L. W. Reeves  
Diamagnetic Anisotropy Lyotropic Liquid Crystals  

J. B. Robert and L. Wiesenfeld  
Chemical Shifts in Solids [or, "Plus ça change..."]

U. Edlund  
Ockham's Razor Again - SSP vs DSP

J. Schaefer  
Nitrogen Metabolism by CPMAS N-15 NMR

V. Bystrov  
Selectively Fluorine and Spin Labeled Neurotoxin

M. W. Baum  
$^{13}$C Investigation of Mesoionic Compounds

R. K. Harris, K. J. Packer and C. J. Groombridge  
Solid-State $^{13}$C NMR of Platinum Compounds

E. T. Samulski  
Pinch 'em, Squeeze 'em, Stretch 'em... They Like It! $^{2}$H NMR in Rubber Bands

G. Gurato, G. Lunardon and G. Rigatti  
$^{13}$C Chemical Shift Assignments, Spin-Lattice Determination and Quantitative Analysis of a Model Elastomeric Polyurethane

P. J. Sadler, I. M. Ismail, G. E. Hawkes and M. J. Buckingham  
$^{35}$Cl/$^{37}$Cl and $^{79}$Br/$^{81}$Br Isotope Effects

P. Stilbs  
Size-Resolved NMR

L. Ernst  
Determination of the Relative Signs of Interbenzylic $^{31}$P, $^{31}$P Spin Coupling Constants from SPI Difference Spectra

H. C. Charles and M. J. Albright  
Use of the Dixon Sequence for Suppression of Spinning Sidebands in Solid State Spectra

H. Günther and J. Wesener  
More $^{2}$H/$^{1}$H Isotope Effects on $^{13}$C Chemical Shifts

A. de Jager and C. Mooney  
Improvement of the Redfield Sequence

T. W. Bentley  
NMR Studies of Reaction Kinetics

W. C. Still  
Position Available

G. C. Levy  
Bruker HX-60 Spectrometer for Sale

T. C. Wong  
Positions Open

A monthly collection of informal private letters from Laboratories of NMR. Information contained herein is solely for the use of the reader. Quotation is not permitted, except by direct arrangement with the author of the letter, and the material quoted must be referred to as a "Private Communication". Reference to the TAMU NMR Newsletter by name in the open literature is strictly forbidden.

These restrictions apply equally to both the actual Newsletter participant-recipients and to all others who are allowed access to the Newsletter issues. Strict adherence to this policy is considered essential to the successful continuation of the Newsletter as an informal medium of exchange of NMR information.
Consummate care in the storage and preparation of spectroscopic samples is just as integral a part of good spectroscopic practice as running the investigation or analyzing the spectra. And consummate care, of course, begins with equipment.

Our new, expanded Wilmad line of vials, storage and septum bottles, and a broad variety of stoppers, caps, and septa help materially to simplify the handling, storage, and preparation of samples...eliminate expensive sample loss...and save unnecessary waste of time and money.

Wilmad vials and bottles are manufactured of top-quality borosilicate glass to prevent any pH modification of the contents. The variety of caps available match any sampling or storage need. Snap caps of polyethylene, open-top types with elastomer septa, aluminum seals with Teflon-faced septa...whatever you need we now carry in stock.

Write or call for our new Catalog 781.
FT NMR was never "hard," only certain samples were.

Now with the low cost JEOL FX60QS System
High Resolution Solid State NMR becomes routine
I am hampered by our regular seasonal postal strikes, but now the University has opened up an illegal mail run to Niagara Falls twice a week. The mounties have not yet stopped the truck. Following on from the last letter, I can release the abstract of a paper to appear in J. Phys. Chem. in November, which shows that we really did know how to manipulate the diamagnetic anisotropy of disk shaped micelles.

**Abstract**

The diamagnetic anisotropy of magnetically aligning disc micelle lyotropic liquid crystals has been reversed by the inclusion of aromatic amphiphiles. This reversal occurs without a phase change and at the point of transition a non-aligning Type O disc micelle mesophase is formed. Different host mesophases have been taken through the change in sign of the diamagnetic anisotropy, and the effects of temperature variation investigated. The rate of alignment of the Type I disc micelle mesophases is a linear function of aromatic amphiphile concentration.

The figure from the paper is instructive. It shows the velocity constant of alignment of a type I DM mesophase as a function of mole-fraction of potassium heptyloxy-benzoate (an aromatic amphiphile) in sodium lauryl sulphate. The dependence is linear, extrapolating to 0 rate constant at a mesophase that is diamagnetically isotropic, a Type O DM. Diamagnetism is a molecular property and mixing aromatic and aliphatic chains in a micelle, with of course appropriate relative alignment, leads eventually to diamagnetically isotropic micelles that dispose in orientational order to give a liquid crystal. Kind regards.

Yours sincerely,

L.W. Reeves
Professor of Chemistry
ALIGNMENT RATE VS. AROMATIC CONTENT

![Graph showing alignment rate vs. aromatic content](image-url)
Dear Dr. Shapiro,

More and more data concerning the n.m.r. chemical shift are obtained now from solid state studies. Thus, using the measurement of the constant of spin rotation and ab initio calculation on a reference compound, it is possible to determine the magnetic shielding tensor components of the recorded nuclei on an absolute scale with respect to the bare nucleus. In a n.m.r. experiment, the bare nucleus $N_0$ experiences a field $B$ which will in general be greater than the one experienced by the same nucleus $N_C$ involved in a chemical bond. With spectrometers operating in the F.T. mode, i.e. at constant field and variable frequency, the bare nucleus $N_0$ most frequently resonates at higher frequency than chemically bonded nuclei $N_C$. Thus we suggest to adopt a scale increasing from high to low frequency for the components of the magnetic shielding tensor components $\sigma_{rr}$, even if the values are given with respect to a reference and not in absolute scale. Such a suggestion is not in agreement with the I.U.P.A.C. recommendation for the chemical shift sign scales.

