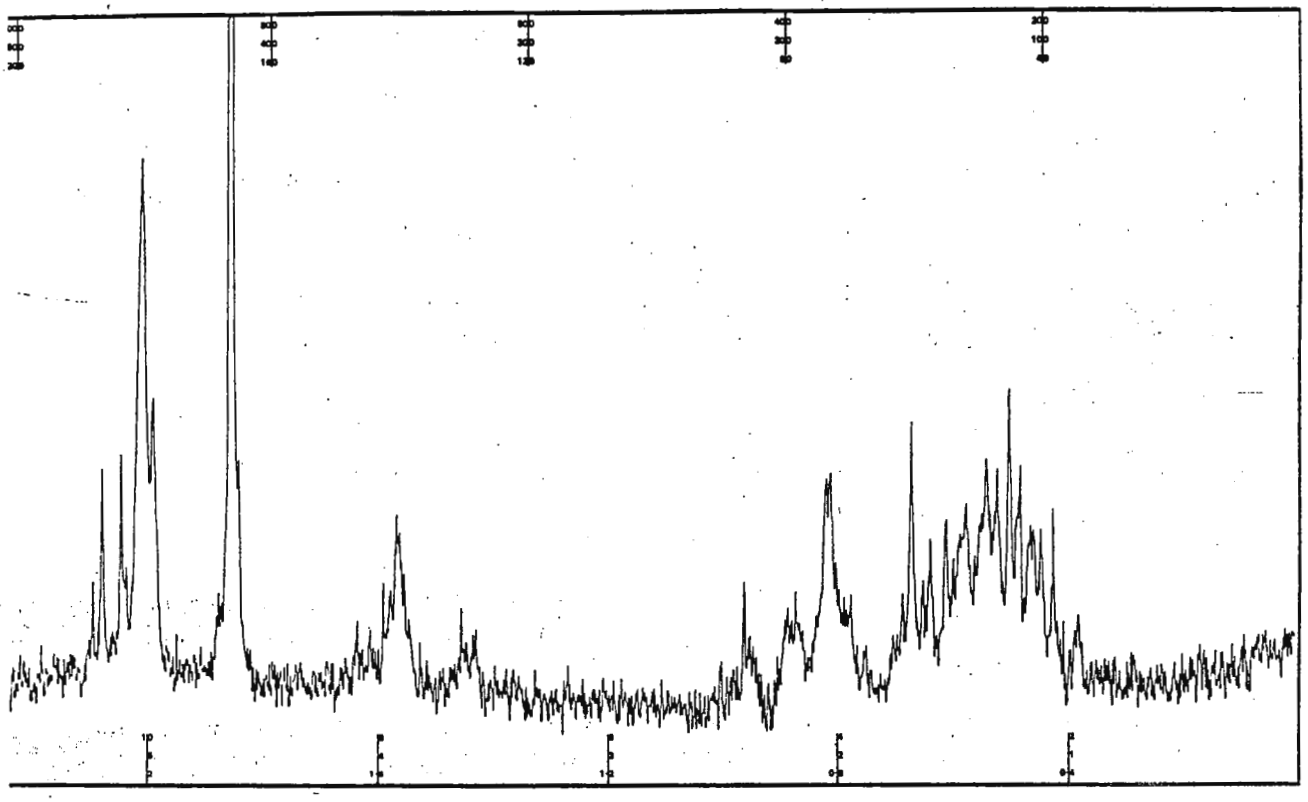


Fig.1 ^{13}C NMR spectrum of defatted chicken as a gel in deuterated aqueous 6M guanidinium chloride.

