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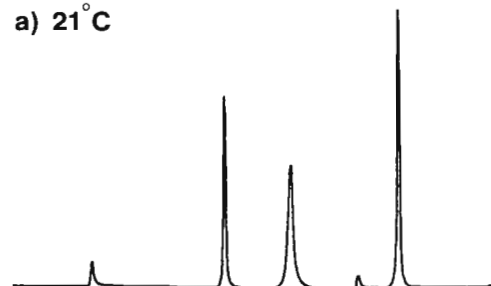
Professor Bernard L. Shapiro
 Department of Chemistry
 Texas A&M University
 College Station, Texas 77843 U.S.A.

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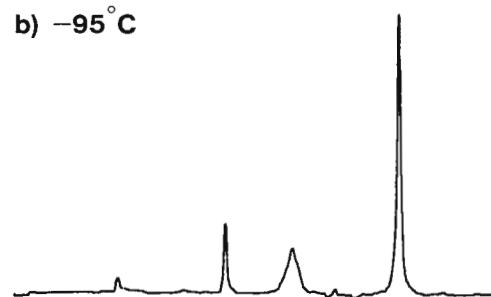
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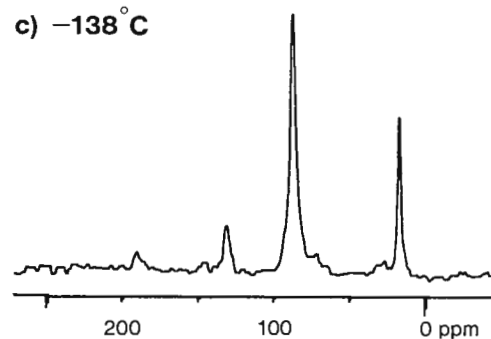
a) 21°C



b) -95°C



c) -138°C



¹³C (50.1 MHz) VT/MAS spectra of hexamethylbenzene. a) and c) ¹H-¹³C cross polarization. b) Bloch decay. The peak at ~90ppm is due to the Delrin rotor.

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August 31, 1983

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

³¹P NMR Spectra of Model
and Biological Membranes

Dear Barry:

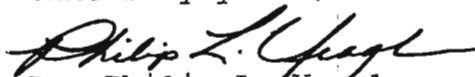
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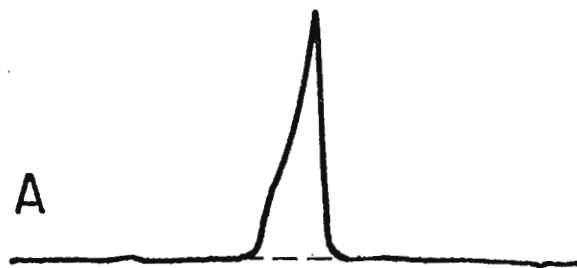
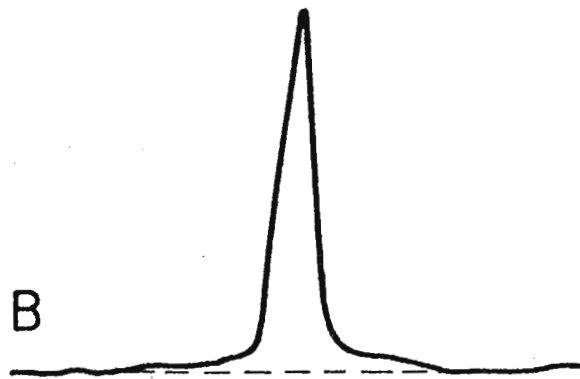
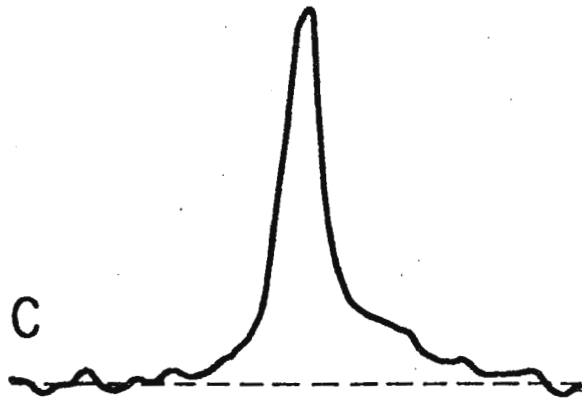
We have for some time been interested in the P-31 NMR spectra of model and biological membranes. When we recently began using our JEOL FX 270 to observe spectra from unsonicated systems, the problems inherent in using a high resolution spectrometer to observe broad resonances immediately became apparent. The resonance seen from pure phospholipid bilayers is a pseudo-axially symmetric powder pattern about 45 ppm wide. The single pulse experiment produced distorted spectra.

Recently a fully phase cycled spin echo experiment was described (Rance and Byrd, J. Mag. Res. 52, 221-240 (1983)). This we wrote into the PG200 pulse programmer, and thereby began producing undistorted spectra, as predicted, using 20 μ sec echos. An example of this is seen in figure 1a for unsonicated egg phosphatidylcholine bilayers.

Upon applying this technique to membranes containing proteins, a considerably different resonance shape was obtained. Examples are seen in figure 1b and 1c. These membranes contain the photopigment protein rhodopsin at increasing concentrations. Spectral simulations show that these spectra consist of overlapping resonances. One is a fairly normal appearing "bilayer" resonance. The other is much broader and dominates the spectrum of the membrane of highest protein content. Apparently the presence of protein causes a second phospholipid headgroup environment to be created that is motionally restricted compared to a protein-free phospholipid bilayer. The echo sequence allows a view of this phenomenon not previously available to us and it is now under intensive study.

Sincerely yours,


Dr. Philip L. Yeagle
Associate Professor



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August 31, 1983

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

Dear Barry:

MAGIC ANGLE SPINNING CAN IMPROVE SPECTRA OF LIQUIDS

The non-destructive nature of NMR as an analytical tool makes it of special interest in the analysis of living organisms. One example of this is the measurement of the composition of the lipid fraction of seeds, i.e., the various fatty acid components of the triglycerides, without altering the viability of the seeds. Single seed analyses, in particular, would allow much more direct selection of particular traits associated with incorporation of unsaturation of the fatty acids, and thus greatly improve breeding programs aimed at improving oil quality.

Analysis of animal and vegetable oils by ^{13}C NMR has been reported (1) for the separated oils, and spectra of oils in seeds have been reported by a number of workers (2-5). In all cases the resolution has been disappointing. This is not surprising considering that the oil is trapped within the cells of the seed and may not only be partially immobilized by binding to the cell membranes, but is certainly subjected to an inhomogeneous field due to variations in susceptibility throughout the seed.

In magnetic fields up to about 20 Kgauss the line widths of the ^{13}C peaks from the oil have typically been about 8-10 Hz, and with drastic resolution enhancement the various fatty acid components could just be resolved. However, the sensitivity at such field strengths required data acquisition for an hour or more for each seed. I attempted measurements at 47 Kgauss but reported (6) no improvement.

I have just carried out a series of ^{13}C measurements at 20, 50, 75, and 100 MHz which show that not only are the T_1 's in the oil in seeds independent of field up to 75 MHz, but are unchanged from the values found in the pure triglycerides. Apparently, the oil is not immobilized by binding to the membranes, and the line widths, which reach 50 Hz at 94 Kgauss, are due to spatial variations in susceptibility. Spinning the seed about the magic angle axis for which $3\cos^2\theta - 1 = 0$ should average such broadening to zero (7) and Figure 1 shows that this is indeed the case. Line widths of 2.5 Hz were achieved which could be further narrowed by resolution enhancement as shown.

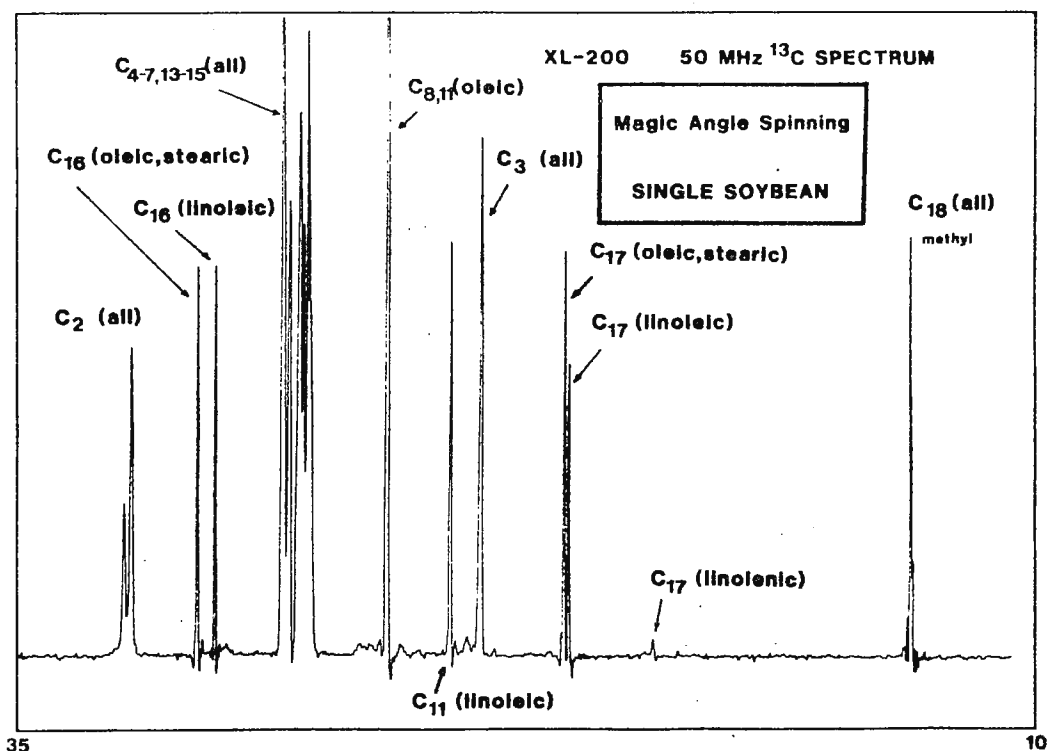


Figure 1. CH_3 and CH_2 peaks in soy oil within a viable seed.
1500 pulses at 0.4 sec intervals with 45° tip angle.

Narrowing the lines results in recovery of the improved sensitivity expected at higher magnetic fields, and it appears that analyses of the needed accuracy could be accomplished with as little as 1-2 minutes of data acquisition per seed. This should be of considerable interest to agronomists concerned with improvement of vegetable oil quality.

The technique of magic angle spinning (MAS) is normally thought of in connection with studies of rigid solids, but in biological samples with liquids trapped in cellular compartments it may have wide applicability.

Sincerely,

J. N. Shoolery

J.N. Shoolery
Applications Chemist
NMR Applications Laboratory

References:

1. Shoolery, J.N., Prog. in Mag. Res., 79-93 (1977)
2. Shoolery, J.N., Varian Assoc. Applic. Note, NMR 73-3 (1973)
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Anion Binding to Proteins by ^{14}N M R

Studies of anion binding to proteins usually involves the physiologically omnipresent chloride and thus utilizes the ^{35}Cl nucleus. The bound anion is presumed to have a very much enhanced quadrupolar relaxation, and by rapid exchange with unbound ions causes the observed linewidth to broaden. Analysis of the width versus concentration curves then allows binding constants to be inferred¹.

The iron transport protein transferrin requires the binding of a synergistic anion in order for the iron to be complexed by the protein. The simplest of these, and the most physiologically relevant, is the bicarbonate (or carbonate) anion². Unfortunately, bicarbonate does not contain an abundant quadrupolar nucleus. However, we have been able to synthesize a ^{14}N containing pseudo-carbonate ion, which we call the "nitrate ion". This is "isostructural" and isoelectronic with bicarbonate which is probably the form of the anion bound to the protein³.

Initial binding data from ^{14}N linewidths (analogous to ^{35}Cl studies using Cl^- or ClO_4^-) indicate that nitrate indeed satisfies the fast exchange limit. Our present data with the metal free protein point towards a specific binding which is considerably stronger than that of either Cl^- or ClO_4^- despite the fact that nitrate lies intermediate between these anions on the lyotropic series.

We are currently using nitrate ion to examine binding sites on chemically modified transferrins. We believe it to be a useful probe of synergistic ion effects in the transferrins and can perhaps be employed in studies of other proteins as well.

N. Dennis Chasteen

N. Dennis Chasteen

Carl P. Thompson

Carl P. Thompson

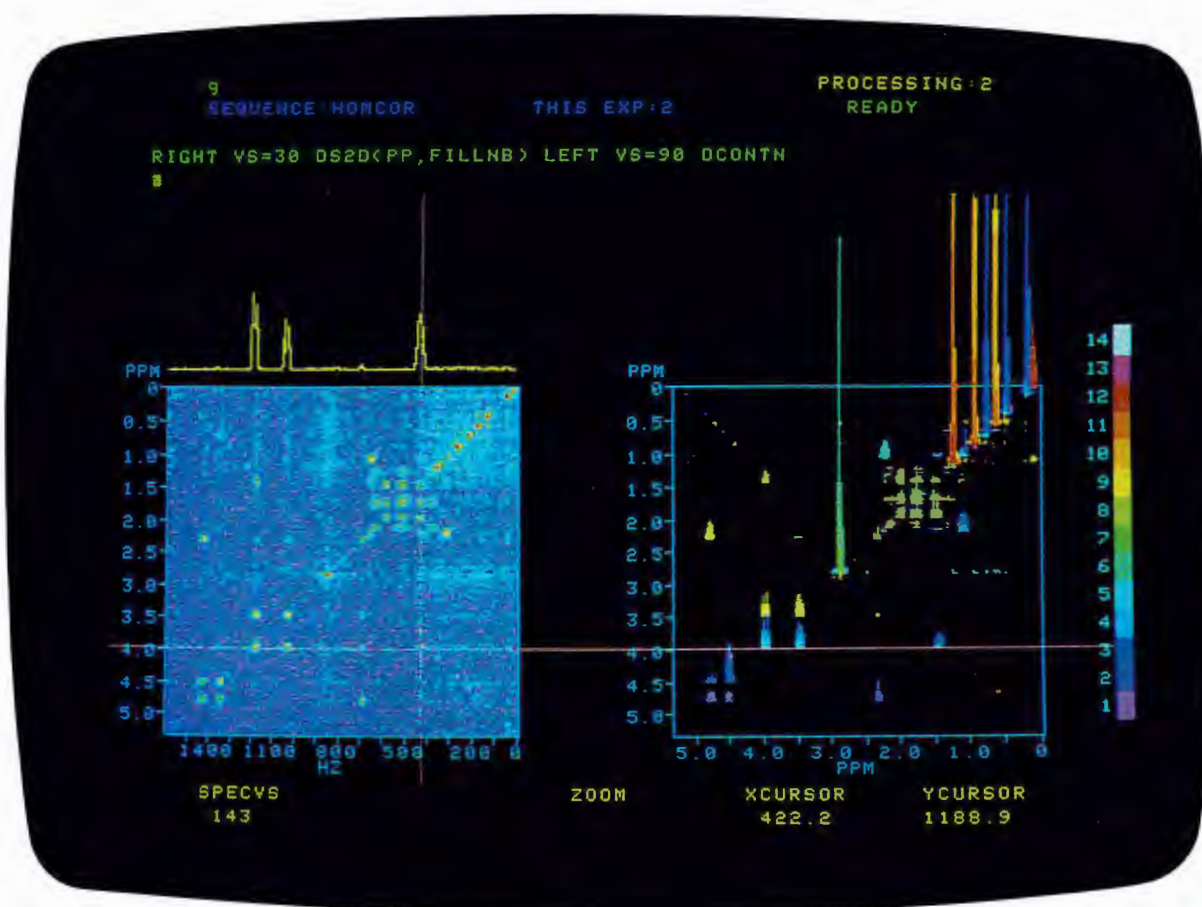
C. E. Holloway

Clive E. Holloway

Please credit this to the account of Kathleen Gallagher, University of New Hampshire.

