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ADDITIONAL LISTINGS OF MEETINGS, ETC., ARE INVITED.

DEADLINE DATES

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.
Dear Prof. Shapiro,

Present expectations are that high resolution NMR spectroscopy may offer powerful approaches to investigate some metabolic alterations associated with either neoplastic growth and/or with effects of antitumoral treatment. In particular experimental evidence is now growing pointing to the interest of investigating alterations in tumors of the pool sizes of phospholipid metabolites, as they may represent well defined biochemical responses of cells to factors regulating their conditions of growth and proliferative capability (1).

It is known that tumor 31P NMR spectra generally exhibit conspicuous resonances arising from phospholipid metabolites, such as phosphorylcholine (PCho) and phosphorylethanolamine (PEtn) in the phosphomonoester region and glycerophosphorylcholine (GroPCho) and glycerophosphorylethanolamine (GroPEtn) in the phosphodiester region. On the other hand, the N(CH3)3 peak of choline can also be clearly detected in 1H NMR spectra.

Previous studies in our laboratories, carried out on solid tumors grown in DBA/2 mice after s.c. injection of Friend erythroleukemia cells (FLC), and in vivo treated with murine tumor necrosis factor (TNF) showed that this antitumor agent induces considerable decreases in the pool sizes of intratumoral GroPCho, GroPEtn, PCho and PEtn, and a conspicuous increase in the concentration of α-glycerophosphate. Combined 31P and 1H NMR spectroscopy recently demonstrated that significant alterations also occur in TNF treated tumors at the level of the intratumoral concentrations of choline and of the ratio [PCho]/[Cho].

Figure 1 (top) shows 31P and 1H NMR spectra obtained at 9.4 T from ethanolic extracts of FLC (clone 3Cl-8) tumors, 14 days after implantation, dissected from mice 6 h after intratumoral injection of TNF (4 µg/mouse). The spectra of control tumors (treated with bovine serum albumin, BSA) are shown at the bottom. Tissues were frozen at liquid N2 temperature immediately after excision and then extracted by EtOH:H2O, 60:40 vol/vol.

Analysis of the 1H NMR peak areas of choline N(CH3)3 group (normalized to one gram of tissue wet weight) indicated a 4-fold increase in the concentration of this phospholipid precursor in TNF-treated tumors vs control tumors. The combined analysis of 31P and 1H NMR spectra showed that the [PCho]/[Cho] ratio decreased by a factor of about 18 in TNF-treated tumors.

These results would suggest that the antitumor effects of TNF are associated with substantial alterations of several reactions of the major metabolic pathways for the biosynthesis of phospholipids, both at the level of end-products (GroPCho, α-glycerophosphate) formation as well as at that of precursors metabolic conversions (such as that controlled by choline kinase).
We thank Prof. W. Fiers, the State University of Ghent, Ghent, Belgium, for kindly providing tumor necrosis factor. This work was partially supported by CNR Special Projects Biomedical and Clinical Engineering n. 85.01516.57 and Oncology n. 85.02560.44.

References
September 22, 1986

(Received October 2, 1986)

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

Dear Dr. Shapiro:

We have recently employed 2D Homonuclear Hartmann-Hahn (HOHAHA) experiments to aid in the assignment of the 1-H spectrum of the 33 amino acid antimicrobial protein NP-5. These experiments were performed on our Varian XL-400 spectrometer. The observe and decoupler channels on the spectrometer are phase locked which allowed us to use different power levels for the 90° pulse and the spin lock pulse. The 90° pulse was generated by the observe channel using the high power pulse amplifier. A 7.8 kHz RF field spin lock pulse was generated on the decoupler channel by adjusting the output of the broadband 1-H decoupling amplifier on the spectrometer. Our initial 2D experiments employed simple phase alteration of the spin lock pulse along the tX axis (1). This method proved unsatisfactory since it gave no better coherence transfer than a RELAYED-COSY experiment. We next employed a MLEV-17 pulse sequence (2) to reduce the decay of magnetization from T1R during the spin lock period. The standard XL-400 spectrometer cannot perform multipulse experiments, but Varian provided us with the necessary information to modify the output board so that it can execute "hardware looping". With this modification and Version 6.1 software the XL400 is able to perform the MLEV-17 pulse sequence. Figure 1 shows a contour plot of a portion of the phase sensitive 2D HOHAHA experiment on NP-5. A 72 ms spin lock pulse was used to transfer coherence through a number of protons on the amino acid side chains in NP-5. The connectivities on two amino acids, Leu-29 and Val-25, are illustrated in the figure. Coherence was transferred from the Cα protons through the Cβ protons to the Cγ and Cδ protons on these two residues. We were unable to observe any significant transfer of magnetization from Cα to Cδ protons in a DOUBLE RELAYED-COSY experiment and thus conclude that the MLEV-17 based HOHAHA experiment is superior to multiple-relay experiments for spin system assignments in proteins. A detailed discussion of the complete resonance assignment of NP-5 will appear elsewhere.
J. Am. Chem. Soc. 107, 2820-2821.


Figure 1. Contour plot of a phase sensitive 2D ROESY spectrum of 7mN P5 in 
D2O at 20°C. Quadrature detection was used in both the $t_1$ and $t_2$ time domains. 
The spectrum was acquired with 512 complex points in $t_2$, 256 complex FIDs in $t_1$ 
and 64 transients for each FID. The $t_1$ and $t_2$ data sets were zero-filled to 1K 
complex points before Fourier transformation. The CO resonances appear 
along the $\omega_2$ axis while the $\text{C}^\beta$, $\text{Cy}$ and $\text{C}^\delta$ chemical shifts appear along the $\omega_1$ 
axis. Connectivities from the $\text{Ca}$ proton to the $\text{C}^\beta$ and $\text{C}$ methyl protons are 
shown for Val25. Similarly, connectivities from the $\text{Ca}$ proton to all the side 
chain protons are shown on Leu29.

Sincerely yours,

Arthur Pardi

Alvin C. Bach, II
Dear Barry,

Now that the popularity of phase sensitive COSY spectra [D. Marion and K. Wuthrich. BBRC 113 967 '83] is spreading, I'd like to draw attention to the potential advantages of the double dispersion mode [for a picture see Prog. NMR Spectrosc. 17 p341 '85]. There is little difference in the S/N ratio as compared with the double absorption for lines which are not well digitised [J. Magn. Reson. 61 p48 '85]. However, if spin coupled multiplets are poorly resolved, the pure absorption lineshapes interfere destructively [cf. J. Magn. Reson. 53 p269 '83] whereas the pure dispersion builds up intensity at the centre of the multiplet and therefore gives better sensitivity [Prog. NMR Spectrosc. 17 p350 '85]. The biggest advantage may be that while the cross peaks are double dispersions, the diagonal peaks are double absorptions [W. P. Aue, E. Bartholdi and R. R. Ernst, J. Chem. Phys. 64 2229 '76] and the tails are therefore much less obtrusive. As a result, the spectra look a little like the double-quantum filtered COSY, but the sensitivity is much higher. The example below is taken from the aromatic region of a 10kD protein and shows both positive and negative levels. In closing, I should stress that this information can be pieced together from the literature and that it does not apply to experiments with net magnetisation transfer such as NOESY.

With best wishes,

David Turner

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

"A Novel Equilibrium"

Dear Barry:

As you well know, one of my hobbies is the examination of complex equilibria by NMR methods. The most practical application of this in my work is the examination of the fast-exchange equilibria encountered with LSR's.