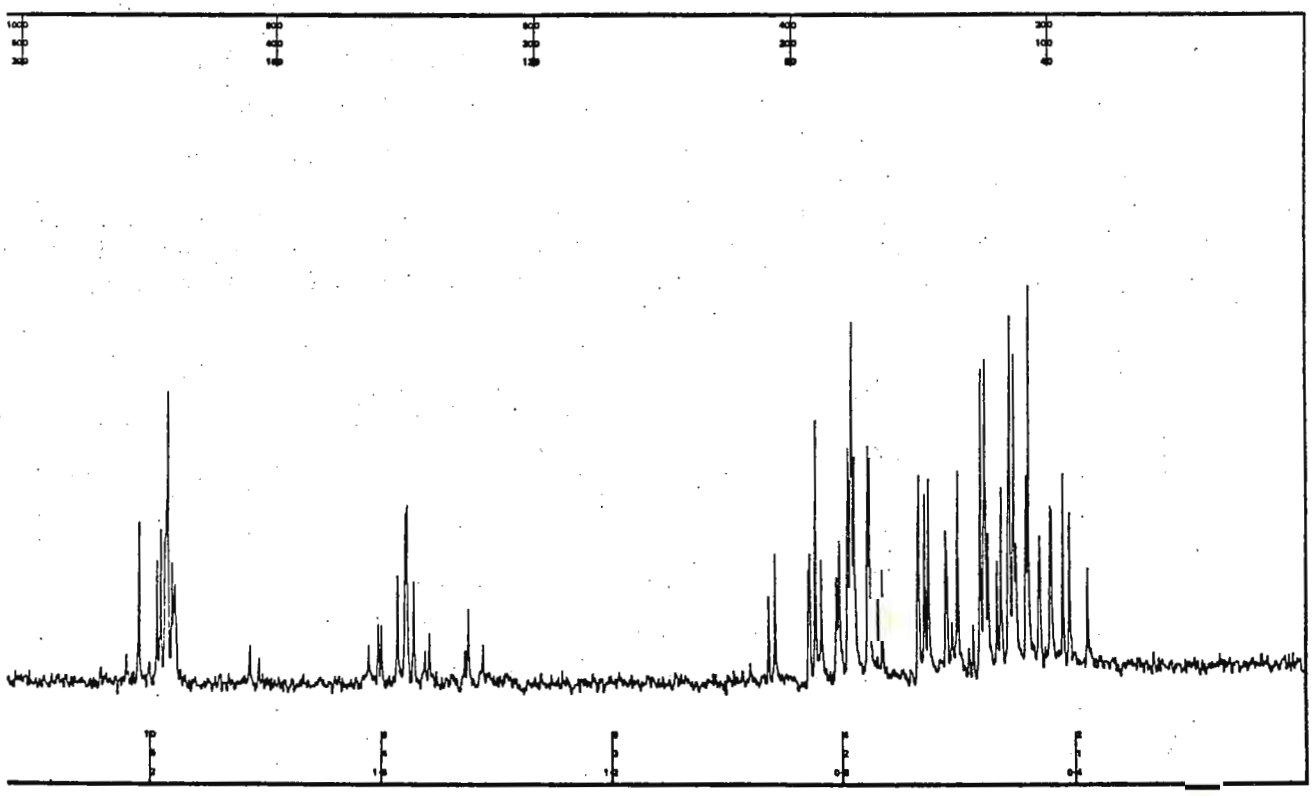


Fig. 2 ^{13}C NMR spectrum of hydrolysed defatted chicken in D_2O/DCl.

THE UNIVERSITY OF NORTH CAROLINA
AT
CHAPEL HILL
27514

DEPARTMENT OF CHEMISTRY

September 6, 1974

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

TITLE: DOWNFIELD C-13 SHIFTS IN ORGANOLITHIUM COMPOUNDS

Dear Barry:

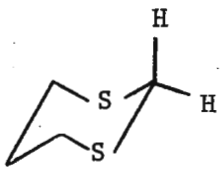
Charles Johnson, Slayton Evans and myself have finally arranged a symbiosis with respect to the TAMU Newsletter, so please credit this contribution to C.J.'s subscription.

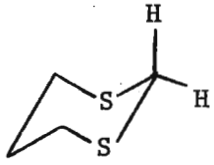
As my first contribution to TAMU-NMR from Chapel Hill, where I moved two years ago, let me report an observation regarding downfield shifts in the ^{13}C spectra of certain anionic species (organolithiums).

When one compares the 13-C spectra of n-butyllithium and sec-butyllithium in benzene with that of butane,[1] one finds that only the alpha carbon undergoes the "expected" upfield shift, whereas the more remote carbons are shifted downfield.

$CH_3-CH_2-CH_2-CH_2Li$	$CH_3-CH_2-CH_2-CH_3$
13.8 31.3 31.8 11.3 (C_6H_6)	13.2 25.0 25.0 13.2
13.9 34.2 36.1 12.1 (TMEDA)	$CH_3-CH_2-CHLi-CH_3$
14.3 34.6 36.3 12.5 (C_6H_6-TMEDA)	19.2 31.2 15.8 15.8

The shifts are not due to oligomeric aggregates, for they are also seen in butyllithium disaggregated by tetramethylethylene diamine (TMEDA) and at the C-4 and C-5 positions in 2-lithio-1,3-dithianes which are not oligomeric:[2]



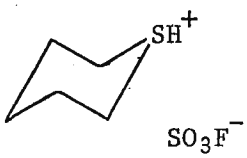


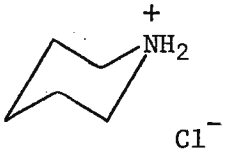
THF (TMEDA)		THF (TMEDA)
32.2 (31.9)	C-2	26.7 (26.9)
30.4 (29.5)	C-4/6	34.1 (34.0)
26.3 (27.0)	C-5	31.2 (31.1)

The phenomenon appears to be the reciprocal of the upfield shifts seen at C-β and C-γ in ammonium salts, $\overset{\gamma}{C}-\overset{\beta}{C}-\overset{\alpha}{C}-\overset{+}{N}H_3$ compared to their parent amines C-C-C-NH_2 seen by Grant and Duch;[3] we[4] have seen comparable shifts at the β- and γ-carbons of thianium ternary salts[5] as well as thianes dissolved in strong acid. Downfield shifts appear at C-α in the cases of the thianium salts:

29.1	27.8	26.5
C-2,6	C-3,5	C-4
32.1	24.8	22.4

47.7	27.6	25.8
C-2,6	C-3,5	C-4
44.6	22.5	22.5





(ext. TMS)

Sincerely,

Ernest

Ernest L. Eliel

A. Abatjoglou.

Anthony G. Abatjoglou

ELE:AGA:bs

REFERENCES

1. J. B. Stothers, "Carbon-13 NMR Spectroscopy," Academic Press, New York, 1972, p. 56.

2. cf. E. L. Eliel, A. A. Hartmann, and A. G. Abatjoglou, J. Amer. Chem. Soc., 96, 1807 (1974).

3. Michael W. Duch, Ph.D. Dissertation, University of Utah, Salt Lake City, Utah, 1970.

4. R. L. Willer and E. L. Eliel, unpublished observations.

5. cf. E. L. Eliel, R. L. Willer, A. T. McPhail, and Kay D. Onan, J. Amer. Chem. Soc., 96, 3021 (1974).

193-19

THE PENNSYLVANIA STATE UNIVERSITY

152 DAVEY LABORATORY
UNIVERSITY PARK, PENNSYLVANIA 16802

College of Science
Department of Chemistry

September 13, 1974

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

Dear Dr. Shapiro:

Lanthanide Porphyrin Complexes

In an effort to develop a potent new dipolar nmr probe for use in biological systems, we have sought to combine the desirable qualities of the heme moiety (rigid, well-defined structure; specific, tight binding site in proteins; resistance to demetallation) with the propitious electronic and magnetic properties of trivalent lanthanide ions. As a start in this direction we have synthesized lanthanide complexes of meso-tetraphenylporphin (TPP) and some of its derivatives. Despite considerable current interest in metalloporphyrin chemistry, lanthanide complexes of this class of macrocycle have not previously been reported.

