

TEXAS A&M UNIVERSITY

NMR

NEWSLETTER

No. 341

February 1987

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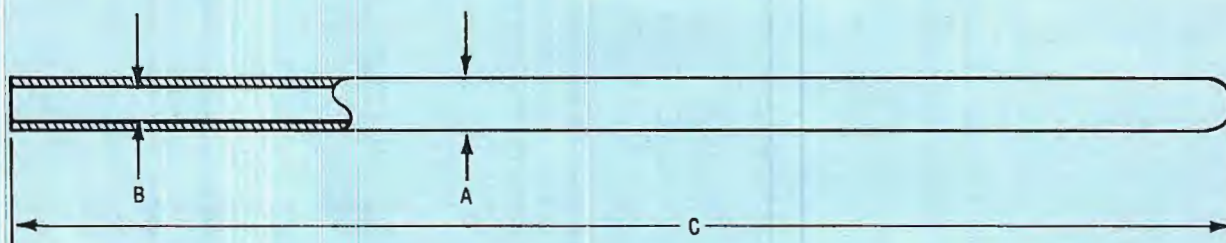
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FORTHCOMING NMR MEETINGS

Conferences on In Vivo NMR - April 3-5, 1987; Veterans Administration Medical Center, San Francisco, California; For further information, see page 15 of this Newsletter.

28th ENC (Experimental NMR Conference) - April 5-9, 1987; Asilomar, Pacific Grove, California; Chairman: Dr. Lynn W. Jelinski, (AT&T Bell Laboratories); Detailed information appears on pp. 54-57 of Newsletter #340.

8th International Meeting "NMR Spectroscopy" - July 5-10, 1987; University of Kent at Canterbury, England; For information, contact Dr. John F. Gibson, Royal Society of Chemistry, Burlington House, London W1V 0BN, England. See Newsletter #338, p. 55 for information and application.

29th Rocky Mountain Conference - August 2-6, 1987; Radisson Hotel, Denver, Colorado; Program Chair: Michael Reddy, U.S. Geological Survey, 5293 Ward Road, Arvada, Colorado 80002, (303) 236-3617; Nuclear Magnetic Resonance Symposium: James Haw, Department of Chemistry, Texas A&M University, College Station, Texas 77843, (409) 845-1966; Preliminary program and pre-registration information available from Sandy Grande, 8780 W. Quarto Circle, Littleton, Colorado 80213.

26th Eastern Analytical Symposium - September 13-18, 1987; New York Hilton Hotel, New York, New York; For information, contact J.P. Luongo, AT&T Bell Laboratories, Room 1A-352, Murray Hill, New Jersey 07974, (201) 846-1582.

FACSS XIV - October 4-9, 1987; Detroit, Michigan; For information, contact Dr. Stephen J. Swarin, Publicity Chairman, Analytical Chemistry Department, General Motors Research Labs, Warren, Michigan 48090-9055, 313-986-0806.

Fritz Haber International Workshop on Modern Techniques in Magnetic Resonance - December 13-17, 1987; Weizman Institute of Science, Rehovot, Israel; See page 7 of this Newsletter for additional information.

29th ENC (Experimental NMR Conference) - April 17-21, 1988; Rochester, New York; Chairman: Professor Stanley J. Opella, Department of Chemistry, University of Pennsylvania, Philadelphia, Pennsylvania 19104, (215) 898-6459. For information, contact Dr. Charles G. Wade, ENC Secretary, IBM Instruments, Inc., 40 West Brokaw Road, San Jose, California 95110, (408) 282-3641.

Additional listings of meetings, etc., are invited.

All Newsletter Correspondence
 Should be Addressed to:

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

DEADLINE DATES

No. 343 (April) ----- 27 March 1987

No. 344 (May) ----- 24 April 1987

UTHSCD/ Radiology
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11 December 1986

(received 30 December 1986)

Bernard Shapiro, Ph.D.
Dept. of Chemistry
Texas A&M University
College Station, Texas

Dear Professor Shapiro:

We report on the initial results obtained on our "home built" one meter bore, 1.8 Tesla in vivo spectroscopy system. The current configuration (which changes almost from week to week) is an OMT magnet with a twelve term resistive shim set, Novex broadband transceiver and preamp, an Amplifier Research model 2000 L rf power amp, and a Nicolet 1280/293B CPU and pulse programmer for data acquisition, experiment control, and data processing. All of the rf coils are made in the laboratory. The patient table is a highly modified wooden design with two separate sets of "crosshair" laser beams for accurate positioning. Despite some effort in the positioning problems, we still do not have a fully acceptable design.

Preliminary results on human volunteers are shown in Figures 1 and 2. We were quite pleased to see the splittings (as others have reported) on the three ATP resonances obtained from the calf muscle of one of us (JTV). The spectrum was obtained using a 4 cm i.d. surface coil. The brain spectrum (Fig. 2) was acquired using a 12 cm. i.d. single surface coil with the pulse length adjusted to produce the largest intensities of the PME and PDE signals. Again, our results compare well with those published by other investigators. In fact, this is our "reference" brain spectrum demonstrating the outstanding properties of this brain (EEB).

We have tried to implement a concentric surface coil design for performing rotating frame spatial localization, but have encountered significant problems when relying upon passive diode detuning methods. The first configuration selected was a "20/10" probe consisting of a 20 cm i.d. transmit coil stacked over a 10 cm i.d. receive coil. Despite our best efforts, the detuning of the transmit coil is not enough to prevent significant signal pickup from the sample and the receive coil which is then added back to the receive channel with "wonderous" phase distortion of the various resonances. We are currently awaiting the appropriate PIN diode drivers so that we can implement a more effective detuning scheme on the concentric coil probe.

With the latest from Dallas...

Sincerely yours,

Evelyn E. Babcock
Evelyn E. Babcock, Ph.D.

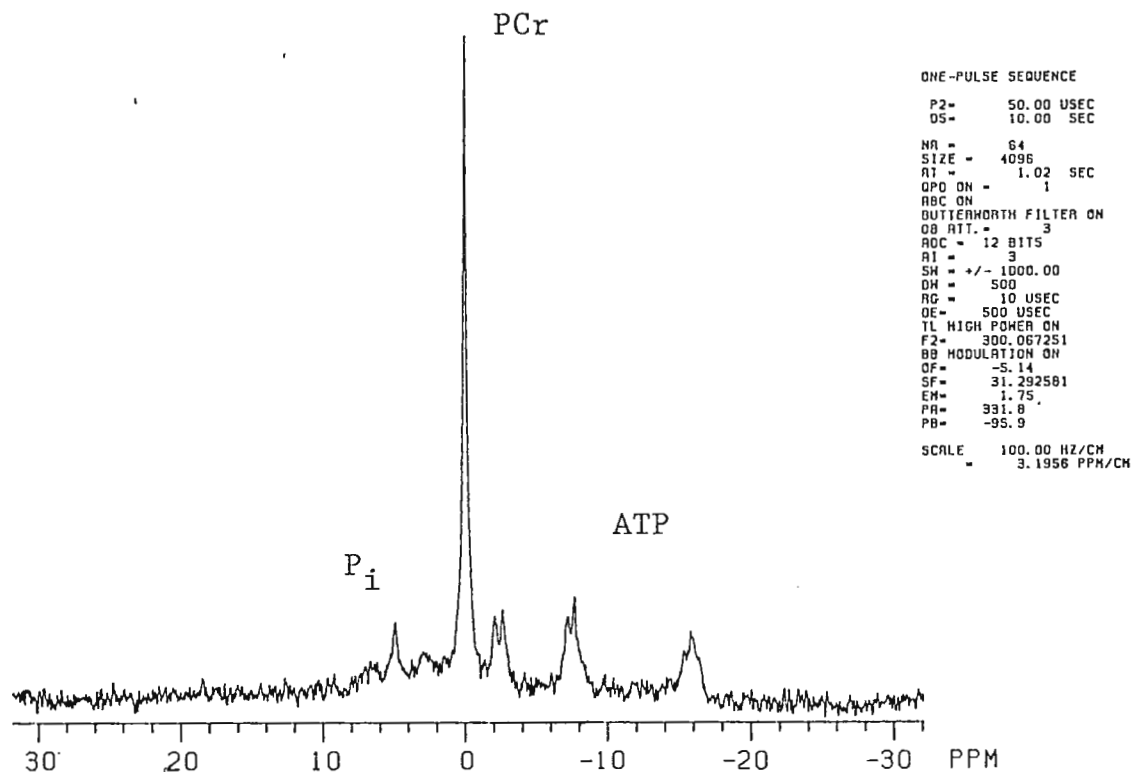
Ray
Ray L. Nunnally, Ph.D.

Tommy
J. Tommy Vaughan, BSEE, MSBME

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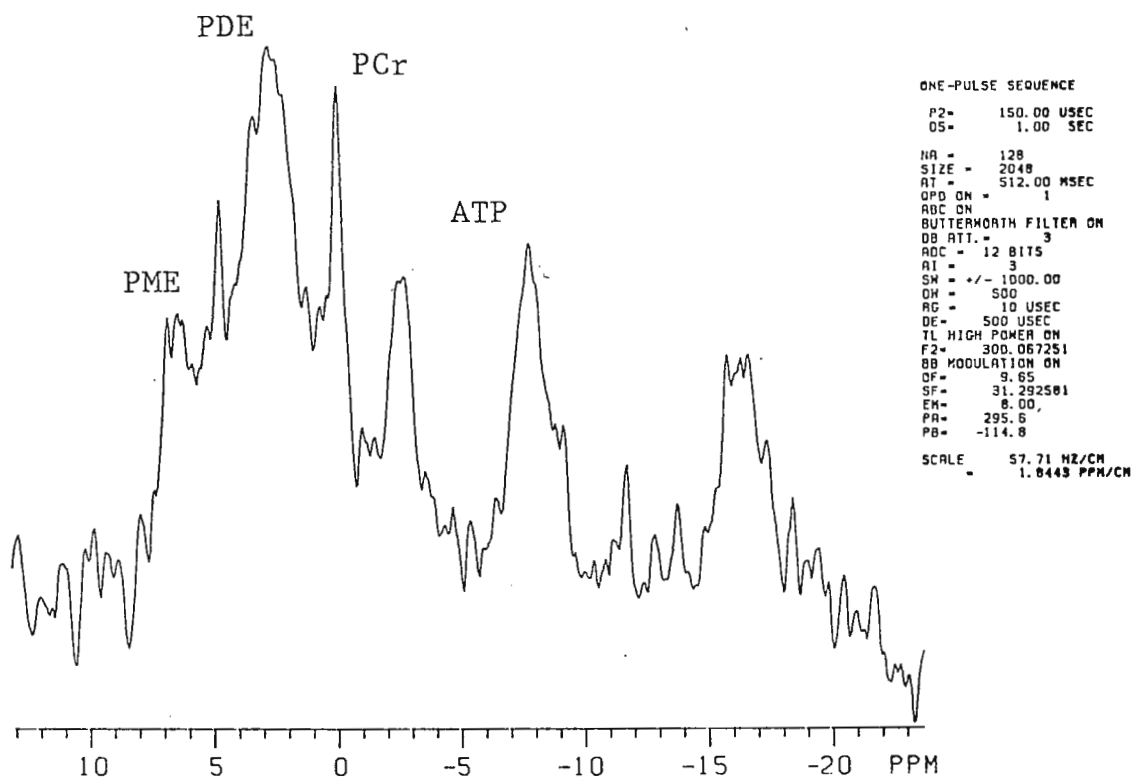
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Figure 1



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P-31 OF EEB'S BRAIN

Figure 2



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December 22, 1986 (Received 2 January 1987)

Problems with Samarium Acetate NMR Thermometer, CMD Drive Crashes

Dear Barry,

Samarium acetate tetrahydrate has been reported as a CP/MAS NMR chemical shift thermometer (Campbell, Gordon, Crosby, Haw *J. Magn. Reson.*, **69**, 191-195 (1986)). We have experienced difficulty in using this material in the temperature region of 25° to 80°C where we observe a very different functional dependence of $\delta(^{13}\text{C})$ with $1/T$ as compared to the published relationship. Our results show a chemical shift dependence on $1/T$ with a slope of $\frac{1}{3}$ to $\frac{1}{4}$ of that reported. A second, independently obtained sample from the same supplier (Morton Thiokol Alpha Products) gave similar results. Both samples were verified by X-ray diffraction to be primarily the tetrahydrate.

Our measurements were made on a Bruker CXP spectrometer operating at 50.3 MHz for carbon. The sample temperature in our Doty probe was calibrated by a procedure similar to that described by English (*J. Magn. Reson.*, **47**, 491-493 (1984)) using the proton shifts of a sealed ethylene glycol containing sphere embedded in the SmOAc powder in the MAS rotor. The ethylene glycol calculated temperatures were 30°C and 81°C for setpoints of 30°C and 100°C, respectively, on our Bruker BVT-1000 controller. This discrepancy results from the location of the thermocouple 5 cm upstream of the rotor in the drive air supply line. SmOAc shifts were determined relative to polyethylene, adamantane and hexamethylbenzene, all of which showed no temperature dependent shifts from 30°C to 80°C. For the same setpoint temperatures on our Bruker BVT-1000 controller, carbon chemical shifts obtained for SmOAc were identical with or without the ethylene glycol sphere.

We believe that SmOAc is not suitable as a chemical shift thermometer above room temperature. Differences in our results with those published may be explained by subtle sample differences, such as crystal packing or preferred crystal orientations. We have not attempted measurements at lower temperatures, where SmOAc is claimed to give a more accurate calibration (Haw, Campbell, Crosby, *Anal. Chem.*, **58**, 3172-3177 (1986)). We are interested in hearing of other's experiences with this and other possible standards.

On another note, we read with curiosity the TAMU note (336-22) by J. Loo regarding CDC-CMD disk crashes. We have followed the procedure, which is suggested in the CDC manual, of purging the system with the voice coil disconnected after each filter change only to be rewarded by our second head crash after the last maintenance. We work in a relatively clean environment and have concluded that occasional crashes somehow related to filter changes are inevitable. Our "solution" (with the endorsement of the local CDC service representative) is to reduce the opportunity for a crash by decreasing the frequency of filter changes to 1 year intervals from our present 4 month schedule. We also back up data on floppy far more frequently than before in order to lessen the pain of any such future catastrophes.

Sincerely,



Fay Crandall



Sam Kaplan

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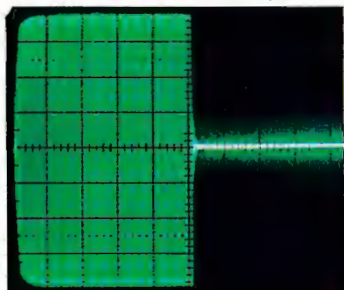
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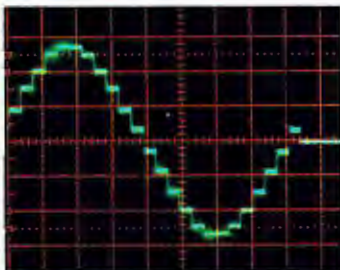
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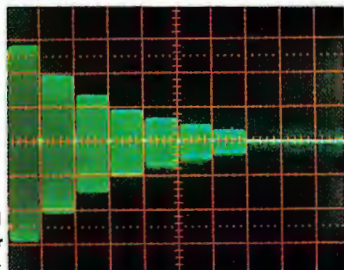
A 5 μ sec ^1H observe pulse. ▾



15° phase shift through 360°. ▸

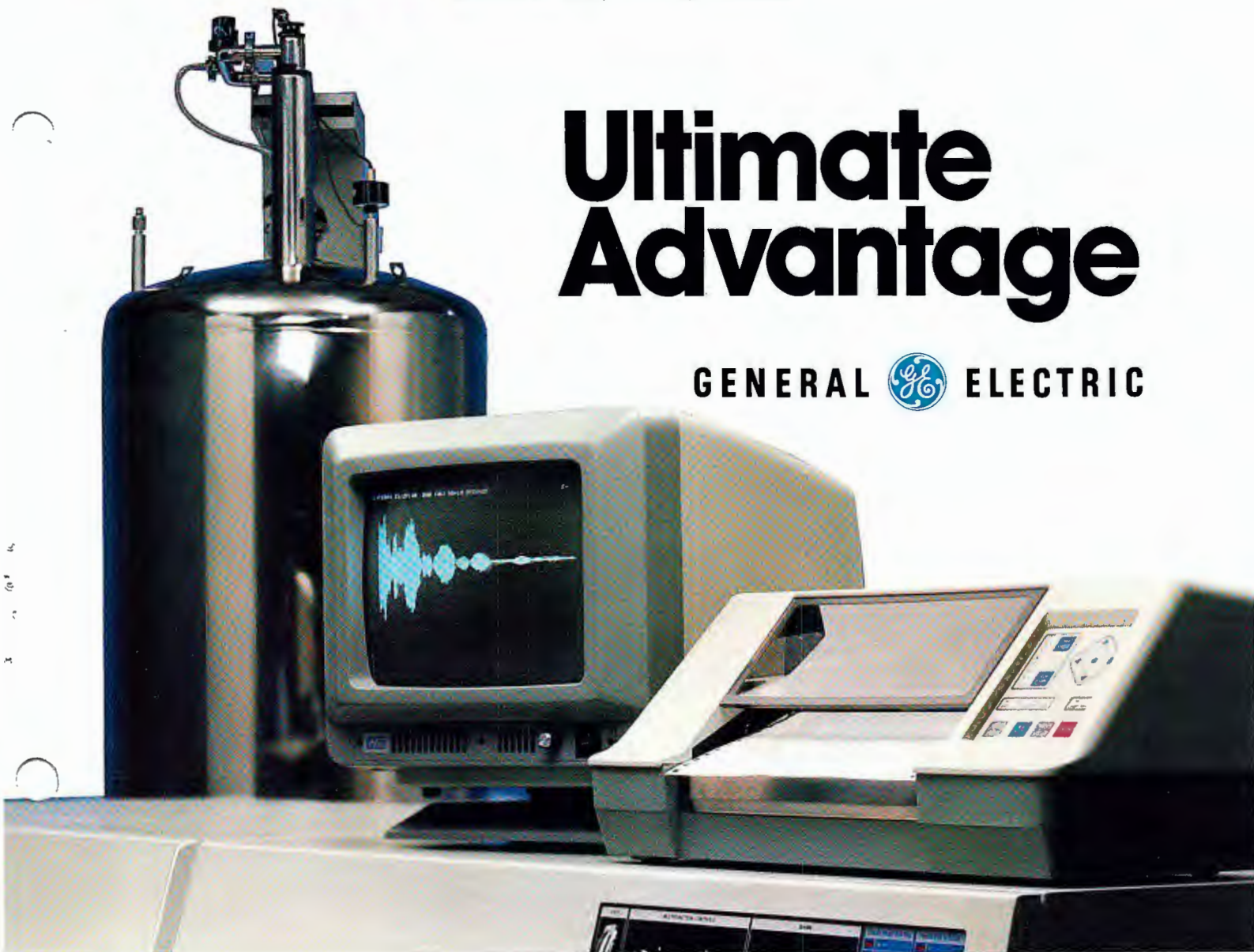


Shaped decoupler pulse. ▸



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Fritz Haber International Workshop
on Modern Techniques in Magnetic Resonance
Israel, December 13-17, 1987

During this workshop experimental magnetic resonance techniques of common interests will be discussed. The workshop will take place at the Weizmann Institute of Science in Rehovot and the Ganei Hamat Hotel in Tiberias, on the shore of the Sea of Galilee.