LSR's complex via a simple two-step mechanism, $L + S = LS$ and $LS + S = LS_2$. Most studies carried out in analyzing this equilibrium system are routine and straightforward and, as would be expected, $K_1$ is greater than $K_2$. That is, one would expect the second ligand to be less well-bound than the first. Exceptions to this rule are quite rare.

In some work I am doing in collaboration with Alan Marchand at North Texas State University, we have found an exception to this rule. In this case the LSR was Eu(fod)$_3$ and the substrate was the following:

![Diagram of Eu(fod)$_3$ complex]

In this case it was found that $K_1 = 4$ and $K_2 = 30$. Statistical analyses of these data shown these numbers to be well within experimental error. Thus, a rather novel equilibrium system has been uncovered. We are currently working on explaining of this phenomenon.

Sincerely yours,

Milton D. Johnston, Jr.
September 26, 1986 (Received October 13, 1986)

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

Dear Dr. Shapiro:

In our first letter to your newsletter, we will describe our magnetic resonance spectroscopy and imaging program at the Veterans Administration Medical Center, University of California, San Francisco.

I am a physician interested in developing and using NMR spectroscopy techniques for investigating the regulation of metabolism in animals and human subjects; specifically, to determine the metabolic alterations which occur in disease and to develop NMR techniques for clinical investigation and diagnosis.

We have been fortunate to acquire excellent instrumentation which is well suited for this purpose. We have a Philips Medical Systems 1 meter bore 2.0 Tesla NMR imaging/spectroscopy system which is broad-banded and capable of obtaining high resolution NMR spectra from localized volumes within the human body. We also have a General Electric CSI 2 Tesla animal system.

Dr. Gerald Matson, Ph.D., recently recruited from the University of California, Davis, is directing an effort to develop further spectroscopic localization techniques. These studies are being performed in close collaboration with Dr. Jan den Hollander at Philips Medical Systems in Eindhoven, Holland, who is working on pulsed gradient localization techniques such as SPARS and ISIS. These techniques have been implemented in our laboratory. Furthermore, Dr. Robin Bendall of Griffiths University in Australia will be spending three months in our laboratory this winter and will be working with Dr. Matson to implement a variety of $B_1$ localization techniques such as the rotating frame experiment and depth pulsing. Other scientists in our laboratory are working on computer simulations of these various localization techniques, and development of specialized coils for spectroscopy on human subjects. This year we have several scientists joining us on sabbatical leave including Dr. Klaus Roth from the Free University of Berlin and Dr. Yasuo Nagai from Takeda Pharmaceuticals in Osaka, Japan. The program is supported by the National Institutes of Health, the Veterans Administration Research Service, and Philips Medical Systems.

We are interested in recruiting postdoctoral fellows and junior faculty who have a background in NMR spectroscopy and who wish to...
become involved in various aspects of in vivo spectroscopy and imaging. I am collaborating with a number of cardiologists, neurologists, neurosurgeons and radiologists to apply these techniques to a wide variety of human diseases and animal models. We also have a particular need for physicists or engineers with an emphasis in instrumentation, especially rf coil design.

The acquisition of the equipment described above, and a team of physicians and spectroscopists, provides us with an interesting opportunity to explore the many possibilities of NMR to the investigation of human metabolism, and ultimately for clinical diagnosis.

In future letters we will provide progress reports on our work.

With kindest personal regards.

Sincerely yours,

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

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Succinic Acid Conformations as a Function of pH

Dear Barry,

Your strident call for the mandatory contributions to TAMU NMR came during continuing magnet and computer problems with the GE 4.7-T CSI unit installed at the Huntington Medical Research Institutes, which we had hoped to employ for some stirring research this summer. It has not been possible to stir anything, so this will be a report on a minor NMR project.

For some years, I have been interested in conformational changes which occur in acid-base ionizations and, lately, I decided I needed to know what happens in the case of succinic acid. V. Gil and coworkers, *Tetrahedron*, 37, 611-614 (1981), did a nice job of measuring the proton-proton couplings in the $^{13}$C satellites as a function of pH, but in the analysis of their results, did not determine the couplings which are characteristic of the monoanion. Indeed, their analysis of the % of the various conformations as a function of pH shows rather large error bars in the region where the monoanion is expected to predominate.

Gil reports that one of the proton-proton couplings changes by 6 Hz over the pH range and the other by only about 1.3 Hz. This coupling with the larger pH change, designated by Gil as $3J_{14}$, could be readily analyzed by the procedure we used earlier for the ionization of urocanic acid, *J. Am. Chem. Soc.*, 104, 3945-3949 (1981), where the only inputs are the respective couplings at each pH and the couplings at the extremes, which correspond to un-ionized and fully ionized acid. The computer takes this input and thrashes around trying for the best possible fit to the data for the first and second ionizations and the couplings of the monoanion. Ten data points yielded $pK_1 = 4.1$ and $pK_2 = 5.3$, and $3J_{14}$ for the monoanion as 6.1 Hz. The fit was excellent, with a correlation coefficient of 0.997. The literature $pK_A$ values are 4.1 and 5.5, respectively. Forcing the literature $pK_A$ values onto the fit, made $3J_{14} = 6.6$ Hz and the correlation coefficient 0.995. The change of $3J_{24}$ with pH is too small to treat very accurately in the same way, but assuming $pK_1 = 4.1$ and $pK_2 = 5.3$, then $3J_{24}$ comes out as 7.7 Hz, with a correlation coefficient of 0.973.
The increase in $^3J_{14}$ suggests that monoionization of succinic acid leads to an increase in preference for conformation 1 with carboxyls trans, which is somewhat surprising because of the possibility of hydrogen bonding between un-ionized CO$_2$H and CO$_2^-$.

The Gil analysis of the conformational equilibria has some further difficulties in that it assumes that the coupling constants of 1 and 3 are independent of whether the carboxyls are un-ionized, monoionized or diionized. Certainly, 3 could well have an expanded CO$_2^-$–CO$_2^-$ distance when ionized, as compared to monoionized or un-ionized 3. There are also some possible additional ambiguities about the assignments of the couplings, and it would seem desirable to have a look at the couplings in meso-1,2-dideuteriosuccinic acid as a function of pH, which I hope we can do.

With all good wishes,

Very truly yours,

Jack

Fit of experimental points for change of $^3J_{14}$ for succinic acid with pH.
September 25, 1986 (Received October 13, 1986)

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

Dear Professor Shapiro:

SUBJECT: Early experiences on a new AM500

We have recently completed the installation of a Bruker AM500 at the Kodak Research laboratories. Here are a few notes from our early days.

We have received VAXCOM from ABACUS (c/o Syd Brownstein and Jack Bornais) to allow communications between our microvax II and the AM500. Vaxcom is written for the Silent 700 terminal; whereas we have the LCD terminal. To overcome this problem, we operate the console off port B and attached a VT200 to channel A. Channel A is converted to a baud rate of 300 on the I/O board. The cable connections are shown below.

For most operations we run on channel B from the keyboard, but for vax communications we switch port B to the vax and use the VT220 as a vax terminal running the program VAXCOM. I admit this is somewhat of a band aid but it got us going.

[Diagram of cable connections between VT220 and CHANNEL A]
Although veteran AM users know this the new user will fall into this one! On the new console, the alpha lock key is easily hit while adjusting the A, B, C, and D knobs. This causes approximately 1/2 of DISNMRP to fail to respond most notably in EP mode. For the uninitiated this takes a day to solve! As a note, IBM tells me they hard-wire the alphalock key.