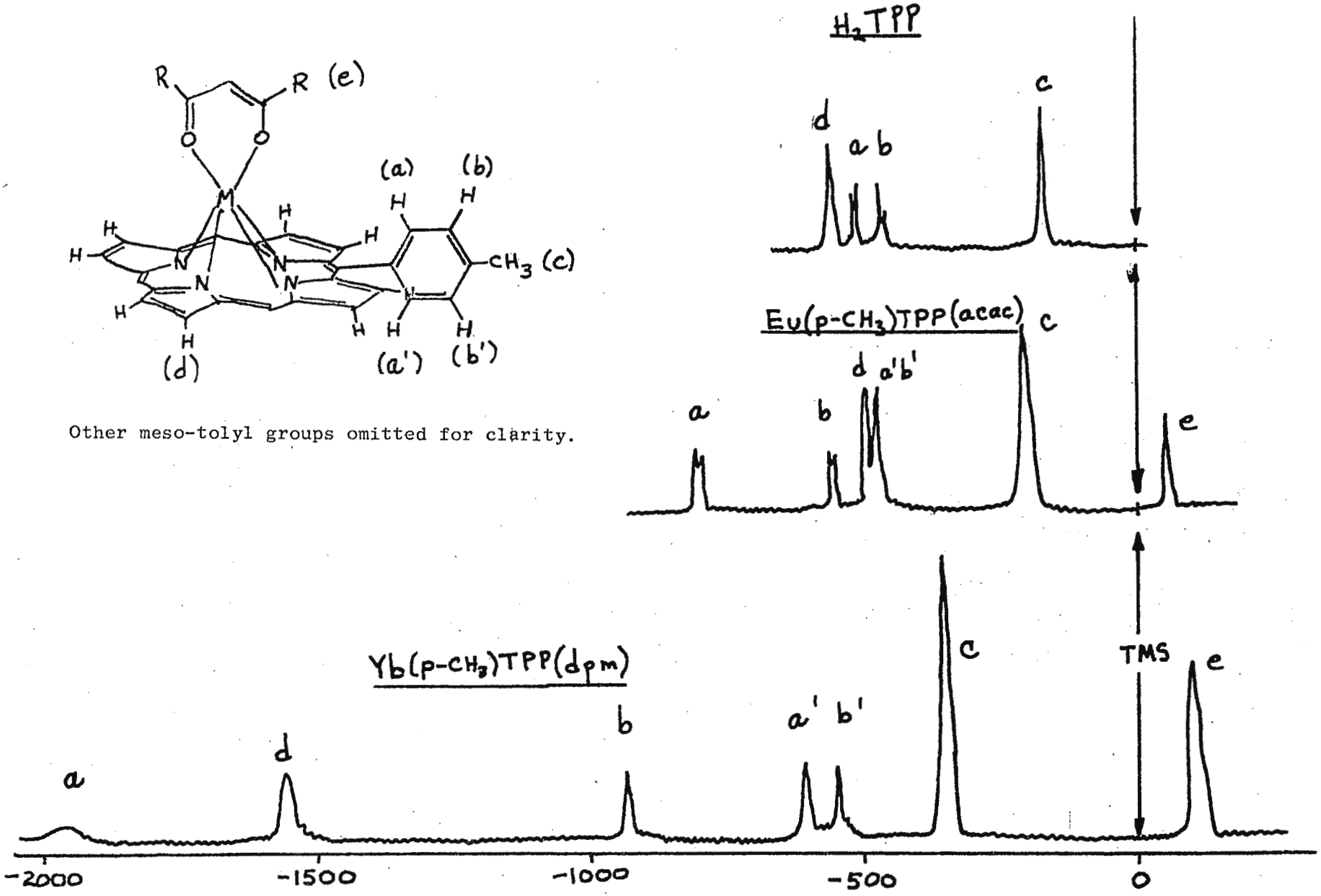
The nmr spectra confirm the formation of paramagnetic metalloporphyrins with the metal ion considerably displaced from the porphyrin plane. The proton nmr spectrum of the tetra-p-tolyl derative, Eu(p-CH_3TPPacac, at -21° exhibits peaks (ppm from TMS) at -13.31d-(ortho-endo); -9.33d(meta-endo); -8.30(pyrrole); -8.12(ortho- and meta-exo); -3.44(para-CH_3); 0.88(acac-CH_3); with areas consistent with this assignment. These may be compared with diamagnetic free-base resonances at -9.22(pyrrole); -8.43d(ortho); -7.80d(meta); and -2.82(para-CH_3); (d signifies a doublet with 8 Hz splitting). The signals shift linearly with T^{-1} as expected for isotropically shifted resonances. Shift ratios for the phenyl protons (ortho-endo:meta-endo:ortho-exo:meta-exo:para-CH_3 = 10.00:-3.14; 0.61:-0.68:-1.28) agree well with those calculated assuming axial symmetry (-10.00: -3.09:0.80:-0.68:-1.23). The ratios agree best for a model with the europium atom 1.0 Å out of the porphyrin plane. This implies a Eu-N distance of 2.83 Å which is 0.18 Å larger than the pyridine nitrogen to europium distance in a shift reagent system. No attempt was made to account for phenyl ring oscillation, porphyrin skeleton doming, or Fermi contact contributions, so the above estimate, which is extremely sensitive to the small ortho-endo and meta-endo shifts, must be considered highly uncertain and is probably too large. The same structural parameters predict a relative dipolar shift of -4.77 for the pyrrole protons, while this resonance is observed to shift upfield by 0.92 ppm at -21°. We attribute this to a sizeable Fermi contact interaction. It is for the pyrrole protons that contact shifts are particularly evident in both low- and high-spin iron(III) porphyrin systems. Preliminary nmr experiments with an impure sample of Pr(p-CH_3)TPPacac show a well resolved p-CH_3 resonance shifted upfield by 1.15 ppm at -21° which may be compared with the -0.63 ppm shift for this resonance in the analogous europium complex. This implies a larger magnetic anisotropy of opposite sign for praseodymium, consistent with results in shift reagent systems.