Sincerely,

J.B. ROBERT

L. WIESENFELD
August 3, 1981

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

Dear Barry,

Ockham's Razor Again - SSP vs DSP.

As expected, there has been some response to my last contribution in TAMU (no 265), which concerned the necessity of testing the degree of parameterization using NMR SCS correlation models. Sardella suggested (TAMU no 269), that if a single substituent parameter equation (SSP) and a dual substituent parameter model (DSP) gave an identical fit, one should use the latter, since the DSP model permits a detailed partitioning of effects. This proposal was exemplified by treating the individual C4-C7 C-13 NMR shifts of 1-substituted azulenes.

First of all, five 1-substituted azulenes are too few compounds to be a basis for a meaningful SCS data analysis (minimum 6-7) and besides, correlation coefficients get larger with decreasing sample size. Moreover, his choice of substituents is hardly representative for the whole substituent domain (CH₃, H, Cl, CH₃CO, NO₂). However, ignoring this sad fact for the moment, I consider the azulene data set as a typical multivariate data analysis problem, i.e., we want to find out how many "uncorrelated effects" are needed to account for all four C4-C7 C-13 NMR SCS. If one analyses such a matrix (preferably a larger data set than the 5x4 matrix) it could very well be that on a statistical basis (F-test) a two-component model is superior to a single component model. Of course, this does not exclude that one could find individual positions showing an acceptable correlation to a single scale. I can mention, that in a very similar study of 2-substituted indenes we found, that a two-component model was necessary to account for the total C4-C7 C-13 SCS variation, although for some individual carbons only one "effect" was significant. So I do not think there is a need for a re-examination of "Ockham's Razor". If a two-component
is necessary, it will be revealed when we treat the complete shift matrix with a multivariate data analysis method.

One must also keep in mind that correlation models can operate either on a descriptive or explanatory level. Especially in the first case there exists statistical tools for the selection of those variables or shifts with relevance to the actual problem. We have exemplified this approach by considering three classes of \( \alpha \)-substituted styrenes (\( \alpha \)-H, \( \alpha \)-Me and \( \alpha \)-t-Bu series)\(^4\), each class consisting of seven 4-substituted derivatives. Based on those NMR parameters (Cl, C\( \alpha \) and H\( \alpha \)\(^1\) and H\( \beta \)\(^1\)) which had relevance to our classification problem, we then tried to classify "unknown \( \alpha \)-substituted styrenes" and to probe the substituent trend in each class. A single component (\( \Theta \)) model was found to be sufficient in each class. Since the selection of variables partly was determined by our classification problem and since we were afraid that the component values (\( \Theta \)) should be misused as a general substituent scale, we did not publish these values for the separate classes. The flood of scales is large enough without our contribution. However, in one case, after having merged the \( \alpha \)-Me and \( \alpha \)-H classes, the component values were given. We thought, that component values based on such a structurally heterogenous class (14 \( \alpha \)-H and 7 \( \alpha \)-Me structures), based on both H-1 and C-13 variables with relevance to our classification problem etc., would prevent the use of these values as a general substituent scale. But contrary...

Reynolds et al\(^5\) recently used these component values in a study of a subset of our matrix (12 \( \alpha \)-H structures) to prove the superiority of the \( \sigma_i \), \( \sigma_o \) model to SSP scales (\( \Theta \) and \( \sigma_p \)).\(^5\) Our \( \Theta \) values for the merged class are of course of a very local validity and strongly related to our classification problem. Therefore, these values should not be used on other data sets or subsets of our shift matrix. Reynolds concluded from this study that "a DSP treatment is not only sufficient but also sufficient to interpret the styrene magnetic resonance parameters".

A completely contradicting result was quite recently reported by Laszlo et al\(^6\) They analysed four series of styrene derivatives and they found by a rigorous statistical treatment, that
there exists no statistical superiority in general of a DSP to a SSP treatment in these four series. Moreover, if DSP approaches were applied, the inductive effect was found to be more sensitive to configurational differences.

Does it seem confusing? No wonder that one sometimes have a wish, that organic chemists should not go beyond variable-by-variable plots.

Best regards

Ulf Edlund

August 12, 1981

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

Dear Barry:

Cross-polarization magic-angle spinning (CPMAS) $^{15}$N nmr of intact lyophilized Neurospora crassa grown in $^{15}$N-nitrate medium as a function of time showed the incorporation and subsequent metabolism of label, with, for example, individual lines observed for nitrogen in lysine, arginine, and histidine residues. The time dependence of the intensity of each of these lines over an eight-hour period resulted in a detailed budget for nitrogen metabolism impossible to achieve with a nitrogen radiolabel ($^{13}$N half-life of 10 minutes). We have extended these experiments to studies of nitrogen fixation by Klebsiella pneumoniae introducing label uniformly via $^{15}$N$_2$. An unexpected result of some early experiments is the observation of a separate resonance associated with purine nitrogen of ribosomal RNA ($\delta_N=200$ ppm, top spectrum). We are in the process of performing various relaxation experiments which will allow our using this peak to quantify the extent of label directed into RNA synthesis. Ultimately we hope to perform these experiments on intact viable cells.