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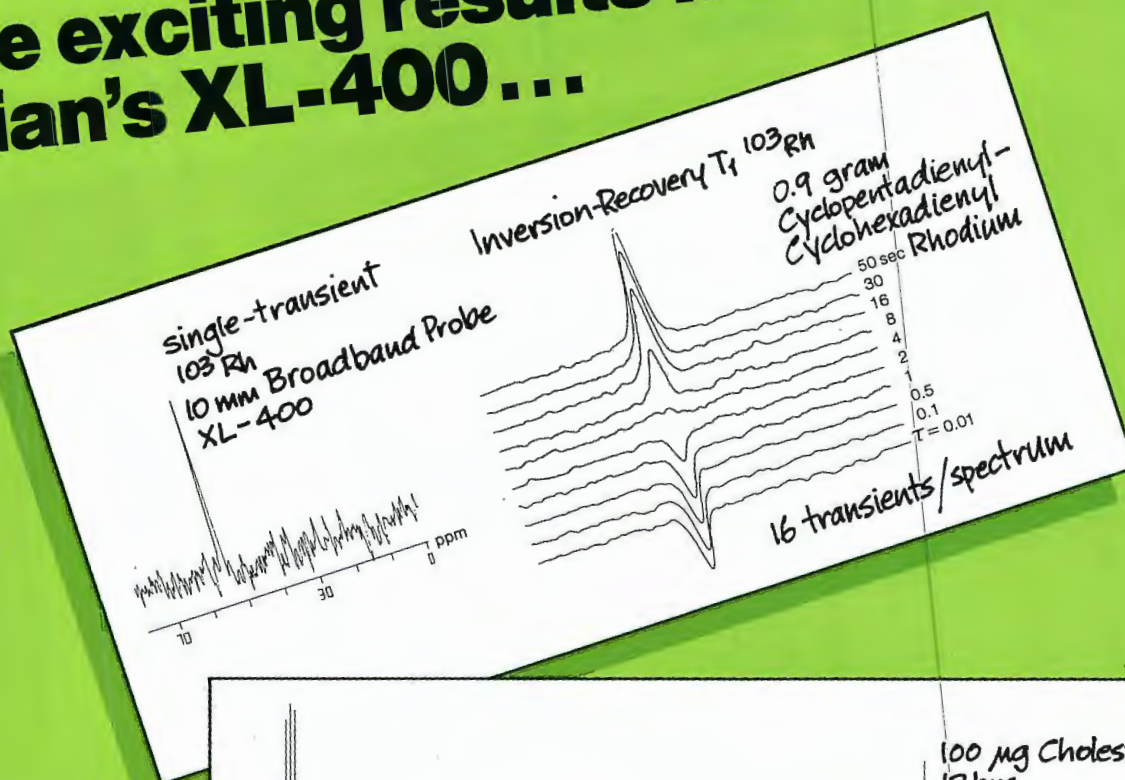
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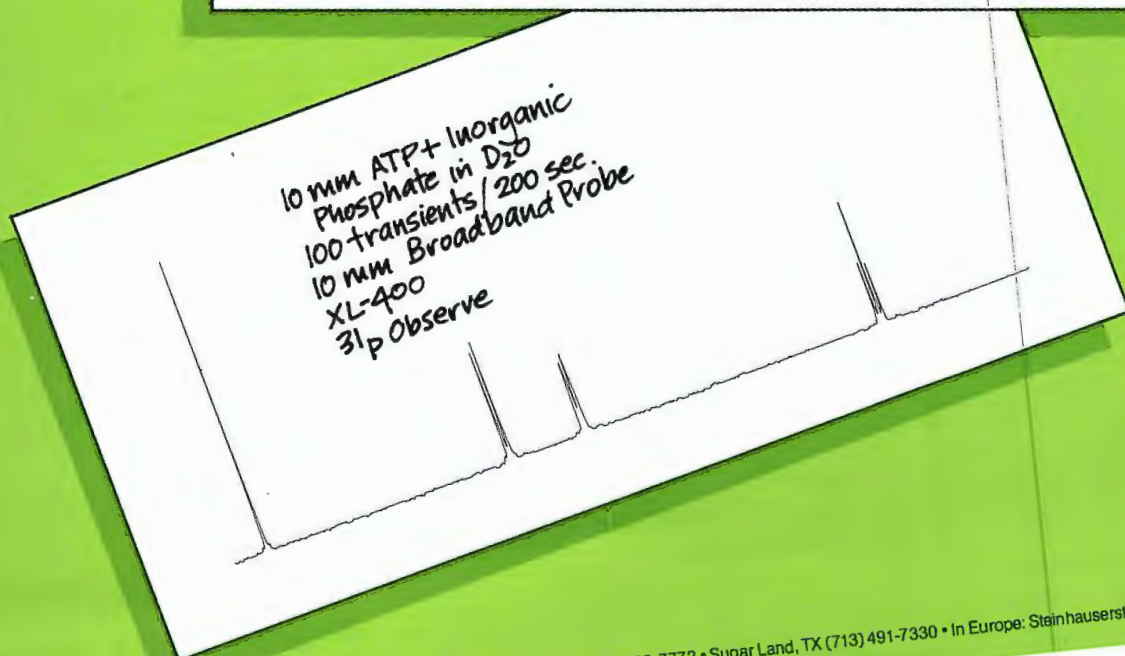
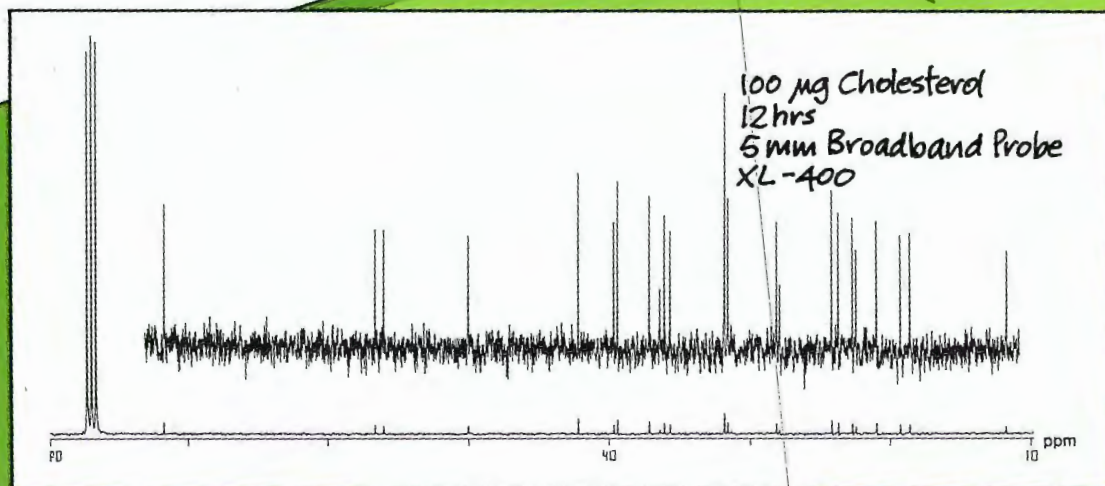


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Montpellier, le September 1, 1983.

Professor B.L. SHAPIRO
Department of Chemistry
Texas A and M University
College Station, Texas 77843
USA

NMR Studies of Conducting
Polymers

Dear Professor SHAPIRO :

Solid state High Resolution NMR spectroscopy is now of common use to study the properties of "conducting polymers". The existence of conduction electrons in systems like polyacetylene doped with iodine (giving the compound $(CH I_{0.14})_x$ for instance) should be characterized by a typical shift of the ^{13}C resonance for instance (Knight shift) whose magnitude and sign are related with the electronic properties of the system. Nevertheless in such cases the shift is expected to be very small, in the range 10 to 100 ppm.

Any attempt to evidence such a shift on doped polyacetylene or polyphenylene has been either unsuccessful (1) or strongly criticized (2, 3). One important reason for such a negative situation is the particularly unstable nature of most of the conducting polymers : contact with air or moisture, heating, hard mechanical constraints of the systems.

We have made the following observation. Small pieces of polyacetylene films doped with iodine up to 14 molar percent (conductivity $\sim 100 (\Omega \text{ cm})^{-1}$) have been observed using ^{13}C MAS NMR. No Knight shift could be clearly evidenced. But after a 30 minutes spinning (2.7 kHz) of the sample the remaining polymer had a conductivity close to 1 implying that 80% of the iodine had been removed from the sample. And it has been otherwise established that such a loss of dopant is possible only if the temperature of the sample is close to 140°C during a significative period of time (4) we then conclude that the existence of Eddy currents during the spinning of the metallic polymer in the magnetic field is not a secondary effect and can strongly modify the nature of the system under study.

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- (4) M. ROLLAND, S. LEFRANT, M. ALDISSI, P. BERNIER, E. RZEPKA, F. SCHUE, J. of Elect. Mat., 1981, 10, p 619.

Sincerely yours.


Patrick BERNIER



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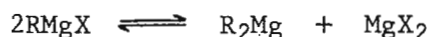
5th September, 1983.

Professor Bernard L. Shapiro,
Department of Chemistry,
Texas A. & M. University,
COLLEGE STATION. TEXAS. 77843.
U.S.A.

Dear Professor Shapiro,

Sealing n.m.r. tubes under high vacuum

The University of Adelaide has recently entered the medium field n.m.r. world with a CXP300 spectrometer. Most of our time has been taken up in starting new experimental programs to take advantage of this (for us) much higher field than that to which we were accustomed. One project that had nearly been completed on our HX90E spectrometer was an investigation into the nature of the Grignard reagent, especially with respect to the kinetics of the Schlenk equilibrium.



One problem area remaining was that of the nature of the species present in t-butyl magnesium bromide solutions in THF.

We had mapped the behaviour of the ^1H resonances at about 0.8 ppm (cf. Fig. 1) as a function of temperature (Fig. 2). As can be seen in Figure 1, there are 4 major resonances, two of which (A & B) were assigned to be RMgX like, and the other two (C & D) to be R_2Mg like. The nature of this system was, however, quite unlike any other Grignard, having 4 distinct major species rather than 2.

Imagine our consternation when we re-ran our sealed tube on our brand new CXP300 and obtained Figure 3! Eventually the light dawned. Our mystery two species with their field dependent chemical shifts turned out to be nothing more than the methyl doublet of $(\text{CH}_3)_3\text{CH}$ which has a coupling constant of 5.7 Hz and is represented by peaks B & D. The true RMgX is peak A and R_2Mg is peak C. The alkane is formed by reaction of t-butyl MgBr with water produced in the pyrolysis of THF (the solvent) when the n.m.r. tube is sealed off on the high vacuum line.¹

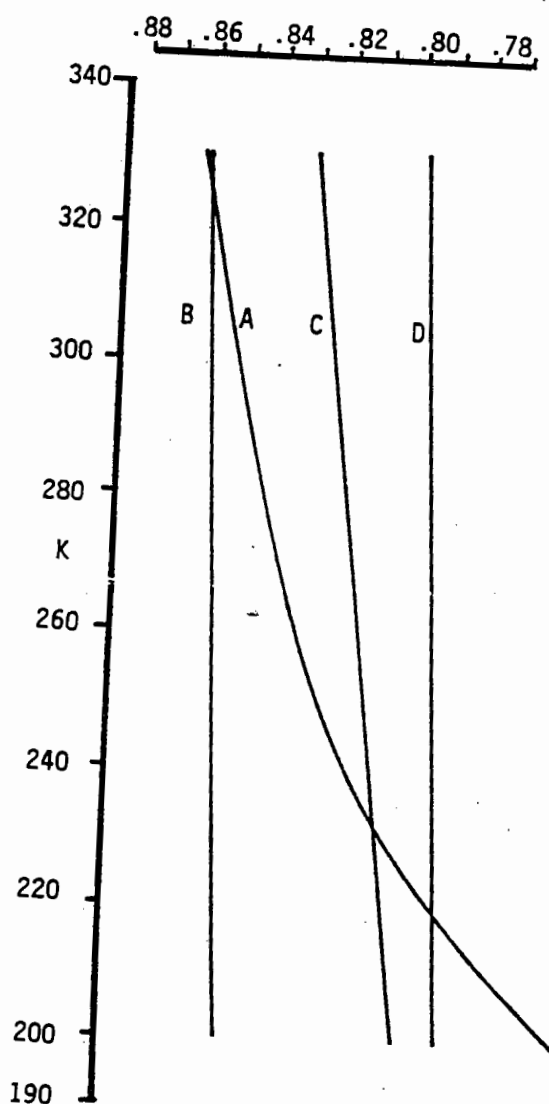
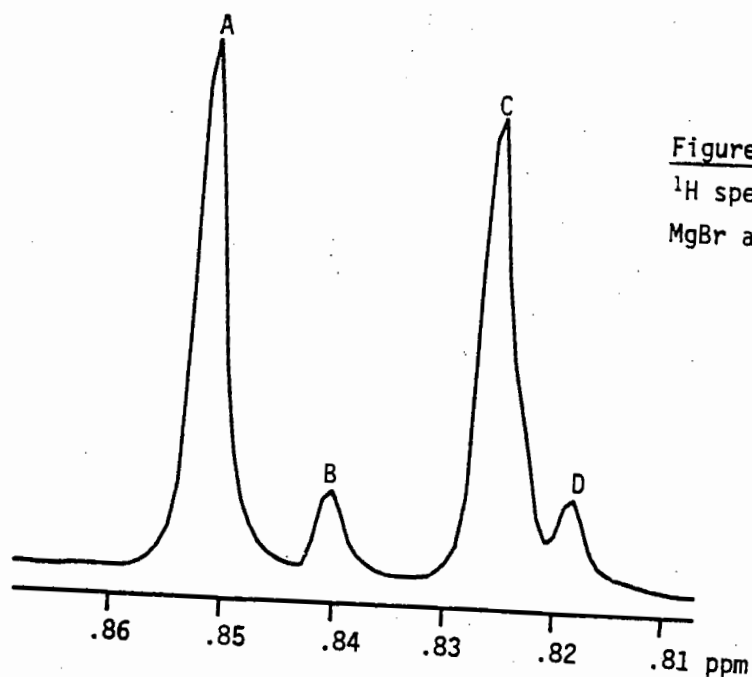


Figure 2: Shift of t-butyl MgR system resonances with temperature at 90 MHz ^1H frequency.