These preliminary findings, and particularly the observed stability of lanthanide porphyrin complexes in neutral aqueous organic environments, suggest that the analogous complexes of naturally occurring porphyrins may serve as potent and specific nmr shift and broadening probes in biological systems.

Please credit this contribution to the account of Joseph J. Villafranca.

Sincerely, Ching-Ping Wong, Robert F. Venteicher, William DeW. Horrocks, Jr.

Other meso-tolyl groups omitted for clarity.



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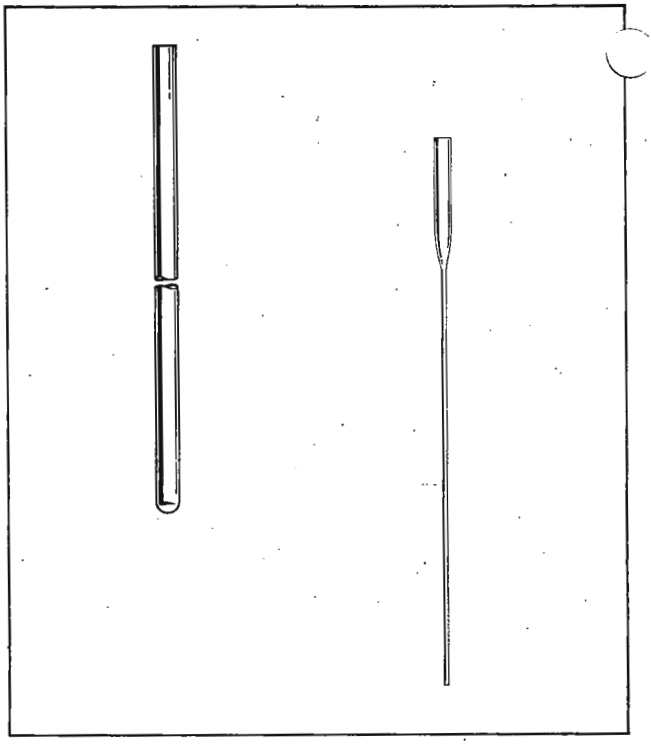

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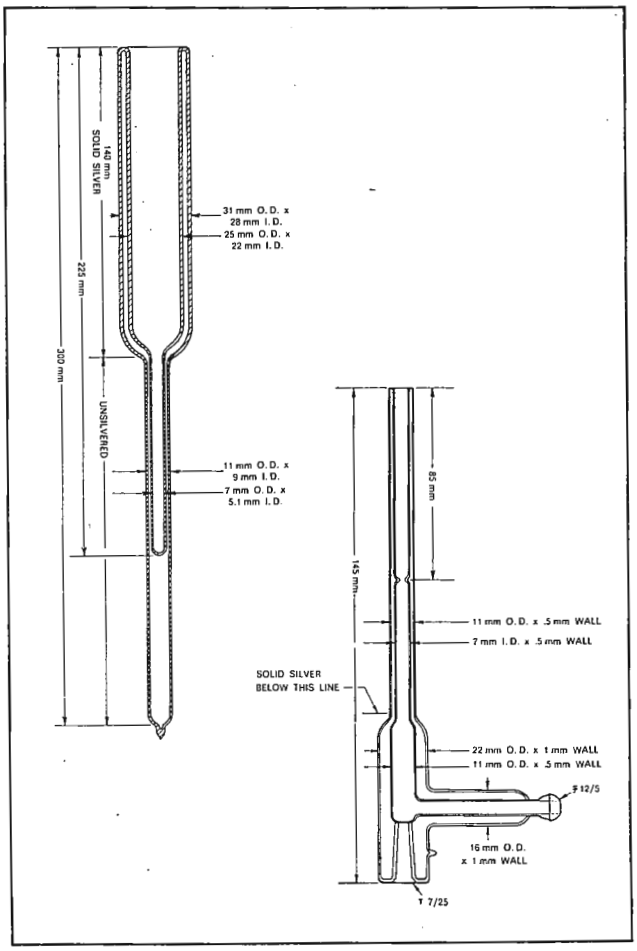

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October 1st, 1974
OC/VJ/53015

Dear Professor SHAPIRO,

Isotope effects on ^{19}F - chemical shifts

In low-field ^{19}F - NMR experiments, $CFCl_3$ is comonly choosen as a reference for measurements of chemical shifts ; now, it is well known that the broad resonance signal of $CFCl_3$ arises from the isotopic effect of the chlorine atoms.

We have observed this isotopic effect (fig.) on the RMN 250 MHz CAMECA.

In general :

- the isotopic chemical shift is approximatly proportionnal to the number of atoms in the molecule that have been substitued by the isotope.
- the field increases with the mass of the isotope.

In this compound there are 4 possibilities of substitutions :

$3\ Cl_{35}$

$2\ Cl_{35} - 1\ Cl_{37}$ } 3 arrangements

$1\ Cl_{35} - 2\ Cl_{37}$ } "