Sincerely,

Jacob Schaefer

Lyophilized intact cells (6-hr derepressed, with L-serine)

RNA (isolated)
Varian's new Zens Probes double NMR sensitivity

Varian's new high-sensitivity probes, available in 10-mm or 16-mm sample sizes and in the frequency range from $^{14}$N through $^{31}$P, provide double the sensitivity of any other commercial NMR system at 200 MHz.

This superior sensitivity of the XL-200 allows you in just a few hours to complete experiments that would previously have taken overnight.

Get all the facts.
For detailed information on the unique capabilities of the XL-200 and the new high-sensitivity Zens Probes, contact your nearest Varian Magnetics Sales Specialist or the Palo Alto Magnetics Product Team.

Research Magnetics Sales Specialists
East 201-822-3700
301-772-3683
Midwest 216-261-8035
312-825-7772
South 713-783-1800
404-955-1392
West 415-968-8141
Ext. 2196
213-927-3415
303-425-0413

Research Magnetics Products Team
Palo Alto 415-493-4000
Ext. 3047

$^{13}$C Sensitivity Test: Cholesteryl acetate, 100 mg/ml, 10 mm broadband probe. Transients accumulated using 90° pulses every 2.28 seconds with 0.5 Hz line-broadening.

Additional spectra appear on the following page
**Chemical Sensitivity Tests:**

- **Carbon Sensitivity Test:** 0.02 molar cholesteryl acetate in a 16 mm tube, 200 transients.

- **Nitrogen Sensitivity Test:** 90% Formamide in DMSO-d6, 10 mm 20-81 MHz broadband probe. Upper trace: single-transient (with NOE) proton-decoupled. Lower trace: eight transients, coupled (with NOE) 8-second acquisition time, 20-second delay time.

---

**Varian U.S. Sales Offices**

**CALIFORNIA**
9901 Paramount Boulevard
Downey, CA 90240
Phone: (213) 927-3415
375 Distel Circle
Los Altos, CA 94022
Phone: (416) 986-8141

**GEORGIA**
6550 Powers Ferry Road
Suite 100
Atlanta, GA 30339
Phone: (404) 955-1992

**ILLINOIS**
205 W. Touhy Avenue
Park Ridge, IL 60068
Phone: (312) 825-7772

**COLORADO**
4665 Kipling, Suite 1
Wheatridge, CO 80033
Phone: (303) 425-0413

**MASSACHUSETTS**
83 Second Avenue
Burlington, MA 01803
Phone: (517) 272-4152

**MICHIGAN**
3721 W. Michigan, Suite 300
Lansing, MI 48917
Phone: (517) 321-5000

**MARYLAND**
4701 Lydell Drive
Covington, MD 20781
Phone: (301) 772-3633

**NEW YORK**
6489 Ridings Road
Syracuse, NY 13206
Phone: (315) 427-6454

**OHIO**
333 Babbitt Road
Euclid, OH 44123
Phone: (216) 261-8035

**TEXAS**
25 Hanover Road
Florham Park, NJ 07932
Phone: (201) 822-3700

**WASHINGTON**
300 120th Avenue
Bellevue, WA 98005
Phone: (206) 454-2910
Title: Selectively Fluorine and Spin Labeled Neurotoxin

Dear Barry:

To evaluate intramolecular distances in the spatial structure of polypeptide neurotoxin in solution (1) we incorporate selectively two different labels in the Central Asian Naja naja oxiana neurotoxin II (NT-II).

Firstly, the mono-spin labeled (SL) derivative was prepared by treating NT-II with one equivalent of 2,2,6,6-tetramethyl-4-carboxymethylpiperidyl-1-oxyl N-hydroxysuccinimide ester. The individual [SL-Lys-27]NT-II product was treated with excess of trifluoroacetic acid phenyl ester and two derivatives were isolated: (A) tetra-trifluoroacetyl[Leu-1, Lys-15, Lys-26, Lys-47]-[SL-Lys-27]NT-II, and (B) penta-trifluoroacetyl[Leu-1, Lys-15, Lys-26, Lys-45, Lys-47]-[SL-Lys-27]NT-II.

Fluorine resonances in the spectra of the spin label quenched A and B compounds were identified by comparison with assigned signals and their chemical shift vs. pH dependencies of hexa-trifluoroacetyl-NT-II (2).

The fluorine spectra of A and B derivatives were decomposed by iterative procedure on a HP-computer with plotter as shown in the Figure. Calculated line widths of the individual resonances were used in calculation of apparent distances between the spin label on Lys-27 and corresponding fluorine groups by Bloembergen equation. The results are shown in the Table together with the data obtained previously from EPR spectra of di-spin labeled NT-II derivatives at liquid nitrogen temperature (3) and
from the X-ray crystallography of homologous erabutoxin b (4).

The pronounced difference in the Lys-27 — Lys-15 distances could be easily realised on the three-dimensional molecular model as being due to local conformational mobility of the corresponding side chains.

Sincerely yours,

Vladimir Bystrov

References:


FIGURE: a) and c) — F-19 NMR spectra of A and B derivatives, respectively.
b) and d) — Their computer decomposition.
August 5, 1981

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

Title: 13C Investigation of Mesoionic Compounds

Dear Professor Shapiro:

A certain J. S. Baum (well-known to this author) published some work on cycloaddition reactions of activated isocyanates with anhydro-4-hydroxythiazolium systems (1). These reactions were claimed to give cycloadducts of structure 2, but a recent paper (2) cast some doubt on the structure of these compounds, and further head scratching afforded two possible mesoionic structures 3 and 4 for the products of these reactions. As these compounds proved to be quite stable over the years, and since Baum and Potts have a good pack rat instinct and never discard anything, it was decided to reinvestigate the 1H, and investigate the 13C NMR spectra to clear up all doubt.