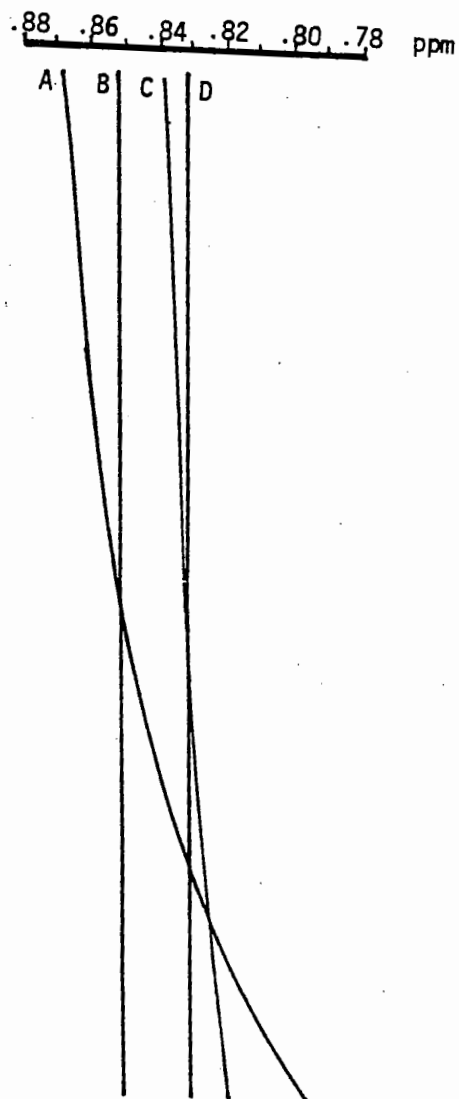


Figure 3: As for Figure 2 but at 300 MHz ^1H frequency.

The moral of this tale is that sealed tubes are not necessarily a more "pure" environment than a stoppered one. We have found that in order to avoid such contamination we must warm the seal-off point for 20-30 minutes before raising to glass-melting temperatures. All this time, the vacuum line must be pumped down hard to avoid condensing volatiles in the frozen contents of the tube (always a hazard in sealed tube work).

Such an occurrence also shows the merit in acquiring n.m.r. spectrometers at as many different field strengths as possible.

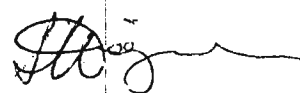
Yours sincerely,



E.H. WILLIAMS.



P.E.M. ALLEN.



S. HAGIAS.

Ref: 1. M. Szwarc, "Carbanions, living polymers and electron transfer processes", p. 167, Wiley, N.Y. (1968).

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

Dear Barry:

^{57}Fe Chemical Shifts and Relaxation

Last year, after the arrival of our Nicolet 500 MHz instrument we started a project on ^{57}Fe NMR ($I=1/2$, natural abundance 2.2%, relative sensitivity 3.3×10^{-5}) with the ultimate objective of studying iron in biological systems. Since very little is published on ^{57}Fe NMR, the first task was to define the chemical shift scale. Von Philipsborn, et al.,¹ have reported on a large number of compounds, but they are all of the type where iron is coordinated to carbon. We therefore used the model compound $^{57}\text{Fe}(\text{bipy})_3\text{Cl}_2$ (bipy = bipyridyl) in D_2O and set out to find its chemical shift using a non-spinning probe (built by Dennis Torchia), fitted with a solenoid coil. After some unsuccessful attempts, we finally found it 9701 ppm downfield of t-butylferrocene (80% in acetone- d_6). That is 8907 ppm downfield of $\text{Li}_4\text{Fe}(\text{CN})_6$. For comparison $^{59}\text{Co}(\text{bipy})_3(\text{ClO}_4)_3$ resonates 6620 ppm downfield of $\text{K}_3[^{59}\text{Co}(\text{CN})_6]^{2-}$ and $^{99}\text{Ru}(\text{NH}_3)_6^{2+}$ resonates 7680 ppm downfield of $^{99}\text{Ru}(\text{CN})_6^{4-}$.³ t-Butylferrocene (80% in acetone- d_6) is 1568 ppm downfield of $\text{Fe}(\text{CO})_5$, the reference used in ref. 1.


The high field is advantageous, not only from the standpoint of sensitivity, but also because of the shorter T_1 . We have investigated the field dependence of T_1 in t-butylferrocene and find that chemical shift anisotropy is the dominant relaxation mechanism.

Lars has recently left after two years to take up a position at the University of Goteborg in Sweden. Otto is moving to the National Cancer Institute as Chief of the Section on Inorganic Chemistry.

Sincerely yours,

Lars Baltzer


Otto A. Gansow


Edwin D. Becker

1. Jenny, T., Von Philipsborn, W., Kronenbitter, J. and Schwenk, A., J. Organomet. Chem. 205 (1981) 211-222.
2. Dharmatti, S. S. and Kanekar, C. R., J. Chem. Phys. 31 (1960) 1430.
3. Dykstra, R. W. and Harrison, A. M., J. Mag. Res. 46 (1982) 338-342.



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PROGRESS IN SOLID STATE NMR STUDIES

Some 16 months ago we entered the field of solid state NMR by installing a JEOL/Chemagnetics accessory on our FX200 spectrometer. Apart from some initial rotor wear problems the system has worked well and we have obtained many hundreds of spectra from coals, shales, zeolites and many miscellaneous samples. The system was delivered with two probes, one for ^{13}C and one for ^{29}Si . We were pleased to find that the ^{13}C probe would tune to the ^{27}Al frequency and retain a good performance. Further, we have found that without any padding capacitors the probes will tune to 80MHz enabling us to examine samples by ^{31}P NMR. These spectra can be surprisingly informative as the example of cis dichlorodi-(triphenyl phosphine) platinum shows (Figure 1). The central group of bands can be interpreted as showing the presence of three (A, B and C) crystallographically different species (1). About 33% of platinum atoms are the 195 isotope and these couple with the phosphorus to give doublets. Again, due to solid state effects, three coupling constants (e.g. J_{AP}) are observed and have the values 3725, 3715 and 3546 Hz. Such spectra are also useful in detecting impurities.

Our Laboratory also has a JEOL GX400 system (^1H and ^{13}C) and the quite close proximity of the 9.4T magnet to the FX200 console prompted us to consider using it for solid state studies. Initially, one can use the existing probe to examine nuclei that now resonate in the tuning range of ~35 to 80 MHz. One good sample for system tuning is $^{133}\text{CsCl}_3$. The ^{133}Cs signal is very strong and is easily detected. Recently, we have attempted to obtain some ^{15}N spectra, initially from inorganic samples since, as yet, we do not have ^1H 400 MHz decoupling. Figure 2 shows the ^{15}N spectrum from enriched $\text{Na}^{15}\text{NO}_3$. For such a system we find a relaxation time of 38.5 ± 5 seconds and the side band intensity pattern indicates a significant chemical shift anisotropy*. It appears to us that for practical systems a polarisation enhancement process is necessary for successful ^{15}N studies.

To examine nuclei such as ^{29}Si and ^{27}Al at high frequencies we have constructed a probe using a Beams/Andrew/Fyfe type rotor system. This has worked quite well and Figure 3 shows a ^{27}Al signal (one pulse) from a 13X sieve sample. In the construction of these high frequency - high voltage MAS probes we find

it is vital to use components of the highest quality. It seems one cannot make any compromises here.

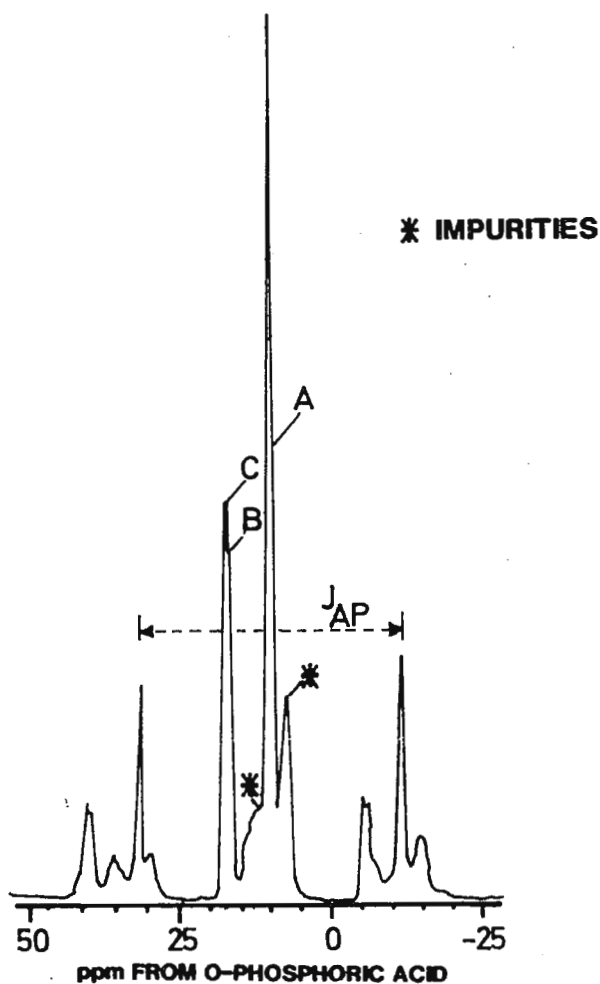
S. Knight

I.J.F. Poplett

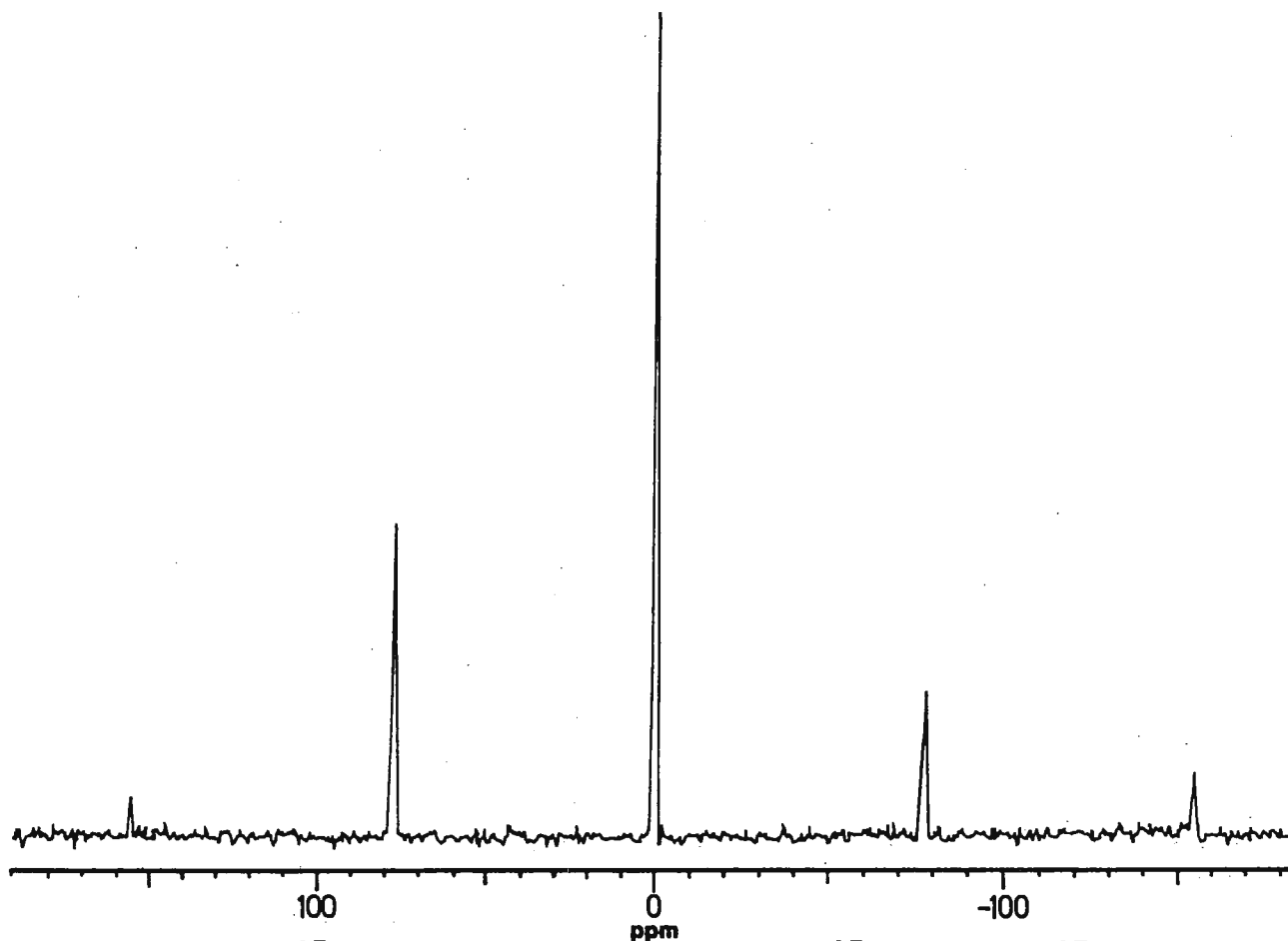
Reference

- (1) Berni, L., Clar, H.C., Davies, J.A., Drexter, D., Fyfe, C.A., and Wasylishen, R., J. Org. Metal Chem., 1982, 224, 65 - 69

* Approximate values are $\sigma_{11} = -94$, $\sigma_{22} = -47$, $\sigma_{33} = 140$, assuming for NO_3^- $\sigma = 0$



**FIG 1 SOLID STATE PHOSPHORUS-31 MAS NMR
SPECTRUM OF ~~cl₂~~ DICHLORO DITRIPHENYL
PHOSPHENE PLATINUM**



**FIG 2 ^{15}N MASNMR SPECTRUM OF $\text{Na}^{15}\text{NO}_3$ (95% ^{15}N)
40 PULSES 120s PULSE INTERVAL**

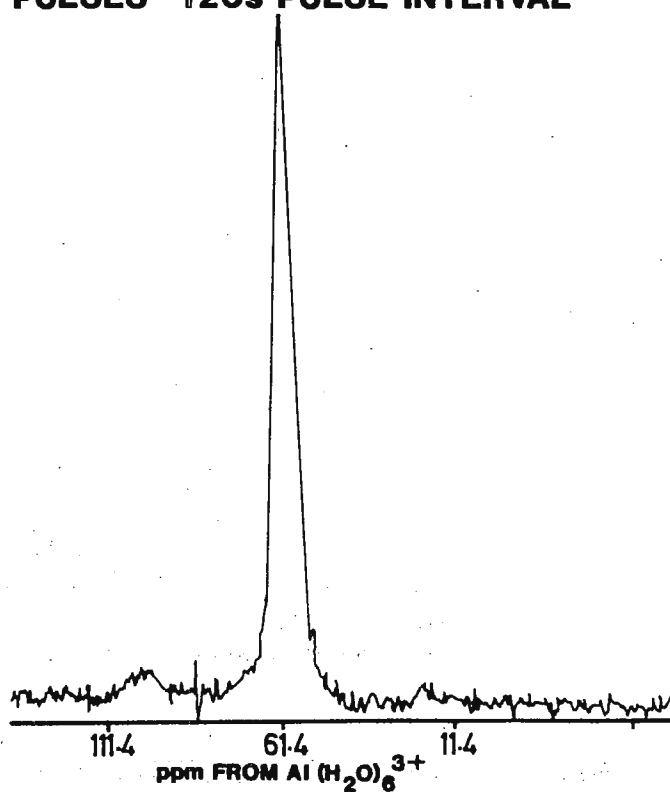


FIG 3 104.14 MHz ^{27}Al SPECTRUM FROM 13 x SIEVE - 1 PULSE EXPERIMENT



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Ns. Rip. Prot. N.

Photo-CIDNP Experiments on
Bovine Pancreatic Secretory
Trypsin Inhibitor

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

Dear Prof. Shapiro:

In the last months our lab has gone through some major changes. Dr. G. Gatti, to whom your circulars have been addressed, has been on a leave of absence for one year, and will most likely remain on leave for at least another year. Since we would not like our institution to be dropped from your list of contributors, we would ask you to address any future correspondence to either one of the undersigned.