Finally, a note on probes. Dennis Hare had mentioned to me that different style proton probes at 500 MHz had shown different abilities to handle presaturation of 90% H2O solutions. Bruker kindly lent us one of their older probes and I'm pleased to say our new probe was significantly better at producing a good suppression of the water peak without humping on the low field side. This is not a statistical survey but for those thinking of handling high water containing samples it may be worth noting.

Sincerely,

Richard N. Moore
Corporate Research Laboratories

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

TITLE: SOLID STATE NMR STUDY OF BISMUTH(III) XANTHATES

Dear Prof. Shapiro,

As a part of an ongoing study of the coordination of the xanthate ion (S2CO2-) with the main group elements, the xanthates of bismuth(III) have been prepared and their properties investigated.

In the solid state Bi(S2CO2Pr)3 exists as a polymer, as evidenced by an X-ray study; the polymer arising as a result of substantial intermolecular Bi-S interactions. Further, two of the S2CO2Pr anions (which do not participate in the generation of the polymer) are related to each other across a crystallographic mirror plane and therefore in the solid state there are only two unique S2CO2Pr moieties. If the methyl analogue, Bi(S2CO2Me)3, is isomorphous with the polymeric Bi(S2CO2Pr)3 derivative then, the solid state 13C nmr spectrum would be expected to show two resonances due to the methyl carbon atoms, and similarly only two thiocarbonyl resonances should be present.
The proton enhanced solid state $^{13}$C spectra were run on a Bruker CXP300 spectrometer with magic angle spinning, in macor rotors with delrin caps.

The spectrum (Fig. 1) of $\text{Bi(S}_2\text{COMe)}_3$ shows three methyl resonances ($6.62.6$, $63.7$, $64.8$ PPM) and two thiacarbonyl resonances ($6.224.1$, $225.7$ PPM); an observation inconsistent with the presence of a crystallographic mirror plane. A subsequent X-ray analysis has revealed that $\text{Bi(S}_2\text{COMe)}_3$ does possess a pseudo mirror plane but is not polymeric. The two xanthate moieties related across this plane coordinate the bismuth centre in a similar manner; and together are different from the third xanthate. We suggest that the thiacarbonyl carbons of the former two are insufficiently different to be resolved in the nmr experiment whereas the methyls (crystallographically inequivalent) give rise to three separate signals. It's nice to know that X-ray crystallographers and nmr spectroscopists can agree sometimes!

Please credit this contribution to the account of the NMR Centre, University of Adelaide.

Regards,

Ms A.M. Hounslow

Dr E.R.T. Treilink

Fig. 1. (SSB = spinning side band; D = delrin)
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Acknowledgement: Obtained in cooperation with the Department of Molecular Biophysics and Biochemistry, Yale University.

Bruker MR systems designed to solve problems.
BULK SUSCEPTIBILITY EFFECTS AGAIN IN SHIFT REAGENTS

Charlie Springer's recent letter to you concerning the importance of bulk magnetic susceptibility effects in determining the efficacy of shift reagents is borne out by some results we have recently obtained.

As an organic chemist it irks me (FGR) that the best aqueous shift reagents for the alkali metals are inorganic, with tripolyphosphate being the best of all. The three main essentials for the design of a good shift reagent are now known: there must be a ligand containing a large number of anionic sites that are relatively close together, the ligand must coordinate well with a triply charged lanthanide ion but less well with the singly charged alkali metal species, finally the complexes must be fairly water soluble. Further considerations are that the molecular weight of the complex should not be so big as to lead to too large a correlation time and the EFG it creates at the metal ion should not be too large. These factors lead to rapid quadrupolar relaxation and broad lines. We have one case where $^{23}$Na has a 3000Hz linewidth! Finally, the reagent should not be too expensive, a factor which precludes extensive synthetic work. We have been looking at some further possible organic contenders.

Butane 1,2,3,4-tetracarboxylic acid (BTCA) should be interesting as it fulfills the conditions laid down above. We perform our experiments in a slightly different way to Springer in that we add our lanthanide to the solution of the alkali metal salt of the shift reagent. When we added Dysprosium to the sodium salt of BTCA we observed small shifts to high frequency. We thought that we possibly had a new but poor shift reagent. However, checking back on the bulk magnetic susceptibility of the corresponding concentration of sodium chloride showed that what we were observing was totally accounted for by the bulk susceptibility.

Like Springer we have also looked at diethylenetriaminepentaacetate (DTPA) and find that it gives up to 4ppm shifts to high frequency at a molar ratio of just over 1:1 Dy$^3+$DTPA (300K). For triethylenetetraminehexaacetate we observe a maximum shift of ca 3.5ppm to low frequency again at a molar ratio of just over 1:1. These shifts are small in comparison with what is available with tripolyphosphate (ca 40 ppm) which makes it unlikely that they will replace the tripolyphosphate shift reagents.

We haven't yet given up hope of finding suitable organic ligands and are still searching for suitable simple candidates. We still have one or two tricks up our sleeves!

Best wishes,

(Frank G. Riddell)

(S. Arumugam)
A DUTY-CYCLE LIMITER FOR HIGH-POWER NMR

Dear Barry:

A recent letter (TAMU 331, p. 32) described a circuit designed to protect high-power amplifiers and probes from rf pulses which have been inadvertently set too long. We had need for similar protection for our home-built solid state spectrometer in the Regional Facility. However, simply limiting an individual pulse length was not a suitable approach, since in the cross-polarization experiment (and others) long pulses are acceptable, as long as the duty cycle is not too great. Therefore, we (JMY) designed a circuit (see Figure) which limits the total length of all pulses in a given interval (set to ca. 4s) to a preset value (ca. 100ms). When this duty cycle is exceeded, the pulses are disabled and a panel light comes on until manually reset.

This was most conveniently accomplished by utilizing the 5v signals which are used to blank our two high-power AR amplifiers, which go to zero when the amplifier is turned on. The circuit integrates these two blanking pulses and converts them to a DC level, which is fed to a comparator which in turn drives a TTL latch. The output from the latch is used to create a forced blanking condition.

The integrator A1 samples two TTL signals through R1,R2, which together with C1 determine the time constant of the circuit (in our case, approximately 4s, which is a typical recycle delay). P1 and P2 provide an adjustment to bias the ramp starting point and comparator A2 has a threshold level adjustment; all three are used to adjust the maximum total pulse length permitted (ca. 100ms in our case). A Zener diode is used to clamp the comparator to positive values of less than 5v, protecting the input of U1C. U1D is used as a power on reset and allows resetting of the latch by switch S1B.

In operation the TTL blanking levels are at +5v, causing the integrator output to remain near zero. During "unblanking" the output begins ramping towards -8v. If the unblanking continues beyond the preset comparator threshold value, the latch will trigger. Once the latch is triggered it remains on until the reset switch S1 is depressed.

Any questions about this device should be addressed to John Yehle at (818) 356-6069.

I hope that this communication will prevent our excommunication.