The 1H spectra pointed toward the mesoionic compounds right away, with the N-CH3 chemical shifts at 4.208 for the a structures, and 4.218 for the b structures, leading one to postulate a methyl group on positively charged nitrogen. The 13C spectra (coupled and decoupled; chemical shifts are shown on the structures 4a and 4b) are consistent with structures 4a and 4b, as the singlets at 113.18 and 113.08 showed. The JCH coupling constants for the N-CH3 groups again indicated substantial positive charge on nitrogen, these values being 145.8 Hz for 4a and 145.7 Hz for 4b.

I trust this contribution will serve to reinstate me in the good graces of the TAMUNN after a too-long hiatus.

Sincerely yours,

Mary W. Baum

*On each compound, shifts bearing * may be
incorrectly assigned.
SOLID-STATE $^{13}$C NMR OF PLATINUM COMPOUNDS

Dear Barry,

For a number of years we have been operating a home-built spectrometer for high-resolution $^{13}$C NMR studies of solids at 22.6 MHz using the high-power-decoupling, cross-polarization and magic-angle rotation suite of techniques [1]. Recently we have been looking at a number of samples of platinum complexes supplied by the Johnson Matthey Research Centre. We have now become accustomed to seeing effects which differ from those in solution, and, indeed, we are puzzled by some of the features displayed by the platinum compounds. However, we feel the attached spectrum might interest TAMUNMR readers, since it provides a clear NMR example of the freezing of molecular motion in the solid state, and we believe it is the first observed example for metal compounds using high-resolution $^{13}$C NMR. In this case the solid-state structure is known from X-ray studies [2], and the ring conformation ensures the two CH$_2$ groups bonded to the six-membered ring are non-equivalent, giving $^{13}$C resonances at 35.7 and 28.4 ppm from TMS. In solution, of course, rapid inversion of the six-membered ring results in equivalence of these two CH$_2$ carbons. The solid state thus gives better structural information and also allows shielding effects to be better related to molecular geometry.

Our spectra should show splittings due to (Pt,C) coupling, but clearly this is not so for the attached Figure. However, splittings of 72 Hz and 46 Hz are shown for the methine and methyl carbons, respectively, of platinum bisacetylacetonate. These values relate well to those found [3] in solution.

We hope this keeps us "solvent" with respect to TAMUNMR.

Best wishes

R.K. HARRIS K.J. PACKER C.J. GROOMBRIDGE


CP-MAR $^{13}$C NMR SPECTRUM
of cis-(diammino)(1,1-cyclobutane dicarboxylato)Pt(II)

CT 10 ms
Recycle 2 s
12000 cycles
Single contact
Flip-back
August 13, 1981

Dear Professor Shapiro:

Recently we reported the observation of quadrupolar splittings for deuterated solvents in swollen, uniaxially strained elastomers.\textsuperscript{1} We are currently restricted to examining such materials in a single orientation with our iron-magnetic high-resolution spectrometer: the strain direction is at right angles to the field $H$. However, this summer Bertrand Deloche and I were able to devise a technique for examining such samples in uniaxial compression. Moreover, we could rotate the sample within the 5mm sample space to specified orientations of the compression direction $C$ relative to $H$. When $\theta = 90^\circ$, $H$ is in the plane of the compressed elastomer and the translational diffusion of the solvent appears to be insufficient to completely average the quadrupolar interactions to a discreet doublet. The resulting spectrum is reminiscent of those observed in cholesteric liquid crystals for slowly diffusing species.\textsuperscript{2} We are pursuing these studies to probe "nematic-like" arrangements of the polymer chains in deformed elastomers.

Very truly yours,

Edward T. Samulski

References:

Never before in the history of NMR has time so optimally been shared between processes. Bruker's DISNMR, the first true time-sharing NMR data system allows you to process several data sets simultaneously. For example: you may perform more than one Fourier transformation while executing a PASCAL program at the same time. With the virtual memory capability of DISNMR and multi-tasking architecture acquisition of data never interferes with any I/O devices or whatever jobs are performed by the system. It permits disc acquisition and transformation of up to 256K data tables. This is illustrated by the ultrahigh-resolution 500 MHz spectrum showing the expanded ethylbenzene methylene quartet at 2.65 ppm, obtained by disc acquisition of a 128K FID and subsequent transformation of 256K data points, revealing a stunning amount of fine structure. DISNMR does not require new hardware; it is fully compatible with all ASPECT data systems. The new DISNMR puts Bruker's WM series of spectrometers in a class by itself.

For complete facts simply write "DISNMR" on your stationery and mail it to Bruker Instruments, Inc., Manning Park, Billerica, MA 01821.

In high-field NMR there is simply no alternative.
For information on NMR and EPR instrumentation and accessories your prime source is the nearest Bruker office:

Bruker Instruments, Inc.
Manning Park, Billerica, MA 01821
(617) 667-9580

201 San Antonio Circle, Suite 152
Mountain View, CA 94040
(415) 941-3804

539 Beall Ave., Rockville, MD 20850
(301) 762-4440

1603 Darwin Court, Wheaton, IL 60187
(312) 668-4441

Call or mail this coupon to the nearest Bruker office.

Please send me more information on the new DISNMR

The information is needed for future planning □ for purchase after 6 months □ for immediate purchase □ Please have your specialist contact me □
My telephone number is: (               )

I am also interested in NMR systems □ My field of application is: ________________________________

Name: ____________________________________________
Institute/Company: __________________________________
Address: ___________________________________________
City/State/Zip: _______________________________________
Dear Professor Shapiro,

Title: $^{13}$C chemical shift assignments, spin-lattice determination and quantitative analysis of a model elastomeric polyurethane

We have analyzed in our laboratory a number of linear elastomeric polyurethanes by $^{13}$C pulsed NMR spectroscopy using a Bruker WH90.