Among the invited lecturers will be: W. Aue (Basel), A. Bax (Bethesda), G. Bodenhausen (Lausanne), W.T. Dixon (Atlanta), G.P. Drobny (Seattle), R. Dwek (Oxford), R.G. Griffin (Cambridge), J.S. Emsley (Southampton), U. Haeblerlen (Heidelberg), J.A. den Hollander (Best), R. Kaptein (Groningen), H. Levanon (Jerusalem), M.H. Levitt (Cambridge), Z. Luz (Rehovot), M. Mehring (Stuttgart), K. Möbius (Berlin), J.R. Norris (Chicago), D. van Ormondt (Delft), A. Pines (Berkeley), B.R. Reid (Seattle), R.G. Shulman (New Haven), H.W. Spiess (Mainz), P. Styles (Oxford), W.S. Veeman (Nijmegen) and A.J. Vega (Wilmington)

For further information please write to the Organizing Committee Secretariat:
Shimon Vega, Department of Isotope Research
Fritz Haber Workshop on Modern Techniques in Magnetic Resonance
The Weizmann Institute of Science, Rehovot 76100, Israel



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RE: Employment Opportunities at GE-NMR Instruments

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Sincerely yours,

Paul J. Kanyha
Shop Operations Manager



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Telex: 83-5479

December 16, 1986

(Received 8 January 1987)

Professor Bernard L. Shapiro
Department of Chemistry
Texas A&M University
College Station, Texas 77843-3255

Dear Professor Shapiro:

Relaxation Agents in ^{29}Si NMR

The higher intrinsic NMR receptivity for ^{29}Si in comparison to ^{13}C (2.09:1) suggests that the former should be more sensitive. In practice, this advantage is offset by the longer spin-lattice relaxation times and negative NOE's that are exhibited by ^{29}Si . These problems can, of course, be minimized by the use of relaxation agents. However, while the guidelines for the use of relaxation agents for ^{13}C spectroscopy are well known, considerably less has been published for ^{29}Si . Recently, a systematic study was carried out in our laboratory to measure the concentration dependence of several different paramagnetic metal acetylacetonates (Cr, Fe, Mn and Co) on the ^{29}Si T_1 's, NOE's and linewidths in a series of silanes.

The results shown in Figure 1 on phenyltriisopropoxysilane indicate that the iron and chromium compounds performed the best, lowering the T_1 from 58 seconds to approximately 2 seconds at a concentration of 4 mmoles of metal(acac)/mole silane. We also investigated the reduction in T_1 as a function of the concentration of Fe(III) acetylacetonate for several different silane compounds. These results (Figure 2) indicate that all of the compounds tested approach a T_1 minimum when the optimal concentration of approximately 4 mmoles of relaxation agent per mole of silane is used.

NOE enhancement factors and linewidths were also measured as a function of the concentration of the relaxation agent. NOE effects were wiped out with the addition of as little as 0.05 mmoles of metal(acac) per mole silane. At the same time, the linewidth for phenyltriisopropoxysilane shows a linear increase from 1.5 Hz (without relaxation agent) to only 1.8 Hz at the optimal concentration of $\text{Fe}(\text{acac})_3$.

In conclusion, the best relaxation reagent for silane measurements is Fe(III) acetylacetonate, added in the ratio of 4 mmoles per mole of silane. Furthermore, our data show that this relaxation agent virtually eliminates NOE effects without a significant increase in linewidth. By the use of Fe(acac)₃, typical quantitative ²⁹Si NMR experiments that used to require 8 hours in our laboratory have been reduced to less than 1 hour.

Figure 1

T_1 v.s. Metal(acac) Concentration
for Several Metal(acac) Compounds and
Phenyltriisopropoxysilane

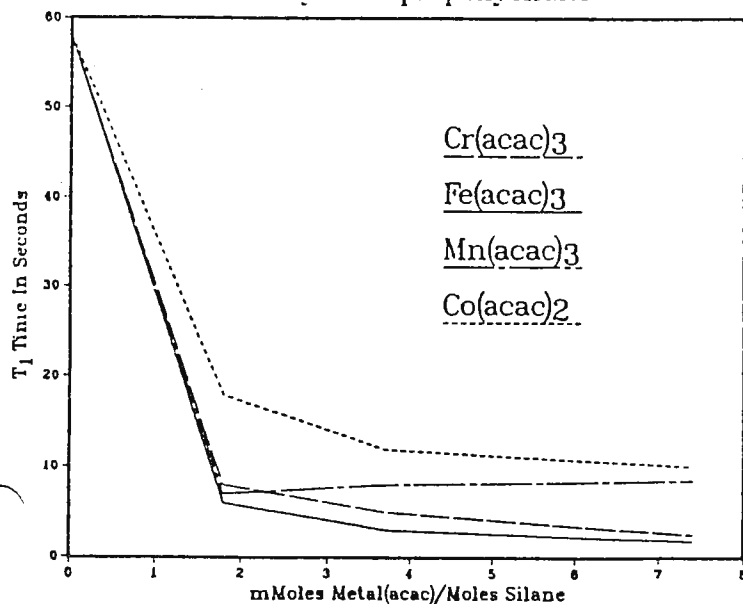
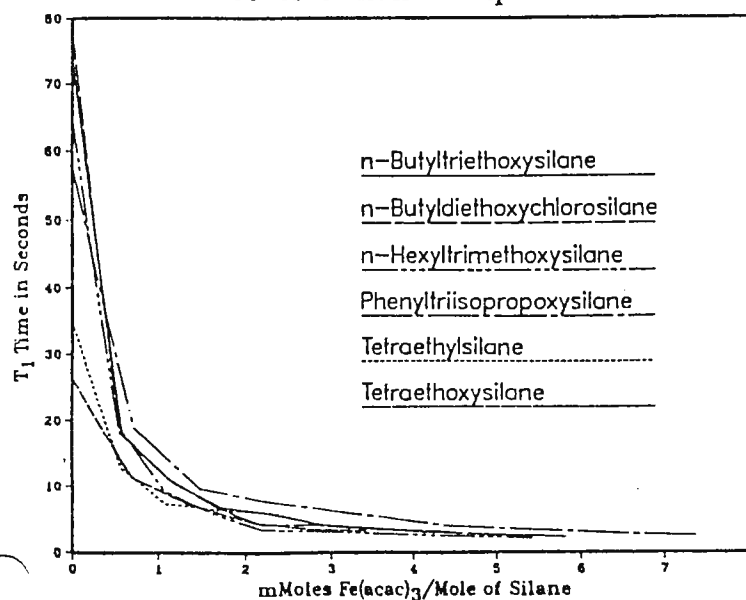


Figure 2

T_1 v.s. Fe(acac)₃ Concentration
for
Several Silane Compounds



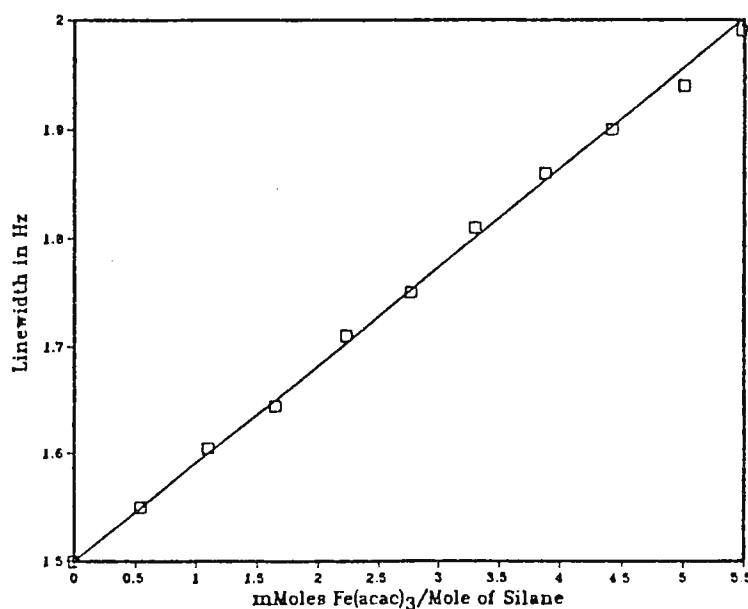
Sincerely yours,

David A. Smith

David A. Smith
Analytical Science Division

Figure 3

Linewidth v.s. Fe(acac)₃ Concentration



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The University of Melbourne

Department of Inorganic Chemistry

Parkville, Victoria 3052

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

December 20th 1986
(Received 6 January 1987)

Dear Professor Shapiro,

Studies on Five Coordinate Tin(IV) Compounds

We find from tin-119 NMR studies that both PhSnCl_3 and PhSnBr_3 form previously unreported 1:1 adducts with OPBu_3 in dichloromethane solution as shown in Fig 1a, with the chloro compound having the higher frequency. A solution containing PhSnCl_3 , PhSnBr_3 and OPBu_3 in approximately 1:1:2 proportions shows halogen exchange at RT, but at -70°C four broad resonances are observed (Fig 1b) corresponding to PhSnCl_3L , $\text{PhSnCl}_2\text{BrL}$, $\text{PhSnClBr}_2\text{L}$ and PhSnBr_3L ($\text{L} = \text{OPBu}_3$). At -90°C (Fig 1c) the resonances are much sharper with obvious phosphorus coupling, but importantly the signals due to the mixed halide compounds are each split into two resonances of unequal intensities, while the trichloro and tribromo compounds still give simple doublets. At -100°C (Fig 1d) all signals are doublets and although becoming broader due to viscosity effects, no other change occurs showing that all exchange processes have become slow on the NMR timescale.

The splitting of the mixed halide compound signals at low temperature is due to the slowing down of the Berry pseudorotation of the five coordinate complexes which allows observation of two isomers for each mixed halide compound. In fact, these spectra allow the structures of the adducts to be determined which could not be achieved from the spectra of either PhSnCl_3L or PhSnBr_3L alone.

There are three possible distributions of the ligands in the trigonal bipyramidal adducts PhSnX_3L as shown in structures (1) to (3) with one, two or three halides in the equatorial plane (the structure (2') with L and Ph interchanged is not distinguished from (2) at this stage). The spectra immediately eliminate (3) since it would not give rise to isomers in the mixed halide compounds. In order to distinguish between structures (1) and (2) let us consider the adduct $\text{PhSnCl}_2\text{BrL}$. The isomers possible with structure (1) and (2) are given in (1a,b) and (2a,b). The statistical distributions would be expected to be a:b = 1:2 in each case since there are two non-equivalent chlorides in each (b) isomer which may be interchanged. It is also known that in five coordinate tin compounds of this kind it is the ligands in the equatorial plane which largely determine the tin chemical shift. Thus (1b) and (2a) would be expected to have resonances at higher frequencies than (1a) and (2b) respectively since chloro derivatives resonate at higher frequencies than their bromo analogues. The spectrum shows that for $\text{PhSnCl}_2\text{BrL}$ the resonance at lower frequency is

the more intense which shows that the compound has structure (2). If the compound had adopted structure (1) the intensities of the signals would be expected to be reversed. This assignment is confirmed by consideration of the resonances for $\text{PhSnClBr}_2\text{L}$, in this case the resonance at higher frequency is the more intense, again as expected for structure (2).

In fact, structure (2) is the stereochemistry expected for these adducts. It is known that $\text{Ph}_2\text{SnCl}_2(\text{OPPh}_3)$ and other oxygen donor adducts of Ph_2SnCl_2 have a trigonal bipyramidal structure with the oxygen donor axial and the phenyl groups equatorial, thus it is not surprising that a similar ligand distribution is found for these new compounds. It is this analogy alone, however, which leads to structure (2) as drawn (rather than (2')) with Ph and L interchanged) since the NMR evidence cannot distinguish between these forms although it does identify the geometrical structure as (2).

Yours sincerely,

R. Colton *D. Dakternieks*

R. Colton

D. Dakternieks*

*Present address: Department of Chemical and Physical Sciences, Deakin University, Waurn Ponds, Victoria 3217, Australia.

P.S. Please credit this contribution to the account of Dr. D.P. Kelly.

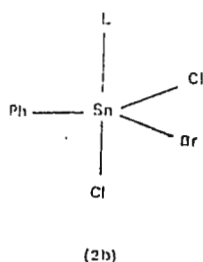
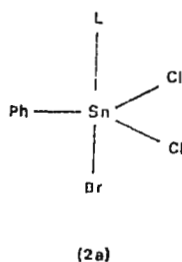
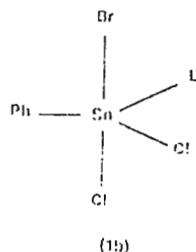
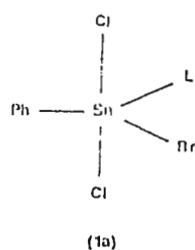
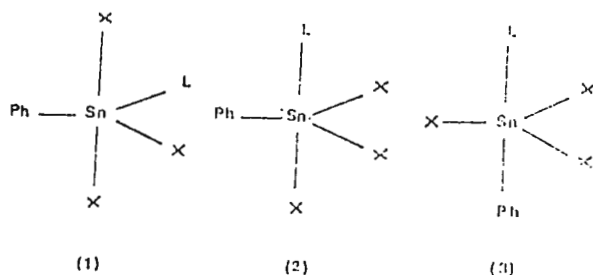
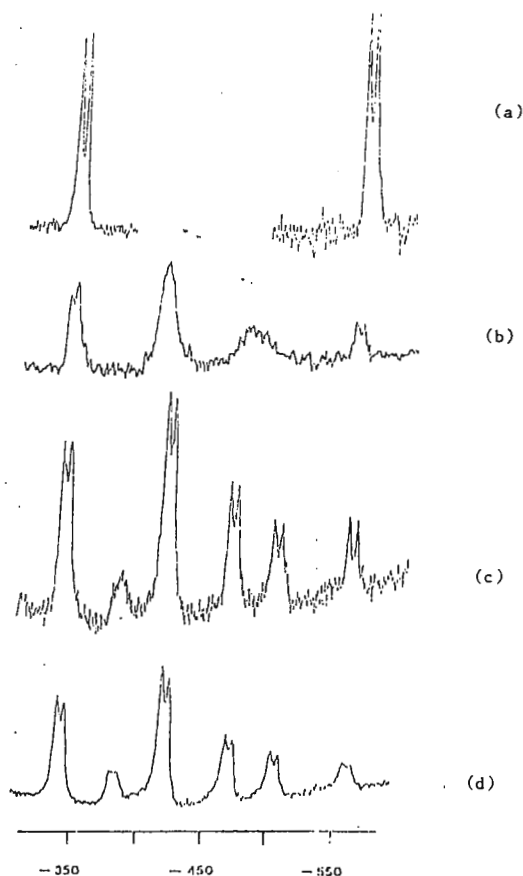


Figure 1



"Ionic Fluxes Through Red Cell Membranes"

Dear Barry,

(Received 5 January 1987)

The presence of an ionophore such as lasalocid A induces extra ionic fluxes through erythrocyte membranes. In order to determine them, nmr spectra are measured in the presence of a paramagnetic marker, Dy(PPP)_2^{7-} , placed in the outer compartment. A simple integration then suffices to determine the relative amounts of outer and inner ions of a given type ¹. The only cations transported significantly under our experimental conditions were Na^+ , K^+ , and H^+ .

The poor sensitivity of ^{39}K nmr prevented direct determination of the potassium fluxes with enough precision. We could obtain them indirectly however. Since cationic fluxes affect chemical shifts of the outer cations, we could resort to ^{23}Na nmr in order to determine not only sodium but also potassium fluxes. First, we defined calibration curves for the paramagnetic shift of the sodium nuclei as a function of Na^+/K^+ values over the whole range appropriate for transport across erythrocytes. Since this shift also depends weakly on proton concentration ² we applied a pH correction.

Variations in chloride ion concentration were derived from intensity changes of the outer ^{35}Cl resonance. The inner chloride signal was invisible under these conditions ³. The overall ionic balance thus determined experimentally shows that, following the perturbation induced by the ionophore, ionic fluxes evolve to maintain electroneutrality in both the inner and the outer compartments. A continual entry of sodium ions parallels the exit of potassium ions. The latter is initially faster. Chloride ion transport tends to compensate this difference. From 30 data points we could obtain the following time evolutions, where concentrations are given in mmol.kg^{-1} of hemoglobin, and time is expressed in minutes :

$$(\text{Na}^+) = 85 \exp(-0.156t) + 132 \exp(-0.009t) + 236$$

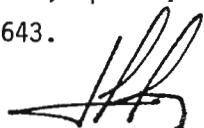
$$(\text{K}^+) = -153 \exp(-0.192t) - 22 \exp(-0.036t) + 203$$

$$(\text{Cl}^-) = -208 \exp(-0.282t) + 310 \exp(-0.028t) + 289$$

Best personal regards,

references:

- (1) Gupta, R.K., & Gupta, P. (1982), J. Magn. Reson. 47, 344.
- (2) Chu, S.C., Pike, M.M., Fossel, E.T., Smith, T.W., Balschi, J.A., & Springer, C.S. Jr. (1984) J. Magn. Reson. 56, 33.
- (3) Brauer, M., Spread, C.Y., Teithmeier, R.A.F., & Sykes, B.D. (1985) J. Biol. Chem. 260, 11643.



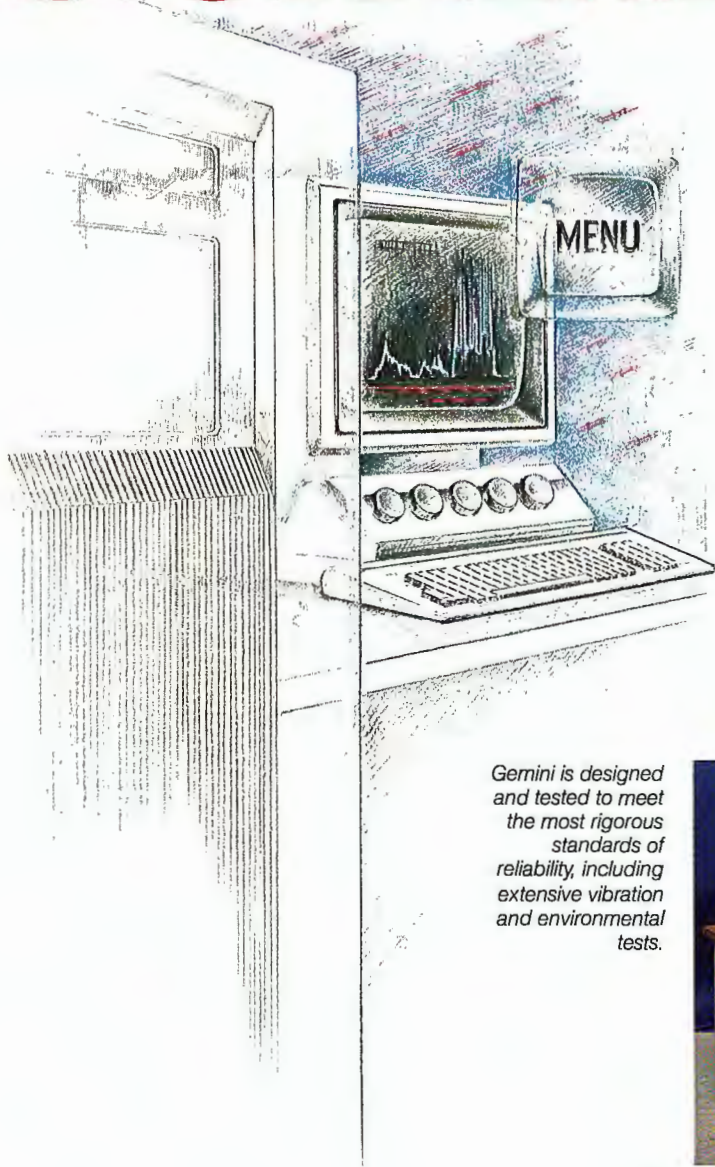
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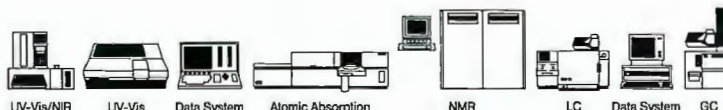
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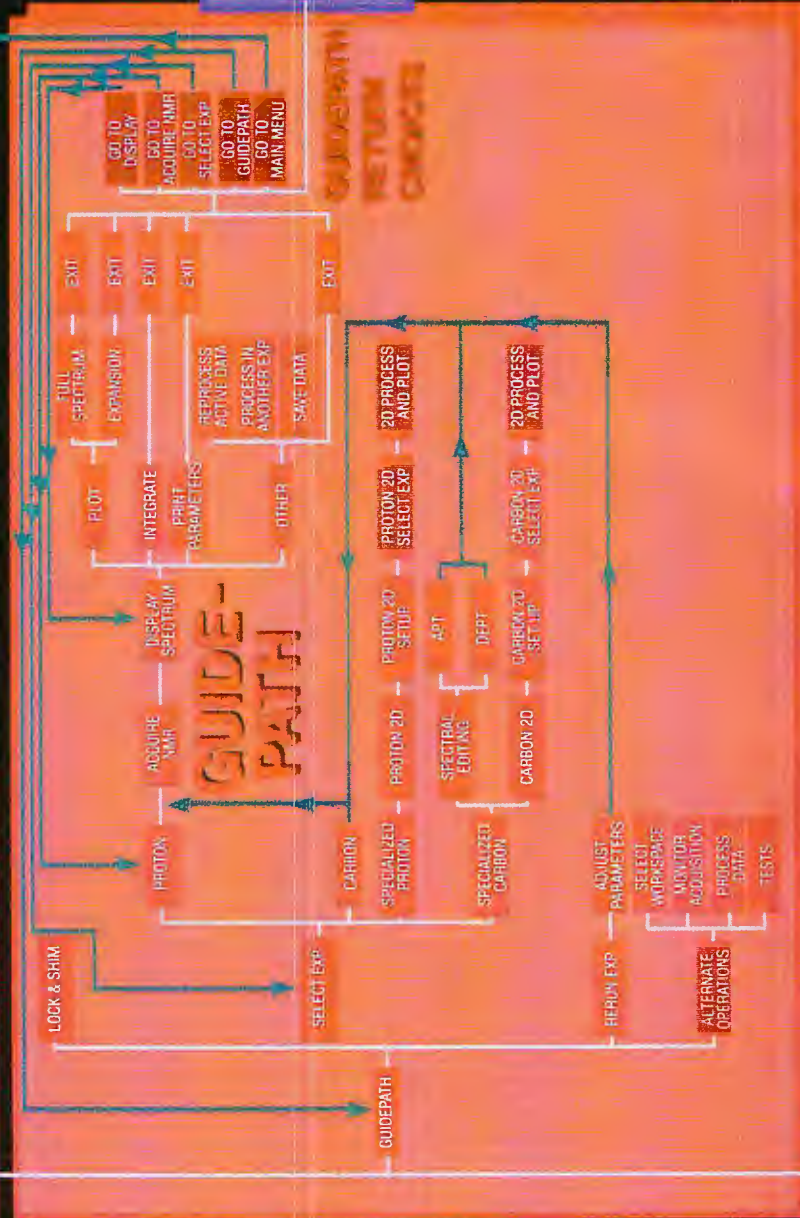
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December 9, 1986 (Received 27 January 1987) University of California Service
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San Francisco, California 94121
(415) 750-2146

Dr. Bernard L. Shapiro
Texas A & M University
Chemistry Department
College Station, TX. 77
Ottawa, Ontario K1A 0R6

Re: Conference on in vivo NMR

Dear Dr. Shapiro:

We call your attention to two conferences we will be holding in San Francisco over the weekend of April 3-5, 1987, just prior to the ENC in Asilomar, Ca.