Sincerely yours,

James P. Yesinowski, Manager
John Yehle, Electronics Engineer
Southern California Regional NMR Facility
Carbon-13 Differential Line Broadening and the Absolute Sign of Carbon-Hydrogen Spin Coupling Constants

Recent literature reports of "strange asymmetries" in NMR spectra (and your pink reminder) prompt this letter. These asymmetries are sometimes the result of multiple relaxation processes which can constructively or destructively interfere with one another. In Figure 1 we have shown the proton-coupled $^{13}$C NMR spectra for the acetylene carbon in phenyl acetylene at 25°, -18° and -28° C. The integrated intensities of the two lines are equal for all temperatures, but the peak heights and line widths become increasingly different as the temperature decreases. As the temperature becomes lower, the molecular correlation time becomes longer and the differential line broadening (DLB) for the two lines of the doublet increases. This DLB has been observed with increasing frequency in the chemical literature (especially at magnetic fields of 9.5 tesla and higher). It arises from interference effects between dipolar and chemical shift anisotropy (CSA) relaxation. In the present case the broad line (at the higher frequency) has a much shorter $T_2$ value, and hence a greater width, than the narrow line.

These DLB effects occur under the following conditions:

1) High magnetic fields ($B_0 > 4.7$ tesla)

2) Long correlation times ($10^{-8} > \tau_c > 10^{-11}$) such as occur in polymers, at low temperature, or absorbed molecules (with restricted motion).

3) For nuclei with large CSA (e.g. $^{13}$C, $^{15}$N, $^{19}$F, $^{29}$Si, $^{31}$P, etc.), and in situations where the magnitude of the dipolar and CSA interactions are comparable and have the same correlation time.

(continued on page 25)
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If the sign of the carbon CSA is known, as it is for the acetylene carbon in phenylacetylene, then the absolute sign of the carbon-hydrogen spin coupling constant can be determined: it is positive, \( J_{C-H} = +252.1 \) Hz. The details of such observations and the theory of DLB will be published in 4 or 5 months in J. Am. Chem. Soc. Preprints will be available in a few weeks for anyone interested.

Best regards.

Thom C. Farrar
Professor of Chemistry
Professor Bernard L. Shapiro
TAMU NMR Newsletter
Chemistry Department
Texas A & M University
College Station, Texas 77843-3255

Postdoctoral and Visiting Scientist Programs at NRL

Dear Barry:

The Naval Research Laboratory has programs for both postdoctoral research fellows and visiting scientists.

There are two postdoctoral programs at NRL: the National Research Council (NRC) associateship and the Office of Naval Technology (ONT) fellowship, administered through the American Society for Engineering Education (ASEE). Each program selects applicants on a competitive basis. Relocation and professional travel allowances are provided. An immutable requirement is that the applicant be a U.S. citizen.

The NRC appointment is for two years; the stipend is expected to be $26,35K. A third year of contractual support from the Office of Naval Research may be available for NRC associates who continue research at an academic institution. There are two selection cycles yearly with deadlines of 15 January and 15 April 1987. Applications are available from:

Associateship Office, JH 608
National Research Council
2101 Constitution Avenue, N.W.
Washington, D.C. 20418
(202) 334-2760

The ONT program is newer and smaller than the NRC program. The ONT research projects are somewhat more applied than those of the NRC. ONT appointment is for one year, renewable for a second and possibly third year. The ONT stipends start at $31K. There are four award cycles with deadlines of 1 January, 1 April, 1 July and 1 October 1987. For applicant's packages, contact:

ASEE
Projects Office
11 Dupont Circle, Suite 200
Washington, DC 20036
(202) 293-7080
The Laboratory has a program for visiting faculty members on sabbatical or leave, under the provisions of the Intergovernmental Personnel Act (IPA). Depending on the circumstances, supplemental or full support may be available. An IPA agreement may cover any period from a few months to two years. Allowances are provided for moving expenses and travel to scientific meetings. There is also an ASEE summer fellow program which brings university faculty to NRL for a 10 week period.

Within the Polymer Diagnostics Section we address a wide range of problems through magnetic resonance. Present efforts and interests include: adsorbed molecules; NMR imaging in solids; mechanical properties of polymers; hydrodynamics of polymers in solution; NMR and ESR in phospholipids. We have a new joint program involving NMR on archaebacteria with the NRL Molecular Bioengineering Branch: someone with a more biological bent would be very helpful.

Please encourage anyone interested to contact me or Henry Resing directly and informally.

A. N. Garroway (202) 767-2323
H. A. Resing -2025

The January NRC deadline approaches rapidly.

Your assistance in publicizing these postdoctoral and visiting scientist programs is greatly appreciated.

Sincerely,

A. N. Garroway, Head
Polymer Diagnostics Section
Code 6122
Polymeric Materials Branch
Chemistry Division
In Vivo Surface Coil NMR Probe for Studies on Rats

We have recently designed an in vivo surface coil NMR probe (1) with a simple and efficient support system (Figure 1) for studies on rats. This probe was used (on our 4.7T Bruker CXP-200 system) for cardiac allograft rejection studies by $^{31}$P NMR (2,3) and the neurology pilot studies on the effect of total cerebral ischemia on water proton relaxation times (4).

The system consists of a single-sided, glass-epoxy printed circuit board (PCB) and a copper tube (hard-drawn 1/4 inch copper pipe). A small electronics shelf is soldered to the copper tube and the PCB to provide rigidity and a place for the various matching and tuning capacitors. The copper foil on the upper portion of the PCB may be etched away. The remaining copper foil provides a convenient ground for the coil and also shields the electronics of the probe from the animal's body.

If double-sided glass epoxy board is used, the side toward the animal may be used as a shield (if properly grounded) and the circuit running to the coil may be etched on the reverse side. Alternatively, a coaxial cable may be used for this circuit.

The method we used for etching away the copper foil uses plastic tape and a saturated solution of FeCl$_3$. The plastic tape masks the area of copper not to be removed. The PCB is agitated in a small beaker with the iron chloride solution until the etching is complete. After cleaning, the tape is removed and the board is ready for use.

The subject rat is placed against the coil which is attached to the flat side of the PCB. A small piece of nylon string or suture is then placed behind the two upper front incisors of the rat's mouth and passed through a small hole in the copper tube. A few pieces of masking tape are then placed around the rat's body. Alternatively, small holes may be drilled along the periphery of the PCB. Small pieces of nylon line may be placed through the holes and then around the animal's body. This provides maximum rigidity and produces very little trauma to the animal. The rigid support system improved the reproducibility of the NMR measurements.

University Station / Birmingham, Alabama 35294
An Affirmative Action / Equal Opportunity Employer
The support system uses very little of the precious room in the probe, is easy to construct from commonly available materials, and requires very little machine work.

Sincerely,

Mike E. Brown

N. Rama Krishna


Dear Professor SHAPIRO,

Agar and agarose gels eventually doped with paramagnetic substances are often used as "standard" tests objects for Magnetic Resonance Imaging. Since these standard samples are supposed to be used to test imagers working in a range of magnetic fields extending between 0.02 T and 1.5 T, we measured the Nuclear Magnetic Relaxation Dispersion of water protons in agarose gels containing paramagnetic ions over almost four decades of proton Larmor frequencies. The measurements have been done on a field cycling relaxometer built by S.H. KOENIG et al. at the IBM T.J. Watson Research Center. (We take this opportunity to mention that, thanks to Drs S.H. KOENIG and R.D. BROWN, further work will take advantage of the fact that our lab is now equipped with one "clone" of their relaxometer). The figures 1 and 2 represent the NMR dispersion curves at 20° C of gels containing 3% of agarose and 0.0005 M/L of MnCl₂ and GdCl₃ respectively.