Recently, by running photo-CIDNP experiments on the bovine Pancreatic Secretory Trypsin Inhibitor (PSTI, 56 aminoacids), we noticed that the solvent accessible, fast flipping Tyr-20 (1), not only gives a neat response to the laser excitation in the aromatic region, but also shows a strongly enhanced, well-resolved multiplet from the β -CH₂ resonances in the difference spectrum (AB part of an ABX spin system, see figure). Analysis of the multiplet gives $^3J_{\alpha\beta} = 2.5$ and 13.8 Hz, implying that only one of the staggered conformers about χ^1 (i.e. g⁺t or g⁻t) is essentially populated. This indicates, rather surprisingly, that the side chain freedom does not extend beyond χ^2 . Hence, the photo-CIDNP technique appears to be suitable for investigating the conformation of Tyr, Trp, and His side chains in proteins, by enhancing resonances otherwise not easily detectable (see "dark" spectrum).

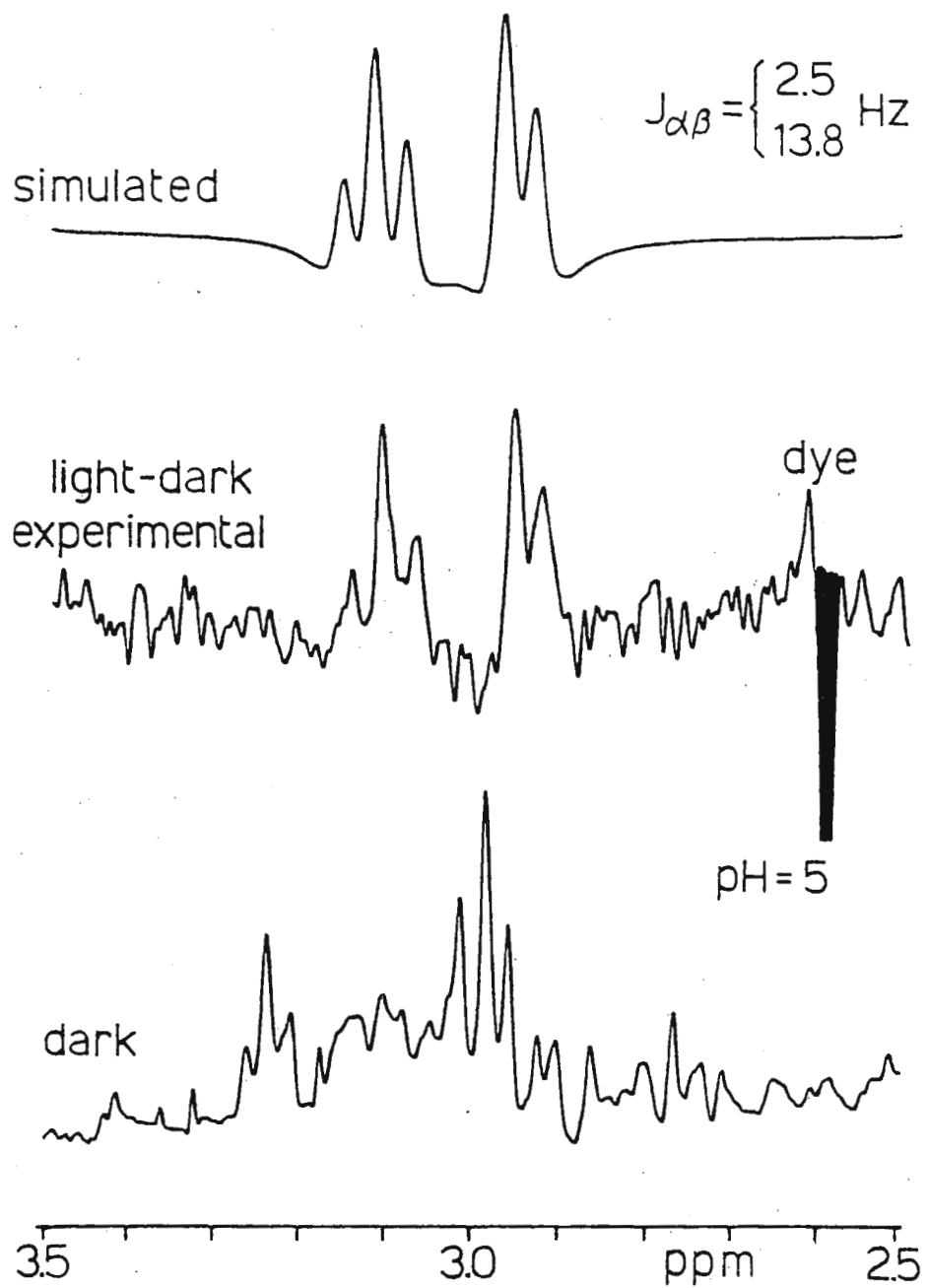
Best regards,

(Dr. Lucia Zetta)

(Dr. Antonio De Marco)

(1) A. De Marco, E. Menegatti and M. Guarneri (1979) Eur. J. Biochem.
102, 185-194

Y-20

 β -CH₂ resonances

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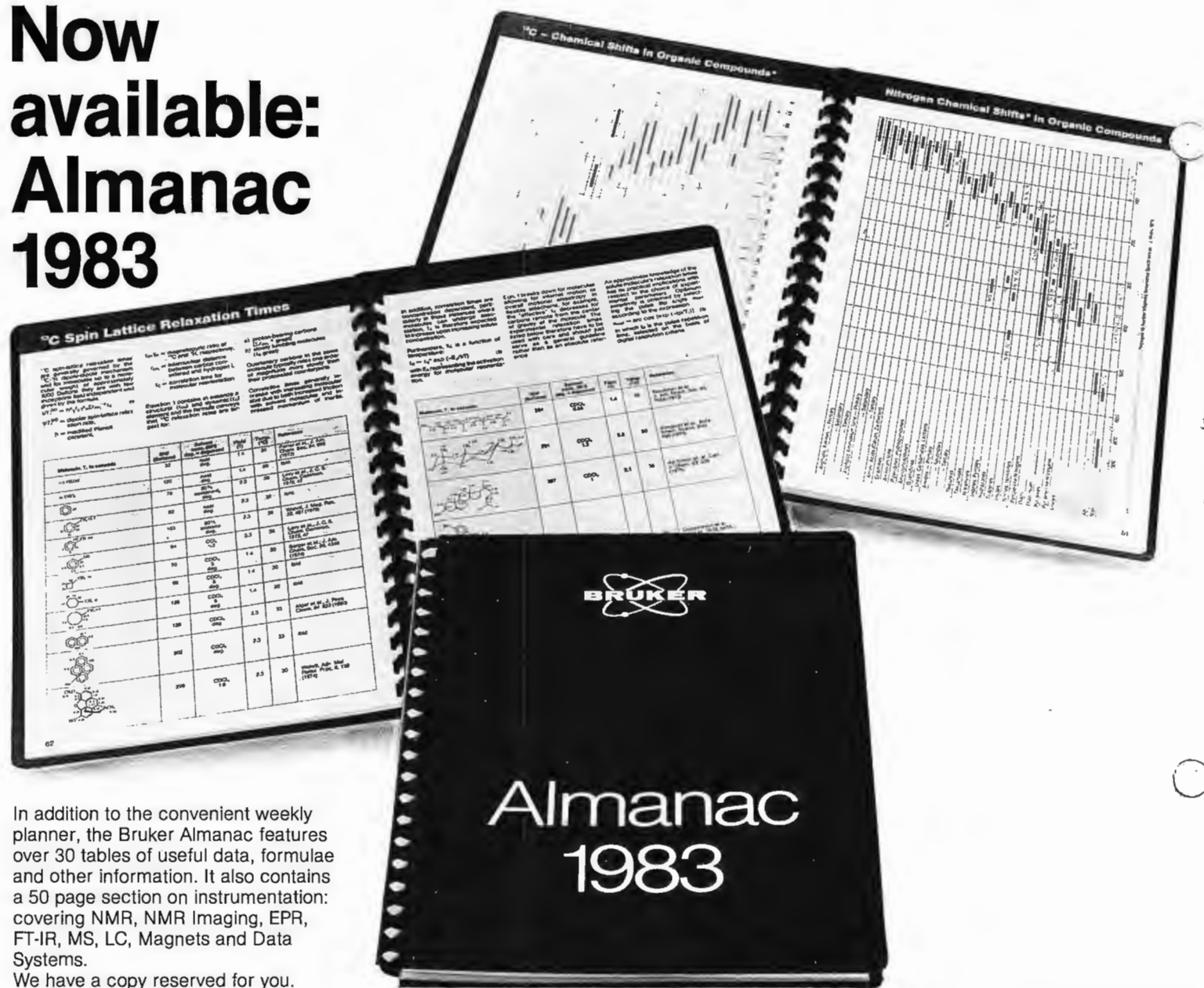
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Department of Chemistry

Evanston, Illinois 60201

September 6, 1983

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

Dear Barry:

Spin Rotation in Trifluoromethyl Groups

We have measured carbon-13 and fluorine-19 spin-lattice relaxation times for the trifluoromethyl groups in $\text{CF}_3\text{CH}_2\text{OH}$ and $\text{CF}_3\text{CO}_2\text{H}$. Plots of $\log T_1$ vs. $1/T$ for both ^{13}C and ^{19}F in the CF_3 groups showed the characteristic maximum for a changeover from dipolar relaxation at low temperature to spin-rotation relaxation at high temperature. Interestingly, proton and carbon-13 relaxation times for the CH_3 group in $\text{CH}_3\text{CH}_2\text{OH}$ or $\text{CH}_3\text{CO}_2\text{H}$ did not show these maxima but were almost entirely dipolar.

Normally, spin-rotation relaxation is associated with small molecules (overall rotation) or small groups such as CH_3 that rotate rapidly (internal rotation). Here we have the heavier but not the lighter group (CF_3 vs. CH_3) exhibiting spin-rotation relaxation. The observations are similar for both the acids and the alcohols.

If anybody has an explanation of or knows of precedent for these observations, I would like to hear. The measurements were made in connection with a study of complexation by these solvents, so the observed spin-rotation relaxation did not help us toward these ends.

Sincerely,


Joseph B. Lambert

Sharon Vlasak Simpson



University of Cincinnati

Cincinnati, Ohio 45221
(513) 475-2263

DEPARTMENT OF CHEMISTRY

September 14, 1983

Principal Values of the Methanol ^{13}C Chemical Shift Tensor

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

Dear Barry:

In our work involving surface chemistry, we typically observe powder patterns for molecules physisorbed on silica gel. Upon first examining methanol, we noticed a discrepancy between the reported chemical shift tensor principal values and our own results. We find the tensor to be somewhat asymmetric, in contrast to the original report.¹

The natural abundance ^{13}C spectrum of neat methanol was obtained by cross polarization. Our pulse program includes two-level proton irradiation (about 42 KHz match, 50-60 KHz decouple), a carbon spin echo to allow acquisition well after the receiver ring down and thereby obtain good powder pattern fidelity, and a proton flipback pulse to conserve proton magnetization.² 350 transients were acquired for the spectrum shown (Figure 1); the mix time was 5 msec, recycle delay 10 sec, temperature 140 K. Spectra obtained over the range of 77K to nearly the melting temperature never displayed evidence of axial symmetry.

Table 1. Methanol ^{13}C Shift Principal Values in ppm

	σ_{11}	σ_{22}	σ_{33}	$\bar{\sigma}$
reference 1	73	73	10	52
our results	72 \pm 2	63 \pm 1	7 \pm 1	47 \pm 2
liquid				49

Expressing the traceless tensor components σ_x , σ_y , σ_z in terms of anisotropy and asymmetry parameter ($\delta = \sigma_z$, $\eta = (\sigma_y - \sigma_x)/\sigma_z$ with $|\sigma_z| > |\sigma_x| > |\sigma_y|$) we find $\delta = -42$ ppm and $\eta = 0.21$. This anisotropy is identical to that reported by Pines, Gibby and Waugh.¹ Perusing the tensor component values tabulated in Mehring,³ we note that the anisotropy is in the usual upfield direction for methyl groups (upfield shifts are tabulated in Mehring as positive). The asymmetry parameter falls in the low end of the typical range for methyl groups.

Holly B. Ragle

Holly B. Ragle

Sincerely,

Jerry Ackerman

Jerry Ackerman

1. A. Pines, M. G. Gibby and J. S. Waugh, Chem. Phys. Lett. 15, 373 (1972). The shifts in Table 1 have been adjusted to TMS reference using $\delta_{\text{TMS}}(\text{C}_6\text{H}_6) = 128$ ppm.
2. J. Tegenfeldt and U. Haeberlen, J. Magn. Reson. 36, 453 (1979).
3. M. Mehring, "High Resolution NMR Spectroscopy in Solids," Springer-Verlag, Berlin, 1976.

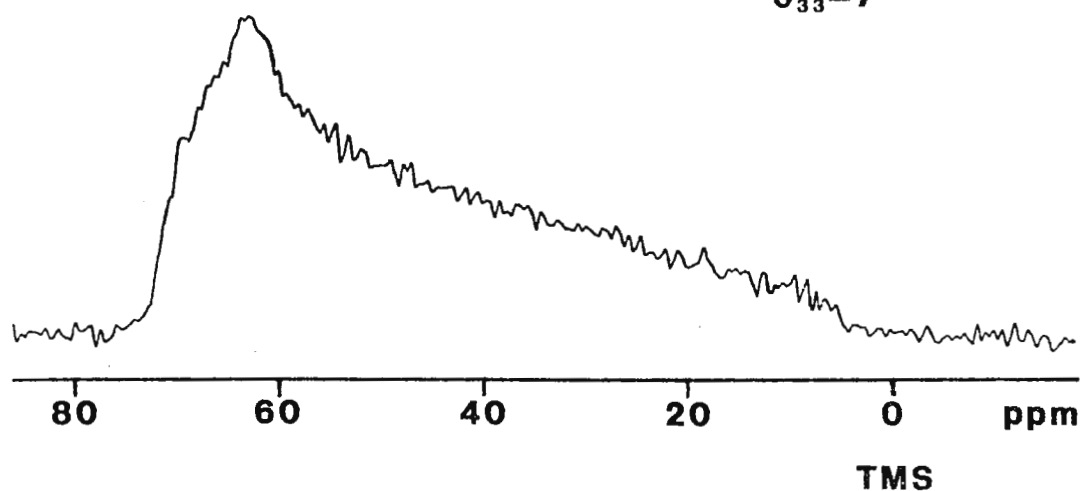
METHANOL

$\bar{\sigma} = 47$ ppm

$\sigma_{11} = 72$

$\sigma_{22} = 63$

$\sigma_{33} = 7$





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(317) 923-1321

September 14, 1983

Professor B. L. Shapiro
Department of Chemistry
Texas A & M University
College Station, TX 77843Deuterium Substitution Decreases Linewidth of Immobilized,
Protonated Carbon-13 Nuclei in High-Molecular Weight Proteins

Dear Professor Shapiro:

Fructose 1,6-bisphosphate is the glycolytic enzyme which catalyzes the reversible cleavage of fructose 1,6-bisphosphate to dihydroxyacetone phosphate and D-glyceraldehyde 3-phosphate. The mammalian variant forms an imine between Lys-227 of the enzyme and the carbonyl group of dihydroxyacetone phosphate which is reducible with sodium borohydride (1). Since all of the steps in the aldolase mechanism involve carbon, carbon-13 spectroscopy should be a useful tool to elucidate the mechanism. However, a limit of about 100,000 daltons on the size of a protein in which ^{13}C NMR signals of immobilized, protonated carbons would still be narrow enough to observe has been suggested (2). We have corroborated this estimate in studies of the interaction of rabbit muscle aldolase (M.W. = 160,000) with 90% ^{13}C -enriched substrates, in which signals of protonated carbons are extremely broad, if observable at all. This extreme broadening is attributable to dipolar interactions between the labeled carbon and a directly bound proton and can be decreased by replacing the directly bound proton with deuterium. Comparison of the spectra (see figure) of the borohydride- and borodeuteride-reduced complexes of aldolase with dihydroxyacetone phosphate shows the improvement achieved in the case of aldolase.