$3\ Cl_{37}$

with the natural abondance $Cl_{35} = 75.4\ \%$

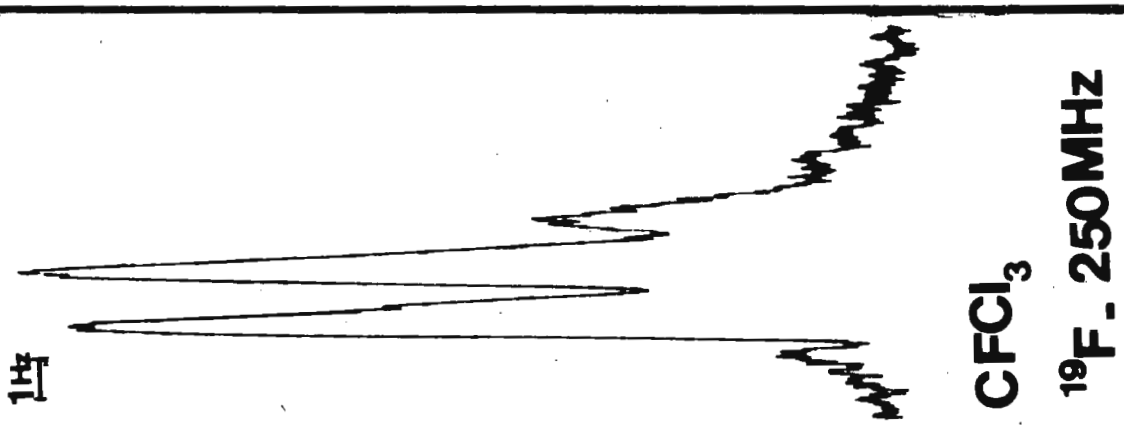
$Cl_{37} = 24.6\ \%$

we obtain 4 lines with relative intensities approximatly 1 1 1/3 1/9 ⟶ H

The magnitude of the shift is about 1 to 1.5 Hz (0.004 to 0.006 ppm).

Sincerely yours

D. CREPAUX



193-23

MRC

Medical Research Council

National Institute for Medical Research
The Ridgeway, Mill Hill
London NW7 1AA

telegrams Natinmed LondonNW7
telex 922666(Natinmed London)
telephone 01-959 3666

reference

25th September, 1974.

Interactions of -D-Ala-D-Ala Peptides with Vancomycin

Dear Barry,

We have recently been using NMR to study the complex formation in vitro between vancomycin and acetyl-D-Ala-D-Ala. Perkins (Biochem. J. 111, 195 (1969)) has found that mucopeptide precursor molecules containing the terminal-D-Ala-D-Ala fragment bind strongly to the antibiotic vancomycin. This would be expected to interfere with bacterial cell-wall formation and offers a possible mechanism of action for the antibiotic. The ^{1}H resonances of the three methyl groups in acetyl-D-Ala-D-Ala are all shifted upfield in the complex, probably due to the methyl groups being bound in the proximity of aromatic rings in vancomycin. An analysis of the binding as a function of pH shows that the most stable complex is formed between the peptide anion and the vancomycin cation. The interacting cationic group in vancomycin is identified as an N-terminal N-methyl leucine: from a titration of this N-Methyl ^{1}H resonance in the complex (see fig.1) it is seen that its pK value is shifted to 8.7 ± 0.2 (from 7.7 in free vancomycin). This is in excellent agreement with the pH dependence of the binding constant (fig.2).

The interactions of vancomycin with many peptides related to acetyl-D-Ala-D-Ala have been examined by measuring the proton chemical shifts of the peptides bound to vancomycin. The C-terminal alanine group shows least perturbation in chemical shifts when the structures of neighbouring groups are modified, whereas the N-terminal acetyl group shows the largest changes. The lability of the chemical shifts gives some indication of the contribution of the parts of the molecule to the overall binding energy of the complex.

Yours sincerely,

Jim Feeney.

J.P. Brown, A.S.V. Burgen, J. Feeney.

FIG. 1

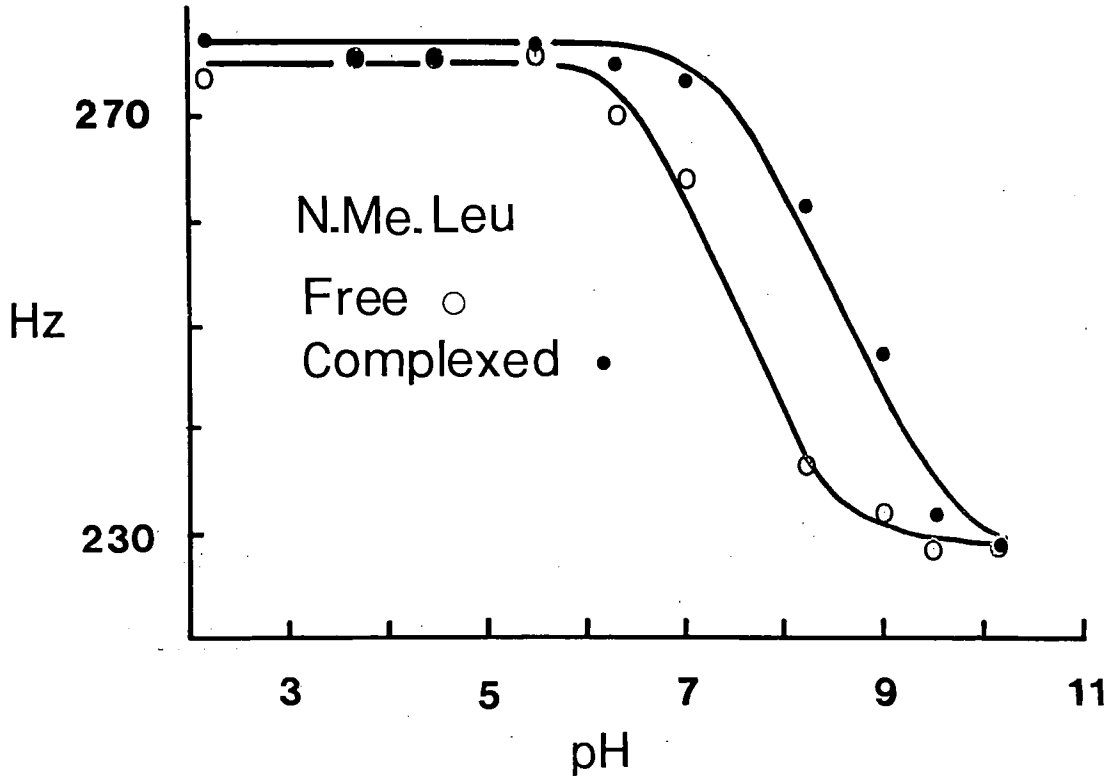
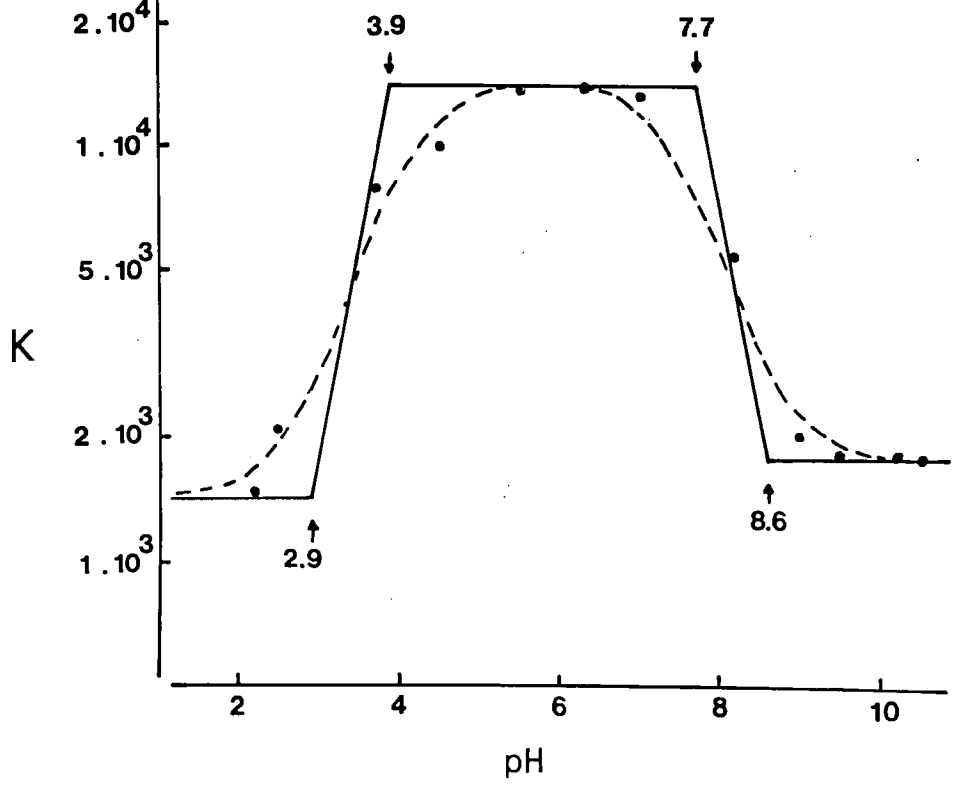