Figure 1

Figure 2

(continued on page 33)
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Integrated solutions for the laboratory

IBM Instruments Inc.
(continued from page 30)

The T1 dispersions are clearly different for both solutions. The analysis of the results shows that the Mn2+ aquoion reorientates nearly at the same rate in the gel than in the aqueous solution whereas the tumbling of the Gd3+ ion is much slower in the gel than in the aqueous solution. This observation is related to the bigger size of the Gd3+ aquoion as compared to the Mn2+ one.

Profiles recorded on agar gels doped with Gd3+ exhibit between 1 and 50 MHz larger variations than those observed for agarose gels. These different behaviors of gels do not preclude their use as standard materials but suggest the necessity to record their NMRD profiles in the range of magnetic fields in which they are supposed to be used.

We also want to point out the importance of the choice of the glassware of the NMR tube used for this kind of studies.

In a normal glass tube, the T1 at 20 MHz of a 5.3 $10^{-4}$ M GdCl3 aqueous solution initially at pH 3.5 varies from 0.22 s to 0.56 s after 71 hours. In a pyrex tube, the T1 remains constant. This is explained by the pH variation of the solution contained in the glass tube due to the Na+/H+ exchange. The Na+ ions go from the glass to the solution while the protons from the solution enter the glass. The resulting pH increase induced the precipitation of gadolinium hydroxyde and a concomittant increase of the T1 values.

Sincerely,

Prof. Y. VAN HAVERBEKE.

Dr. R.N. MULLER.

Dr. L. VANDER ELST.

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

Oct. 9, 1986  
(Received October 16, 1986)

PC-TALK III to Aspect 3000 and Nicolet 1180/1280
Dear Barry,

Recently we needed to extract data from a Nicolet 1180 for analysis using a PC. The DTL routine in GENT can be used to dump signed decimal numbers in ASCII (select BO, even parity). The PC is running PC-TALK III* (version 830909; use shift-tab to check). Using the Receive File command (Alt-R) the dump is captured to disk.

PC-TALK III is written in BASIC and easily modified. To speed things up we patched PC-TALK to run at 19200 and 38400 Baud. The 8250 UART chips can be programmed for these rates but MS-DOS or PC-DOS (and most application programs) do not use rates higher than 9600 Baud.

We have been successful in dumping at 19200 Baud. At 38400 Baud we experience transmission errors. We are not yet certain as to where these occur. Due to recent pink mail we were not able to find out more at this time. An XON/XOFF modification to GENT is being worked on.

To receive ASCII files from a bruker Aspect 3000, the RS-232 channel B is used ("ON B"). Only pins 1, 2, 3, and 7 are wired between the Aspect and the PC. To capture a file, e.g. DEPT.AU, use the Alt-R command in PC-TALK and then issue the ADAKOS command "LIS DEPT.AU" from the PC. The file can now be edited on the PC using a text editor, e.g. EDIX (word processors are not recommended).

To send edited (or new) files back to the Aspect we activate TECO from the PC (e.g. TECO<cr>, EWDEPT1.AU<esc><esc>, I<cr>). "I" enters the insert mode. Then using the Transmit File (Alt-T) command in PC-TALK send DEPT1.AU in ASCII format. Close the TECO generated file by sending "EX<esc><esc>, H<esc><esc>". This permits several versions of an experiment to be prepared off line without tying up the instrument, and without having to learn more than four TECO commands. The changes (lines 4045-4405) cause PC-TALK to wait for the character to be echoed; this avoids overflow of the buffer in TECO, which was a problem with this method. We are working on a patch to interface to SPECNET to further simplify transfers.
The listing included below can be typed in on top of PC-TALK III or put into a separate merge file. Lines 5300-5380 are the subroutine which resets the UART to the higher Baud rate after the serial port is opened for 9600 Baud. Lines 427, 5027-5283, and 5672-5673 support the entry of the two higher Baud rates, which must be entered as 19K (upper case K) and 38K.

Sincerely,

Rudi Nunlist

Changes to PC-TALK III:

427 IF BAUS$="19K" OR BAUS$="38K" THEN GOSUB 5300
4045 FOR I=1 TO 128:PRINT INPUT$(1,#1):NEXT
4240 PRINT#1,Z$:IF TR$="P" THEN IF Z$=CR$ THEN PRINT INPUT$(1,#1);:GOSUB 4425:GOTO 4210
4250 PRINT INPUT$(1,#1);:GOTO 4210
4405 FOR I=1 TO LEN(Y$):Z$=MID$(Y$,I,1):IF Z$=LF$ THEN 4415 ELSE IF Z$<>CR$ THEN PRINTl1,Z$;:PRINT INPUT$(1,#1);:GOTO 4415 ELSE PRINT#1,",",CR$;:PRINT CR$;:BS$="":GOSUB 4420
5027 PRINT"5 - 19K,N,7,1 (Bruker) 6 - 19K,E,7,1 ( Nicolet)
5060 Q=VAL(Q$):IF Q<1 OR Q>6 THEN BEEP:GOTO 5045 ELSE PRINT Q
5081 IF Q=5 THEN BAUS$="9600":PAR$="N":BAUS1$="19K"
5082 IF Q=6 THEN BAUS$="9600":BAUS1$="19K"
5088 IF Q=4 THEN GOTO 5300
5283 IF BAUS1$="38K" OR BAUS1$="19K" THEN GOSUB 5300
5100 ' set high Baud rate
5385 COMBASE=&H3F8 ' for COM1
5310 IF COMMPORT$="COM2:" THEN COMBASE=&H2F8
5320 IF BAUS$="19K" THEN LOW=6
5325 IF BAUS$="38K" THEN LOW=3
5340 HIGH=0
5345 DIVLSB=COMBASE
5350 DIVMSB=COMBASE+1
5355 LINECTRL=COMBASE+3
5360 OUT LINECTRL,INP(LINECTRL) OR &H80 ' set DLAB
5365 OUT DIVLSB,LOW:OUT DIVMSB,HIGH
5370 OUT LINECTRL,INP(LINECTRL) AND &H7F ' clear DLAB
5375 COMM$=COMMPORT$+BAUS1$+,"*PAR$*","*DTA$*","*STP$+COMMINIT$ 5380 RETURN
5572 BAUS1$=BAUS
5673 IF BAUS$="19K" OR BAUS$="38K" THEN BAUS$="9600"

2. A. Fluegelman, The Headlands Press, Inc., P.O. Box 862, Tiburon, CA 94920.
Preliminary Studies of Vascular Smooth Muscle Physiology and Metabolism by 31P NMR.

Dear Professor Shapiro:

In preliminary studies of vascular smooth muscle physiology and metabolism by 31P NMR, we have compared a variety of hog carotid artery preparations. Under all conditions studied, comparisons of metabolite concentrations have yielded a phosphocreatine (PCr) to ATP ratio of less than one. This is true whether the tissue is relaxed or contracted, whether the tissue strips are mounted isometrically or isotonically, whether the superfusate is bubbled with room air or 100% oxygen and at superfusate flow rates varying from 5.0 to 15.0 ml/min. per gram of tissue. Furthermore, we have found that oxygenation of the sample with 100% O2 prior to and during perfusion is critical to obtaining high PCr concentration. The spectra shown in figure 1 illustrate this fact. The low phosphocreatine concentration in spectrum A is obtained from tissue oxygenated only with room air, whereas in spectrum B the tissue is oxygenated with 100% O2 and shows a larger PCr concentration. As indicated, the PCr/ATP ratio is always less than one with the magnitude dependent on the conditions during perfusion.