Please credit this submission to the account of B. D. Nageswara Rao.

Sincerely,

A handwritten signature in cursive script, appearing to read 'Bruce D. Ray'.

B. D. Ray

E. T. Harper

W. K. Fife

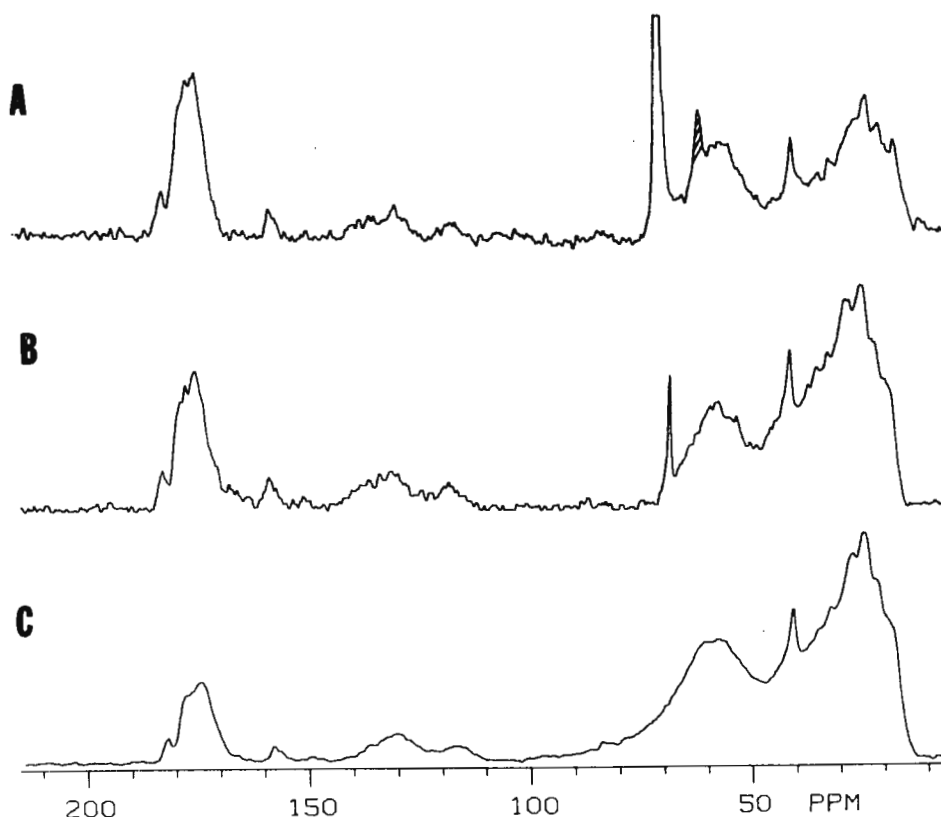
BDR:mrp

References:

1. Horecker, B. L., Rowley, P. T., Grazi, E., Cheng, T., and Tchola, O., Biochem. Z. 338, 36-51 (1963).
2. O'Leary, M. H., and Payne, J. R., J. Biol. Chem. 251, 2248-2254 (1976).

Comparison of ^{13}C spectra of Derivatized Aldolases

- A: Aldolase derivatized by reduction of the aldolase-dihydroxyacetone phosphate complex with NaB^2H_4 . The shaded peak is attributed to labeled carbon.
- B: Native aldolase with internal dioxane reference at 67.4 ppm.
- C: Aldolase derivatized by reduction of the aldolase-dihydroxyacetone phosphate complex with NaBH_4 . The signal from labeled carbon is apparently sufficiently broadened that the protein envelope masks its presence.




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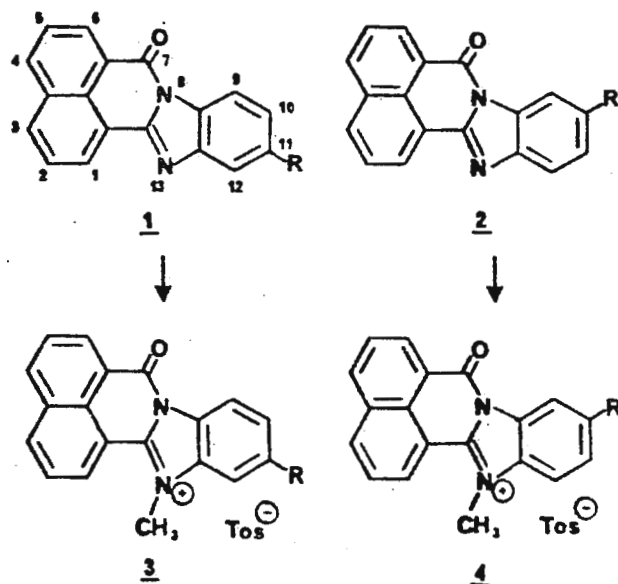
 Dr. Barry L. Shapiro
 Department of Chemistry
 Texas A and M University
 College Station, TX 77843

Structural Assignments on Aromatic Isomers Using NOE

Dear Dr. Shapiro:

With the advent of multipulsing capabilities, we tend to think of NMR as an all powerful technique. However, on occasion we find a compound(s) which defies analysis (usually structural for us at Lilly). When this happens, it is nice to be able to fall back upon the "ancient" technique of derivative making. One case in point is differentiation of isomers 1 and 2. The NMR spectra of these compounds are very similar, differing only in values of their chemical shifts.

The methyl toluene sulfonate salts 3 and 4 were prepared and, with the relatively simple NOE technique, the isomers were distinguished. A strong NOE was observed from the N-methyl to H-12. The magnitude of H-12's coupling served to give the answers (~ 2 Hz for 3 and ~ 8 Hz for 4) which we sought. Another NOE was observed from the N-methyl to H-1. This allowed the assignment of the protons for the naphthalene ring system.



Sincerely,

LILLY RESEARCH LABORATORIES

 Jon W. Paschal
 Assistant Senior Physical Chemist
 Physical Chemistry Research

JWP:JCW

- a. R = Cl
- b. R = CF₃
- c. R = Br

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CONCORDIA UNIVERSITY

DEPARTMENT OF CHEMISTRY



September 15, 1983

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

Dear Barry,

^1H CORRELATION SPECTROSCOPY ON AN HA-100

We have been interested in getting more useful life out of our old HA-100 spectrometer (vintage 1968). Recently, we have added ^1H correlation spectroscopy capability, largely using existing hardware and some existing software. There is nothing new in converting an HA-100 to correlation mode, but we thought that some of the details might be of interest.

The heart of the system is a Hewlett-Packard 1000 computer. We had previously built a dual 12-bit DAC for the HP-1000. One of the DAC's is used to drive a Wavetek 110 voltage-to-frequency converter. The other is used for spectral display. The Wavetek and associated electronics are part of an old Varian V-3530 CW ^{13}C unit, originally designed to be driven by a C-1024 CAT. The Wavetek provides the swept frequency for the analytical channel in frequency-sweep mode, via the V-4354 "lock-box", almost exactly as Varian handled the CW ^{13}C sweep. Sweep rate, and sweep ramp start and stop positions are under computer control.

The sweep ramp, timing, and acquisition software is written in Hewlett-Packard assembly language. All other software is written in Fortran IV. Correlation is carried out with a theoretical rather than with an experimental function. A Zeta 1553 plotter is used for spectral presentation.

The correlation system is intended to be used for some projects where large solvent signals would cause severe dynamic range problems on a pulse FT spectrometer.

The example shows a fast passage spectrum and the same spectrum following correlation for a compound prepared and run in H₂O solution. In D₂O solution, the formyl proton (Ha) is replaced by deuterium. There is a clear advantage in this case in making spectral assignments when an H₂O solution is used, and the correlation mode eliminates dynamic range problems.

Best regards,

Yours sincerely,

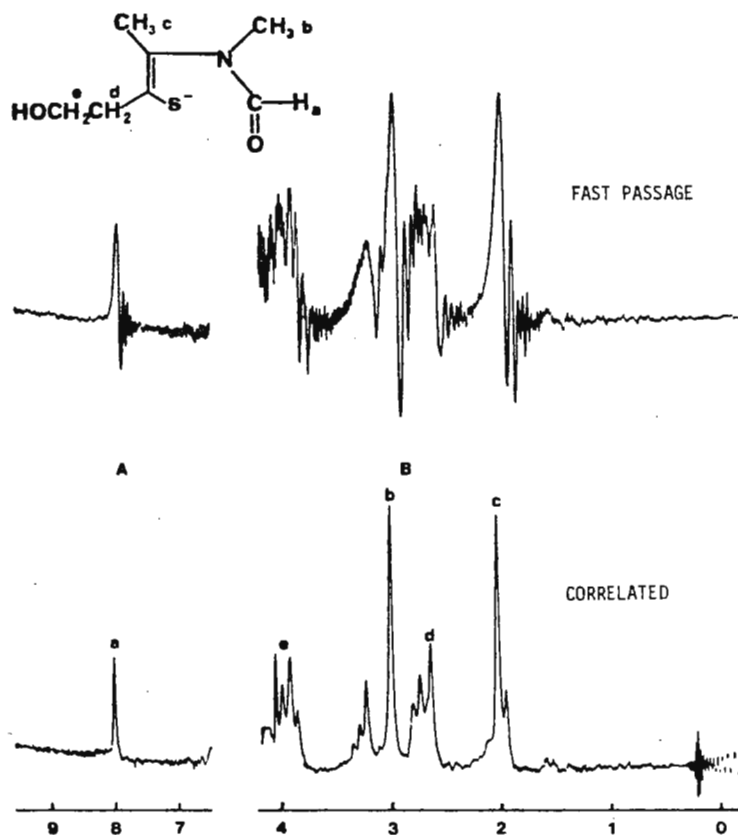
G. Nesbitt

Laurie

LDC/dg

G. Nesbitt

L.D. Colebrook





The Ohio State University

Campus Chemical
Instrument Center116 Johnston Laboratory
176 West 19th Avenue
Columbus, Ohio 43210

Phone 614 422-3446

9 September, 1983

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

Proton 500 MHz NMR in H₂O: Base-
Pair Spectra of Ribosomal 5S RNA's

Dear Barry,

Our new NIC-500 spectrometer was installed this month, and we have been using it to look at the downfield imino base-pair proton signals from several small ribosomal RNA's in 95% H₂O (see Figure). The sensitivity of the instrument on "real" samples is outstanding--the sample concentrations here are only about 0.5 millimolar, with natural line widths of ca. 30 Hz. The FID's have been resolution-enhanced by converting from Lorentzian to Gaussian line shape, and minor baseline curvature has been corrected.

Among several possible water-nulling schemes, we have been most successful so far with a Redfield 21412 soft pulse,¹ with alternating delayed acquisition.² Tune-up is facilitated by Nicolet's user-interactive knob-adjustment of the lengths of the middle and other pulses for optimal water-nulling. We use a 90° phase shift (rather than the original 180° shift) for the "1" pulses of the sequence. Quadrature detection is on, but with no phase cycling. We are typically able to reduce the water peak height by a factor of about 10,000, so that a 12-bit ADC can be used if desired.

We have begun a series of selective homonuclear proton n.O.e. experiments, in order to discover the base pair type (A:U, G:C, or G:U) and sequence (e.g., A:U followed by G:U followed by...). It is already clear that we will be able to deduce at least partial base-pair sequences from n.O.e.'s of the imino base-pair protons alone. Final sequence determinations will be based on combination experiments (e.g., proton NMR of RNA's spin-labeled at known sites).

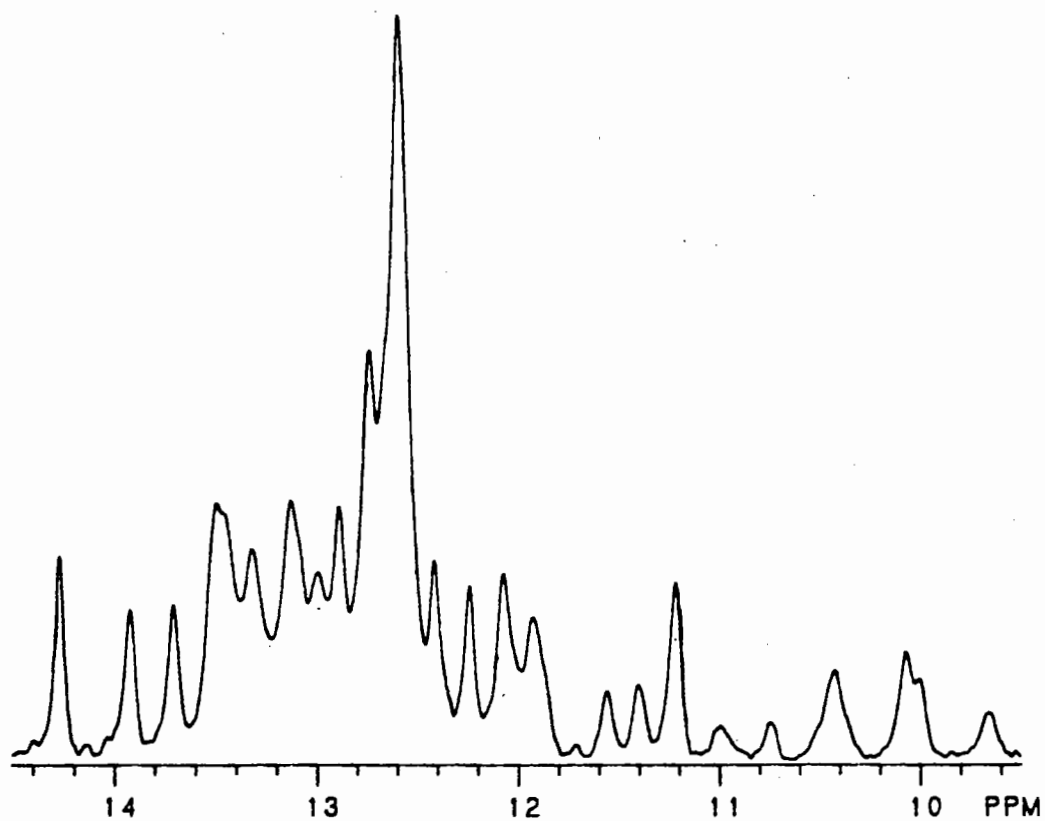
The spectra on the next two pages were obtained from 1024 transients (neurospora, *B. subtilis*), 5120 transients (yeast), and 9614 transients (wheat germ). The RNA's were isolated by Lee-Hong Chang (*B. subtilis*), Shijiang Li (wheat germ), Kai Mon Lee (yeast), and Shiow-Meei Chen (*Neurospora crassa*) in my laboratory.