270
230
Hz
N.Me.Leu
Free ○
Complexed •
3
5
7
9
11
pH

FIG. 2


2.10⁴
1.10⁴
5.10³
2.10³
1.10³
K
3.9
7.7
2.9
8.6
2
4
6
8
10
pH

193-25

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DEPARTMENT OF CHEMISTRY
REVELLE COLLEGE

POST OFFICE BOX 109
LA JOLLA, CALIFORNIA 92037

October 7, 1974

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

Dear Barry,

The accompanying proton spectrum is that of a mixture of 2,5-dibromothiophene and benzene. The chemical shift between the two peaks is 36.3 Hz (.66 ppm) but as far as information content goes, the only interesting thing is that it was obtained on our pulse spectrometer by FT of a very conventional free induction decay after a 90° pulse. We admit to feeling a certain amount of nostalgia when we got this spectrum, and think that it might present a proper challenge to the spectrum simulators. Have fun!

Best Regards,

Howard

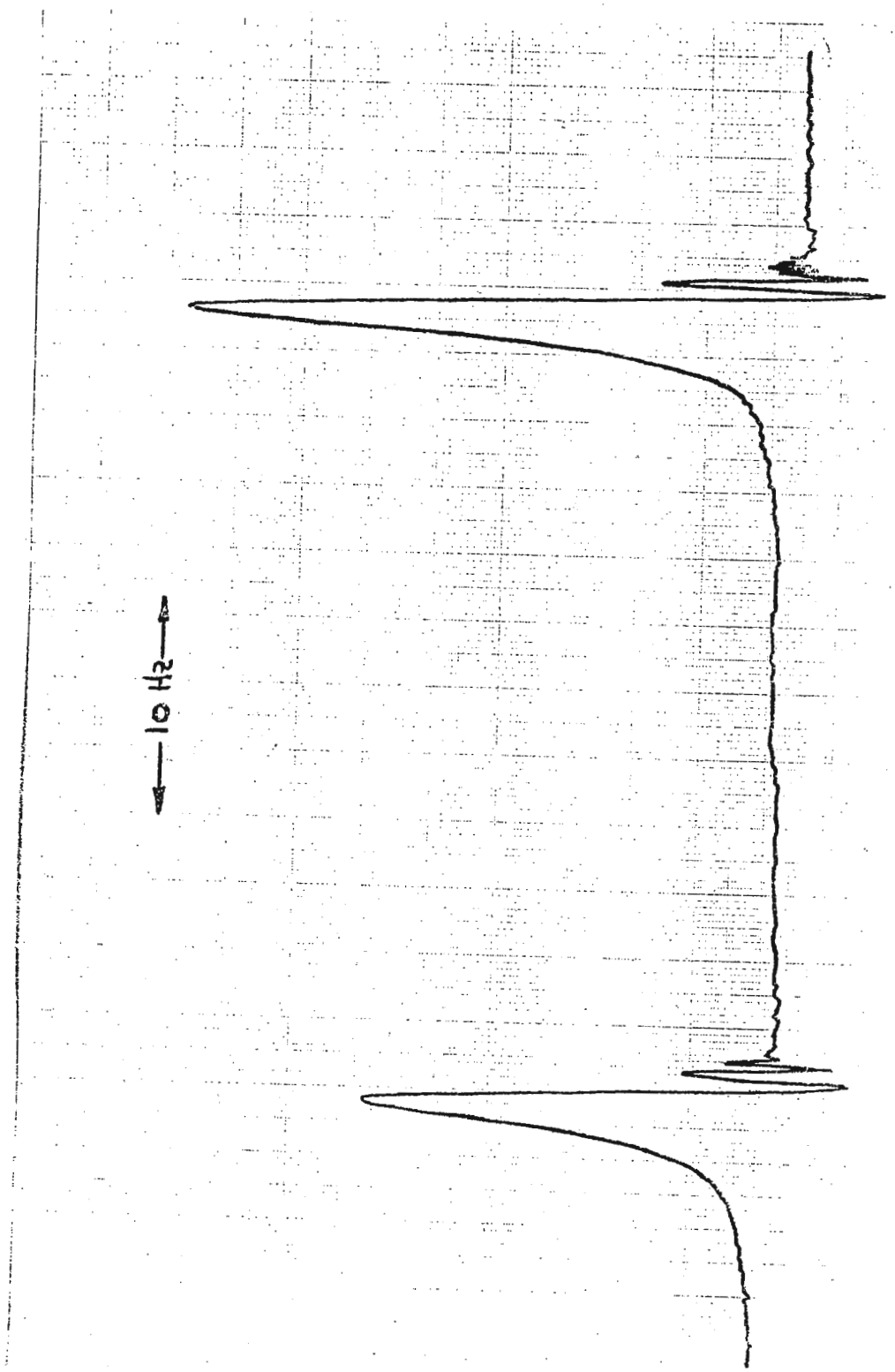
Bob

Gitte

Howard Simon
Bob Vold
Gitte Vold

mb
enclosure

P.S. We assure you that the spectrum was recorded quite slowly – it is not pen overshoot!


10 Hz

Department of Chemistry

The Florida State University
Tallahassee, Florida 32306

October 11, 1974

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

Dear Barry,

Unlikely Molecular Complexes

At last I have tasted all that life can offer, having received my first pink letter.

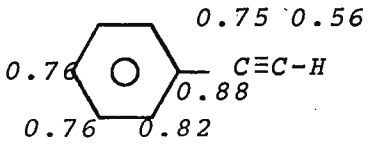
In the absence of anything straightforward to present I thought I'd let you know about some interesting but challenging studies we are doing in the area of electron-nuclear relaxation (in this case ^{13}C relaxation resulting from dipole-dipole interactions with unpaired spins).

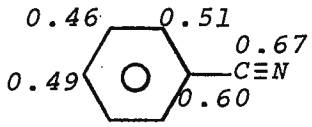
We have already reported that acidic hydroxyl protons interact with "inert" paramagnetic relaxation reagents such as chromium tris-acetylacetonate $[Cr(acac)_3]$[1,2]. Hydrogen bonding (presumably to the oxygen ligands of the metal chelate) is indicated when ^{13}C T_1's for the interacting species are (a) shorter than for "inert" molecules in the same solution and (b) graduated, with the shortest T_1's observed for carbons located close to the hydroxyl group.