The overall low PCr/ATP ratio is in agreement with other studies of vascular smooth muscle but contrasts with results obtained from smooth muscle from other organs. This result could be explained by the fact that distribution of smooth muscle in the body is more heterogeneous than skeletal and cardiac muscle. Therefore, the relative concentrations of intracellular phosphagens may be more varied as well.

Sincerely,

Nina C. Gonnella, Ph.D.
CIBA-GEIGY Corp.

Thomas Grieder
University of Medicine and Dentistry of New Jersey
Figure 1. Spectrum A: Hog carotid artery oxygenated with room air. Spectrum B: Hog carotid artery oxygenated with 100% O₂.
Selective Suppression of Particular Signals
in homonuclear 2-D NMR

Dear Dr. Shapiro:

We have been experimenting with incorporating the homo-nuclear bilinear rotation pulse into COSY-type experiments to effect selective observation of certain signals. The results are not as clear as in the heteronuclear case. However, the following pulse sequence was found to be useful for certain types of molecules (e.g. oligosaccharides, polyaromatics).

\[
\begin{align*}
&\text{MIS (1)} \\
&\text{acquisition (signals added)} \\
&t_1/2 \quad t \quad t \quad t_1/2 \quad t_2 \quad t
\end{align*}
\]

In these types of molecules, the spin system (or a part of the spin system) follows the condition

\[
J = J(I_1, I_2) - J(I_2, I_3) \cdots - J(I_{n-1}, I_n)
\]  

[1]

and all other coupling constants being substantially smaller.

The results from this pulse sequence with \( \tau \) set equal to \( 1/2J \) will give a homonuclear CSCM map in which the signals of

1. single spins are eliminated (the solvent peak is also suppressed);
2. inner spins (\( I_2, I_3 \cdots I_{n-1} \) in [1]) are eliminated from the diagonal;
3. terminal spins (\( I_1 \) and \( I_n \)) show up prominently in the diagonal. The patterns for spin-pairs (\( n = 2 \)) and for terminal spins in a chain (\( n > 2 \)) are different and can be easily distinguished.
4. The off diagonal peaks, which are "spurious" in this experiment provide J-connectivity information (even between signals which are absent from the diagonal).

These results (illustrated in Fig. 1) should be useful in providing a valuable starting point for the assignment of COSY maps for very congested spectral regions by eliminating selected signals.

(continued on page 41)
MG-5DP Peak Reading Portable Laboratory Gaussmeter

GENERAL:
The MG-5DP is a general purpose peak reading portable Hall effect gaussmeter designed to measure both DC & AC (RMS) magnetic fields.

Three full-scale bipolar ranges of ±100.0 gauss, ±1.000 KG and ±10.000 KG with 100% over-range and resolution of 0.05% provides DC & AC field readings from ±100 milligauss to ±19.99 KG with true RMS readings from 3 Hz to 20 KHz; readings are displayed on a 3½ digit ±0.05% bipolar LCD meter.

In the Peak Mode, the MG-5DP will sense and display the most recent peak magnetic field level from DC to 20KHz.

This instrument can be set to either detect the peak value when the field is bipolar (varying from positive to negative) or it can be set to exclusively detect either the positive peak or the negative peak of a varying field. Because of the unique digital circuit design, there is no decay in the peak field reading.

A wide selection of precalibrated transverse and axial Hall probes is available to meet most every application, including probes which will extend the measuring range of this instrument to 150.0 KG.

The MG-5DP operates either from AC or from sealed lead acid batteries. During AC operation, the batteries receive a floating charge which keeps them fresh until the instrument is required for portable use. Freshly charged batteries will continuously operate this instrument for approximately 10 hours.

In addition, analog outputs are also provided for simultaneous external monitoring of both the instantaneous field level and the peak field level.

APPLICATIONS:
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• Analyze Magnetic Circuits and Components
• Classify Magnets
• Measure Absolute & Differential Fields
• Plot Field Uniformity
• Measure Stray & Leakage Fields

FEATURES:
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• Range Extendable to 150.0 KG with Select Probes
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• One Year Warranty

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Reversing the phase of the last 90° pulse will reverse the signals preserved and the signals suppressed.

Sincerely yours,

Venceslav Rutar
Wei Guo
Tuck C. Wong

Fig. 1: The contour map resulted from the pulse sequence shown in the previous page. Only part of the correlations has been illustrated.
Dear Barry,

Subject: Multiquantum NMR of solids on a CXP

We have been working on some multiple quantum NMR experiments in solids using time reversal and "phase domain" data acquisition. To do this it was necessary to install a variable phase shifter on our CXP-200. We used a Merrimac 8-bit digital phase shifter model PSDW-84-50.33 which operates at 50.33 MHz (i.e. $^{13}$C at 4.7 T) with a phase shift resolution of 1.4 degrees. It is rather difficult to give a detailed description of the entire installation and operation of this phase shifter within the limits of a letter so we will give only a functional description here. Any CXP users interested in details can contact us for a more complete description.

The rf installation is straightforward, with the phase shifter being installed between the "rf modulator" and the "preamplifier" with BNC cables. We found it necessary to insert a small, broadband amplifier to make up for the $\approx 3$ dB loss in the phase shifter. Although the phase shifter only gives a linear 1.4° phase shift per control bit when operated at 50.33 MHz, we have performed proton experiments at 200.13 MHz by mixing the CXP's 60 MHz intermediate frequency down to 50 MHz, phase shifting and then mixing back to 60 MHz. This corresponds to using two IF's for proton operation.

It was difficult to get flexible and fast communication between the spectrometer and the phase shifter to set the 8 control bits. We wanted to be able to switch phase quickly during a pulse program and to be able to switch back and forth between a manually preset phase and a pulse programmer controlled stepped phase for the phase domain data acquisition. This means that the phase shifter must be controlled by the pulse programmer, Z17c, rather than the Aspect computer. Basically, we use the levels of the first 8 bits of counter 4 in the pulse programmer (which are available at a 25 pin connector labelled "counter out" at the rear of the Z17c) for rapid advancing of the phase. We have built an interface with 8 switches providing TTL levels for preset, manual switching of the phase shifter. To switch rapidly between the "preset" phase and the phase set by counter 4 we use the receiver blanking pulse (which is on the Burndy connector at the rear of the pulse programmer) to switch between the two phases. The receiver in our CXP is sufficiently well protected by the duplexer and preamp protection circuits that giving up the receiver blanking function doesn't affect our data.
The interface with the pulse programmer was relatively easy to build and in operation the system is very simple. The biggest problem was sorting out the errors in our schematic for our Z17c pulse programmer. We have been quite successful in getting multiple quantum spectra with magic angle sample spinning as can be seen in the paper coming out this month. It would be nice to have more steps in our pulse programs because even with looping and phase lists, we have an insufficient number of steps for a few sequences that we would like to try.

We have documented the details of this system for our own future use so anyone needing a detailed description can contact us.

REFERENCES


Sincerely,


Los Alamos
Los Alamos National Laboratory
Los Alamos, New Mexico 87545
Group INC-4, Mail Stop C345

I would like to point out that we have a labwide, competitive, postdoctoral program at Los Alamos with many nice features including salaries near 30 k per year. I would be interested in hearing from any potential applicants. Our present interests are primarily in solid state NMR especially in molecular dynamics and solid state chemical reactions and in some curious and difficult inorganic compounds. The detailed projects would depend on the interest and background of the applicants. Applications and final acceptance are made through a labwide competition so there is a delay of several months in the application process. Interested applicants can contact me at the address above or by telephone at (505) 667-0773. They should include a curriculum vita, transcripts of undergraduate and graduate grades (not necessarily an official transcript), a simple, informal description of interests, a list of publications (including those submitted but not yet published), and the names of at least 3 references. Applicants should know NMR but not necessarily solid state NMR.