Sincerely,

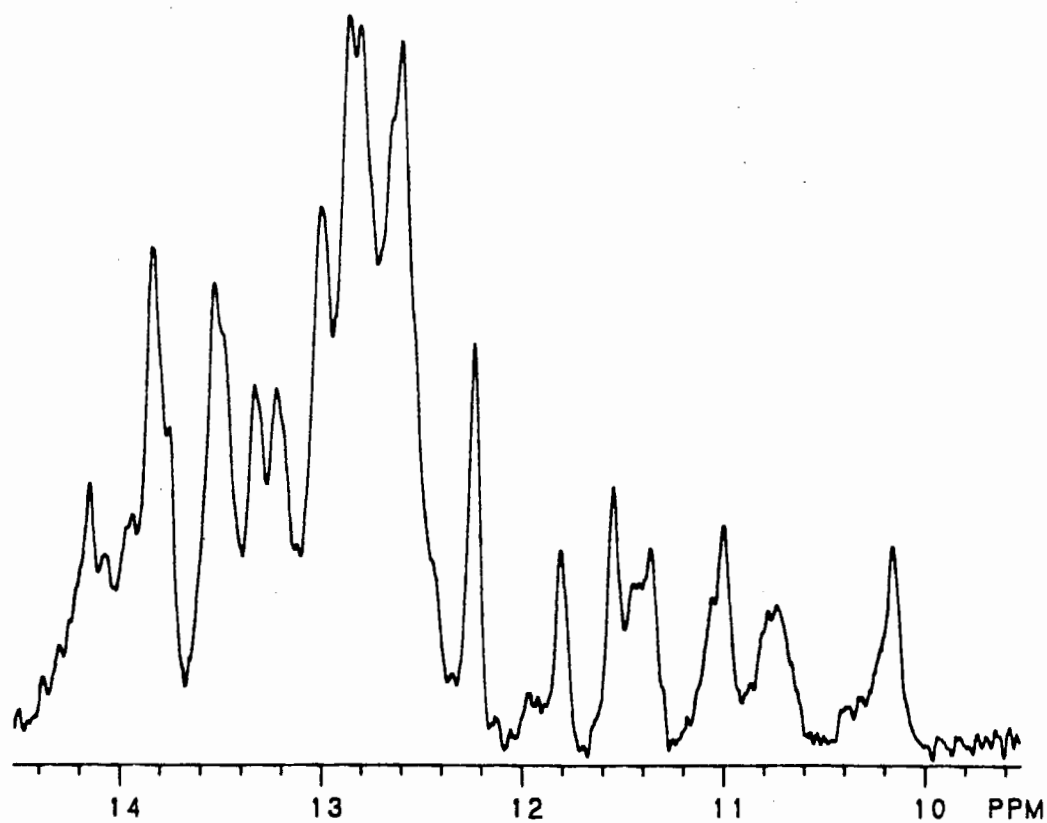
Alan G. Marshall
Professor, Chemistry & Biochemistry

1. Johnston, P. D., and Redfield, A. G. (1977) *Nucleic Acids Res.* **4**, 3599.
2. Roth, K., Kimber, B. J., and Feeney, J. (1980) *J. Magn. Reson.* **41**, 302.

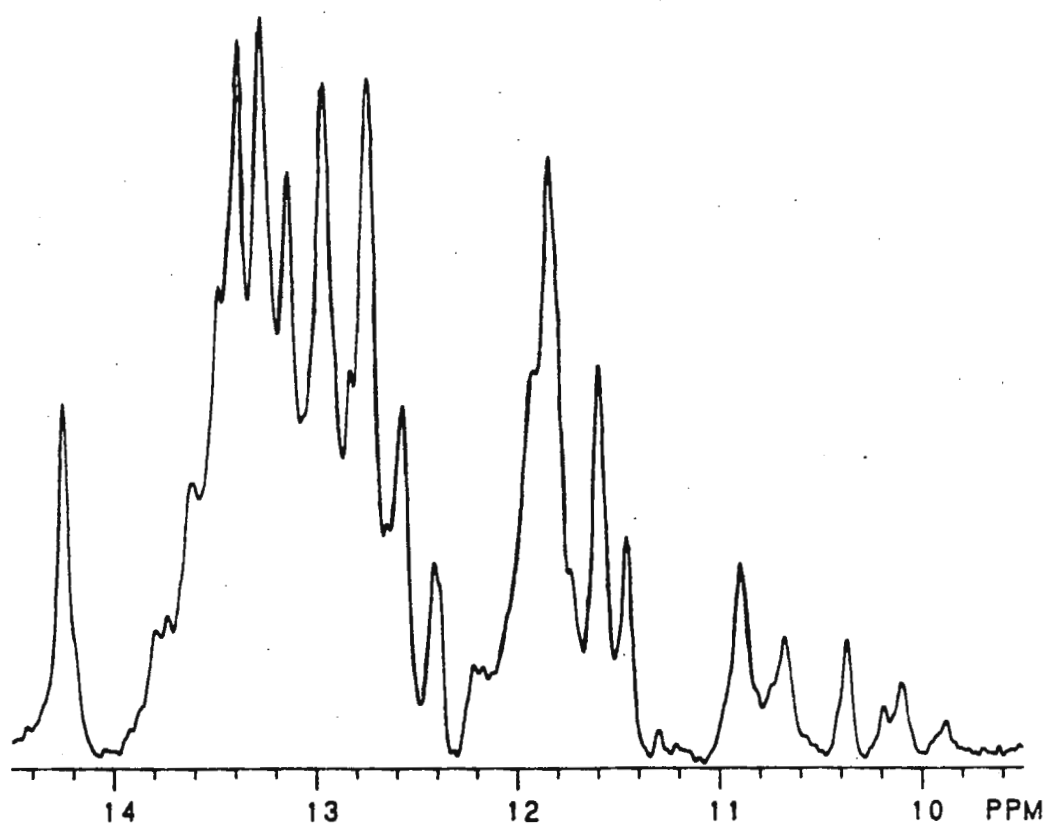
WHEAT GERM 5S RNA. NO MG. 20C. 20 MG/ML



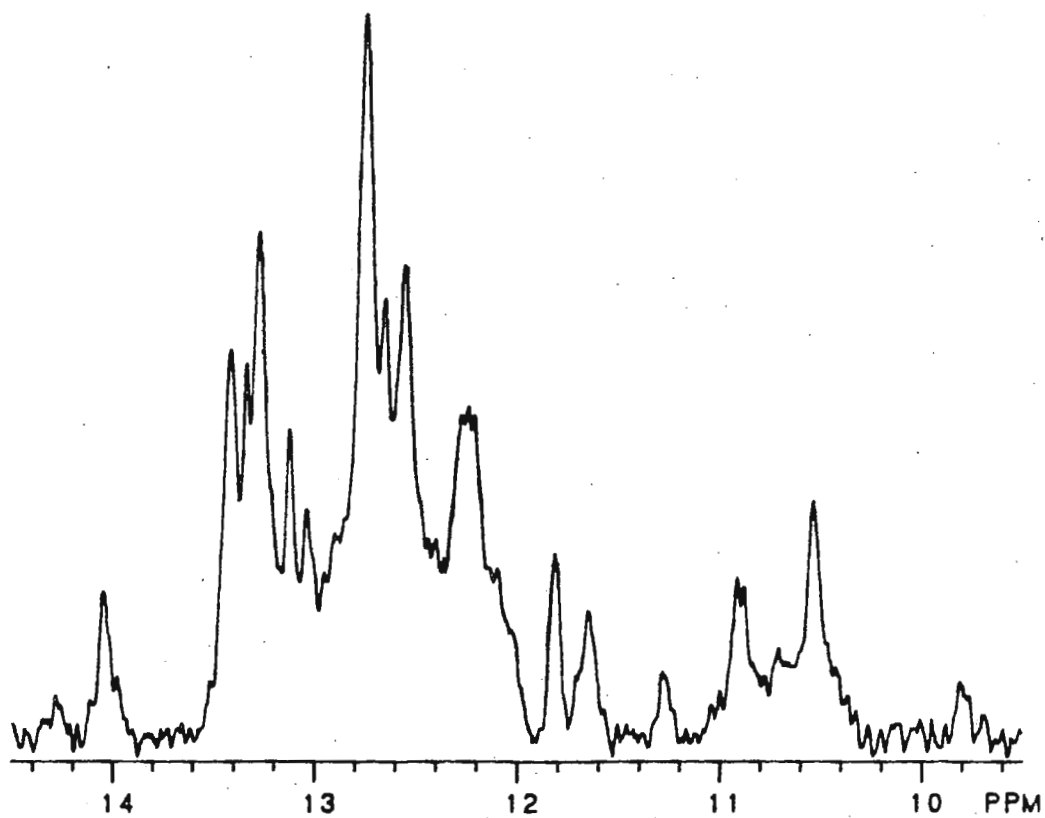
BACILLUS SUBTILIS 5S. NO MG. 30C. 20 MG/ML



YEAST 5S RNA. NO MG. 20 C. 15 MG/ML



NEUROSPORA CRASSA 5S. NO MG. 20C. 20 MG/ML



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Switching: 5-20 μ s
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: -75dB

Phase Noise: -70dBc, (0-15KHz)
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par. or GPIB
Size: 19"W, 5 1/4"H, 18"D
Price: \$5,400.00*

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nx10MHz output 20-140MHz
or any 10MHz multiple
from 20-140MHz.

PTS 200

Range: 1-200MHz
Resolution: 0.1Hz-100KHz (opt.)
Switching: 5-20 μ s
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: -70dB

Phase Noise: -70dBc, (0-15KHz)
Freq. St'd: Oven, TCXO, Ext.
Interface: BCD par. or GPIB
Size: 19"W, 5 1/4"H, 18"D
Price: \$6,150.00*

Other Options:
Progr. Attenuator, 0-90dB
(or 0-99dB with GPIB)
nx10MHz output 20-140MHz
or any 10MHz multiple
from 20-140MHz.

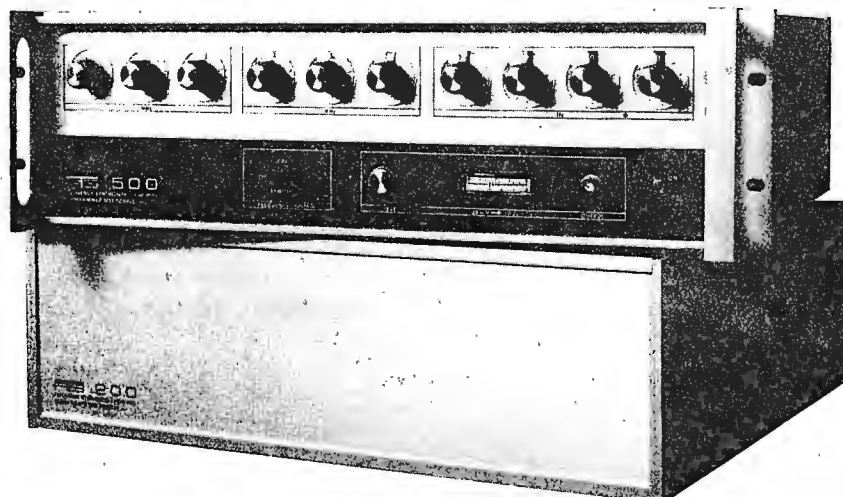
PTS 500

Range: 1-500MHz
Resolution: 0.2Hz-100KHz (opt.)
Switching: 5-20 μ s
Output: +3 to +13dBm: 50 ohm
Spurious Outputs: -70dB

Phase Noise: -63dBc, (0-15KHz)
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from 20-140MHz.

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N.S.W. 2006
Department of Organic Chemistry

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TELEPHONE: 692 1122

DX 1154

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

September 23, 1983

Dear Barry,

"Ideal" High-temperature ^{13}C NMR thermometer

The ideal thermometric compound for NMR should (i) be a single, stable and easily purifiable substance, (ii) be a liquid over the relevant temperature range, and (iii) possess an adequate temperature variation in some NMR parameter.

While substances suitable for ^1H probes have long been known, no ideal ^{13}C thermometer has so far been reported.¹ A graduate student in this Department, Mr Wayne Veigel, has now established that dichloroacetic acid meets the ideal specifications for a high temperature range ^{13}C NMR spectrometer. Using a JEOL FX60Q spectrometer and a 5 mm sample tube filled with DMSO d_6 and containing sealed capillaries of dichloroacetic acid and a melting point calibrant, he demonstrated an excellent linear relationship (Fig 1) between probe temperature and the chemical shift separation ($\Delta\nu$) of the ^{13}C signals of the two carbon atoms in CHCl_2COOH between 50 and 190°C. Expressing this in dimensionless units, gives:

$$T (^{\circ}\text{C}) = 5758.4 - 53.878 \Delta\delta (\text{ppm})$$

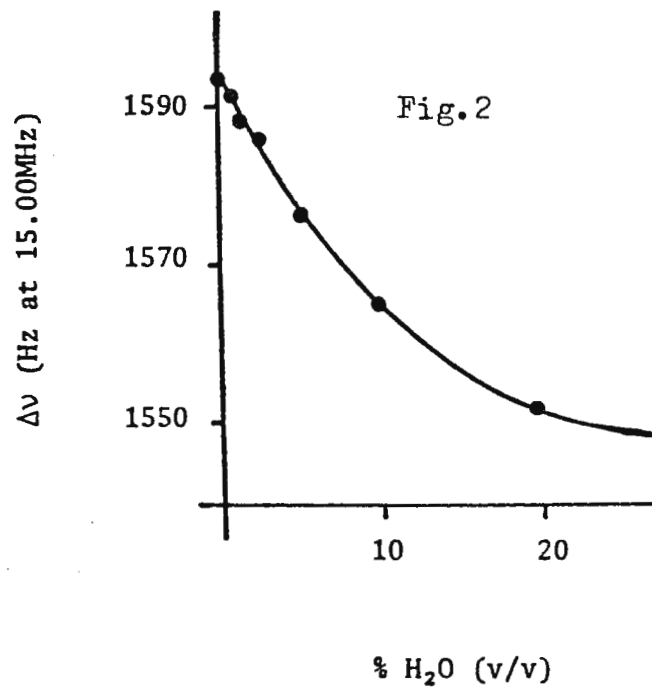
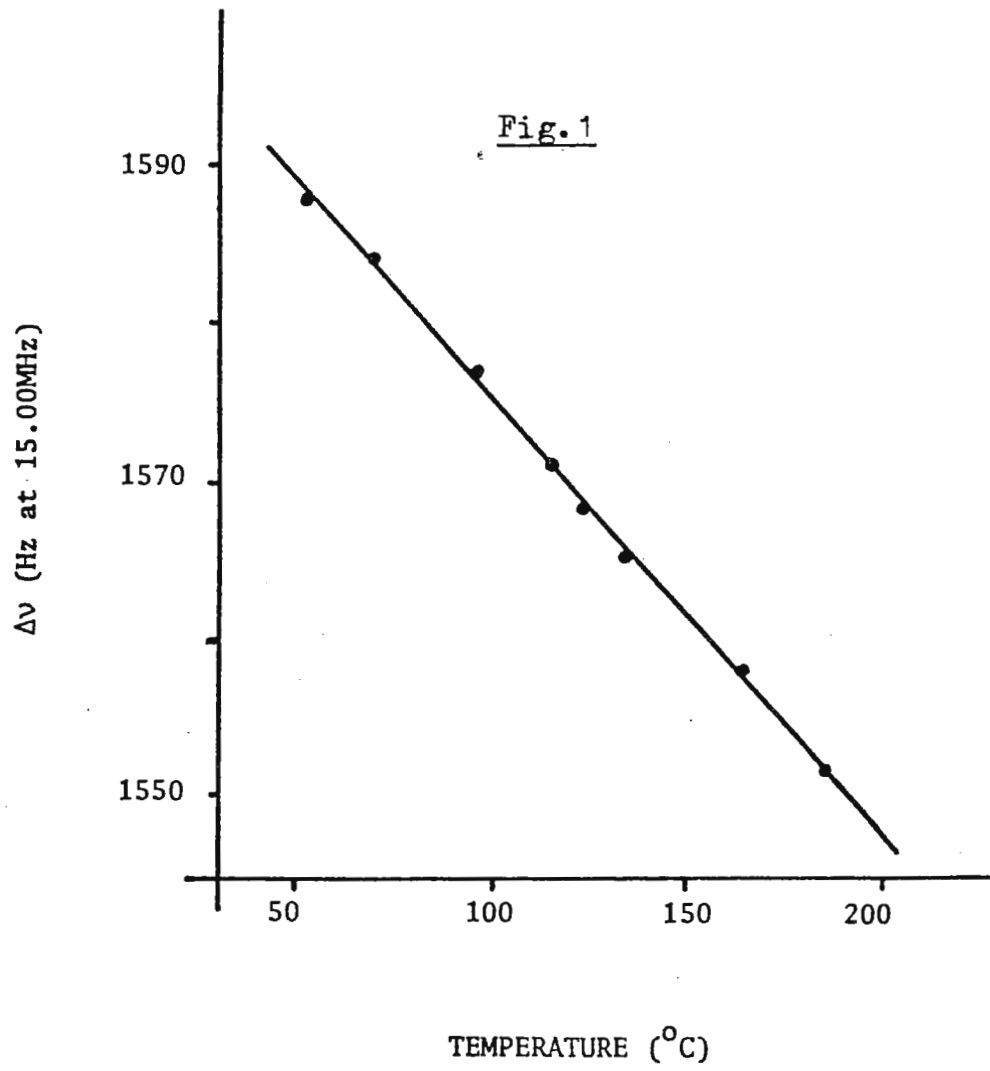
Moreover, simple distillation gives dichloroacetic acid of sufficient purity although addition of controlled amounts of water has a pronounced effect on the chemical shift separation in Hz at 35°C (Fig. 2). The chemical shift separation is even greater for trichloroacetic acid, but the absence of protonated carbon atoms makes its ^{13}C spectrum difficult to observe.