The aim of this work was to check the quantitative NMR analysis according to the data from laboratory synthesis. We chose a sample amid these polyurethanes (PIPNEG) that was of a particular interest by virtue of its components: neopentylglycol (NEO), hydroxy pivalic acid (PIV), butanediol (BUT), adipic acid (ADA) and methane-bis (p-phenyl-isocyanate) (MDI). A 11% (w/v) solution was used in 10 mm. o.d. tubes at 112°C under the following experimental conditions: pulse angle=55°, cycle time: 7 sec, sweep width: 6000 Hz, memory: 8K. About 2000 sweeps were needed to obtain a satisfactory signal to noise ratio in the decoupled spectrum. To justify the cycle time we report a list of $T_1$ values of the different carbons of the copolymer (Table 1). From the comparison of the experimental chemical shifts with those of model compounds and of other polyurethanes, the lines of the spectrum were assigned and it was possible to obtain a quantitative analysis of the components. For the determination of the $T_1$ values the following experimental conditions were adopted using the usual inversion recovery method: cycle time: 15 sec, number of spectra: 8, delay between 180° and 90° pulse: 12 sec, 12/1.85, 12/1.85, etc. We remember that the $T_1$ values determined at 130°C for another polyurethane sample (BUT/MDI/ADA) of similar molecular weight: $\bar{M}_n=25000$, as reported in a previous letter (1), were much higher for the corresponding carbon atoms.
This was partially justified from the different nature of the copolymer and the higher temperature. The $T_1$ values of the carbon atoms corresponding to the lines used for the quantitative determination are in the range 0.6-1.4 sec. We calculated the molar percentages of the comonomers on the basis of the following chemical shifts: $\delta_1$ for NEO, $\delta_2$ for PIV, $\delta_3$ and $\delta_8$ for BUT, $\delta_2$ for ADA and $\delta_4$ for MDI (Table 2). Data are reported from the $100$ and $10$ Hz/cm integrals together with the synthesis data.

<table>
<thead>
<tr>
<th>$^{13}C$ Chemical shifts $(\text{EMDS} = 0)$</th>
<th>Assignments</th>
<th>$T_1$ (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\delta_1 = 19.43$</td>
<td>$\text{CH}_3$ NEO</td>
<td>1.3</td>
</tr>
<tr>
<td>$\delta_2 = 20.08$</td>
<td>$\text{CH}_3$ PIV</td>
<td>1.4</td>
</tr>
<tr>
<td>$\delta_3 = 22.03$</td>
<td>$\beta$ $\text{CH}_2$ ADA</td>
<td>0.9</td>
</tr>
<tr>
<td>$\delta_4 = 23.53$</td>
<td>$\beta$ $\text{CH}_2$ BUT</td>
<td>1.0</td>
</tr>
<tr>
<td>$\delta_5 = 31.325$</td>
<td>$\alpha$ $\text{CH}_2$ ADA</td>
<td>4.4</td>
</tr>
<tr>
<td>$\delta_6 = 32.885$</td>
<td>$\text{C}$ $\text{NEO}$</td>
<td>0.6</td>
</tr>
<tr>
<td>$\delta_7 = 40.55$</td>
<td>$\text{C}$ $\text{PIV}$</td>
<td>0.6</td>
</tr>
<tr>
<td>$\delta_8 = 61.935$</td>
<td>$\alpha$ $\text{CH}_2$ BUT</td>
<td>0.6</td>
</tr>
<tr>
<td>$\delta_9 = 66.81$</td>
<td>$\alpha$ $\text{CH}_2$ NEO</td>
<td>0.7</td>
</tr>
<tr>
<td>$\delta_{10} = 67.39$</td>
<td>$\text{CH}_2$ O PIV</td>
<td>1.0</td>
</tr>
<tr>
<td>$\delta_{11} = 68.01$</td>
<td>$\text{CH}_2$ O of MDI</td>
<td>1.0</td>
</tr>
<tr>
<td>$\delta_{12} = 117.05$</td>
<td>$\text{CH}_2$ O of MDI</td>
<td>1.0</td>
</tr>
<tr>
<td>$\delta_{13} = 126.86$</td>
<td>$j,j$ of MDI</td>
<td>5.1</td>
</tr>
<tr>
<td>$\delta_{14} = 133.68$</td>
<td>$j,j$ of MDI</td>
<td>4.6</td>
</tr>
<tr>
<td>$\delta_{15} = 135.31$</td>
<td>$\text{OCO NH}$</td>
<td>undet.</td>
</tr>
<tr>
<td>$\delta_{16} = 151.88$</td>
<td>$\text{COO ADA}$</td>
<td>undet.</td>
</tr>
<tr>
<td>$\delta_{17} = 170.21$</td>
<td>$\text{COO PIV}$</td>
<td>undet.</td>
</tr>
<tr>
<td>$\delta_{18} = 170.40$</td>
<td>$\text{COO PIV}$</td>
<td>undet.</td>
</tr>
<tr>
<td>$\delta_{19} = 172.6$</td>
<td>$\text{COO PIV}$</td>
<td>undet.</td>
</tr>
</tbody>
</table>