William L. Earl.
An Efficient Probe for High Field Multinuclear CP/MAS NMR of Solids

Dear Barry:

We wish to inform the readers of the Newsletters on some important improvements of our CP/MAS probe reported earlier (TAMU NMR 322-61) and developed for the Varian XL-300 spectrometer. The improvements concern both the spinning speed and electronic performances of the probe and are illustrated in the accompanying spectra of figures 1 and 2. With our modifications we now have the following features for the probe:

1. The spinning speed of the 7 mm o.d. rotors can routinely and accurately be set at any value up to at least 10 kHz with a stability of a few hertz. To obtain spectra at high spinning frequencies we use rotors machined from PSZ and Si$_3$N$_4$ ceramics, e.g. Si$_3$N$_4$ rotors for 10 kHz spectra. Air drive pressures of 1 and 4 bar are required for 4.5 and 9.0 kHz spinning, respectively.

2. The double-tuned X/$^1$H probe circuit has a broadband observation channel. So far we have observed nuclei in the frequency range from $^{27}$Al (78.16 MHz) to $^1$H (46.04 MHz) using switchable chip capacitors.

3. The isolation of the observation (e.g. $^{13}$C) channel from the $^1$H decoupling channel is about 45 db.

4. With the high Q for the decoupling channel (Q ~ 350) we generate a $^1$H decoupling field $\gamma_B B_1^H/2\pi = 80$ kHz ($B_1^H = 19$ gauss) using only about 25 watts of power. For the observation channel we generate a 90° $^{13}$C pulse in 6.1 µsec ($B_{1C} = 36$ gauss) using 100 watts of power.

5. The sensitivity (S/N) for the observation channel of the probe is illustrated in Figs. 1 and 2. For example for $^{13}$C we obtain a S/N ratio of 200-230/1 for 160 mg of hexamethyldisilazane based on the CH resonance from a CP/MAS experiment using 4 scans.

Sincerely,

Preben Daugaard Vagn Langer Hans J. Jakobsen
Fig. 1. $^{13}$C (75.43 MHz) CP/MAS NMR spectra of hexamethylbenzene (160 mg) recorded on a Varian XL-300 spectrometer at 7.05 Tesla using different spinning rates, $\Omega_R$, and rotor materials. (a) Macor rotor, $\Omega_R = 4860$ Hz; (b) $\text{Al}_2\text{O}_3$ rotor, $\Omega_R = 7000$ Hz; (c) $\text{Si}_3\text{N}_4$ rotor, $\Omega_R = 10080$ Hz. The spectra were obtained using 4 scans and a linebroadening of 50 Hz has been applied.

Fig. 2. $^{113}$Cd (66.54 MHz) CP/MAS NMR spectra of $\text{Cd(NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (390 mg) obtained in 4 scans using (a) $\text{Al}_2\text{O}_3$ rotor, $\Omega_R = 6200$ Hz; (b) $\text{Si}_3\text{N}_4$ rotor, $\Omega_R = 8000$ Hz. A linebroadening of 25 Hz has been employed for both spectra.
Dear Barry,

"Computer Optimized Solvent Suppression"

I am so terrified of being disenfranchised after so many years as a subscriber to TAMUNMR that I have borrowed this story from my colleague Peter Hore who has an independent NMR group in this laboratory.

You will remember that some time ago Peter Hore proposed the 133T sequence for solvent peak suppression (1), and showed that the null condition at the carrier frequency is relatively broad and insensitive to $B_0$ and $B_1$ inhomogeneity. It also tolerates a missetting of the 1:3 ratio of pulsewidths.

The difficulty with this and the related solvent suppression sequences is that the spectral region where there is appreciable excitation is relatively narrow, leading to severe intensity distortions for the observed spectrum. The idea is to make this excitation region broader and flatter by modifying the pulse sequence using A.J. Shaka's method of non-linear computer optimization (2). Peter defines a figure of merit $\psi$ such that

$$\psi = \frac{|v_1 - v_2|}{|v_1 + v_2|} \quad 0 < \psi < 1$$

where $v_1$ and $v_2$ are the offsets defining the region where the excitation is 90% of its maximum value:

We want $\psi$ to be as large as possible, to give a flat excitation region not too distant from the carrier.

(continued on page 49)
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or Steinhausenstrasse, CH-6302 Zug, Switzerland.
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In Latin America (South) Av. Dr. Cardoso de Melo, 1457/1459, CEP 04548, Sao Paulo, SP, Brazil.
In Australia 679 Springvale Road, Mulgrave, Victoria 3170.
In Japan 3rd Matsuda Bldg., 2-26 Ohashi Shinjuku-ku, Tokyo 160.
In Asia (South East) Mandarim Plaza, Rm 1018-29, Tower A, 14 Science Museum Rd., Yiamahasi East, Kowloon, Hong Kong.
To cut a long story short, I will simply say that the search yielded two new pulse sequences A and B that have figures of merit markedly better than that of the $^{133}$I sequence ($\psi = 0.28$).

The A sequence is:

$$7.1 - 4\tau - 10.9 - \tau - 55.1 - \tau - 55.1 - \tau - 10.9 - 4\tau - 7.1$$

where the flip angles are in degrees and overbars indicate phase inversion. This has $\psi = 0.76$.

The other sequence B is:

$$6.8 - \tau - 4.5 - \tau - 7.4 - \tau - 53.3 - \tau - 53.3 - \tau - 7.4 - \tau - 4.5 - \tau - 6.8$$

This has $\psi = 0.66$ but a smaller phase gradient across the spectrum.

Note that these sequences are antisymmetric about their centres; the second half is the same as the first half but reversed in time and inverted in phase. Such sequences behave symmetrically with respect to resonance offset and have a null at the transmitter frequency. Note, however, that unlike the binomial sequences, the pulse phases do not alternate along the sequence.

Interestingly, the computer search also identified two sequences previously proposed by Starčuk and Sklenár (3) for the same purpose.

When the pulse sequences A and B were tested experimentally on our Varian XL-400 there was good agreement with the theory, confirming that they do indeed have a wide excitation bandwidth. Suppression ratios for the solvent peak were in the range 200-400, comparable with that obtained with the $^{133}$I sequence.

Yours sincerely,

Ray Freeman

October 27, 1986
(Received 31 October 1986)

In our work on NMR at very low temperatures it became important to verify that the temperatures of our samples were actually the same as measured by conventional means (i.e., by a carbon resistance thermometer mounted a few cm from the sample, calibrated at "high" temperatures against the vapor pressure of helium). The NMR spectrum of a multi-level system offers an attractive possibility: Consider the classic Pake doublet arising from a 2-spin system

\[
\begin{align*}
\hbar \omega_0 & \\
\downarrow & \quad B \\
\uparrow & \\
\hbar \omega_0 & \\
\downarrow & \quad A
\end{align*}
\]

In the high temperature approximation \(\hbar \omega_0 / kT \ll 1\) the lines are equally intense, but at low temperatures the intensity ratio \(B / A = e^{-\hbar \omega_0 / kT}\) departs from unity.

Here is a proton powder pattern from the \(\text{H}_2\text{O} \) molecular slightly hydrated \(\text{CaSO}_4\) obtained by FT of the FID elicited by an 8° pulse.