L.D. Field

Yours sincerely

S. Sternhell

-
1. F.A.L. Anet in "Topics in ^{13}C NMR Spectroscopy", G.C. Levy, ed. pp.79-83, Wiley, New York, 1979. D.W. Vidrine and P.E. Peterson, *Anal. Chem.*, **48**, 1301 (1976). S. Combrisson and T. Prange, *J. Magn. Reson.*, **19**, 108 (1975). H.J. Schneider, W. Freitag and M. Schommer, *J. Magn. Reson.*, **18**, 393 (1975). G.C. Levy, J.T. Bailey and D.A. Wright, *J. Magn. Reson.*, **37**, 353 (1980). J. Bornais and S. Browenstein, *J. Magn. Reson.*, **37**, 353 (1980). P.J. Smolenaers, M.T. Kelso and J.K. Beattie, *J. Magn. Reson.*, **52**, 118 (1983).





Oklahoma State University

Department of Chemistry / (405) 624-5920 / Stillwater, Oklahoma 74078

September 12, 1983

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

Title: Methyl Isobutyl Ketone—An Excellent Model to Illustrate DEPT Sequence

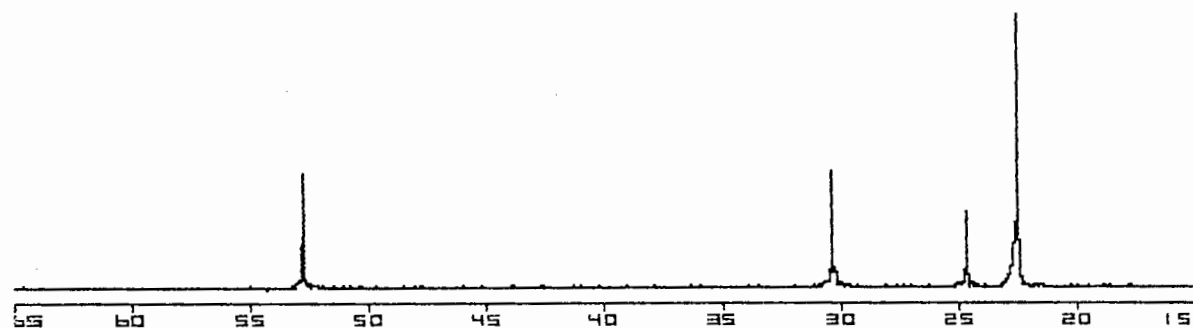
Dear Barry:

Perhaps the following contribution will make up for the last one. We have been searching for a simple system to illustrate the DEPT [Distortionless Enhancement By Polarization Transfer] with our new XL-300 NMR spectrometer. Methyl isobutyl ketone has turned out to be very fine and one can see in the enclosed spectra the easy designation for the methyl, methylene, and methine carbons. Clearly this is of value to the organic chemists as ours are discovering. This approach is the basis for the complete automation in the task of assigning multiplicities to all lines in a broadband, decoupled ^{13}C NMR spectrum. The method has been documented in the literature [D. M. Doddrell, D. T. Pegg, and M. R. Bendall, *J. Mag. Reson.* 1982, 48, 323]. The DEPT method has the advantage over INEPT [G. A. Morris, *J. Am. Chem. Soc.* 1980, 102, 428] in that the $J_{\text{C-H}}$ value does not have to be so precise for the former.

Sincerely yours,

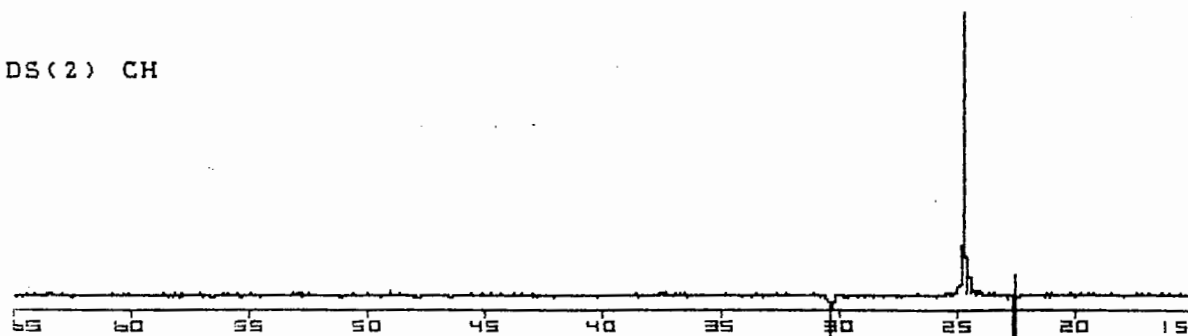
K. Darrell Berlin
Regents Professor

DEPT ON METHYL ISOBUTYL KETONE

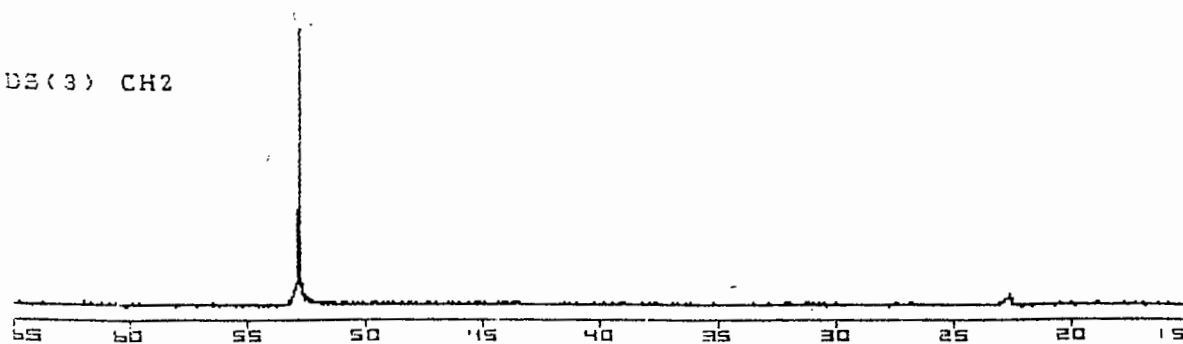


DS(1) ALL PROTONATED CARBONS

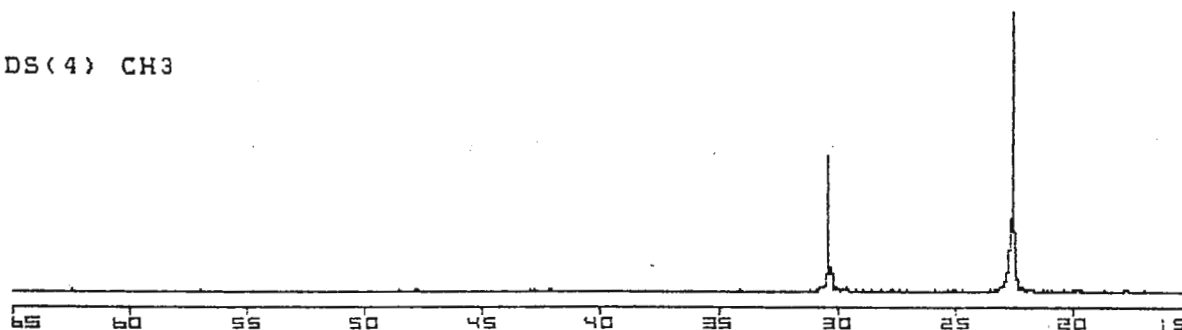
DS(2) CH



DS(3) CH2



DS(4) CH3



D E P T - DISTORTIONLESS ENHANCEMENT BY POLARISATION TRANSFER

NT = NUMBER OF TRANSIENTS, SHOULD BE MULTIPLE OF 16
 PW = X-NUCLEUS (^{13}C) 90 DEGREE PULSE WIDTH
 PP = 1-H 90 DEGREE PULSE WIDTH
 J = AVERAGE X-H COUPLING CONSTANT
 MULT = MULTIPLIER CONSTANT (THETA PULSE IS $\text{MULT} \times \text{PP}$)
 D2 = X-SATURATION DELAY (SET HS=YY AND $\text{D2}=0.05$)

A D E P T AUTOMATIC DEPT ^{13}C SPECTRUM EDITING (PRELIMINARY)

TYPE "DDEPT" THEN FOLLOW INSTRUCTIONS
 THEN DS(1) FOR ALL PROTONATED CARBONS, DS(2) FOR CH,
 DS(3) FOR CH2 AND DS(4) FOR CH3
 DSS(ALL) FOR COMPLETE ARRAY

ACQUISITION
 TN 13.500
 SW 16000.0
 AT 0.200
 NP 6400
 BS 16
 SS 2
 PW 18.0
 D1 2.000
 D2 $5.00\text{E}-2$
 TO -4000
 NT 32
 CT 0
 J 135.0
 SFRQ 75.429

DECOUPLING
 DN 1.500
 DO 0
 DM NNY
 DMM CCE
 DMF 200
 DHP Y
 DLP 60
 PP 40.0


FLAGS
 IL Y
 IN N
 DP N
 HS YY

PROCESSING
 SE 0.318
 LB 1.000
 RE NOT USED
 CD NOT USED
 CCD NOT USED
 AF NOT USED
 FN NOT USED
 MATH I

SPECIAL
 TEMP NOT USED
 VTC 25.0
 PAD 1.000
 D3 $-3.84\text{E}-3$
 MULT ARRAY1

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16



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Department of Chemistry • College of Liberal Arts
575 Buehler Hall • Phone 615 974-3141

September 9, 1983

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

Faculty Positions Available

Dear Professor Shapiro:

We are presently seeking candidates to fill two tenure-track positions in our department. The positions will be at the Assistant Professor level, and candidates with specialization in one of the following areas will be acceptable: experimental nuclear magnetic resonance, organometallic chemistry with emphasis on homogeneous catalysis, catalysis, or chemical instrumentation.

The University of Tennessee, Knoxville offers excellent opportunities for an individual to establish a strong research program. In addition to the usual physical facilities of a large department committed to research, the proximity of the Oak Ridge National Laboratory with its personnel and equipment offers the possibility of cooperative endeavors.

We will appreciate it greatly if you would bring this opportunity to the attention of qualified candidates. Candidates should send their resumes, transcripts, three letters of recommendation, reprints, and descriptions of their research interests to our chairman, Dr. Gleb Mamantov, Buehler Hall 572, University of Tennessee, Knoxville, TN 37996-1600.

The University of Tennessee, Knoxville is an EEO/Title IX/Section 504 Employer.

Sincerely yours,

Fred M. Schell
Associate Professor of Chemistry

RMP/gem

Enclosure



September 23, 1983

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

Title: CP/MAS Time Resolved Spectral Separation on the S-100

Dear Barry:

Time resolved techniques have been used in CP/MAS NMR on composite or multi-phase solid samples (see ref. 1). In this method different proton T_1 's from the different domains of a composite material are used to selectively excite resonances from one of the components while nulling the resonances of the other component. Typically this technique is used to suppress unwanted signals from the rotor. A more interesting (and easier to implement) application of this technique is the separation and reconstitution of the spectra of the individual components of the composite material.

Figure 1 shows an example of this spectral separation technique for a two component, high technology, polymer composite used by most of us in our daily lives. Spectrum A is an ordinary carbon-13 CP/MAS spectrum of the composite material. Spectrum B was obtained by using the proton T_1 CP/MAS experiment outlined in reference 1. The delay, T , was chosen to give a spectrum in which the two components are time resolved via their different proton T_1 's. By simple addition and subtraction of the two spectra A and B, individual component spectra are generated. (A+B and A-B.)

The use of the proton T_1 CP/MAS experiment does not require a very accurate determination of the proton relaxation rates of either of the two components and therefore is less critical to set up than the component suppression mode covered in reference 1. Of course, this technique requires large differences in the proton relaxation rates for the two components.

The "high technology" composite polymer used in this example is an old Colorado credit card of mine. The difference spectrum is of the filler material of the card while the sum spectrum is of the glossy coating material. Spectra were obtained on my new demo S-100 here in Fremont, California.

Best regards,

A handwritten signature in cursive script that reads 'Tom'.