(*) Undetermined because of overlapping with $\text{dmsod}_{d6}$
TABLE 2

<table>
<thead>
<tr>
<th>% Moles</th>
<th>100 Hz/cm</th>
<th>10^1 Hz/cm</th>
<th>Synthesis data</th>
</tr>
</thead>
<tbody>
<tr>
<td>W' MDI</td>
<td>18.5 38.6</td>
<td>20.0 38.7</td>
<td>17.1 37.9</td>
</tr>
<tr>
<td>W' ADA</td>
<td>20.1 38.6</td>
<td>18.7 38.7</td>
<td>20.8 37.9</td>
</tr>
<tr>
<td>W' PIV</td>
<td>24.7</td>
<td>23.5</td>
<td>24.2</td>
</tr>
<tr>
<td>W' BUT</td>
<td>15.1 36.7</td>
<td>13.4 37.8</td>
<td>13.7 37.9</td>
</tr>
<tr>
<td>W' NEO</td>
<td>21.6</td>
<td>24.4</td>
<td>24.2</td>
</tr>
</tbody>
</table>

We can observe that the relationship $W'_{MDI} + W'_{ADA} \approx W'_{BUT} + W'_{NEO}$ is obeyed and that we obtained a satisfactory fit with the synthesis data.

Yours sincerely

Giorgio Gurato  
G. GURATO

Gianluca Lunardon  
G. LUNARDON(*)

Giorgio Rigatti  
G. RIGATTI(**)

(*) Montepolimeri SpA/URI/PM

(**)Istituto di Chimica Fisica of the University, via Loredan 2, Padova

References

(1) G. Gurato and G. Rigatti. TAMUNN n°250 July 1979, pag.41
Professor B.L. Shapiro,
Department of Chemistry,
Texas A&M University,
College Station,
Texas 77843, U.S.A.

Dear Professor Shapiro

35Cl/37Cl and 79Br/81Br Isotope effects.

In response to the letter from Dr. Brevard (TAMU No. 272) we note that the additional fine structure he observed in the 103Rh spectrum of RhCl₆⁻ from 35Cl/37Cl isotope effects is identical to our observation of the same effect in the 195Pt spectra of PtCl₂⁻ and PtBr₂⁻ (1).

It would seem that under favourable conditions small isotope effects such as these can be readily detected in both organic and inorganic molecules. A recent example of this comes from work undertaken in this laboratory on the 31P spectra of PCl₃ and PBr₃ where the isotope effects can be readily seen (Figure 1) (2). The increasing use of high field instruments will undoubtedly reveal more such effects and n.m.r. spectroscopists should consider the possibility of isotope shifts before seeking alternative explanations (mass spectroscopists have recognised these splitting patterns for some time).

Please credit this contribution to the ULIRS/Ed Randall account.

Best Wishes.

Yours Sincerely,

Peter Sadler

Prof. I.M. Ismail

G.E. Hawkes

M.J. Buckingham
REFERENCES


$^{31}P$ spectra at 162 MHz from ULIRS WH-400 NMR Service
at Queen Mary College.

$\text{PCl}_3$ (+ 5\% acetone-d$_6$) at -100$^\circ$.

$\text{PBr}_3$ (+ 5\% acetone-d$_6$) at -70$^\circ$. 

$^{35}\text{Cl}$

$^{37}\text{Cl}$

$^{35}\text{Cl}(^{37}\text{Cl})_2$
Prof. B.L. Shapiro  
Texas A&M University  
Department of Chemistry  
College Station, Tex 77843, U.S.A  

"Size-resolved NMR"

Dear Professor Shapiro,

Thank you for your yellow reminder. The pulsed-gradient spin-echo experiment, when performed in the FT mode (*) is remarkably useful in providing quantitative physico-chemical information in the form of molecular self-diffusion coefficients (D) for complex systems in solution (**) . We have even noted that it may be of use as an analytical tool.

With the $90^\circ$-$t$-$\delta$-$t$-$180^\circ$-$t$-$\delta$-$t$-acquisition sequence ($t-\delta>t=\Delta$) the Fourier-transformed spin-echo signals decay with increasing $\delta$ according to

$$A_i = c_i \cdot \exp\left(-\frac{\gamma G \delta}{2D_i}(\Delta - \frac{1}{2}\delta)\right)$$

if $\Delta$ is kept constant. Regardless of any J-modulation or $T_2$ effects this will be true for each part of an NMR signal if there is only one component in solution, because all nuclei in a molecule necessarily diffuse at equal rates on the time-scale of the experiment. Therefore (apart from a common amplitude factor) the whole bandshape is constant for all $\delta$-values under these conditions. An experiment ($G \approx 1$ Gauss/cm) is illustrated in figure a (neat decanol). Even at high gain the decanol difference spectrum is essentially zero. (These spin-echo spectra are recorded at arbitrary vertical gain, and the difference spectra have been obtained through data manipulation so as to null the $CH_2OH$ signals by subtraction in memory.) Note that the bandshape from a multicomponent sample necessarily must change during an experiment of the present kind (unless all molecules have equal self-diffusion coefficients). This is illustrated in b (50/50 decane-decanol).

Nulling the $1,2 OH$ (and thus the whole decanol bandshape)
leaves only the decane spin-echo spectrum. The same procedure could, of course, be extended to more than two components, provided that each component gives at least one isolated NMR band.

A longer version of this letter will appear in Anal. Chem. in the near future. With best wishes.

Yours sincerely

/Peter Stilbs/

(*) The idea on which FT-PGSE is based originates from a paper by Vold, Waugh, Klein and Phelps, J. Chem. Phys. 48 (1968) 3831. The first experiments were apparently made by James and McDonald in 1973.