The first will be a two-day Workshop on Medical Magnetic Resonance Spectroscopy to be held Saturday and Sunday, April 4 and 5. This workshop will be similar to that previously held by Drs. Chance and Leigh in Philadelphia. This year our emphasis will be on localization techniques for magnetic resonance spectroscopy. However, there will be ample opportunity for presentations and discussions concerning all areas of in vivo NMR. Please advise us if you wish to attend and/or present your work.

Second is a tutorial on Medical Magnetic Resonance Spectroscopy to be held Friday, April 3. This is intended for physicians, NMR spectroscopists, and other individuals (administrators, technicians, scientists) who have little expertise in in vivo NMR for animal studies or clinical investigation. The faculty for this tutorial will be composed of both local scientists and prominent visitors who will also be attending the workshop. Individuals attending the tutorial will be welcome to attend the workshop on Saturday and Sunday. Thus these individuals will have the opportunity for 3 solid days of exposure to in vivo NMR. Both meetings will be held at the Veterans Administration Medical Center, overlooking the Golden Gate in San Francisco. Meals will be provided as well as direct bus transportation to the ENC on Sunday. We have attached announcements of both these events. Please pass them on to interested individuals and post them.

A detailed announcement and registration form will follow, but we would appreciate an early indication of your interest.

Michael W. Weiner

Michael W. Weiner, M.D.
Associate Professor of Medicine
and Radiology

Gerald B. Matson

Gerald B. Matson, Ph.D.
Facilities Manager

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF IOWA
IOWA CITY, IOWA 52242

January 21, 1987

(Received 26 January 1987)

PAINLESS GAUSSIAN RESOLUTION ENHANCEMENT

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

Dear Professor Shapiro:

This note describes a straightforward "recipe" for easily and routinely obtaining near-optimum results with Gaussian resolution enhancement (GRE). GRE was proposed over 20 years ago by Ernst⁽¹⁾, implemented much later by Ferrige and Lindon⁽²⁾, and eventually found its way into the commercial FT NMR data-messaging software of companies such as Bruker. Unfortunately, the commercial implementation is awkward to use efficiently, and the standard "optimization procedure" described in the documentation is not helpful. Setting the maximum of the GRE envelope to the "point the useful signal disappears into the noise" might work OK under certain carefully-specified conditions, but it's usually wrong -- like the proverbial broken clock that's right twice a day. I've seen PhD's with several years of NMR experience read the documentation, try GRE on a few of their FID's, and give up because "It seems to be a hit or miss proposition."

At this point I would like to apologize to any programmer who has written a general NMR data-processing package which correctly implements the convenient set-up of the GRE apodization envelope. I'm sorry, but I wasn't aware of this feature in your package.

GRE transforms a Lorentzian line of width W_0 into a Gaussian line of width ρW_0 , where $0 < \rho < 1$ and the line widths are full widths at half height. This is accomplished by multiplying the FID by a Gaussian envelope which has a maximum at time t_{\max} , where

$$t_{\max} = (2 \ln 2) / (\pi W_0 \rho^2)$$

t_{\max} is in seconds, and W_0 is in Hz. In Ernst's paper, fig. 20 includes an accurate graph of the S/N achieved with the GRE filter as a function of ρ , but eqn. 124 is wrong. Eqn. 124 is apparently one of those malignant spot mutations which seem to appear spontaneously in manuscripts, and which find their way into the pages of books and journals in spite of the author's best efforts at proofreading.

I have submitted a manuscript containing the mathematical details and a more complete discussion of GRE to J.Magn.Reson. The RECIPE is this:

Determine W_0 , the intrinsic linewidth (in Hz) of the sharpest line which you want to "improve". Multiply the FID by a GRE envelope appropriate for W_0 , with $t_{\max} = 1/W_0$ sec. Fourier transform, phase, and plot.

That's all! This recipe sharpens the lines to a relative width $\rho = 0.664$, which is close to optimum in most cases. The procedure is straightforward, reliable, and easy to describe to novice users of NMR spectrometers. If more or less sharpening is desired, then set t_{\max} according to how much S/N you can afford to spend in order to buy resolution.

W_0 can be conveniently measured as follows. Examine the spectrum obtained with conventional exponential apodization, using a small amount of broadening W_L Hz. ($W_L = LB$ on Bruker software.) Measure W_1 , the full width at half height of the signal(s) of interest. If this is not possible because of interference from overlapping lines, then use $W_1 =$ twice the full width at 80% of peak height. Then $W_0 = W_1 - W_L$. On Bruker and IEM systems, $GB = t_{max}/AQ$, where AQ is the FID acquisition time. One should set $GB = 1 / (AQ * W_0)$. Remember to set $LB = -W_0$ before doing the GM.

If you still think that the "FID disappears into the noise" procedure is a good one, then consider the following three situations:

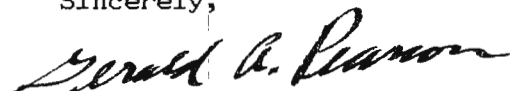
A 90 MHz ^{13}C spectrum was obtained for a dilute solution of a partially ^{18}O -labeled compound ($SW = 24000$ Hz, FID array size 64K). After the lines were sharpened, critical structural assignments could be made on the basis of the now clearly visible doubling of certain signals because of the small ^{18}O isotope effect on the ^{13}C chemical shifts. Since the signals were weak and the splittings of interest were small, careful control of the GRE parameters was essential for success. Had the FID not been totally dominated by the solvent lines (which had a different line width), the entire FID would have been buried in the noise! The data acquisition and processing were done by a graduate student working alone late at night, following a one-page handout sheet describing my GRE recipe. This student follows explicit instructions well, but his main interest is natural-products chemistry and not digital signal processing. Like most operators of NMR spectrometers, he has a low tolerance for ill-defined trial-and-error data massaging. When I asked him about it the next day, he said that it "worked fine -- no problem."

1H FID's of paramagnetic compounds typically have their tails totally obscured by signals from solvent and other sharp lines in the sample.

Take a sample and obtain the FID using a relatively narrow spectral width. Now repeat the data acquisition after increasing the array size, the spectral width, and the analog filter width by a factor of 16. The FID noise will increase by a factor of 4, which will shift the point where the signal "disappears into the noise" to shorter time. Indeed, the increased noise might totally bury the FID. Both the optimum GRE envelope and the resulting S/N ratio of the signals will be the same in both cases, however.

Having said all this, I would urge all NMR applications programmers to modify their software so that the GRE envelope can be specified in terms of W_0 and ρ . There is no good reason why a person sitting in front of a powerful minicomputer should be required to do the arithmetic needed to figure out how to appropriately lie to the software about where the FID "disappears into the noise."

Sincerely,



Dr. Gerald A. Pearson

1. R.R.ERNST, ADVAN.MAGN.RESON. 2, 1 (1966).
2. A.G.FARRIGE & J.C.LINDON, J.MAGN.RESON. 31, 337 (1978).



UNIVERSITY OF MISSOURI-ROLLA

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

College of Arts and Sciences

Department of Chemistry

142 Schrenk Hall
Rolla, Missouri 65401-0249
Telephone (314) 341-4420

January 2, 1987

(Received 9 January 1987)

Dear Barry:

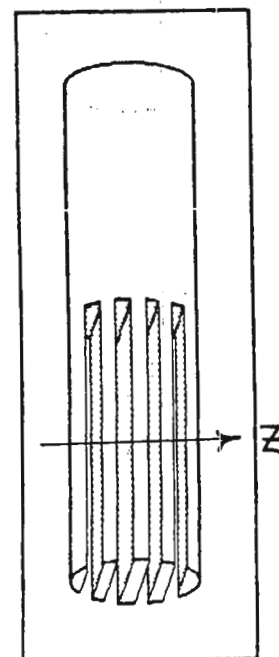
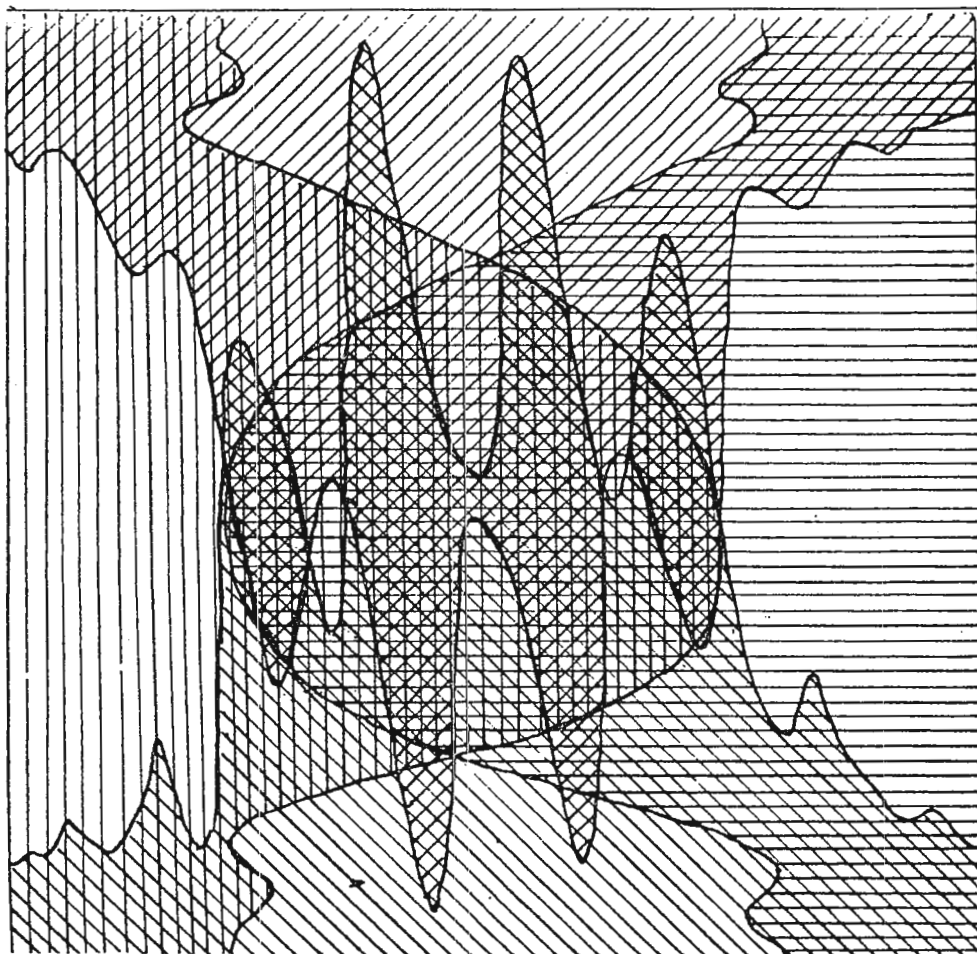
More Imaging on an FX-90Q

Since our last communication to you, I have moved to Rolla. We have purchased a Varian VXR-200 with CP-MAS Solids and Wide-Line Accessory. I'll give you a progress report on the instrument in a later communication. We also have two FX-100's which I will continue to use in my diffusion work.

In our last letter (TAMU 330-24) we reported 1-dimensional imaging spectra taken on an unmodified FX-90Q using the homo-spoil coils. We have extended this work to two dimensions by rotating the sample and using a very simple reconstruction technique. Shown below is the superposition of four spectra, taken at 90° rotations of water and a plexiglass rod with four slits cut into it. The area of overlap of all four spectra clearly shows the two dimensional projection of the rod. A manuscript is currently being submitted to spectroscopy on this and I would be happy to supply reprints to those who are interested. Stephen Pickup at Drexel is a co-author of this work.

Sincerely

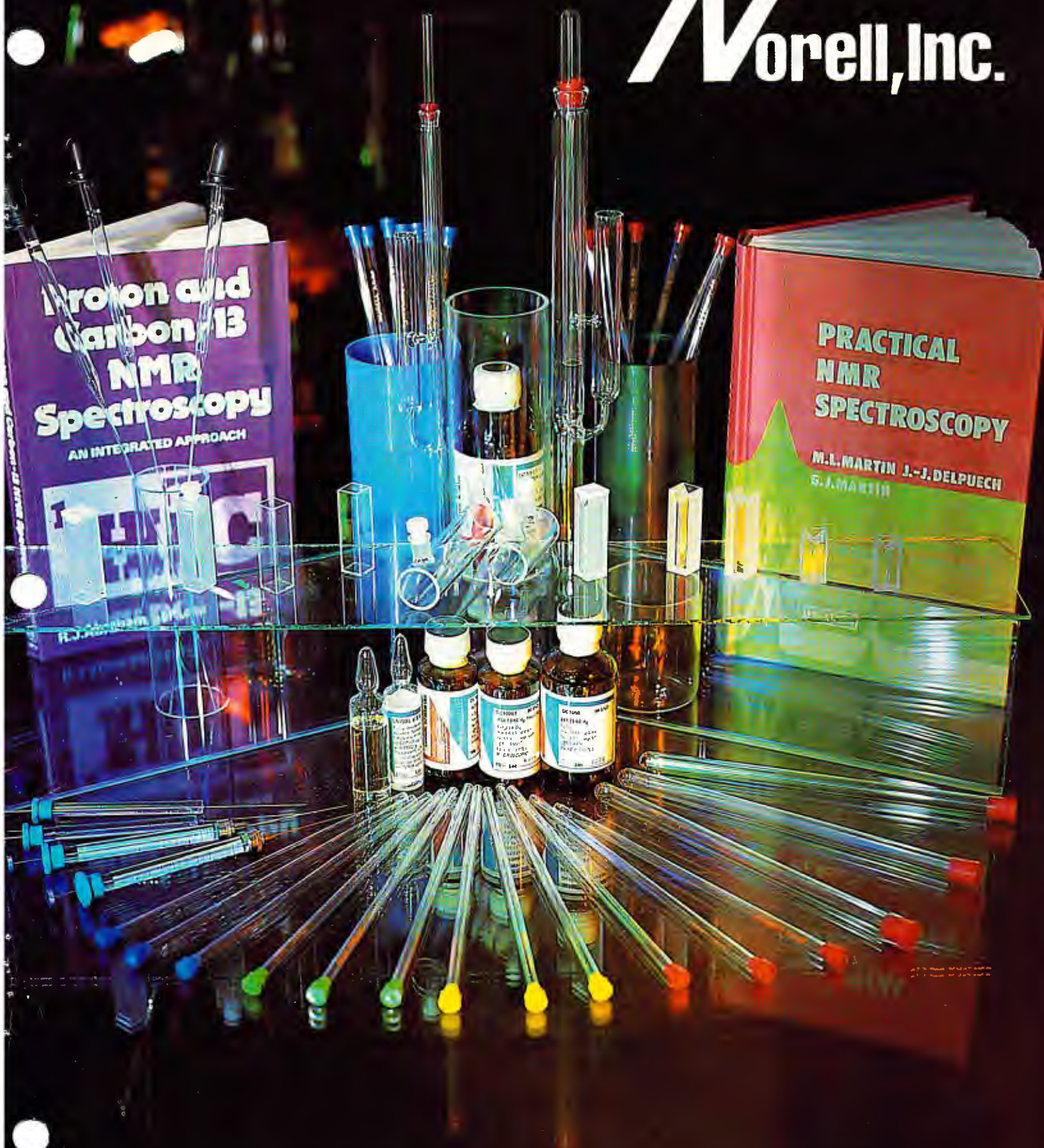
Frank D. Blum
Associate Professor
of Chemistry



PMMA plug.

NMR 2-D Image of PMMA plug taken from four superimposed spectra.

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(Received 15 January 1987)

Postdoctoral Position Available

A postdoctoral position will be available, in April 1987, to study internal dynamics of Staphylococcal Nuclease, a small calcium requiring enzyme, using solid state and solution NMR techniques. Using transformed E. Coli, provided by Professor J. Gerlt, we routinely prepare large quantities of the protein, specifically labeled with ^2H , ^{13}C or ^{15}N . Three instruments (2.3, 6 and 12T) are available to study the dynamics of the labeled sites in protein crystals and in solution.

Level and salary of the position will depend upon the experience and achievements of the candidate. Interested persons should send me a curriculum vitae and the names of three references.

Dennis A. Torchia
30/106
National Institutes of Health
Bethesda, MD 20892
301-496-4563

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Département
Technologie de la Viande

I.N.R.A. THEIX
63122 CEYRAT - FRANCE
Telephone: 73 62 40 00

7 January, 1987 (Received 14 January 1987)

The Meat Science Department of I.N.R.A. (National Institute of Agricultural Research) has an opening for a post doctoral position for one year.

The research programme will include signal processing and chemical shift imaging in order to use low field NMR (0.47T) in wide bore (150 mm) magnet to assess muscle composition and meat characteristics.

The candidate must have a background in NMR instrumentation and technique. Salary will be based on the french Chargé de Recherches level. Those interested should send me a curriculum vitae and names and addresses of two academic referees.

J.P. RENOU

This post doctoral position is not available for french citizens.

Mobil Research and Development Corporation

January 6, 1987
(Received 12 January 1987)

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

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EUGENE L. JONES
MANAGER

CP/MAS OF QUADRUPOLEAR NUCLEI

Dear Barry:

As previously shown (TAMU NMR Newsletter, No. 322 and Zeitschrift fur Phys. Chem. N. F., in press), the CP/MAS line shapes of B-11 in trigonal coordination (large quadrupole coupling constant) and in tetrahedral coordination (small quadrupole coupling constant) in the hydrated mineral kernite, $\text{Na}_2\text{B}_4\text{O}_6(\text{OH})_2 \cdot 3\text{H}_2\text{O}$, are the same as the MAS line shapes. This indicates that the CP experiment excites the central transitions of a given boron coordination equally for all orientation of the crystallite in the powder sample.

One use of the CP/MAS versus the MAS experiment is to reveal details of the hydration of materials, as done by Maciel with the Si-29 spectra of silica gel. Compared to spin 1/2 nuclei, quadrupolar nuclei can be sensitive to the additional parameters of the quadrupole coupling constant (QCC) and the asymmetry parameter (η) of the electrostatic field gradient; these parameters can depend on the hydration of the material.