Thomas A. Early
Solids NMR Product Manager

1. N. Zumbulyadis, J. Mag. Reson., 53, 486(1983).

Nicolet Magnetics Corporation

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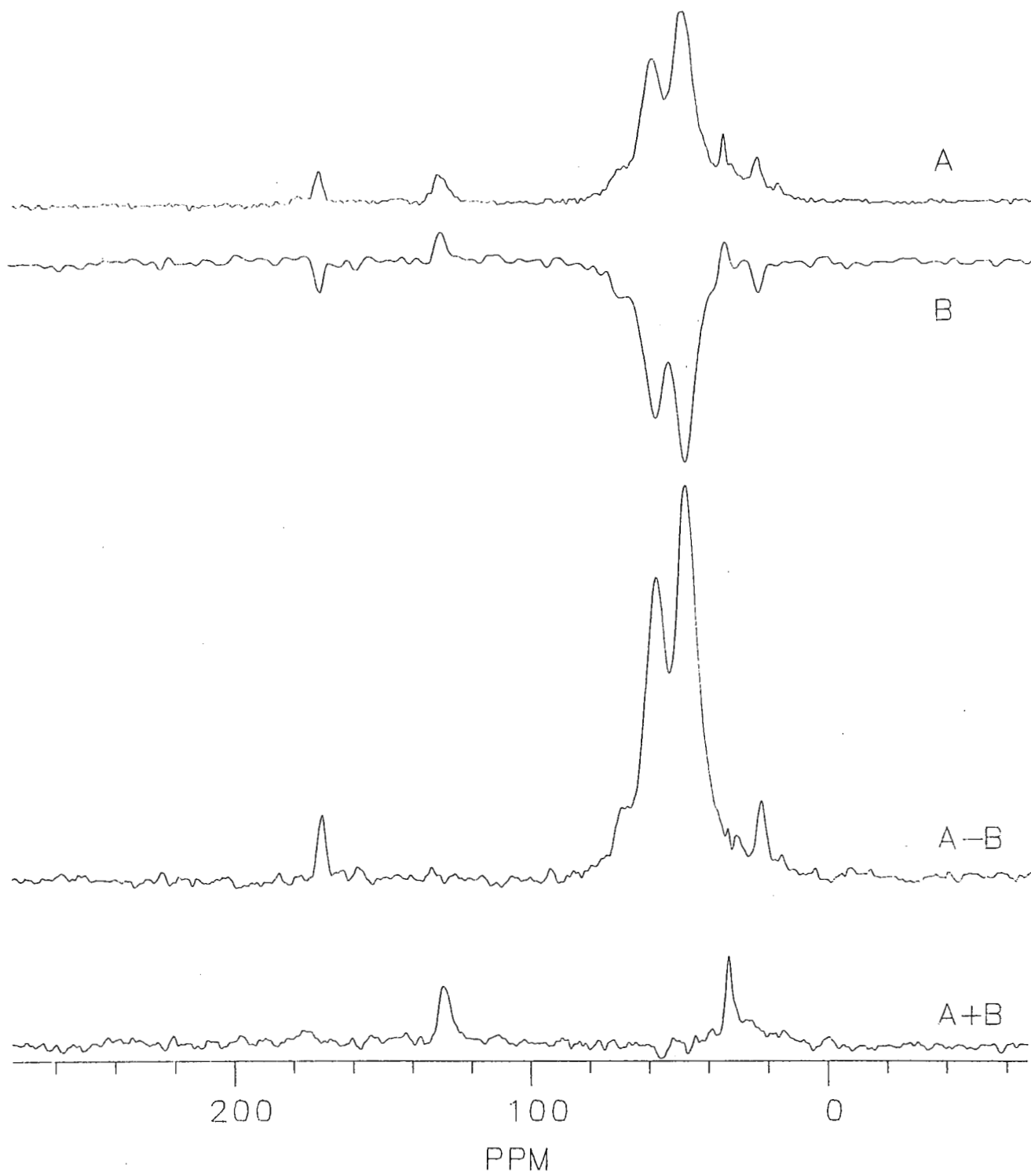


Figure 1

**Weyerhaeuser Company**Tacoma, Washington 98477
(206) 924-2345

September 27, 1983

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

Dear Professor Shapiro:

Re: Slow Movers in Phenolic Resins


For the past several years, Dr. Moon Kim and I have investigated phenol-formaldehyde (PF) resin chemistry, using in particular quantitative NOE-suppressed ^{13}C NMR spectra to measure amounts of substitution on phenol and of the various linkages and reactive methylol groups. Additionally, such spectra yield estimates of mole ratio (F/P) and a measurement of resin advancement. A somewhat upsetting fact is that measuring F/P by directly integrating the carbons of formaldehyde origin and the phenol carbons produces a low result compared with known mole ratios.

Recently we acetylated a phenolic resin and fractionated it by molecular weight. Acetylation makes these fractions extremely soluble in nice NMR solvents like CDCl_3 . Proton and ^{13}C spectra were acquired on each fraction and produced the structural compositions listed in the accompanying table, from which it is seen that the ^{13}C intensities of the $\phi\text{CH}_2\phi$ carbons are not detected fully, whereas the protons on these groups ($\phi\text{CH}_2\phi$) apparently are. Further, the discrepancy between the ^1H and direct ^{13}C measurements of methylene linkages increases with molecular weight. An indirect ^{13}C measurement of these linkages, which does not employ the methylene carbon intensity data, exhibits much better agreement with the ^1H results. For the methylol ($\phi\text{CH}_2\text{O}-$) carbons, the direct measurement comes up short, but not as severely as for the methylene carbons. The direct measurement of total F/P is so low that, at a mole ratio of 1.6, we miss around 0.4 mole of formaldehyde at all but the low-MW end. Fortunately, we obtain more accurate results from either the indirect ^{13}C calculation or from the ^1H data, so all is not lost.

Our present interpretation of this behavior, suitably shaded by theoretical considerations and viscosity data, is that the interunit methylene linkages have severely restricted motions, which are further restricted by crosslinking at higher MW. The resulting fast T_2^* relaxation seems to cause significant signal loss during the ring-down delay. Figuring that if some carbons act like solids we would be wise to study them as solids, CP/MAS spectra were acquired by Dr. Tom Early on two samples using a Nicolet S-100. The direct ^{13}C measurements gave values of 1.10 and 1.00 (CH_2/P), 0.50 and 0.54 ($\text{CH}_2\text{O}/\text{P}$) and 1.60 and 1.54 (F/P) for Samples 1 and 4, respectively, much improved over the direct measurements in solution. We intend to make solution measurements at elevated temperatures to recover our missing intensities.

Dr. Kim discussed the solution results at the Phenolic Resin Symposium at the recent national ACS conclave.

Respectfully,


Larry W. Amos

LWA:jde22/b8

Enclosure

COMPOSITION OF ACETYLATED PF RESIN FRACTIONS BY ^1H and ^{13}C NMR

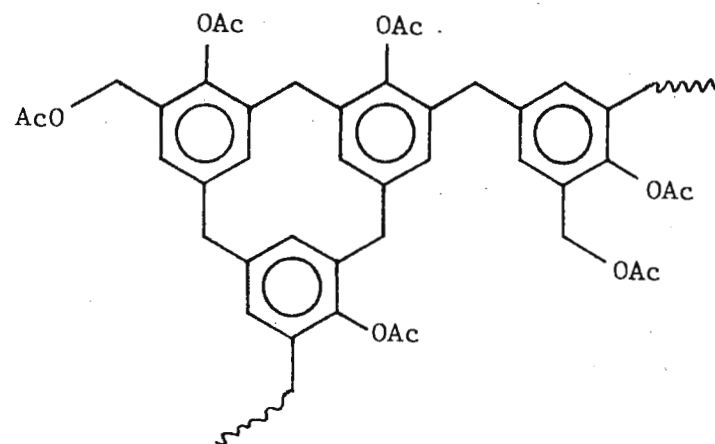
Sample	Methylenes per Phenol (CH_2/P)					Methylols per Phenol ($\text{CH}_2\text{O}/\text{P}$)					Mole Ratio (F/P)				
	Proton	C Dir.	C Ind.	% E D.	% E I.	Proton	C Dir.	C Ind.	% E D.	% E I.	Proton	C Dir.	C Ind.	% E D.	% E I.
1 (High MW)	1.20	0.77	1.01	-36	-16	0.60	0.44	0.53	-27	-12	1.80	1.21	1.54	-33	-14
2	1.04	0.72	1.01	-31	-3	0.58	0.48	0.52	-17	-10	1.62	1.20	1.53	-28	-6
3	1.05	0.80	0.93	-24	-11	0.65	0.50	0.64	-23	-2	1.70	1.30	1.57	-24	-8
4	0.80	0.72	0.94	-19	6	0.50	0.47	0.53	-6	6	1.39	1.19	1.47	-14	6
5 (Low MW)	0.60	0.57	0.65	-5	8	0.86	0.84	0.84	-2	-2	1.46	1.41	1.49	-3	2

Explanation of Headings:

C Dir. - direct ^{13}C measurement

C Ind. - indirect ^{13}C measurement

% E - error of ^{13}C measurement relative to ^1H result, percent, for direct and indirect measurements



Representative Species in Acetylated PF Resins.

EXCESS EQUIPMENT

The Department of Chemistry at Atlanta University has the following equipment available for payment of removal and shipping costs:

Varian A-60D	Console, magnet, and variable temperature probe in operating condition.
Varian A-60	Magnet.
Varian HA-100	Console, high impedance magnet (Model V-3601-2), and magnet power supply. Was operative prior to storage in 1981.
Varian HA-100	Console, regulated power supply (Model V-43001), and high impedance magnet (Model V-4014). Not operated since receipt as a donation.
Varian DP-60	High impedance magnet only (Model V-4013A).
Varian Anaspect EM500	Console only.

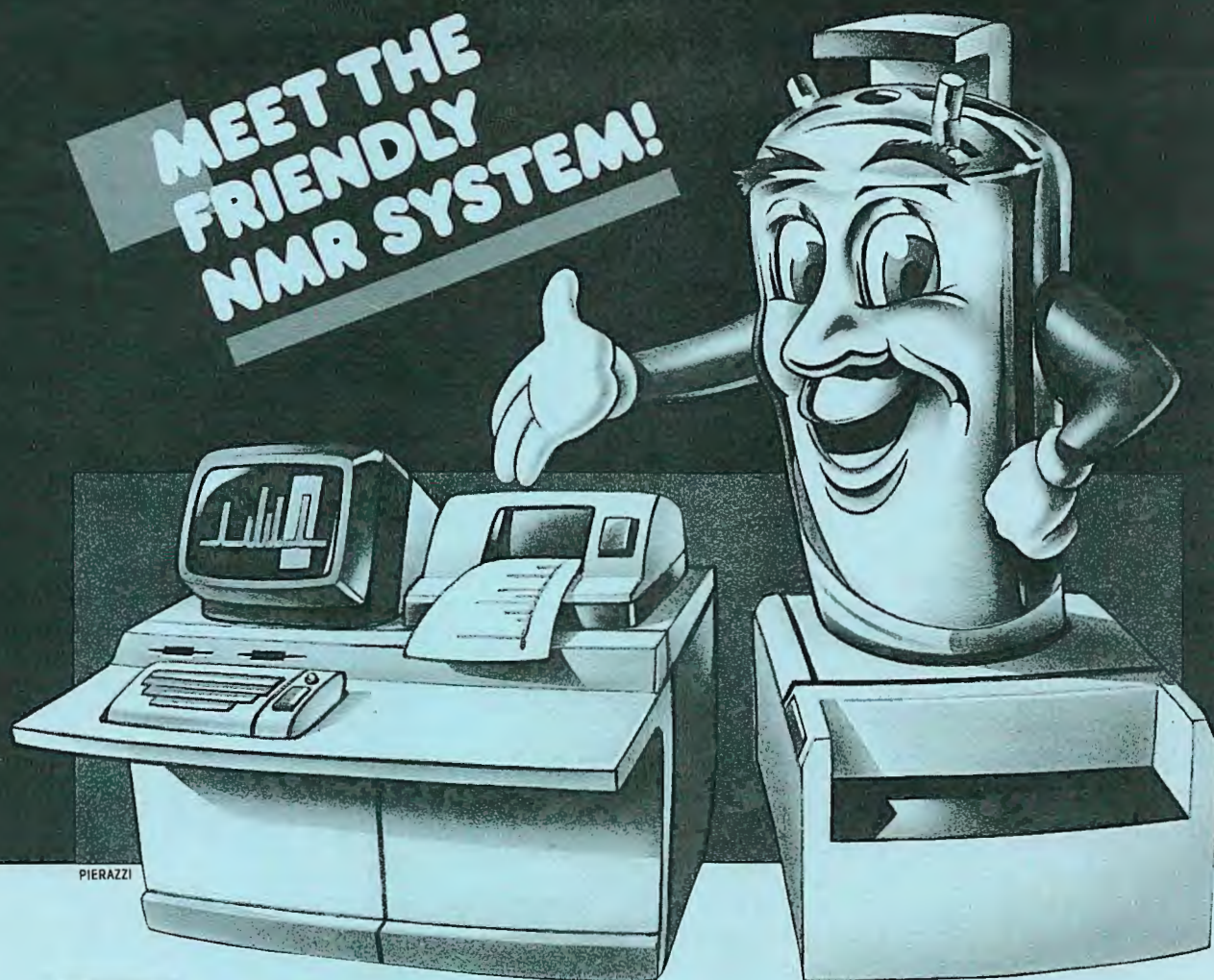
Contact: Dr. Frank E. Cummings, Chairman
Dept. of Chemistry
Atlanta University
Atlanta, GA 30314
tel 404-681-0251 ext 334

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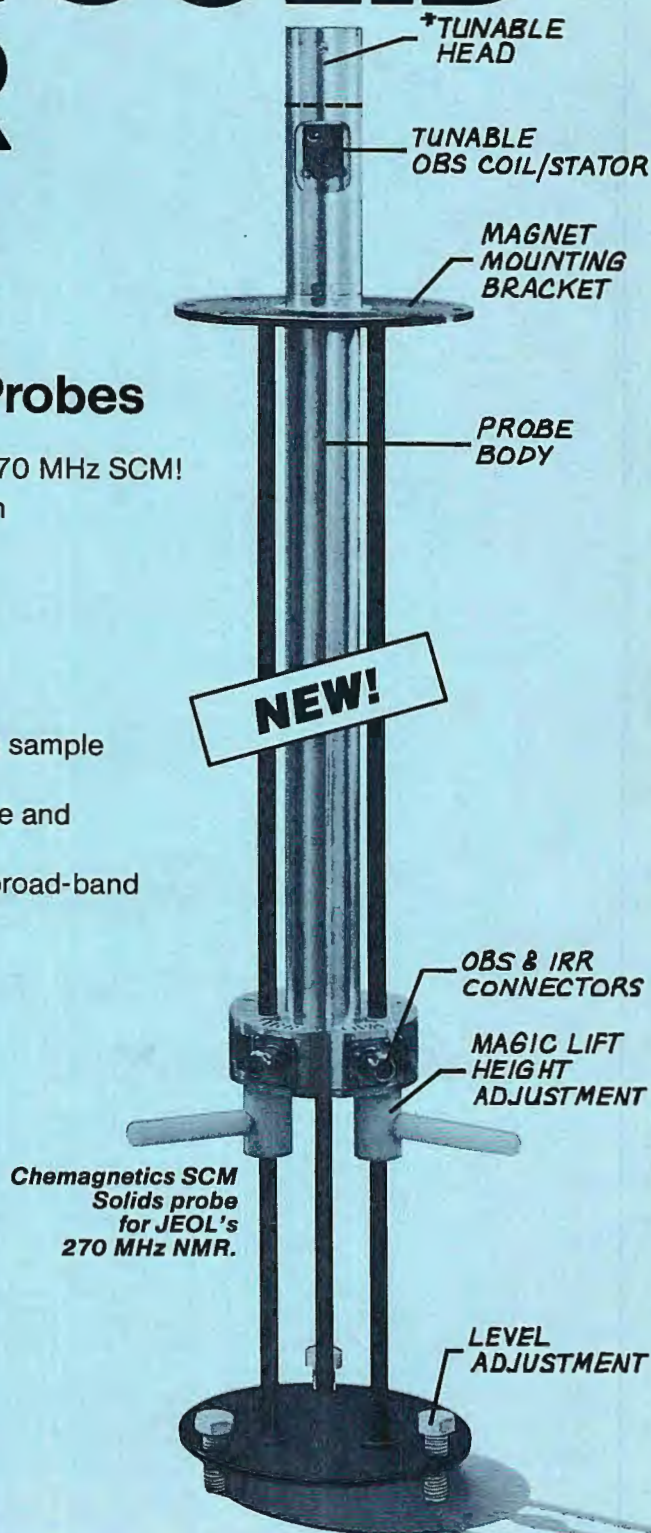
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