Dear Dr. Shapiro:

some time ago we reported interbenzylic $^{31}$P,$^{31}$P spin coupling constants over five, six, and seven bonds in the xylylene diphosphonates.

\[
\begin{align*}
\text{CH}_2\text{P}(\text{OEt})_2 & \quad \text{isomer} \\
\text{CH}_2\text{P}(\text{OEt})_2 & \quad |J_{\text{pp}}| \quad [\text{Hz}] \\
\text{ortho} & \quad 9.0 \\
\text{meta} & \quad 3.1 \\
\text{para} & \quad 7.8
\end{align*}
\]

These $J_{\text{pp}}$ are thought to be transmitted mainly through the aromatic π-system, as are the corresponding $J_{\text{CH}_3\text{CH}_3}$ in the xylenes. One expects opposite signs for the couplings over an even and over an odd number of bonds. Because the spectra of the series are not suitable for double resonance experiments, we synthesized, which contains the three types of $J_{\text{pp}}$ in the same molecule and which gives a nice three spin system of $^{31}$P nuclei when the protons are broadband decoupled (Figure 1a). Having no triple resonance facilities for our NMR spectrometer (at 162 MHz), we used the INDOR-like selective population inversion difference

\[
\begin{align*}
\end{align*}
\]
Fig. 1. (a) 162 MHz $^{31}$P($^1$H) NMR spectrum of $2$ (d$_6$-acetone solution); (b) simulated; (c) SPIDIF spectrum with inversion of transition 12; (d) as in (c) but inverting transition 1.

spectroscopy$^3$ instead. We inverted a $^{31}$P transition by applying a selective pulse (to line 12 in Fig. 1c; to line 1 in Fig. 1d) followed by the nonselective observing pulse. From the resulting FID the normal FID was then subtracted. The broadband $^1$H decoupler was permanently on. The pseudo-INDOR spectra in Figures 1c and 1d show that the sign of the smallest coupling, $J_{meta}$, is opposite to those of the two larger ones, $J_{ortho}$ and $J_{para}$, as expected. The simulated spectrum is shown in Fig. 1b.


Yours sincerely,

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

Dear Barry:

USE OF THE DIXON SEQUENCE FOR SUPPRESSION OF SPINNING SIDEBANDS IN SOLID STATE SPECTRA

We recently have been using the Dixon sequence for spinning sideband elimination on the FX-200 equipped with CP/MAS solids accessory. Our initial experiments indicate this technique will prove quite useful in removing spinning sidebands which are inevitable in high field solid state spectra. The $^{13}$C CP/MAS spectrum of vanillin is depicted in Figure 1a complete with complicating spinning sidebands ($v_{\text{rot}} = 3630$ Hz, Kel-F rotor). Figure 1b shows the addition spectrum of the same sample which removes the first order spinning sidebands as a result of the Dixon sequence. Solid state spectra of hexamethylbenzene in Figures 2a - c show the "normal" spectrum, $180^\circ$ phase inverted 1st order SSB spectrum and the addition spectrum (a-b), respectively.

Note the presence of 2nd order SSB's in the HMB spectrum which may be removed either by additional applications of the Dixon technique or, from a practical viewpoint, by rapid spinning rates ($\sim 4$ KHz). Quantitation using this technique may prove difficult due to the substantial delays required in the pulse sequence and we are investigating these possibilities as well as the use of SSB free spectra for other nuclei (e.g. $^{31}$P).

We feel the Dixon sequence will become a very useful technique in solid state NMR and we tip our rotors to Dr. Dixon for development of this clever approach.

Sincerely yours,

H. Cecil Charles  
Applications Chemist

Michael J. Albright  
NMR Product Manager

HCC:MJA/mjd

Vanillin
"NORMAL" Spectrum

Fig. 1a

SSB FREE Spectrum

Fig. 1b

ppm
Hexamethylbenzene

"NORMAL" Spectrum
\( \uparrow = \text{SSB} \)

Fig. 2a

180° phase inverted SSB spectrum

Fig. 2b

Addition SSB spectrum

Fig. 2c

250 200 150 100 50 0 -50 ppm
PTS 200 FREQUENCY SYNTHESIZER

- 1 - 200MHz
- +3 to +13dBm output
- choice of resolution
- low phase noise
- fast switching, 5-20 micro-sec.
- fully programmable, BCD or IEEE 488 BUS
- modular flexibility
- low power consumption, high reliability
The PTS 200 is a generator of precision frequencies. It transfers the accuracy and stability of a frequency standard (built-in or external) to any output frequency between 1MHz and 200 MHz. Steps as fine as 0.1Hz are available and all functions are remotely programmable.

The PTS 200 is a direct frequency synthesizer of novel design providing high performance for many demanding applications. With its low spurious outputs, fast switching, low phase noise and wide choice of resolution (finest step), it is suited for a range of uses from NMR to communications or ATE.

This new system of synthesis has drastically cut complexity and parts count. The attendant reduction of primary power input and dissipation (less than 50% of that of competitive designs) is a major factor in the reliability which is further enhanced by the use of ceramic ICs, all metal-can transistors and a packaging system maximizing mechanical integrity and stability while keeping weight low. For ease of service most modules are identical and of plug-in design.