Porous Vycor is a boro-silicate glass which has been leached with alkali and has $\sim 55 \text{ \AA}$ diameter pores throughout. It is mainly SiO_2 , but it has several percent B_2O_3 . The B-11 MAS signal of a sample equilibrated with water vapor for twenty years at $\sim 50\%$ relative humidity is shown in Figure 1 for two different spinning speeds. There is a strong trigonal boron signal with $\text{QCC} = 2.4 \text{ MHz}$ and $\eta = 0$ and also a weak tetrahedral boron signal with small QCC. The CP/MAS signals using two different boron r.f. intensities with a two millisecond contact time are shown in Figure 2. Note that the signal from the trigonal borons is different from that obtained in the MAS experiment. Figure 3, with the tetrahedral boron signal appropriately subtracted, emphasizes this difference. The borons which are cross-polarized due to their proximity to surface OH groups and adsorbed H_2O molecules experience different η values. Comparison with simulated spectra indicates a range of η values, probably in the range $\eta = 0.7 - 0.9$.

More details of these experiments can be found in the article in Zeitschrift fur Phys. Chem. N.F.

Sincerely,

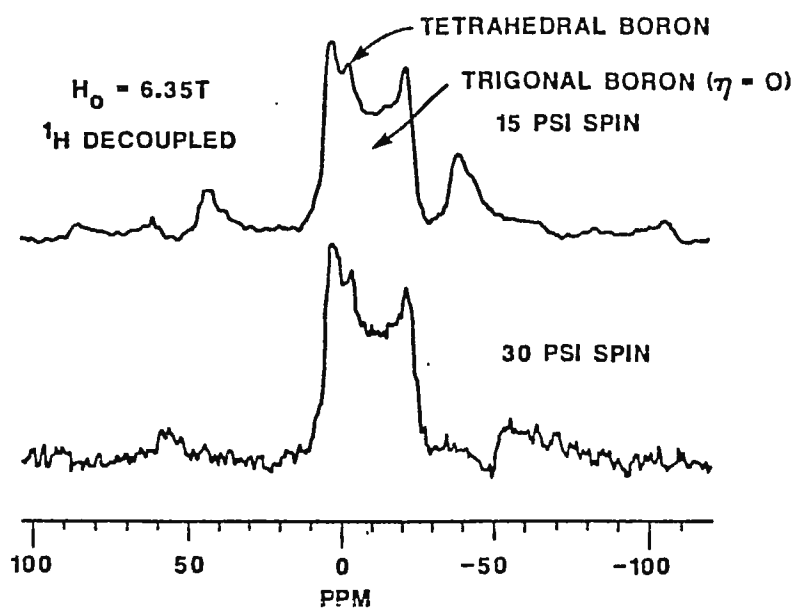


DEW:dpj
cc: B. C. Arnwine
T. H. Bushaw
E. L. Jones
Records Center 602-857

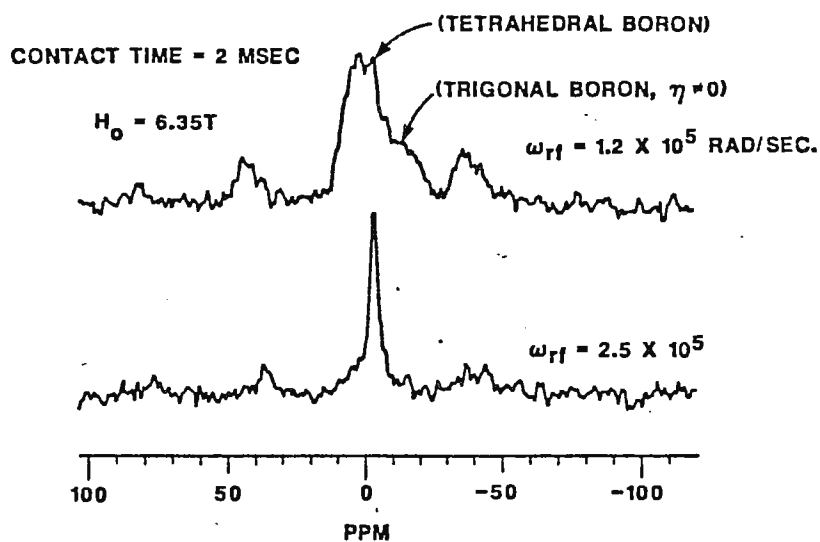
D. E. Woessner
Senior Research Associate

Figure 1.

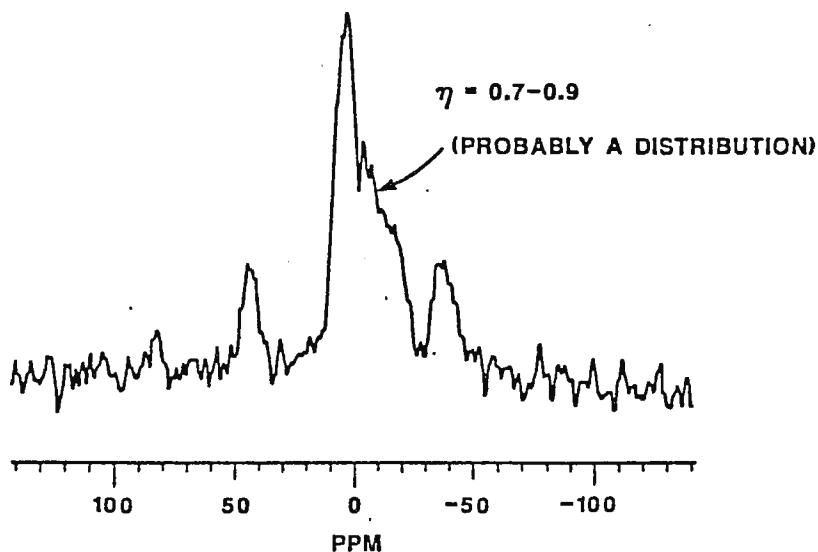
The B-11 MAS NMR of hydrated porous Vycor at two different spinning speeds.

**Figure 2.**

The B-11 CP/MAS NMR at two different B-11 r.f. amplitudes.

**Figure 3.**

The B-11 CP/MAS NMR with subtraction of tetrahedral boron signal.





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January 6, 1987

(Received 12 January 1987)

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

The Compleat Isolator

Dear Barry:

I would give credit to the designer of the basic idea of this circuit if I remembered where and when it was. I think Journal of Magnetic Resonance.

The purpose is to keep the noise and stray signals from the power amplifier getting to the probe and preamplifier during the Receive time, when they would put noise and possible spikes into the spectrum. The old way, at lower frequencies and powers, was to use a couple of crossed diodes in the line.

In this circuit (see figure), if the power is 1 KW, perhaps eight crossed 1N4148's would have to be used. Their stray capacitance, on the order of 6 pF, would pass large amounts of the higher frequency (e.g. 200 MHz) input noise current. Therefore, the noise voltage is inverted by a transmission line transformer (a thin 50 ohm cable wrapped 4 times through a toroid, and the output center conductor grounded) and used to inject current of an equal and opposite phase through a (tuneable) capacitor. The two noise currents cancel over a very large bandwidth, especially if the tuning is optimized at the maximum frequency and then not changed. The extra length of coax in the main leg is equal to the length in the inverting leg to keep the delays equal.

The isolation per stage is typically 30 dB at 200 MHz and is tuned with the other stage bypassed and .2Vpp or so CW on the input.

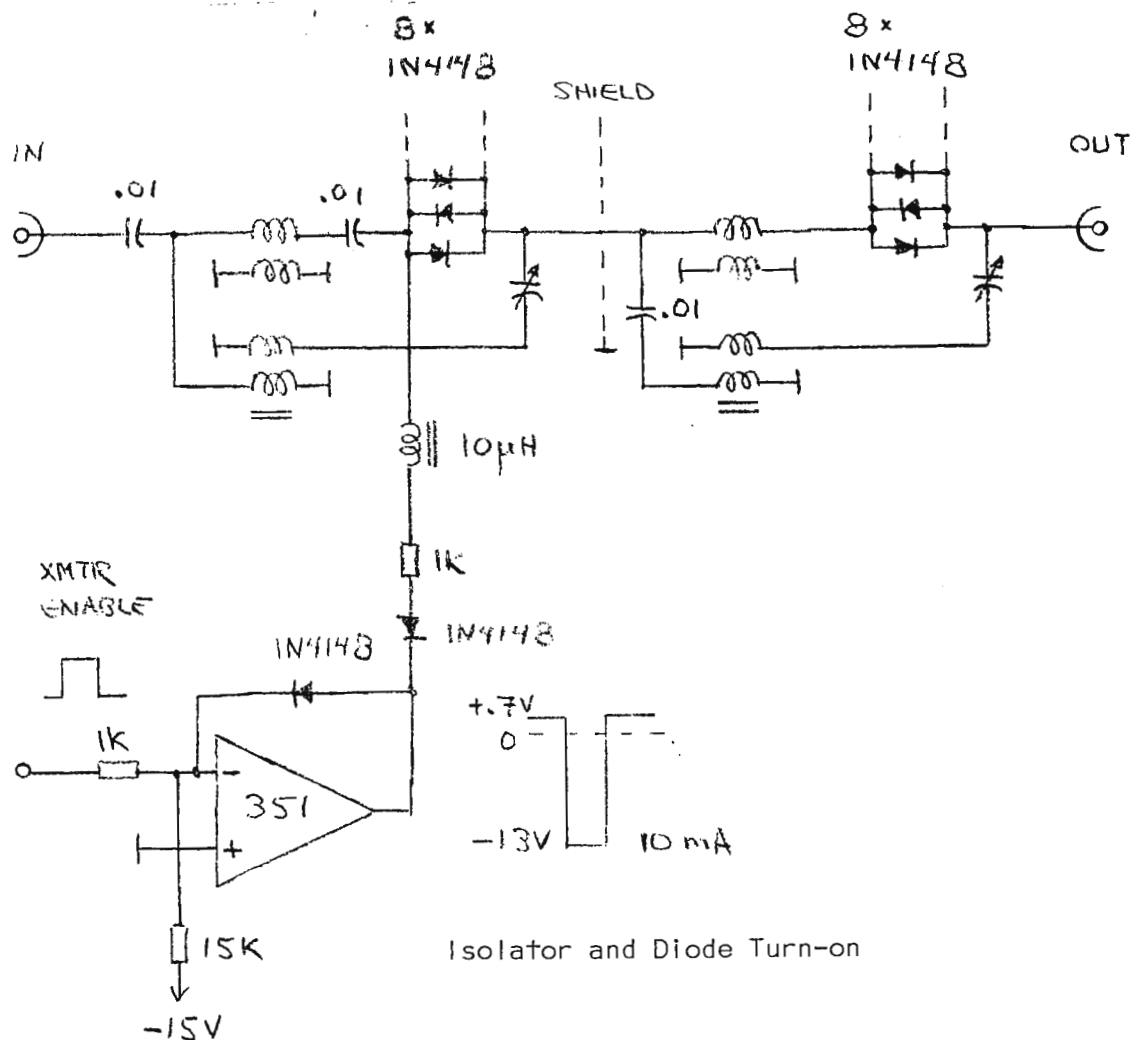
The op amp circuit is an extra feature to make possible the use of low level pulses. It injects 10 MA DC through all of the diodes in the line, including in the probe, and turns them on.

Please credit this to Eugene Poppel's Subscription.

Sincerely,

J.L.

J. L. Engle



**Catalytica*

December 23, 1986

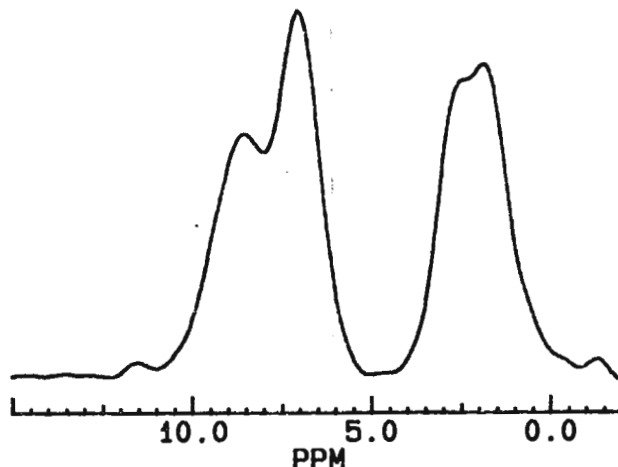
(Received 5 January 1987)

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

High Field CRAMPS

Dear Barry,

DIMETHYLBENZOPHENONE
400 MHZ CRAMPS



In our laboratory, we use a Bruker MSL400 spectrometer to study catalytic systems by a variety of solution and solid state NMR techniques. CRAMPS (combined rotation and multiple pulse spectrometry and MAS ^1H experiments have been useful in directly examining the acid sites in catalysts such as zeolites. These experiments can be done on our system using a standard Bruker double air bearing CP-MAS probe. Such a probe is not necessarily well suited for multiple pulse line narrowing experiments. In particular, the RF homogeneity and pulse ringdown are expected to be less than optimal for typical CP-MAS probe designs. In spite of these concerns, we can obtain quite excellent results, as shown in the attached figure. The spectrum is a CRAMPS spectrum of dimethylbenzophenone using the MREV-8 sequence for dipolar decoupling, with $t_c=34\mu\text{s}$ and an offset frequency of -1.5 kHz. The probe was tuned by standard procedures using a spherical water sample. The sample was contained in an alumina rotor, with a Kel-F plug and end cap used to restrict and center the sample in the RF coil for improved homogeneity. No modifications were made to the probe or spectrometer. The spectrum can be compared to one obtained with the more efficient BR-24 sequence on a low field instrument shown in figure 1 of a paper by Ryan, Taylor, Paff, and Gerstein (*J.Phys.Chem.*, 1980, 72, 508). Our spectrum compares quite well, with better separation of the aromatic and aliphatic resonances evident.

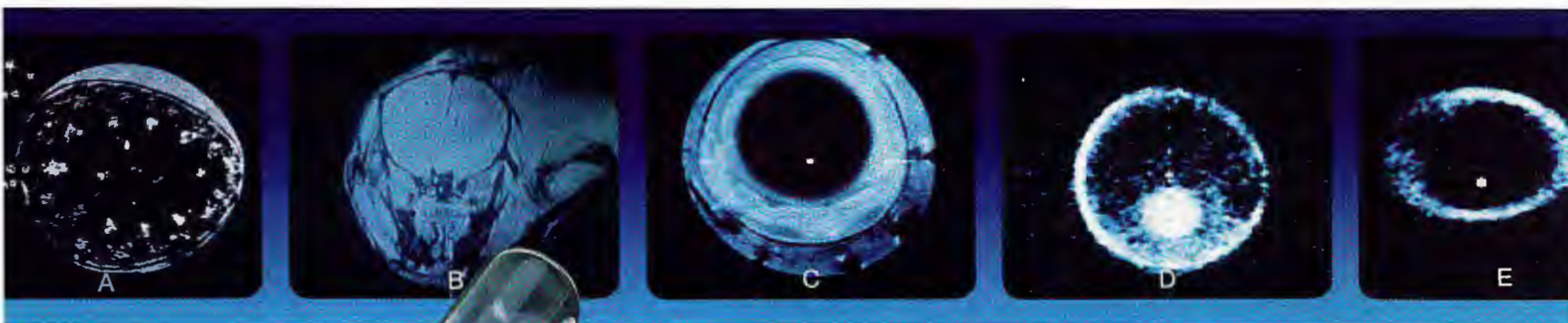
I should point out that Dr. Doug Burum of Bruker Instruments provided both the technical help and the Kel-F plugs needed for setting up these experiments. It is possible that CRAMPS experiments might also be performed with CP-MAS probes on lower field instruments, but these results show again that the brute force of higher magnetic fields can very often prove useful in overcoming many types of experimental deficiencies.

Sincerely,

Charlie

Charles M. Schramm

The NMR evolution continues:



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Fig. C: A cross sectional image of a mouse eye, 3 mm in diameter. Resolution $20\mu \times 20\mu \times 250\mu$.

Fig. D: Image of an ovum from laevis (frog egg). Resolution $10\mu \times 10\mu \times 250\mu$.

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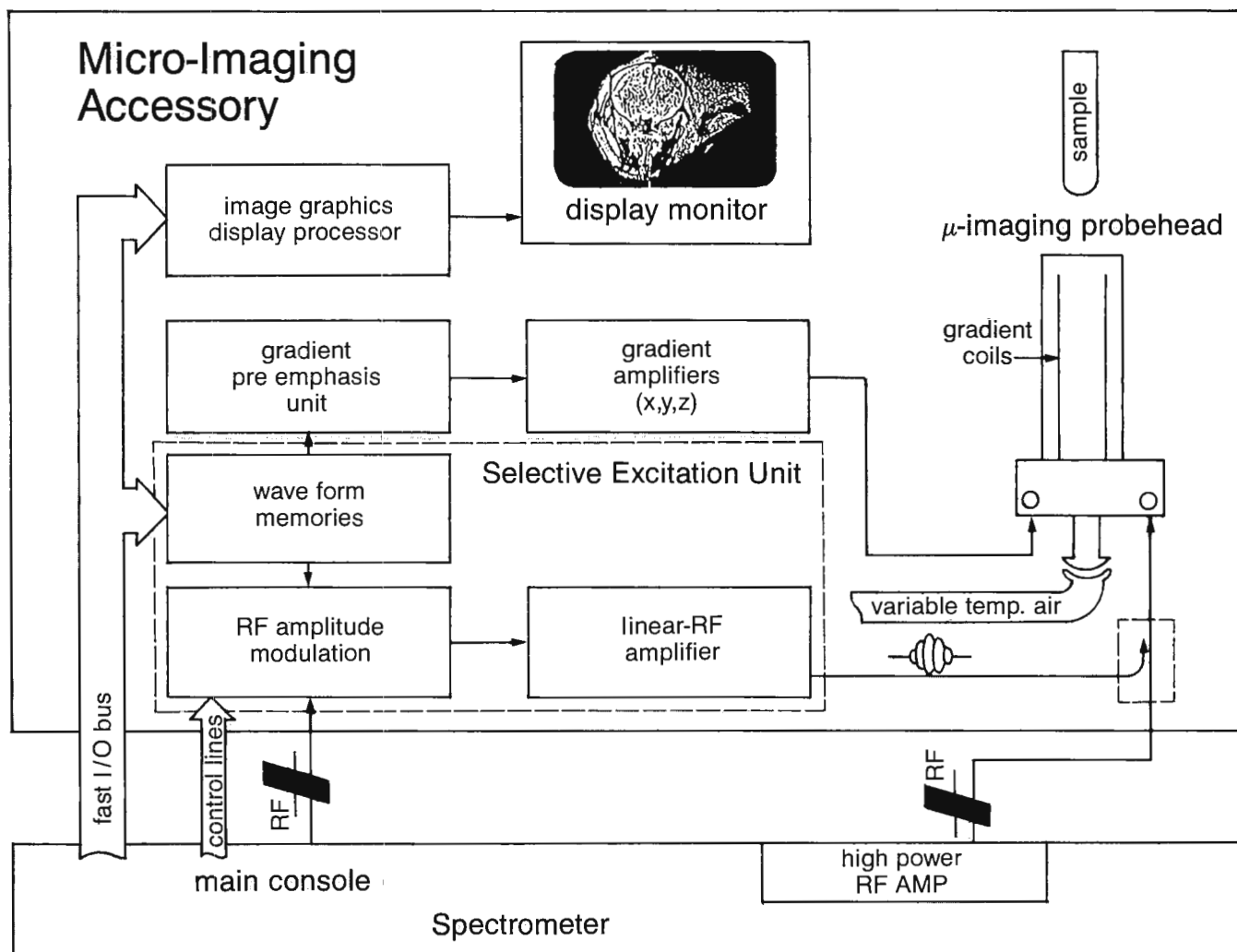
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Professor Bernard L. Shapiro
Texas A&M University
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College Station, Texas 77843, USA

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29.52.78 - 29.54.82 - 29.60.71 - 29.53.10

January 14, 1987

(Received 22 January 1987)

No. Rif. Prot. N. /

WHY DOES MY TRIPLET LOOK LIKE A QUARTET?
(A Warning Against Absolute Value Presentation)

Dear Prof. Shapiro,

it is well-known that a strong resolution-enhancement followed by a "magnitude" calculation (i.e. a very common way of presenting 2D spectra) can affect lineshapes to various extents.