Even though the effect is time and species averaged it is possible to calculate complex geometries, as with LIS reagents.[2] In this case however there is no primary angular dependence; there is an inverse sixth power distance dependence.

We have been especially interested in using these "inert" relaxation reagents to study other weak solution interactions. In principle, we should be able to identify intermolecular interactions as weak as 0.2-0.3 kcal mol^{-1} using this technique.

Acidic C-H hydrogens such as the acetylenic hydrogen in phenyl acetylene can hydrogen bond to the $Cr(acac)_3$. ^{13}C T_1's observed for phenyl acetylene and benzonitrile containing $Cr(acac)_3$ indicate this:





We are currently expanding on these qualitative experiments to determine the exact geometry of the solution complex, etc.

Preliminary experiments on planar aromatic hydrocarbons are indicating that a favorable interaction observed between the acetylacetonates and these aromatics is probably an edge-on interaction and not a π-cloud effect. It may be that a weak "hydrogen bond" between the ring hydrogens and the chelates will have to be invoked, but at this time we are not ruling out other possibilities. Work continues.....

Best regards,

George C. Levy
Associate Professor

[1] *G. C. Levy and J. D. Cargioli, J. Magn. Resonance, 10, 231 (1973).*

[2] *G. C. Levy and R. A. Komoroski, J. Am. Chem. Soc., 96, 678 (1974).*

193-29

TEXAS A&M UNIVERSITY
COLLEGE OF SCIENCE
COLLEGE STATION, TEXAS 77843

Department of
CHEMISTRY

15 October 1974

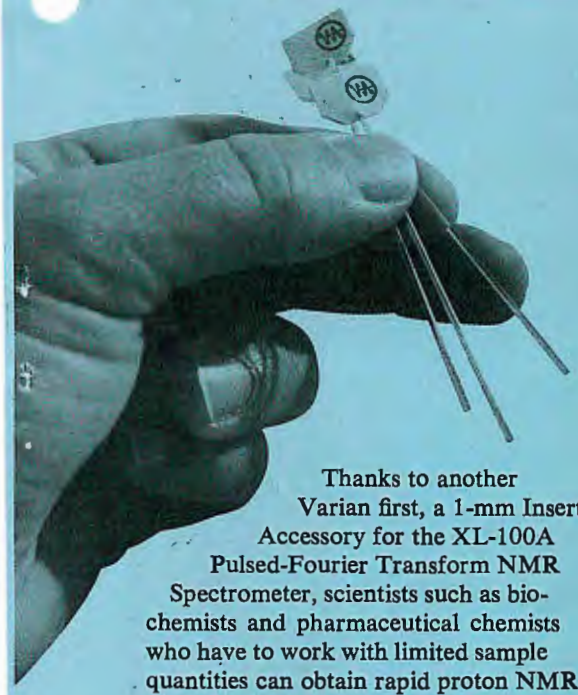
TAMU NMR Newsletter subscribers who have not paid for their annual subscription (or at least written us with a firm indication that payment is coming) will not receive the next issue of the Newsletter.

Needless to say, if you have already paid or sent appropriate word to us, ignore this notice.

I regret very much the necessity of this notice, but our desperate financial situation will no longer permit any other course of action.