The temperature comes out to \(-0.010 \pm .001\, K\).
The following points will be amplified later:

1. A small-angle tipping pulse must be used.

2. Such experiments can determine "absolute signs" of spin-coupling constants.

3. Slightly hydrated CaSO$_4$ is not the thermodynamically expected mixture of CaSO$_4$ and CaSO$_4$ \( \cdot \frac{1}{2}H_2O \).

4. There are "anomalies" in the spin-lattice relaxation of this and related samples.

Yours,

Philip Kuhns

John Waugh

Oded Gonen
Title: $^{185}$Re and $^{187}$Re solid state NMR

Dear Professor Shapiro,

our $^{127}$I solid state NMR studies of metaperiodates with alkyl- or aryl-substituted cations of main group V revealed second order quadrupole effects with quadrupole coupling constants in the range of 2 - 7 MHz [1]. In the special cases of the samples $(CH_3)_4PIO_4$ and $(C_2H_5)_4NIO_4$ the $^{127}$I solid state NMR spectra showed no visible quadrupole effects of first or second order [2].

From these results we calculated maximum values of the $^{185}$Re and $^{187}$Re quadrupole coupling constants of the analogous metaperrhenates. We expected them in an accessible range of our Bruker CXP 200 FT-NMR spectrometer. Despite the very large $^{185}$Re and $^{187}$Re nuclear moments we succeeded in measuring solid state NMR spectra of $(CH_3)_4PReO_4$ and $(C_2H_5)_4NReO_4$. For the first time, it was possible to record $^{185}$Re and $^{187}$Re NMR powder spectra of rhenium compounds apart from a Knight shift investigation of cubic ReO$_3$ at $T = 1 - 4$ K [3].

For polycrystalline $(CH_3)_4PReO_4$ we measured a complete $^{185}$Re and $^{187}$Re solid state NMR spectrum with second order quadrupole splittings. The quadrupole coupling constants of this metaperrenchenate are calculated:

e$^2$Q$^{(185)}$(Re)/h = $18.2 \pm 0.2$ MHz for $T = 307$ K, $n \neq 0$

e$^2$Q$^{(187)}$(Re)/h = $16.9 \pm 0.2$ MHz for $T = 307$ K, $n \neq 0$.
The measured temperature coefficient $\alpha = -1.2 \times 10^{-2} \text{K}^{-1}$ indicates the presence of strong volume effects in the sense of the Kushida-Benedek-Bloembergen theory [4]. The temperature dependent quadrupole splitted NMR signals of polycrystalline $(\text{CH}_3)_4\text{PReO}_4$ do not give any indication of a change of the asymmetry parameter $\eta$.

For $(\text{C}_2\text{H}_5)_4\text{NReO}_4$ we also found $^{185}\text{Re}$-, $^{187}\text{Re}$-solid state NMR spectra. In this case the electric field gradient at the Re-nuclei is so small that no quadrupole effects of first or second order are resolvable. Detailed results will be published soon.

Yours sincerely

P. Burkert

(Dr. P.K. Burkert)

D. Klobasa

(Dr. D.G. Klobasa)

Please credit this contribution to Prof. F.H. Kähler.

References:
Dear Barry:

We have found that the phase programs which we developed for chemical exchange in the rotating frame (1) can also be applied, with the appropriate phase alternation where needed, to CAMELSPIN (2), to ROESY (3), to TOCSY (4), and to HOHAHA (5). We noticed that the diagonal after 2DFT goes from upper left to lower right corner no matter how we modified the basic phase programs. This is due to the fact that spin locking occurs whether spins and rf field are parallel or antiparallel. However, the REV command in the DISNMR software allows for reversal of the F1 dimension and therefore the use of symmetrization through the SYM command.

Sincerely,

Hermann E. Bleich

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

TITLE: F1 Quadrature Detection for Rotating Frame 2D-NMR

October 8, 1986
(Received October 10, 1986)

(5) Davis and Bax, J. Amer. Chem. Soc. 107, 2820-2821 (1985)
Introduction

The eighth international meeting on NMR Spectroscopy will take place at the University of Kent at Canterbury from Monday 6 July to Friday 12 July 1987, with participants foregathering on the afternoon and evening of Sunday 5 July 1987.

The meeting is being organised jointly by the Royal Society of Chemistry, and the Society's NMR Discussion Group.

The meeting is the eighth in the series (now biennial), previous meetings having taken place as follows:


Important Note

Numbers for the meeting may exceed the capacity of the lecture theatre, and as a consequence, arrangements may have to be made for a closed circuit TV link, or even to limit the number of persons registering. Details regarding any such arrangements will be notified in Circular 2.

Scientific Programme

The meeting will deal with selected topics in NMR Spectroscopy and will comprise the following eight symposia:

1. NMR in Organic Chemistry
   Chairman: I H Sadler
   Invited speakers: Dr J Staunton
                   Professor H Günter

2. NMR in Solids
   Chairman: K J Packer
   Invited speakers: Professor C P Slichter
                    Dr C S Yannoni
                    Professor J A S Smith

3. NMR in Industry
   Chairman: G R Bedford
   Invited speakers: Dr D Neuhaus
                    Dr L Jelinski

4. NMR in Inorganic Chemistry
   Chairman: J W Akitt
   Invited speakers: Professor W Klemperer
                    Professor A E Merbach
5. Biomedical NMR
   Chairman: P J Sadier
   Invited speakers: Professor I C P Smith
                    Professor J Seelig

6. Large Molecule NMR
   Chairman: G C K Roberts
   Invited speakers: Professor R G Griffin
                    Professor D J Patel

7. Experimental Techniques in NMR
   Chairman: R Freeman
   Invited speakers: Dr G Bodenhausen
                    Professor R Kaptein

8. NMR in Physical Chemistry
   Chairman: J Emsley
   Invited speakers: Professor R L Vold
                    Dr N Boden

Poster Sessions and Contributed Papers
Two poster sessions are planned for the programme. There will also be
an opportunity for a limited number of short contributions to be presented
orally in the symposia. Anyone wishing to contribute a paper to one of
the poster sessions, or to one of the symposia, should submit, NOT LATER
THAN 26 JANUARY 1987 a title and synopsis (up to 250 words) to Dr John
W1V 0BN, indicating the appropriate symposium topic.

Synopses will NOT be published.

Social Programme
A full programme of evening social events is planned, to include:
Receptions (Sunday and Monday)
Concert in Canterbury Cathedral (Tuesday)
Conference Dinner (Thursday)

As has been the practice with all previous NMR meetings, the scientific
sessions on Wednesday will terminate at lunchtime, so as to enable
persons to participate in organised excursions on Wednesday
afternoon and evening. Excursions proposed include: Leeds Castle,
Hever Castle and other places of historic interest and scenic beauty
in the area.

In addition, an accompanying persons programme will be arranged.

Fees
The fees for attendance at the meeting have not been finalised, but
it seems likely that the conference fee will be around £80 (students
£35), and the accommodation for the full five day period will be around
£100.

TO: Dr John F Gibson
Secretary (Scientific)
The Royal Society of Chemistry
Burlington House
London W1V 0BN

Please send me further details of the eighth international meeting on
NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY, to be held at the
University of Kent at Canterbury, 6-10 July 1987. (Available in February early
March 1987).

PLEASE WRITE NAME AND ADDRESS CLEARLY ON THIS LABEL

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If undelivered, please return to:
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Burlington House, London W1V 0BN, UK.

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*COCONOSY (Haasnoot, et. al., J. Magn. Reson., 56, 343 [1984])