Nevertheless, we were quite puzzled, recently, by comparing ^1H cross sections in a ^{13}C - ^1H heteronuclear correlated spectrum of a cannabinol derivative, Fourier transformed:

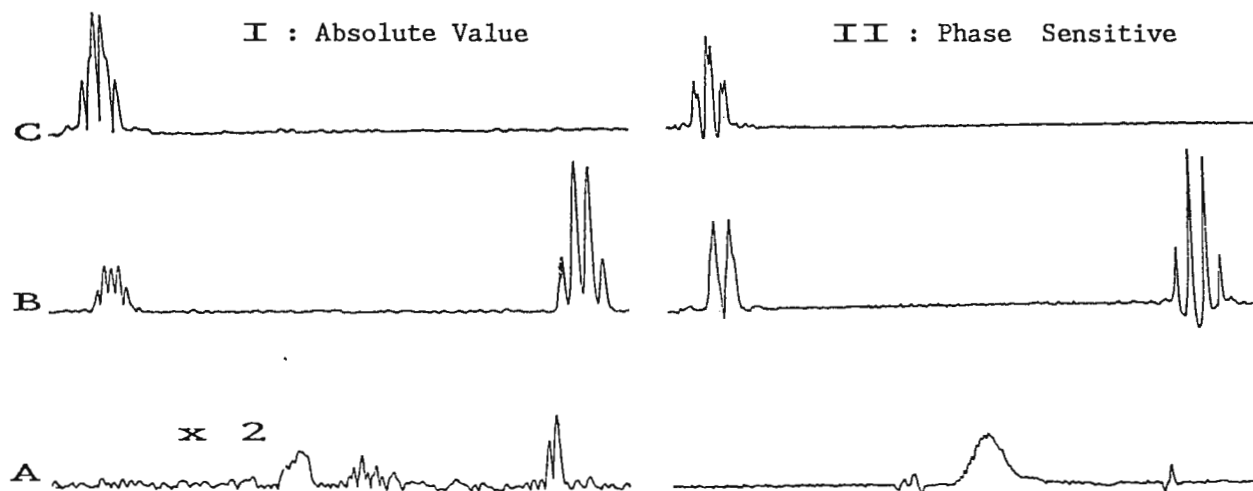
(I) after the application of a squared sine-bell in both dimensions (absolute value presentation)

(II) with a $\pi/32$ and $\pi/8$ shifted sine bell in t_1 and t_2 dimension, respectively (phase sensitive presentation).

Three of such cross sections are shown in the figure.

It is readily noticed that in trace I-A the digital treatment almost wipes out the main signal (broad), while emphasizing the minor (but sharper) resonances from two, non directly bonded protons. In the remaining traces (B,C) it is unquestionable that the true multiplicities are completely distorted in the magnitude spectra.

Although 2D phase correction can be rather cumbersome, in most cases is easily performed on single rows or columns of interest, and certainly worthwhile for the experiments in which the phase properties of multiplets contain valuable information.



Yours sincerely,

Antonio De Marco
Antonio De Marco

Lucia Zetta
Lucia Zetta

BOSTON COLLEGE

CHESTNUT HILL, MASSACHUSETTS 02167

Department of Chemistry
(617) 552-3605

January 15, 1987

(Received 27 January 1987)

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

Dear Barry:

NEW WRINKLES ON A PLANAR RING

Some time ago, Joe Billo, Peter Connolly and I began to look at the proton spectra of a series of Ni(II) cyclam complexes which exist in part as square planar (diamagnetic) conformations. In every case we looked at, the six-membered chelate rings (C_3N_2Ni) were in the chair conformation, as expected. An exception seemed to be bis-(1,5-diazacyclooctane)-nickel(II) perchlorate $[Ni(DACO)_2]^{+2}$, whose coordination geometry had been described as being rigorously square planar, with two planar eight-membered DACO rings folded along their 1,5-axes (i.e., half-chair chelate rings) [Royer *et al.*, *Inorg. Chim. Acta*, 6, 307 (1972)]:

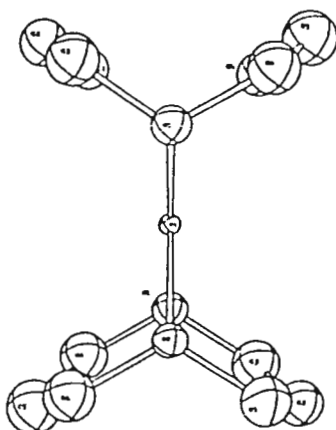


Figure 2. The $Ni(DACO)_2^{+2}$ cation as viewed along the $N1-N2$ axis.

The unusual eclipsed trimethylene bridges were ascribed to the desire of the ion to avoid severe steric repulsions which would result from ring puckering.

The 300 MHz proton spectrum proved easy to fit as an ABCC'DD' spectrum giving vicinal couplings of 4.0 (J_{AC} & $J_{AC'}$), 7.8 (J_{AD} & $J_{AD'}$), 8.8 (J_{BC} & $J_{BC'}$) and 4.9 Hz (J_{BD} & $J_{BD'}$). In comparison with both our experience in the cyclam series and our expectations based on the extended Karplus equation, where syn, gauche and anti couplings should be about 12, 4 and 12 Hz

respectively, the experimental couplings are most consistent with trimethylene bridges interconverting rapidly between boat and chair conformations. Our subsequent molecular mechanics calculations supported this conclusion. However, they have not allowed us to choose confidently among the several alternative puckered forms. Moreover, we have not been able to freeze out the motion, even at -90°C , though we do see what looks like some line-broadening.

Best Wishes,



Dennis Sardella
Professor of Chemistry

*We did encounter a problem with the Varian-supplied spin simulation program, which was unable to handle this degree of magnetic nonequivalence. The spectrum was fitted on a VAX 11/780 using LAOCN5, and the computed and experimental spectra were virtually superimposable. However, the spectrum computed using the Varian software differed significantly from the experimental one.

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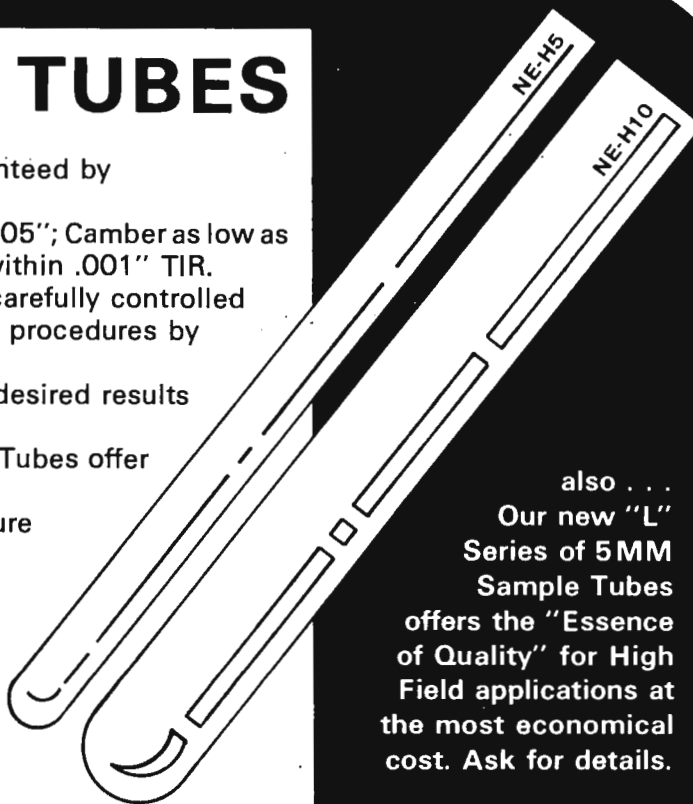
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JR:RO

22 December 1986

(Received 26 January 1987)

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

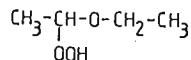
Dear Prof. Shapiro

NMR OF A PEROXIDE?

We have recently been reminded of the danger inherent in accepting samples for NMR without knowledge of their background.

A colleague recently discovered (by GC) that the diethyl ether he was using was contaminated. He took 50 ml and removed the volatiles on a rotary evaporator. This left approximately 10 ml (10 g) of an oily, non-volatile, clear liquid (contamination indeed!). He brought this along to us for NMR - after all, we are forever telling our staff that we can identify anything by NMR. As the 10 mm probe was in, and there was plenty of material, we made up a 50% solution in CDCl₃ and ran H-1, C-13, DEPT-90 and DEPT-135 spectra as shown.

I sat down to look at the spectra while the scientist concerned wandered down the hall with the remaining 8 g of compound for infra-red. A distinct chill came over me and my heart nearly stopped when I began to realize just what we were analysing. The product was not 100% pure but the spectra seemed to be entirely consistent with the peroxide of diethyl ether!!!!



I would be perfectly willing to hear from one of your readers that these spectra are not of the peroxide but, if they are, I suspect we are the first to obtain such high signal to noise C-13, DEPT and proton spectra of this compound. In fact, we can find no literature reference to NMR spectra of this compound. Who else in their right mind would have put 2 g of peroxide into their magnet when microgrammes can detonate so violently.

A standard KI peroxide test on one drop immediately liberated deep purple iodine (a faint yellow colour is sufficient for concern).

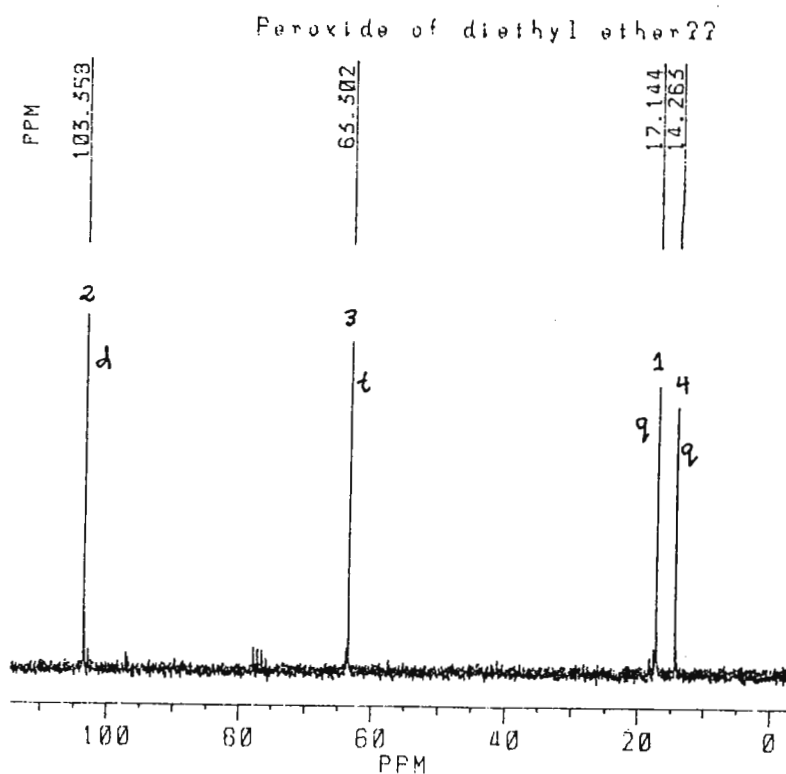
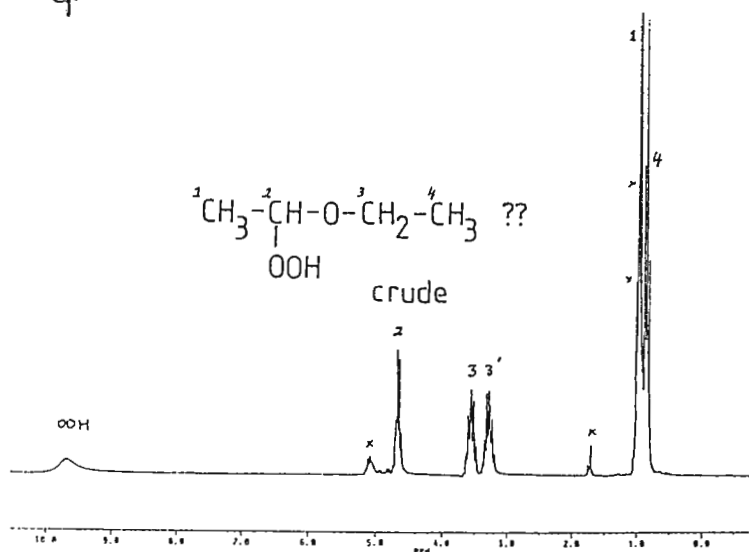
We could have made a more detailed examination, but decided we had tempted fate sufficiently already and disposed of the material and the offending (old can of) ether promptly. And we are now more cautious about accepting unknowns without a little more information on their source!

Yours sincerely

John Ralph

A. H. H. H.

John Ralph
for Director



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AU PROG:
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SY 50.3300000
O1 -1647.000
SI 32768
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HZ/PT .744

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O2 3250.000
DP 17H BB

LB 1.000
GB .500
HZ/CM 541.736
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SR -6667.20

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Telephone: 408-282-3634
800-243-9435 (Danbury, CT)

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

January 20, 1987
(Received 23 January 1987)

DYNAMIC PARAMETERS FROM NONSELECTIVE 1D EXCHANGE SPECTRA

Dear Barry,

We are currently investigating a simple new method for the measurement of rate constants in chemically exchanging spin systems. For two spins A and B the differential z-Bloch equations are

$$\frac{d}{dt} \begin{pmatrix} A-A_o \\ B-B_o \end{pmatrix} = -\underline{R} \begin{pmatrix} A-A_o \\ B-B_o \end{pmatrix}, \text{ where } \underline{R} = \begin{pmatrix} R_{1A} + k_A & -k_B \\ -k_A & R_{1B} + k_B \end{pmatrix}.$$

We seek to measure the elements of \underline{R} . The integrated solutions are

$$(A_t - A_o) = (e^{-\underline{R}t})_{ii}(A_{t=0} - A_o) + (e^{-\underline{R}t})_{ij}(B_{t=0} - B_o) \quad 1]$$

$$(B_t - B_o) = (e^{-\underline{R}t})_{ji}(A_{t=0} - A_o) + (e^{-\underline{R}t})_{jj}(B_{t=0} - B_o). \quad 2]$$

We employ the standard pulse sequence $(90^\circ)-t_1-(90^\circ)-t_2-(90^\circ)-t_2(\text{obs})$ and record spectra for an arbitrarily chosen t_1 value with and without mixing. These spectra provide A_t , B_t and $A_{t=0}$, $B_{t=0}$ respectively. With equilibrium values A_o and B_o we compute two measureables $M_1 = (A_t - A_o)$ and $M_2 = (B_t - B_o)$ and two coefficients $C_1 = (A_{t=0} - A_o)$ and $C_2 = (B_{t=0} - B_o)$. Denoting the four elements of $e^{-\underline{R}t}$ as unknowns X_1 to X_4 , Eqs. 1] and 2] may be rewritten as

$$M_1 = C_1 X_1 + C_2 X_2 \quad 3]$$

$$M_2 = C_1 X_3 + C_2 X_4 \quad 4]$$

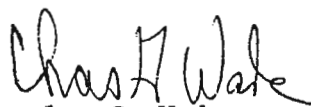
Spectra with and without mixing for a second value of t_1 provide


$$M_3 = C_3 X_1 + C_4 X_2 \quad 5]$$

$$M_4 = C_3 X_3 + C_4 X_4 \quad 6]$$

Eqs. 3] - 6] are used to solve for X_1 to X_4 , which are assembled as a matrix \underline{X} ($= e^{-\underline{R}t}$). Then $\underline{R} = -(1/t) \ln \underline{X} = -(1/t) \underline{S} (\ln \underline{D}) \underline{S}^{-1}$, where \underline{D} and \underline{S} are the eigenvalue and eigenvector matrices of \underline{X} . We are currently applying this kinetic approach to multisite and scalar-coupled systems and hope to report on our results at the ENC.

Sincerely,


Charles G. Wade


Eric R. Johnston

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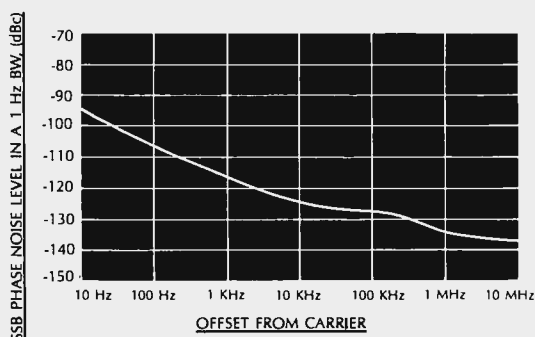
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December 9, 1986 (Received 27 January 1987) University of California Service
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San Francisco, California 94121
(415) 750-2146

Dr. Bernard L. Shapiro
Texas A & M University
Chemistry Department
College Station, TX. 77
Ottawa, Ontario K1A 0R6

Re: Postdoctoral and Faculty Positions in NMR

Dear Dr. Shapiro:

Several postdoctoral positions and potential junior faculty positions, are available to qualified individuals at the University of California, Veterans Administration Medical Center in San Francisco for individuals interested in in vivo NMR spectroscopy. Instrumentation for our human and animal spectroscopy program includes a 2.0 Tesla, 1 meter bore whole body magnet equipped with a VAX 11/750 for both imaging and spectroscopy. We also have a 2.0 Tesla, 30 cm bore NMR spectrometer for animal studies, and we expect a wide bore, 7.0 Tesla imager/spectrometer for additional high resolution studies. We have funds from the VA, NIH, and industry to support efforts to develop NMR localization techniques for ³¹P and ¹H studies in human subjects for biochemical and clinical investigation and ultimately diagnosis.

I am collaborating with a number of medical specialists including cardiologists, neurologists, neurosurgeons, and radiologists. Dr. Gerald Matson, Ph.D., has joined the Department of Pharmaceutical Chemistry at UCSF and is full-time in our facility. Dr. Matson has particular experience with spatial localization techniques. In addition, Dr. Robin Bendall of Griffiths University, Australia, will spend 4 months/year in our laboratory. Dr. Bendall will be developing a number of multipulse NMR techniques for localization and spectral editing. We also plan experiments in collaboration with Dr. Alex Pines across the Bay at U.C. Berkeley.

Finally, we have fully equipped electronics and machine shops. We also have a particular need for individuals with expertise in rf electronics and NMR probe design and construction.

Most importantly, we are eager to recruit physicists, chemists or biochemists who have a strong background or interest in magnetic resonance, and who wish to become involved in this new technology in a medical environment. Interested individuals should send their resume to me at the above address, at their earliest convenience.