B. L. Shapiro

Now the XL-100A NMR Spectrometer lets you think small.



Thanks to another Varian first, a 1-mm Insert Accessory for the XL-100A Pulsed-Fourier Transform NMR Spectrometer, scientists such as biochemists and pharmaceutical chemists who have to work with limited sample quantities can obtain rapid proton NMR analysis of microgram samples.

Using the insert, it's possible to run spectra of 50 μg or less of sample. Spectra run thusly are obtained in less than 17 minutes, yet are superior to 8-hour runs in a 5-mm tube. Sensitivity for a fixed amount of sample can improve from 4- to 6-fold when the 1-mm Insert Accessory is used.

The two spectra of Δ^9-tetrahydrocannabinol (THC) shown here demonstrate the dramatic results possible using the 1-mm Insert. Spectrum A, of a concentrated sample in a 5-mm tube, serves as a comparison for the other spectra. Spectrum B (20 μg of sample in a 1-mm tube) and Spectrum C (20 μg of sample in a 5-mm tube) were run under identical conditions. Note the well-defined peaks in the spectrum run using the 1-mm Insert.

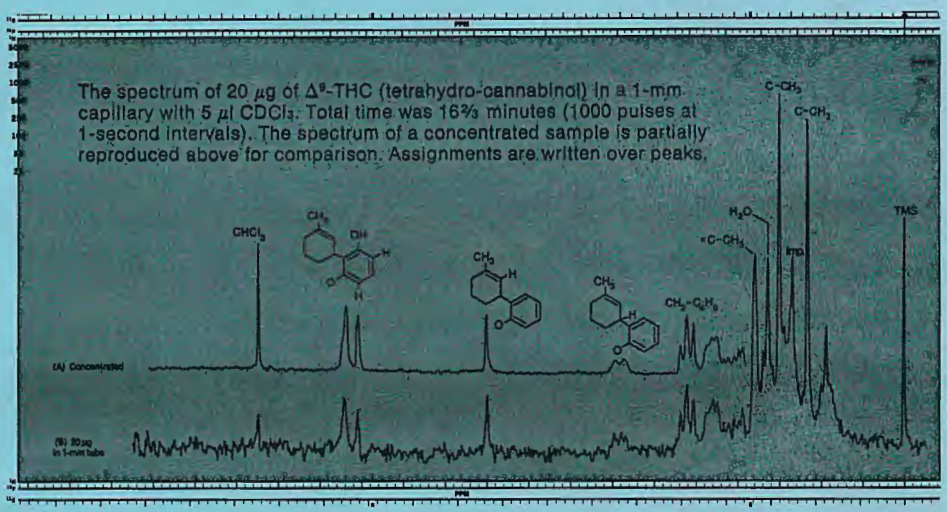
This innovative approach is successful since reducing the sizes of both the sample tube and the receiver coil ensures maximum coupling of the available nuclear magnetic moments with the coil. It permits the use of commercially available capillary tubes costing less than one cent each.

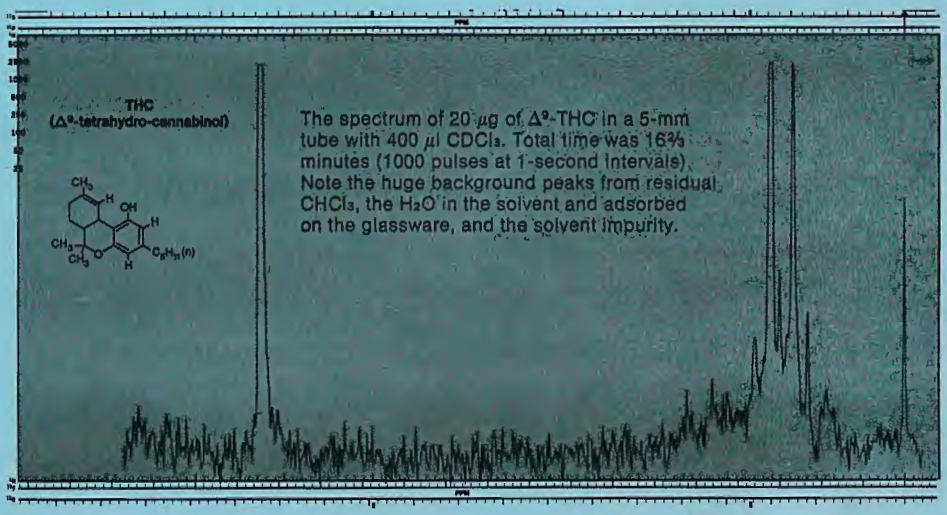
To interchange the 1-mm Insert with standard XL-100A inserts, merely take one out, put in the other, retune and balance. The sample is dissolved in 5 μl of an NMR solvent containing TMS for a reference. It is then transferred into a 1-mm sample tube by using a drawn out glass pipette or a hypodermic syringe. This eliminates the bubble problems which sometimes arise with the use of microcells in larger tubes. The resulting column length is about 10 mm, assuring freedom from line shape distortion. Since spinning produces no vortex, spinning speed is not a critical factor.

The sample volume in the 1-mm Insert is so much less than the 400 μl required for 5-mm tubes that use of deuterated species becomes more economical.

The 1-mm capillary has its own spinner turbine attached. Unlike other existing techniques designed to accommodate small quantities of samples, there are no plugs to adjust and no sample positioning is necessary. Proper positioning is automatic thereby assuring reproducible homogeneity.

Write for a copy of Varian's Application Report NMR-2, which describes the XL-100A Insert Accessory in more detail.





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The FX60 was recently previewed at the ENC Conference, Raleigh, North Carolina, April 28, 1974.

INSTRUMENTATION: NMR Spectrometers/Mass Spectrometers/ESR Spectrometers/Laboratory Computers/Scanning Electron Microscopes/Electron Microscopes/ X-ray Microprobes/X-ray Diffractometers/Electron Beam Apparatus/Amino Acid Analyzers/Sequence Analyzers.