Sincerely,

MICHAEL W. WEINER, M.D.
Associate Professor of Medicine and Radiology



PURDUE UNIVERSITY

SCHOOL OF SCIENCE
at INDIANAPOLISPHYSICS DEPARTMENT
1125 East 38th Street
P.O. Box 647
Indianapolis, Indiana 46223
(317) 923-1321
(317) 274-6900

January 9, 1987

(Received 19 January 1987)

Professor Bernard L. Shapiro
Department of Chemistry
Texas A & M University
College Station, TX 77843-3255

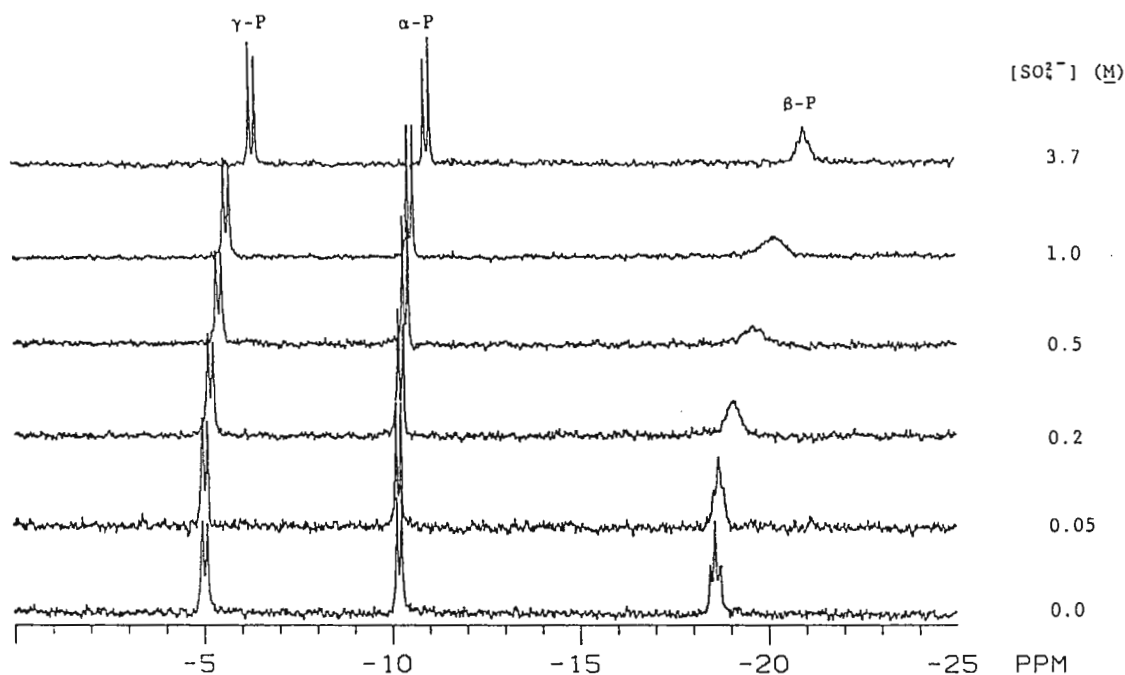
Title: Effect of Sulfate ion on Mg(II) binding to ATP

Dear Barry:

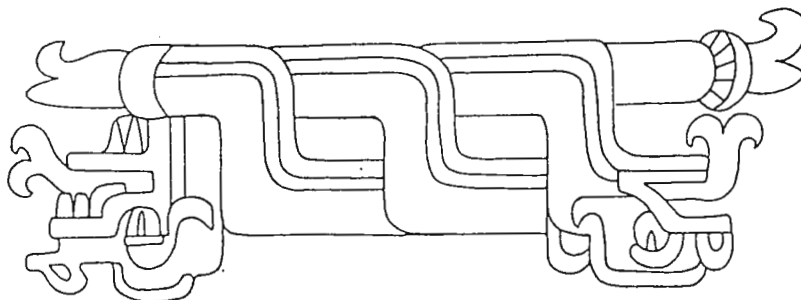
Thank you for the pink reminder. Our interest in the active-site structures of ATP-utilizing enzymes has led us to a discrepancy in the results of NMR and X-ray diffraction on the chelation of the divalent cation to ATP bound to the glycolytic enzyme 3-P-glycerate kinase. X-ray diffraction studies on crystals of horse-muscle enzyme in which enzyme-bound MgATP complexes were formed by soaking the crystal with the appropriate solution, suggest that the cation is positioned near the γ -P and is not within bonding distances to α -P and β -P of ATP (G. D. Banks et al., *Nature* (London) **279**, 773 (1979)). On the other hand, nuclear relaxation measurements in the presence of cation such as Mn(II) or Co(II) made in our laboratory (to be published) and Mn(II)-ESR measurements by George Reed's group at the University of Wisconsin, suggest direct coordination between the cation and all the three phosphate groups of ATP. In order to examine this discrepancy further, we determined the effect of sulfate ion on the Mg(II) binding to ATP and ADP free in solution and bound to the enzyme by ^{31}P NMR. Shown in the figure below are ^{31}P NMR spectra of MgATP in the presence of sulfate ion at different concentrations. The β -P resonance occurs at \sim 21.5 ppm in free ATP, and is shifted down to \sim 18.5 ppm in MgATP. It is clear from these spectra that sulfate ion, in sufficient excess, competes and sequesters Mg(II) from ATP. These and similar results on the other complexes suggest that the discrepancy between the NMR and X-ray results on the positioning of Mg(II) with respect to the phosphate groups of ATP bound to 3-P-glycerate kinase arise from the sulfate ion in the $(\text{NH}_4)_2 \text{SO}_4$ used in crystallization for the X-ray studies.

Sincerely yours,

B. D. Nageswara Rao
Professor of Physics

Effect of SO_4^{2-} on Mg Binding to ATP

Sample conditions: ATP, 3mM; Mg^{2+} , 6mM; pH 8.2 with HEPES, 0.2M; temperature, 20°C
 Reference: 85% H_3PO_4

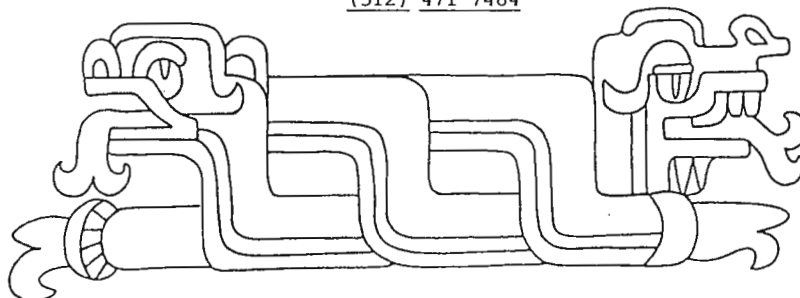


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Alan H. Cowley
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 The University of Texas at Austin
 Austin, Texas 78712
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Department Inorganic Chemistry
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Date December 29, 1986

(Received 5 January 1987)

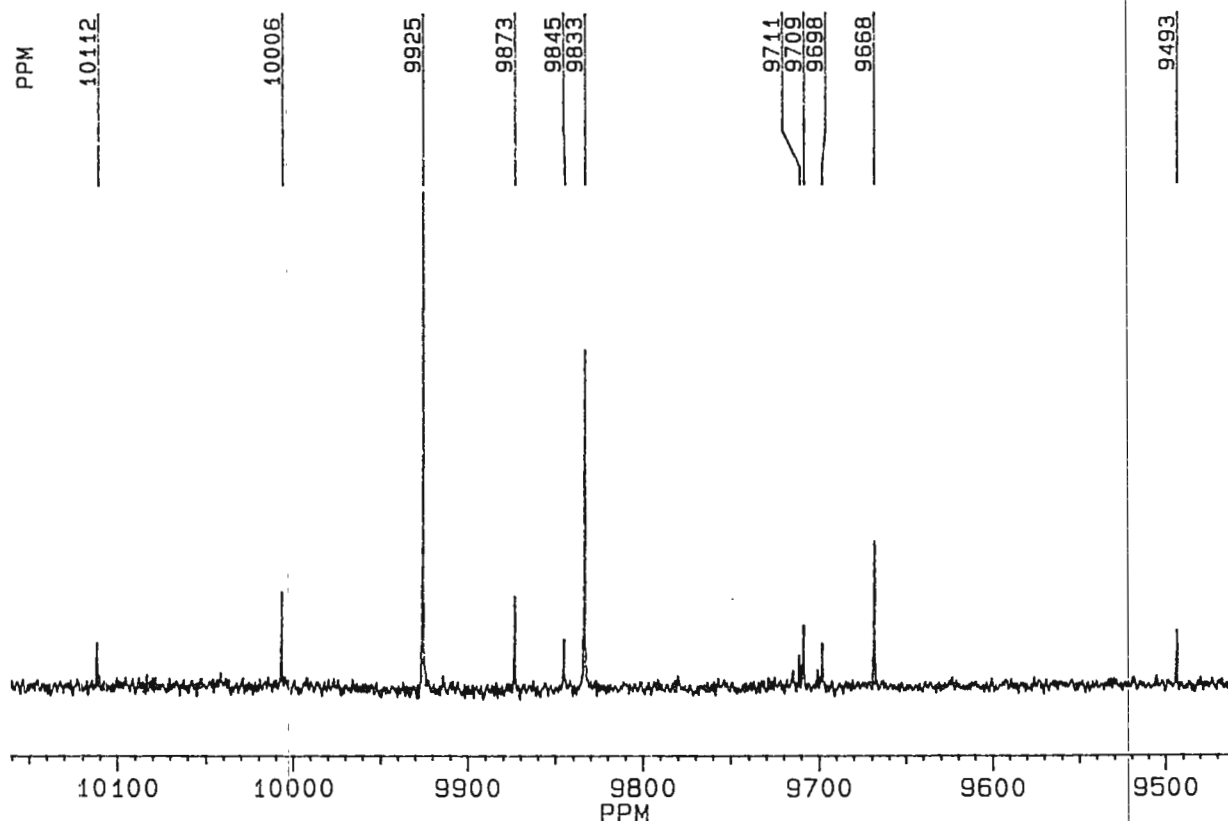
Rh(III) Hydrolysis by ^{103}Rh NMR

Dear Professor Shapiro,

During the last few months we have been happily testing and trying out our new Bruker AM400. We have examined several "exotic" nuclei and have now arrived at ^{103}Rh . In spite of its low sensitivity it gives quite rewarding results because of its several thousand ppm wide chemical shift range and relatively narrow signals (2-3 Hz). Moreover, since the rate of exchange of ligands between rhodium(III) complexes in general is very slow, each species can be observed as a separate signal.

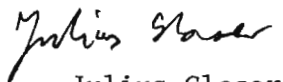
As part of an investigation on rhodium(III) halides we recorded some spectra of an acidic rhodium(III) perchlorate solution, prepared by dissolving hydrated rhodium oxide in perchloric acid. We expected to see a single resonance at 9992 ppm from the $[\text{Rh}(\text{OH}_2)_6]^{3+}$ ion, as previously had been reported (B.E. Mann and C. Spencer, *Inorg. Chim. Acta*, **65** (1982) L57), but instead we obtained the spectrum shown in the Figure.


RH-103 NMR, 1.3 M $\text{Rh}(\text{ClO}_4)_3 \cdot \text{H}_2\text{O}$ AFTER EN VECKAS KOKNING, 308 K



Changing the acidity or heating the solution for several days slightly shifts the peak positions and causes some peaks to decrease and others to appear. What we see is evidently several fairly inert polymeric products of the rhodium(III) hydrolysis. By mixing this solution with another, prepared by dissolving $\text{Rh}(\text{ClO}_4)_3 \cdot 6\text{H}_2\text{O}$ crystals in 1.6 M HClO_4 , and which did give a single signal from the $[\text{Rh}(\text{OH}_2)_6]^{3+}$ ion at 9931 ppm at 25 °C (9866 at 3 °C), we were able to assign the peak at 9925 ppm (which increased) to monomeric $[\text{Rh}(\text{OH})_n(\text{OH}_2)_{6-n}]^{3-n}$ species (because of the fast proton exchange n may have a non-zero value at lower acidity). Since very little is known about the hydrolysis of $\text{Rh}(\text{III})$ we are continuing our studies in greater detail.

Sincerely yours,


Julius Glaser


Magnus Sandström

EMORY UNIVERSITY

Department of Chemistry

1515 Pierce Drive
Atlanta, Georgia 30322
404/727-6585

December 30, 1986

Equipment Available

Because of changing requirements for high resolution solution NMR experiments we are contemplating selling our CXP-300 system except for the magnet and replacing it with a newer console. The system was purchased in 1979 and is in perfect working order. It includes

- 1) The console with full broad band capabilities from 12-120 MHz and ^{19}F and ^1H including high power transmitters.
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- 3) Three Diablo disk drives and one fast paper tape reader
- 4) Bruker room temperature shim coils
- 5) ^1H , and multinuclear probes for solution studies and a CP/MAS probe

If anyone is interested in purchasing this equipment please contact Dr. David Live at the above address or phone (404)-727-0867.

Back issues of the TAMU NMR Newsletter available.

Dr. Live has informed me that a large number of back issues of the TAMU NMR Newsletter and its predecessors are available. These issues, some dating back to the 1950s, formerly were in the hands of the now-retired Jake Goldstein. Anyone interested should contact Dr. Live directly. I suspect his price may be substantially lower than these incredibly important archival documents can be obtained from me.

B.L. Shapiro

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF TURKU
SF 20500 TURKU 50 - FINLAND

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

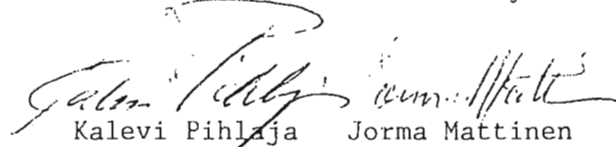
(Received 26 January 1987)

^{129}Xe NMR as a "macro structure probe" of cyclodextrin

Dear Professor Shapiro

^{129}Xe has a spin 1/2 and relatively high natural abundance (26.4 %) and its resonance frequency is near to that of ^{13}C (110.58 MHz on our JEOL GX-400). Since it is also chemically very inert it can be used as a sort of "enviromental" probe for the sample. We tried to apply this idea on cyclodextrin supposing the cavities in it to be large enough for a Xe atom to enter but on the other hand small enough to keep it inside long enough in NMR sense in order to differentiate the Xe-in and Xe-out signals. Xenon gas was first dissolved under normal pressure in chloroform for calibration of the instrument. Then Xe was dissolved similarly in DMSO and the spectra were recorded. Finally the procedure was repeated with DMSO which contained cyclodextrin. However, we could detect only one signal 1 ppm to lower field from the Xe signal in pure DMSO. The signal was also quite sharp. Hence one can conclude the exchange to occur too fast in order to see separately the Xe-in and Xe-out forms even if present.

However, we enjoyed the easy detection of the ^{129}Xe signal since only 50 - 100 scans were needed for excellent S to N. We intend to continue our study on this line in the near future.


Kalevi Pihlaja Jorma Mattinen Ebbe Still Yvonne Biström

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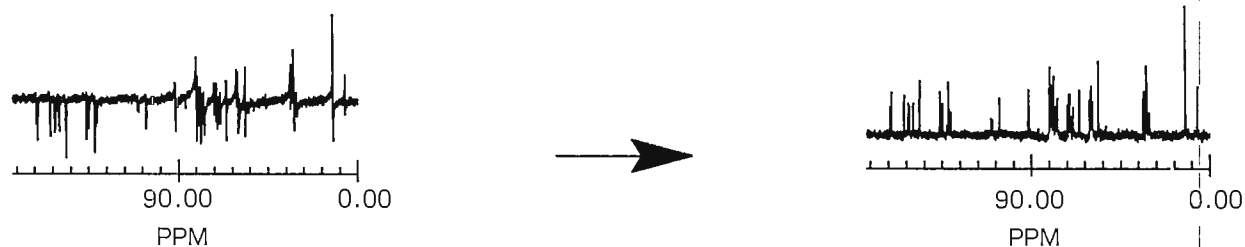
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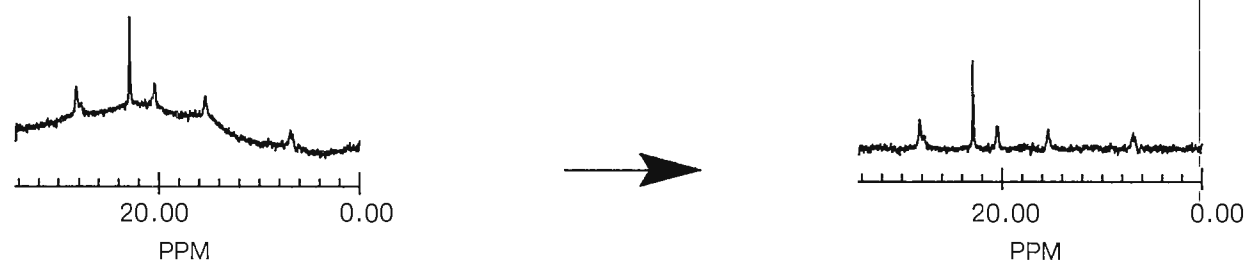
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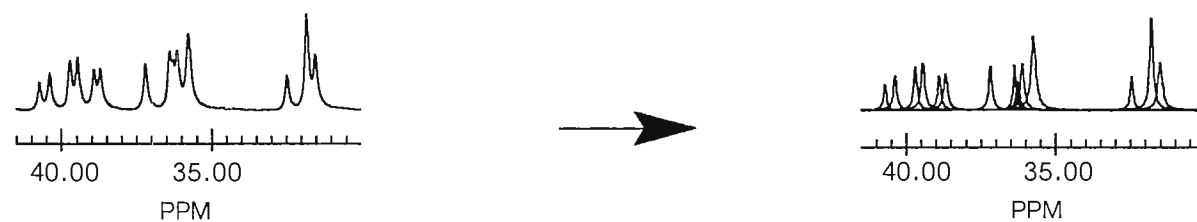
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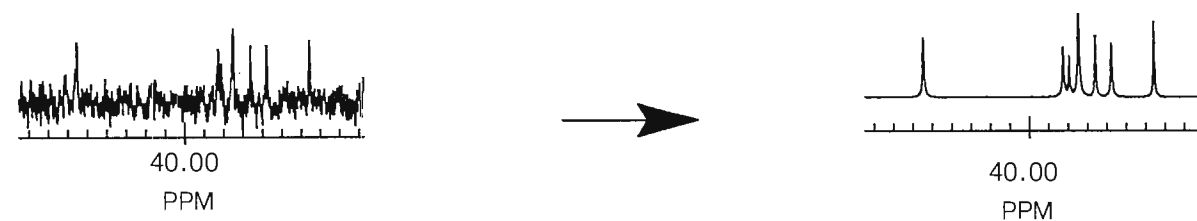
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 Prof. B. L. Shapiro
 Dept. of Chemistry
 Texas A&M University
 College Station, Texas 77843

Swedeland, December 23, 1986

(Received 13 January 1987)

Dear Professor Shapiro,

P.E.COSY: Another means to record exclusive COSY spectra.

I guess it is time to make a contribution. In their original paper, Aue, Bartholdi and Ernst presented explicit expressions for the amplitudes of COSY peaks. These results suggested that particularly simple spectra should arise, when the flip angle of the mixing pulse is set either near 0° or 180° . This is because a short mixing pulse transfers magnetization only between connected transitions, i. e. transitions with one common energy level. The use of a short mixing pulse simplifies the cross-peak pattern which permit more accurate measurement of long-range J-couplings.

Amazingly, this feature was not utilized for a long time until Sorensen and Ernst introduced the E.Cosy technique, which achieves such simplification of cross-peak patterns by a proper linear combination of multiple quantum filtered COSY spectra. The same type of spectra can be obtained with the simple COSY experiment by reducing the flip angle of the mixing pulse to 35° . The major drawbacks in this basic setup are the messed up phases. Diagonal and cross-peaks have different phases in both frequency domains giving rise to streaky spectra.

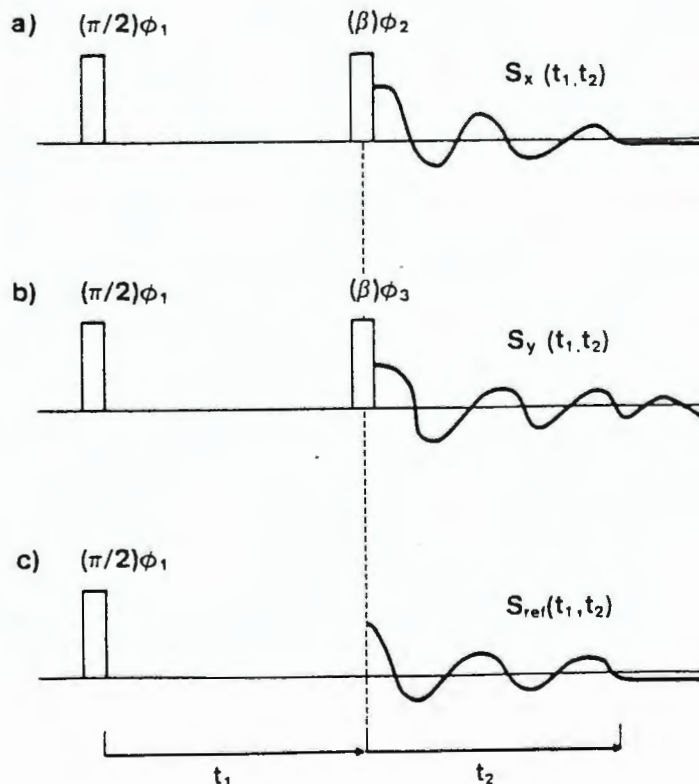
Figure 1:

$$\phi_1 = 0, 0, 90, 90, \\ 180, 180, 270, 270.$$

$$\phi_2 = 0, 180, 90, 270, \\ 180, 0, 270, 90.$$

$$\phi_3 = 90, 270, 180, 0, \\ 270, 90, 0, 180.$$

$$\phi_{\text{receiver}} = \phi_1$$

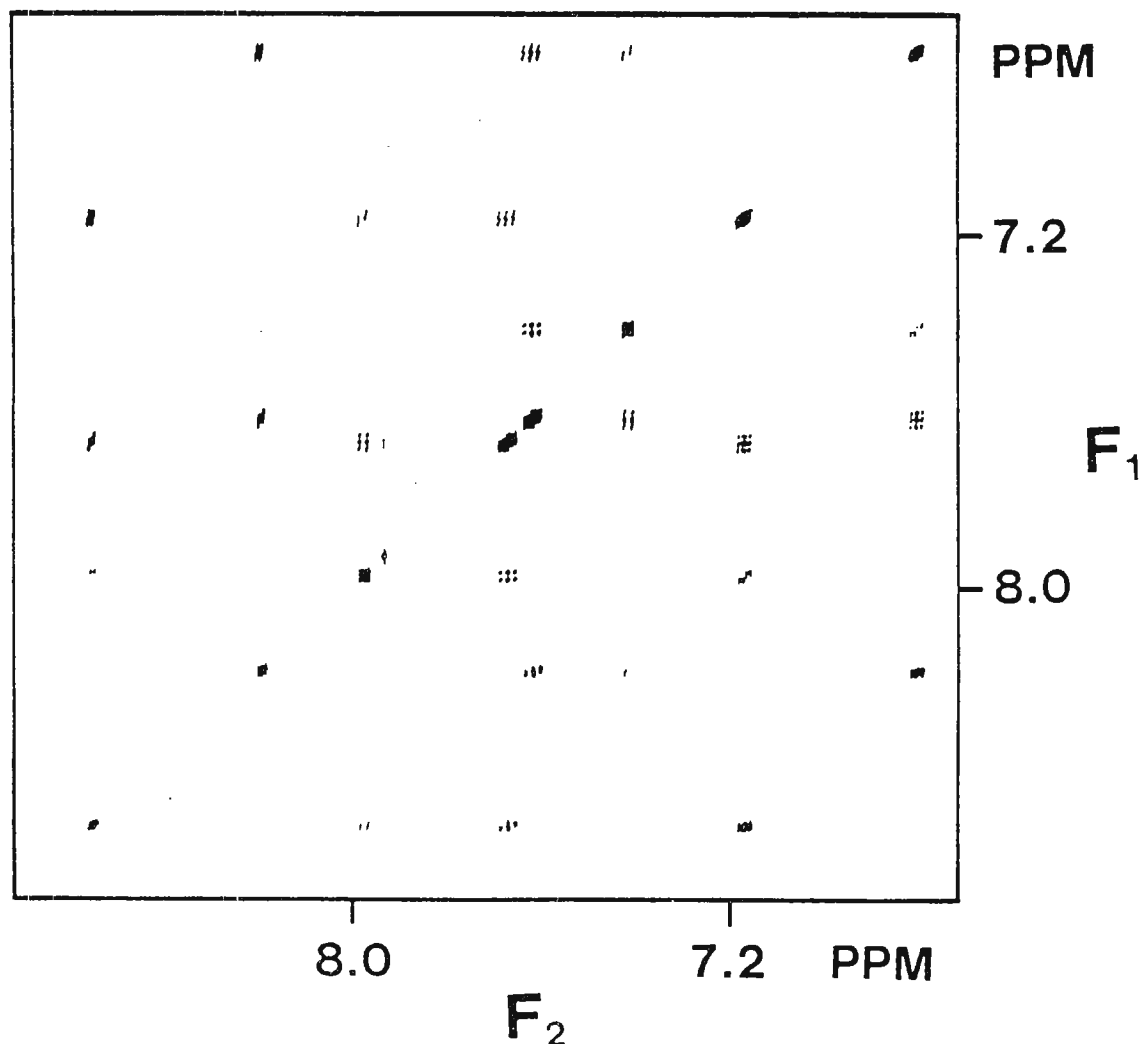


In this letter I wish to show a simple way to purge the phase in these COSY spectra. The basic sequence is depicted in Figure 1. Three separate FIDS are collected per t_1 -value. The 1st two FID's select the x- and y-component of the t_1 -FID as usual. The 3rd trace has no mixing pulse at all, its signal is merely collected to purge the phase of the former two FID's. It is easy to visualize that by subtracting $S_{ref}(t_1, t_2)$ from $S_x(t_1, t_2)$ all residual magnetization must be orthogonal to the phase of the mixing pulse. This allows proper phasing in the resulting difference spectrum $FT\{S_x(t_1, t_2) - S_{ref}(t_1, t_2)\}$. Likewise, subtracting $S_{ref}(t_1, t_2)$ from $S_y(t_1, t_2)$ yields the phase purged y-component of the t_1 -FID.

To be accurate, some dispersive components remain in coupled spin systems along the diagonal. However, they always arise from higher order spin operators such as $I_x * I_{kz}$. These terms yield pairs of antiphase dispersive signals which cancel each other at a distance from the diagonal. By setting $\beta = 35^\circ$ the relative intensity of P.E.COSY is 40% relative to the standard COSY sequence, which is comparable to the double quantum filtered COSY.

Due to its simplicity, this variation of COSY might as well be called primitive E.COSY or P.E.COSY. The spectrum in Figure 2a demonstrates the absence of dispersive streaks in diagonal peaks.

Figure 2a.



Cross-Peak (3,2)

Figure 2b.

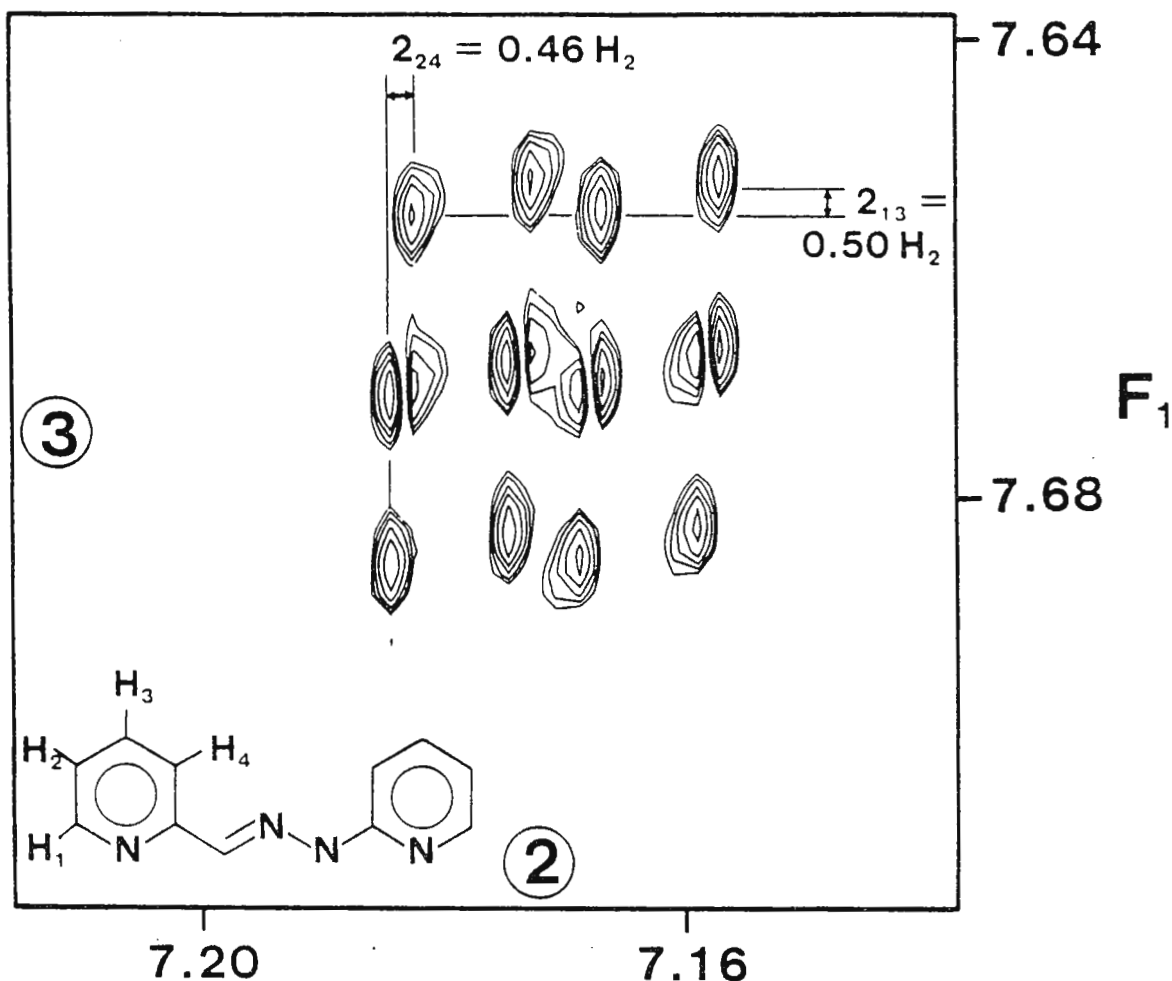


Figure 2 depicts the P.E.COSY spectrum of the compound indicated in the lower left corner of 2b. 2K-FIDS were recorded at 256 t_1 -values. Zero-filling resulted in a 2K * 2K spectrum with a digital resolution of 0.5 Hz. A 30° shifted sinebell was applied in each time domain before Fourier transformation. 2a) full spectrum, 2b) expansion around the cross-section between the resonances of H2 and H3.

This spectrum has been recorded on a JEOL GX500 spectrometer. Data manipulation was performed with D. Hare's magnificent FTNMR on a microVAX II.

Sincerely yours:

Luciano Mueller
Luciano Mueller

References: W. P. Aue, E. Bartholdi and R. R. Ernst, J. Chem. Phys. 64, 2229 (1976).

C. Griesinger, O. W. Sorensen, and R. R. Ernst, J. Amer. Chem. Soc. 107, 6394 (1985).

L. Mueller, J. Magn. Res. in press.

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Mailing Address:
 THE JOHNS HOPKINS HOSPITAL
 Baltimore, Md. 21205

19 January, 1987 (Received 27 January 1987)

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

A Novel method of double tuning an NMR coil for in vivo studies.

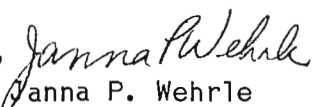
Dear Barry:

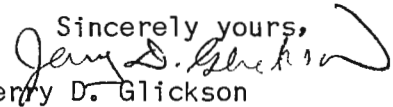
Several methods of constructing double tuned nmr probes have been reported in the literature. All these involve the use of additional LC networks. The use of an extra inductor almost always causes a decrease in the ratio of signal to noise. We report here a novel method of double tuning a circuit which involves the use of only one inductor (nmr coil).

A conventional single tuned probe, with a multiturn coil is first constructed. A variable capacitor is then connected across one or more turns of the coil. Addition of the extra capacitor essentially creates an extra pole in the frequency-impedance chart of the circuit, thereby creating a new resonance at a higher frequency. The performance at the lower frequency is virtually unaltered, whereas slight losses are incurred at the higher frequency. Conventional methods cannot be used to double tune a three turn (diameter 1.6 cm) coil to 145.8 and 360 MHz., due to excessive inductance.

We routinely use this technique to double tune our solenoidal coil to 145.8 and 360 MHz (31P and 1H respectively). This method has also been employed to obtain proton decoupled 13-C spectra. Shown below are representative 31-P, 1-H and 13-C spectra obtained from a murine fibrosarcoma. Figures: 1) 31-P spectrum of mouse tumor (RIF-1), 145.8 MHz., 200 scans, 3 sec recycle time. 2) 1-H spectrum of the same tumor obtained with water suppression echo sequence, 64 scans, 2 sec recycle time and 68 msec echo time. 3) 13-C spectrum obtained with a bilevel proton irradiation (60 mWatts-5 sec for NOE, 1.5 Watts-36 msec for decoupling), 128 scans, 5 sec recycle time.


 Sunder S. Rajan


 Janna P. Wehrle

Sincerely yours,

 Jerry D. Glickson

Please credit this contribution to Dr Glickson's account.

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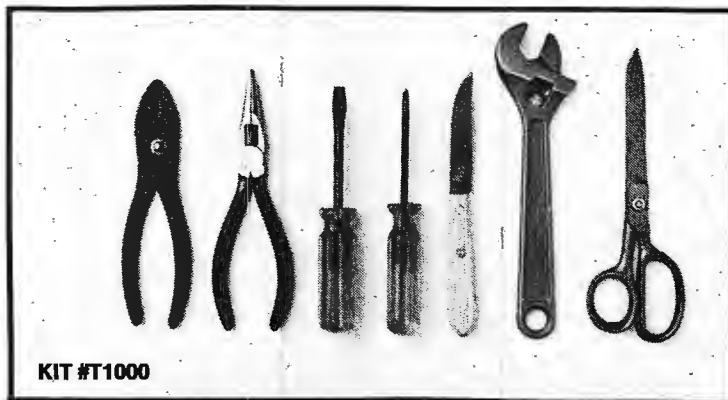
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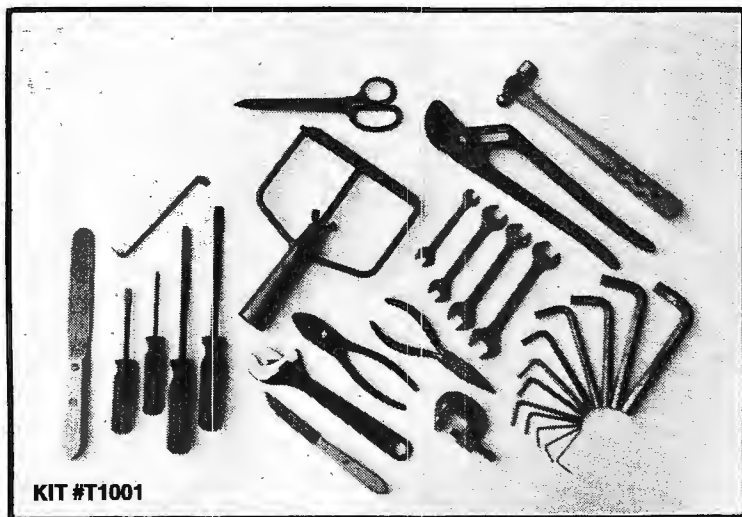
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7 Piece Basic Hand Tool Kit

- Screwdriver, standard, 6½" length
- Screwdriver, phillips, 6½" length
- Wrench, adjustable, 1½" opening
- Pliers, combination, 6½" length
- Pliers, longnose, 7" length
- Scissors, 8" length
- Knife, common, 3½" blade



KIT #T1000



KIT #T1001

KIT #T1001

31 Piece Deluxe Hand Tool Kit

Screwdrivers

- Standard, 6½" blade
- Standard, 10½" blade
- Phillips, 6½" blade
- Phillips, 10½" blade
- Offset, standard, 5" length

Combination

Wrenches

- ¼" X 5/16"
- 3/8" X 7/16"
- 7/16" X 1/2"
- 1/2" X 5/8"

Allen Wrenches

- 1/16"
- 5/64"
- 3/32"
- 1/8"
- 5/32"
- 3/16"
- 7/32"
- 1/2"
- 7/32"
- 1/4"
- 5/16"
- 3/8"
- 7/16"
- 1/2"

**KIT #T1002 - 9 piece hex
nut driver set. SAE**

- 1/8"
- 5/32"
- 3/16"
- 7/16"
- 7/32"
- 1/4"
- 5/16"
- 3/8"
- 7/16"
- 1/2"

**KIT #T1002M - 9 piece hex
nut driver set. Metric**

- 3.5mm
- 4mm
- 5mm
- 6mm
- 7mm
- 8mm
- 9mm
- 10mm
- 11mm

**KIT #T1003 - 10 piece ¼"
drive socket set. SAE**

- Ratchet wrench, reversible
- Extension bar, 4" length
- Socket, 3/16"
- Socket, 7/32"
- Socket, 1/4"
- Socket, 9/32"
- Socket, 5/16"
- Socket, 11/32"
- Socket, 3/8"
- Socket, 7/16"

**KIT #T1003M - 10 piece
¼" drive socket set. Metric**

- Ratchet wrench, reversible
- Extension bar, 4" length
- Socket, 4mm
- Socket, 5mm
- Socket, 5.5mm
- Socket, 6mm
- Socket, 7mm
- Socket, 8mm
- Socket, 9mm
- Socket, 10mm

Pliers

- Combination, 6½" length
- Groove joint, 12" length
- Long nose, side cut, 7" length

Miscellaneous

- Wrench, adjustable, 1½" open
- Scissors, cutting, 8" length
- Mop holder, floor wash, 8"
- Spatula, 1" x 5¾" blade
- Knife, common, 3½" blade
- C clamp, ¾" opening
- Hammer, ball peen, 4 oz. head

KIT #T1001M

**30 Piece Deluxe Metric Hand
Hand Tool Kit**

Same as T1001 with exception of:

- Allen Wrenches 2mm thru 16mm 9 pieces
- Combination Wrenches 6mm thru 16mm 6 pieces

FIG. 1

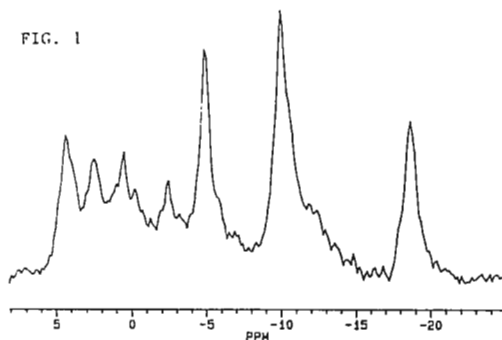


FIG. 2

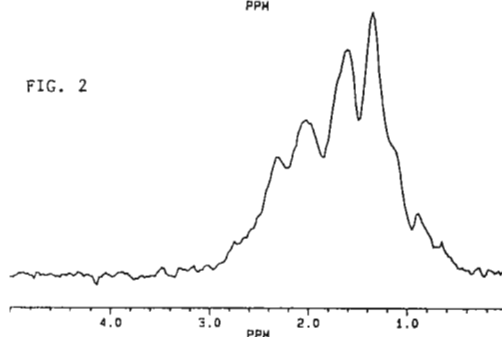
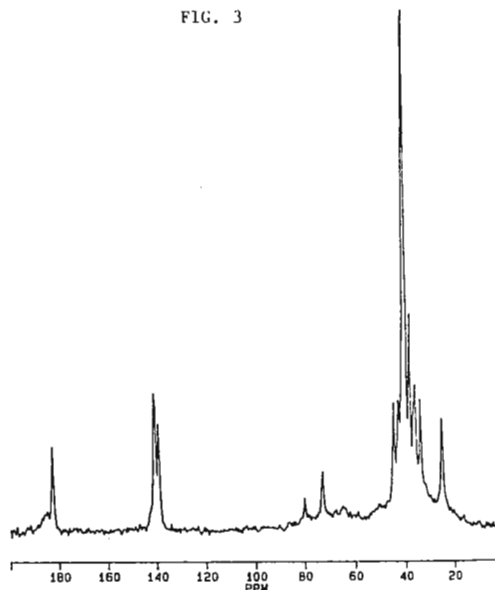


FIG. 3



"Chronology Revisited," or "Too Many Cooksons Spoil the Broth"

memo

To: Barry Shapiro*

DATE: 1/29/87

FROM: Alan Marchand (North Texas State University)

I received the January, 1987 issue of TAMUNMR newsletter with mixed emotions. Whereas I was pleased to note the inclusion of our article on page 10, I also noted with some consternation the substitution of a lengthy IUPAC compound name by the socially regrettable common name "Marchandione". Whereas I sympathize with the space restrictions that led to your action, I must protest that my current title of "Mr. Humble" is jeopardized by your choice of non-euphonious appellations (to paraphrase a well-known organic chemist). Of perhaps greater concern, we were not the first chemists to synthesize this cage dione; (it was first synthesized by Professor Cookson in England in the mid-1960's). Let's hope that an understanding readership will take all of this with a rock of salt.

All best wishes.

*Perpetrator of the "Marchandione" title. Mea culpa. Also the perpetrator of the titles of this clarification memo.



CH-3012 Bern, Freiestrasse 3
Telefon 031 65 43 11

Bern, January 20, 1987
(Received 27 January 1987)

Dr. Barry Shapiro
TAMU NMR Newsletter
Texas A & M University
Dept. of Chemistry
College Station, TX 77843

Postdoctoral NMR position available at August 1, 1987 or later

Dear Dr. Shapiro

In our NMR group at the University of Berne a postdoctoral research associate position is available for an applicant with Ph.D. in chemistry, with extensive experience in magnetic resonance, particularly modern pulse techniques and a strong interest in chemical structure problems. The candidate should be cooperative and experienced in computational analysis as well as in interpretation of NMR data.

The position opens at August 1, 1987 or later and is free for a 1-2 years period, provided that the working permit (obligatory for non-Swiss candidates) can be obtained.

The candidate is expected to participate in NMR research projects and will have to solve complicated structural problems of the various research groups in our institutes. He will further assist in teaching and in maintenance of our NMR systems.

The NMR laboratory includes a BRUKER AM 400 wide bore spectrometer, equipped with multi-nuclear capabilities, with an ASPECT 3000 (process controller) data system and connected to an autonomous data station, a 100 MHz (VARIAN XL-100) machine and several low-field CW-spectrometers.

Interested applicants should submit two letters of reference, a summary of their thesis, a list of publications and a curriculum vitae.

Sincerely yours

Peter Bigler

Mailing address:

Dr. P. Bigler
NMR Laboratory, Institute of Organic Chemistry
University of Berne
Freiestr. 3
CH-3012 Berne, Switzerland

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

(Received 30 January 1987)

DATE: 19870122/kt

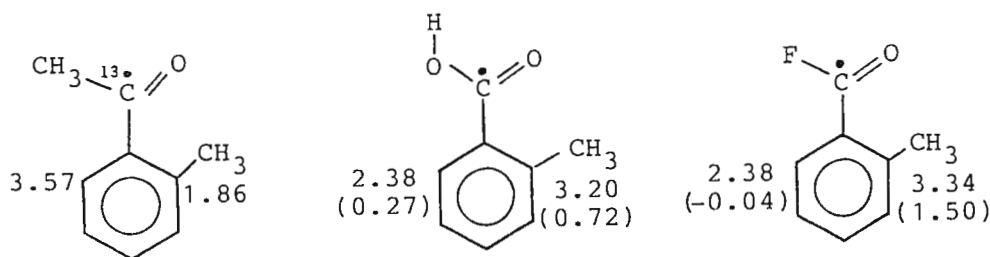
OUR REF:

YOUR REF:

ACID FLUORIDES

Dear Professor Shapiro,

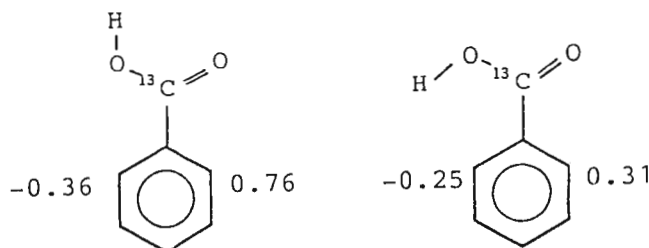
The use of two-bond carbon-carbon coupling constants and their change in magnitude in aromatic carbonyl compounds as a function of the direction of the C=O group has interested us for some time¹⁻³. Recently we (Drs. Schaumburg, Berg and I) have extended the investigation to acid fluorides. The trends observed previously are fully confirmed as seen below



$^2J(C=O, C)$

values in brackets are from theoretical calculation

The change in $^2J(C=O, C)$ is clearly linked to the nature of X-substituent (elektronegativity). Theoretical calculations (INDO-SOS) show that other factors such as the orientation of lone-pairs are important



$^2J(C=O, C)$ theoretical values

ROSKILDE UNIVERSITETSCENTER

We believe that these findings concerning $^2J(\text{CO}, \text{C})$ are valid in general. The results from carbohydrates⁴ seem to confirm this.

In a recent paper we investigated $^3\Delta\text{F}(\text{D})$ and $^2\Delta\text{C}=\text{O}(\text{D})$ of the $\text{CD}_{3-x}\text{H}_x\text{COF}$ isotopomers⁵ and found a directional dependence of $^3\Delta\text{F}(\text{D})$. We have now looked at $\text{CH}_3\text{C}^{18}\text{OF}$. The $^1\Delta\text{C}=\text{O}(^{18}\text{O})$ is small, 0.0385 ppm. This value does not fit well the relation given by Everett⁶. We are presently wondering why νCO is so high and $^1\Delta\text{C}(\text{O})$ so low. Another interesting finding is $^2\Delta\text{F}(^{18}\text{O})$. This isotope effect has only been predicted in COF_2 , but never determined. It turns out to be 0.0383 ppm in acetyl fluoride.

I am still very much interested in isotope effects on nuclear shielding and would be happy to receive any reprint or preprint on this subject.

Happy New Year,



Poul Erik Hansen

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1. P. E. Hansen, O. K. Poulsen and A. Berg, Org. Magn. Reson. 11, 649 (1977).
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Delft University of Technology

Department of Applied Physics

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2628 CJ Delft, The Netherlands
University switch board:
(015) 789111
Telex 38151 bhthd nl(Received 30 January
1987)Prof. B.L.Shapiro
Dept. of Chemistry
Texas A&M University
College Station, TX 77843 USA

Your reference and date

Our reference

Office telephone

Date

(015) 78

Subject

Sub-division

Dear Professor Shapiro,

Getting reproducible in-vivo NMR (NMRS) spectra of the brain of the conscious rat.

To enable reliable quantitative use of NMRS results, one should take care to establish reproducible experimental conditions, for instance:

- preparation and condition of the animal
- size and position of the localized volume
- sensitivity of the receiver coil
- RF homogeneity
- B₀ homogeneity
- chemical shift selectivity
- NMR experimental parameters
- signal processing
- quantification of results

For some of our experiments unanesthetized animals are used for medical reasons. In this case motions of the animal may seriously interfere with getting quantitative NMRS results. In collaboration with drs. Chamuleau and Deutz of the academic hospital of the university of Amsterdam we perform H-1 and P-31 NMRS on the brain of the conscious rat, with the aim of quantifying changes in metabolite concentrations during developing Hepatic Encephalopathy.


For P-31 we use chronically implanted surface coils to minimize the effects of motions, in addition to a good body fixation (1). As phase cycling schemes are rather sensitive to motions of the animal, we most times do not use depth pulses (2) to localize, but simple FID like experiments, with certain combinations of pulse angles and repetition times (3).


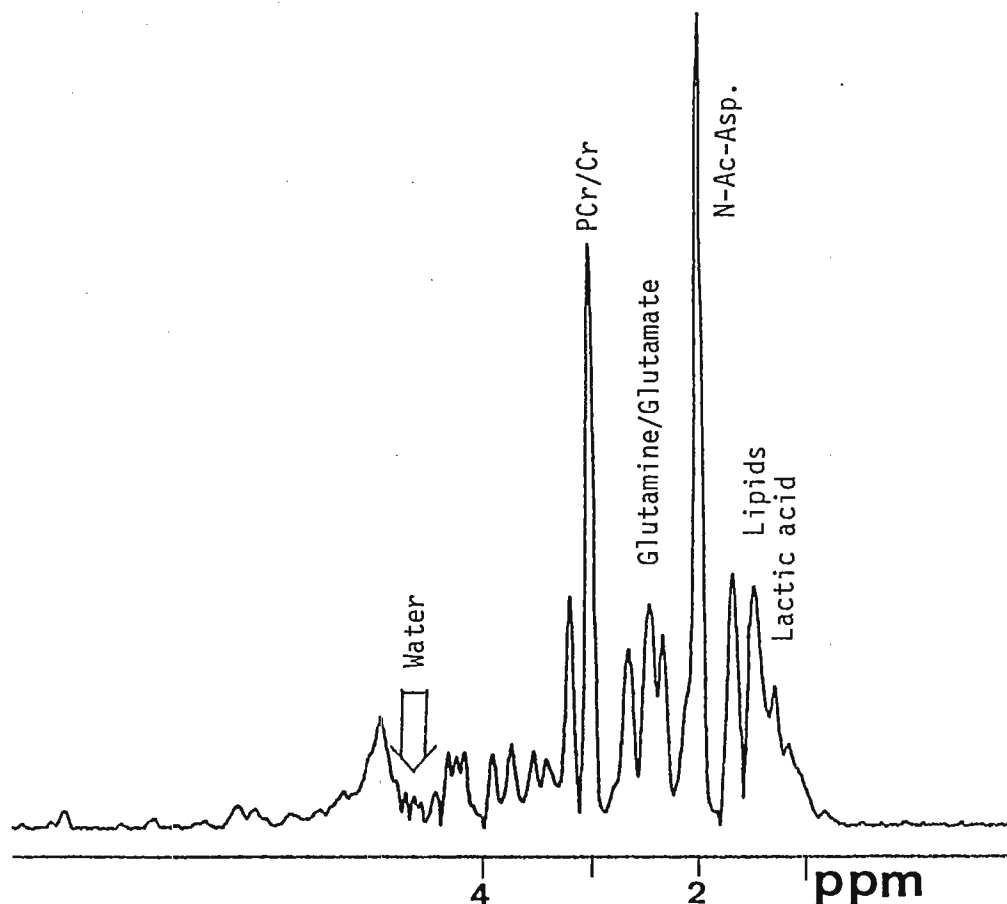
H-1 NMRS is much harder to perform because of the intense water peak and the presence of many overlapping lines. Movements present a major problem. They severely degrade water suppression and spectral editing. For instance, the Hore (4) sequences e.g. 1331-2662 in combination with phasecycling give in this case not only a reduced water suppression, but also misleading spectral amplitude variations in regions with a steep

gradient of the chemical shift selectivity. In the moment we have optimized our technique to such an extent that we get reproducible NMRS spectra of the cortex of the rat brain, using the 11-22 sequence, EXOR- phasecycling, and proper selection of the pulse angles. An example is given in the figure.

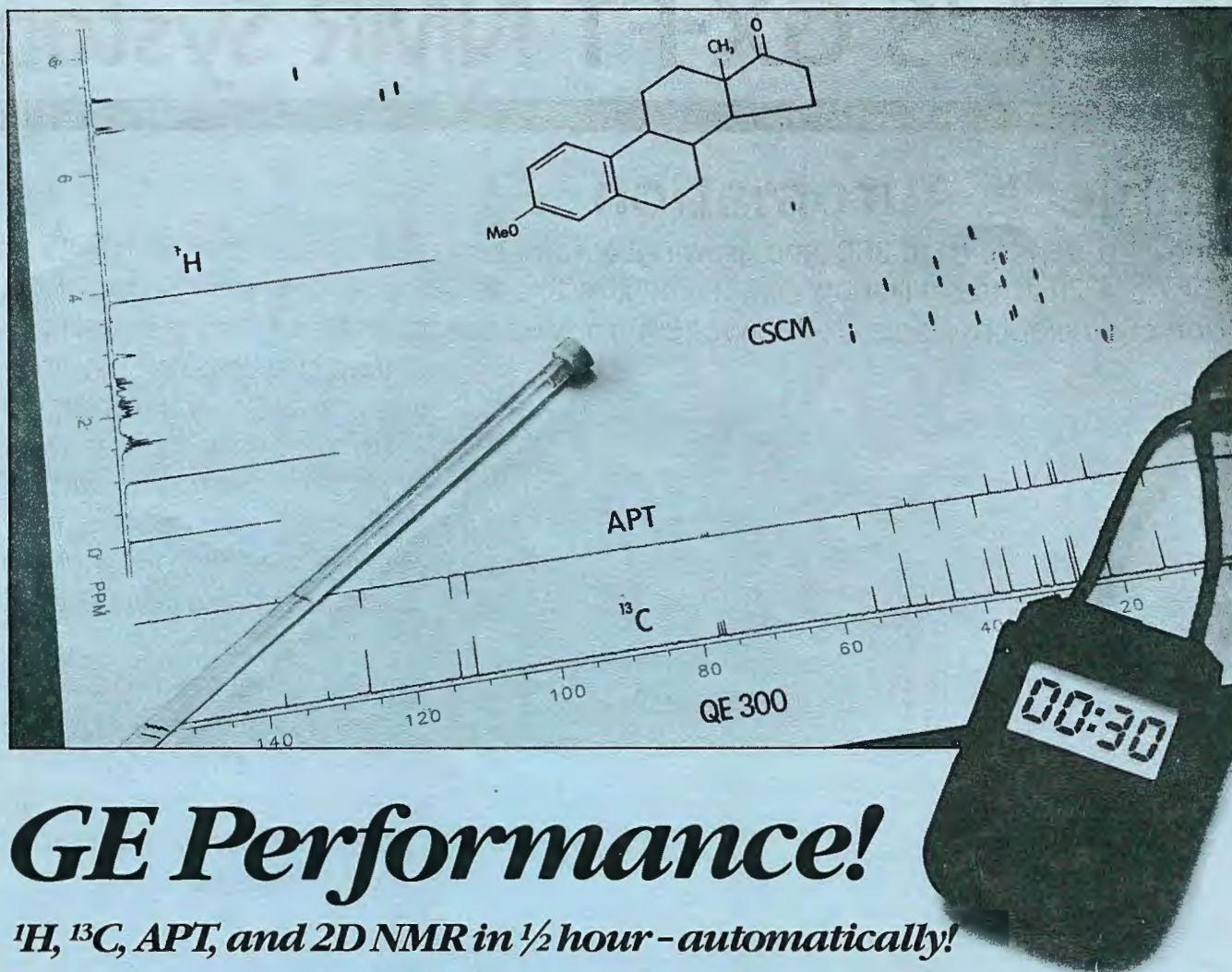
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1. N.Deutz, W.Bovee, R.Chamuleau, J.Neurosci.Meth. 16 157, (1986).
2. M.Bendall, D.Pegg, Magn.Res.Med. 2, 91, (1985).
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4. P.Hore, J.Magn.Res. 55, 283, (1985).


 Wim Bovee


 Albert de Graaf


In vivo proton NMR spectrum of the cortex of the brain of a rat at 7T.



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faster than any other
NMR spectrometer.**

A ¹H spectrum, ¹³C spectrum, an attached proton test (APT), and a ¹H-¹³C chemical shift correlation map (CSCM). All these analyses can be performed in as little as 1/2 hour, on as little as 50 mg. of sample, for most organic compounds. And the QE-300 does them all - automatically.

**With the NMR industry's
most advanced automation.**

This performance is made possible by the QE-300's automated software, hardware, and powerful MACRO programming capability.

Set-up starts with *Autolock*. Lock on as little as 10% CDCl₃ in a 5 mm tube.

Use *Compushim* for touching-up spinning shims or complete shimming with both spinning and non-spinning gradients using

the lock signal or observe FID.

Autogain optimizes the receiver gain independently for sequential ¹H and ¹³C acquisition.

After data acquisition, *Autophase* accurately phases ¹H and ¹³C spectra.

And finally, the analysis is completed with *Autointegrate*.

All these routines can be called up from QE-300 MACROs. In fact, any QE-300 operation, including pulse programs, can be implemented via MACROs for automatic, unattended sample analysis.

**And the most complete package
of hardware accessories.**

The QE-300 is available with the industry's most reliable, highest capacity (100 positions!) *Automatic Sample Changer*. Plus, you can add an array processor, a variety of hard disks, and switchable probes for even higher sample throughput and performance.

**Structural
elucidation simplified.**

For many organic molecules, the four experiments presented above will be all you need to determine or confirm molecular structure. For more complex applications, GE/NMR offers an extensive ¹³C library with outstanding search capability. This library contains data from over 10,000 compounds and is currently being expanded using a QE-300 in operation at the Aldrich Chemical Company.

**High throughput and
performance demonstrated.**

Get all the facts on the GE/NMR QE-300. Better yet, arrange for a demonstration. Call the GE/NMR group at (415) 490-8310. Or write General Electric Company, NMR Instruments, 255 Fourier Avenue, Fremont, CA 94539.

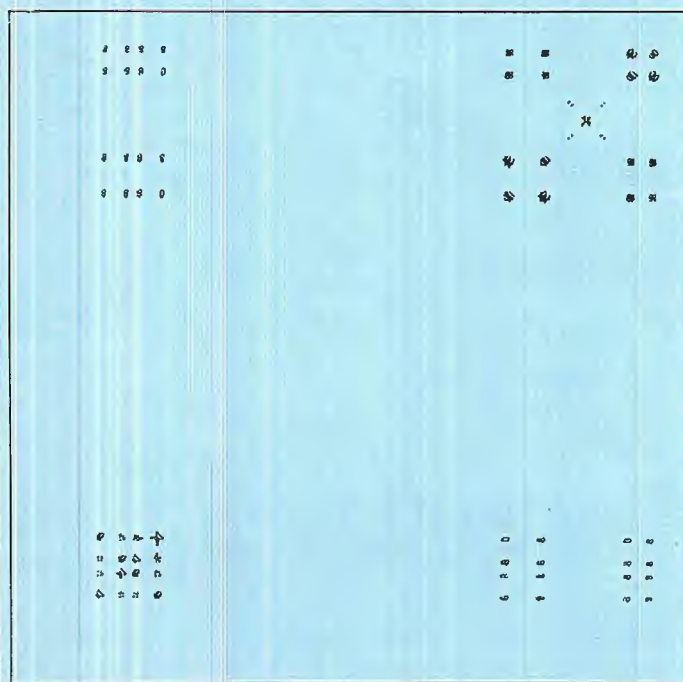
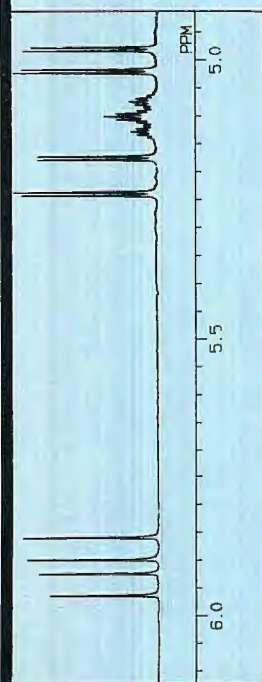
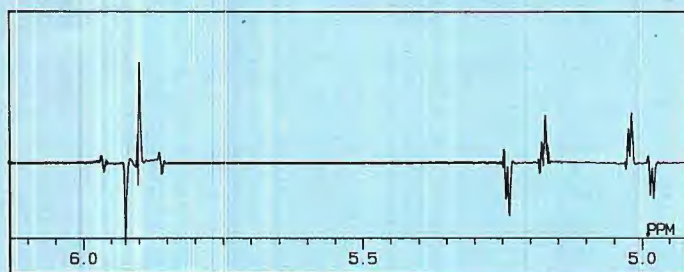
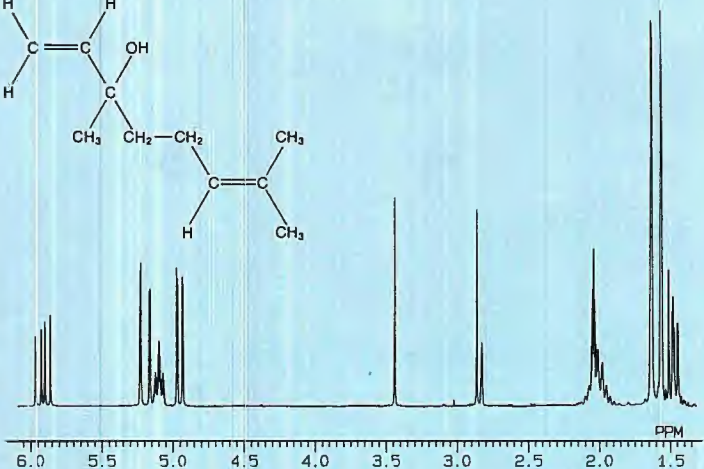
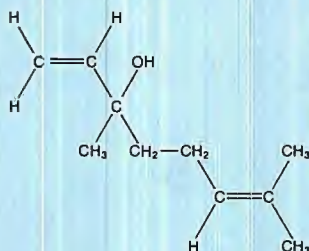
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Subject: Automation

One of the more than 200 (and growing) automated routines available on the GX-Series; the data illustrates a Double Quantum Filter Phase Sensitive Cosy on the downfield section of Linalool run on a GX-270/WB (89mm) MHz spectrometer. From the moment you

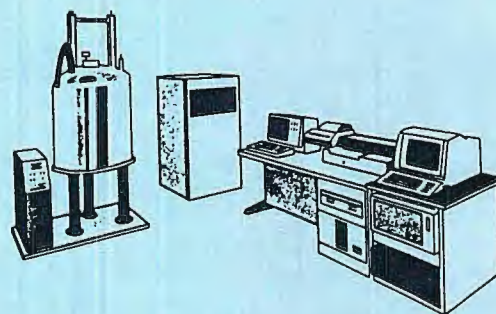
Linalool



load the sample, spinning, lock, shimming, acquisition, transform, phase correction, and plotting are totally automated. Should you need something not already in our menu a few strokes on the keyboard will put it there.

So whether your requirements are for routine or research, the GX-FT NMR is an instrument that you should consider when evaluating FT-NMR Systems.

The GX Series FT-NMR Spectrometer



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