**SPECIFICATIONS**

<table>
<thead>
<tr>
<th><strong>FREQUENCY</strong></th>
<th>Range:</th>
<th>1MHz to 200 MHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resolution:</td>
<td>0.1Hz to 100 KHz steps (optional in decades)</td>
<td></td>
</tr>
<tr>
<td>Control:</td>
<td>Local by 10-position switches. Remote by TTL-BCD, 1248, buffered or by IEEE 488 BUS. R/L transfer programmable.</td>
<td></td>
</tr>
<tr>
<td>Switching Time:</td>
<td>20 micro-sec. (within 0.1 rad at new frequency)</td>
<td></td>
</tr>
</tbody>
</table>

| **OUTPUT**          | Level:                        | +3 to +13dBm, (1V) into 50 ohms, metered in dBm and volt |
|---------------------| Flatness:                     | +, −0.5dB                                    |
|                     | Impedance:                    | 50 ohms                                       |
|                     | Control:                      | Manual by F/P-control, remote by voltage, (+0.63 to +2.00V) |
|                     | Settling Time:                | 20 micro-sec.                                |

| **SPURIOUS OUTPUT** | Discrete:                     | 70dB                                            |
|---------------------| Harmonics:                    | −30dB at full output, (−40dB at lower level)   |
|                     | Phase Noise:                  | −63dBc, (0.5Hz to 15KHz), incl. effects of int. standard |
|                     | L (1Hz):                      | 100Hz/105dBc; 1KHz/115dBc; 10KHz/123dBc |
|                     | Noise Floor:                  | −135dBc/Hz                                    |

<table>
<thead>
<tr>
<th><strong>FREQUENCY STANDARD</strong></th>
<th>Internal:</th>
<th>3 x 10⁻⁹/day or 1 x 10⁻⁸/day (optional)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aux. Drive:</td>
<td>5.000 or 10.000MHz, 0.5V into 300 ohms</td>
<td></td>
</tr>
<tr>
<td>10.000MHz, 0.4V into 50 ohms</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Oper. Ambient:</strong></th>
<th>0 to 55°C, 95% R.H.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Power:</strong></td>
<td>105-125V, 50-400Hz, 45Watts</td>
</tr>
<tr>
<td><strong>Dimensions/Weight:</strong></td>
<td>19 x 5½ x 18” (Relay rack or bench cabinet, 35 lbs.)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>PRICES</strong> (domestic)</th>
<th>Resolution:</th>
<th>100KHz</th>
<th>10KHz</th>
<th>1KHz</th>
<th>100Hz</th>
<th>10Hz</th>
<th>1Hz</th>
<th>0.1Hz</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th><strong>Freq. Standards:</strong></th>
<th>3 x 10⁻⁹/day, (Oven)</th>
<th>$450.—</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>(Option)</strong></td>
<td>1 x 10⁻⁸/day, (TCXO)</td>
<td>$200.—</td>
</tr>
<tr>
<td><strong>IEEE 488 Interface:</strong></td>
<td>$650.—(This option replaces the standard parallel entry BCD interface)</td>
<td></td>
</tr>
</tbody>
</table>

Delete Front Panel Controls: —$200.—
More $^2\text{H}/^1\text{H}$ Isotope Effects on $^{13}\text{C}$ Chemical Shifts

Dear Barry,

we are again behind schedule with our contributions, and I hurry up to inform your readers that another rule generally accepted for deuterium isotope effects on carbon-13 chemical shifts has been found violated. In phenylacetylene we observed that the isotope effect over two bonds is nearly twice as large as that over one bond:

$$\text{C} \equiv \text{C} - \text{D} \quad \Delta = 0.253, \quad ^2\Delta = 0.426 \text{ ppm}$$

This is, to our knowledge, the first exception to the rule formulated by Batiz-Hernandez and Bernheim, and frequently cited by Jameson, that the isotope effect is decreasing with the number of bonds between the two atoms involved.

We are presently engaged in a more complete study of structural effects and hope to find other interesting information from molecules with large structural differences (hybridization, bond angles etc.).

Sincerely yours,

H. Günther

J. Wesener


Subject: Improvement of the Redfield sequence

Dear Professor Shapiro,

In biological samples exchangeable protons are interesting. When the exchange is fast, these protons can only be detected in H₂O as the solvent. The concentration of the relevant protons is very low. The problem is the measurement of these weak resonances in the presence of the nearby, strong water peak.

The Redfield sequence (J. Magn. Res. 19, 114 (1975)) is one of the best solutions for the problem. With this pulse sequence the watertransient on our CXP 300 is about 100 times noise. Our improvement reduces the watertransient to less than 2 times noise. The phase and amplitude of the watertransient is more or less random. This is the reason for the distortion free baseline.

The Redfield sequence consists of a 2(+x)1(-x)4(+x)1(-x)2(+x) pulse. The length of this pulse is optimized for a minimal watertransient. The improvement consists of optimizing the length of the pulse and the phase of (-x). This is easily done on our CXP 300 by minimizing the watertransient by alternately turning the knob of the pulse length and the knob of the phase of (-x).

Please credit this contribution to the account of Prof. T.J. Schaafsma.

Adrie de Jager
Dept. of Mol. Physics

Chrit Moonen
Dept. of Biochemistry

Agricultural University/Transitorium, De Dreijen 11/6703 BC Wageningen/The Netherlands/Tel. (08370) 82044/82634
Figure 1. 1mM Flavodoxine (MW 15,000) in 90% H$_2$O 10% D$_2$O; spinning 5 mm tube; 20,000 transients of 0.5s; no data-manipulating; 12 to -1.5 ppm.

Figure 2. Part of 1 with Gaussian multiplication.
Dr. B.L. Shapiro,
Department of Chemistry,
Texas A & M University,
College Station, TX 77843, U.S.A.

27th August, 1981.

Dear Dr. Shapiro,

We have used n.m.r. to follow the rates of solvolytic reactions over several hours in a thermostatted HA100 n.m.r. probe at 25°C. The "standard" procedure we tried initially utilised liquid nitrogen as the coolant, but careful adjustment of the settings for flow and heater controls was required to obtain specification precision (±0.5°C) over several hours. These settings varied significantly from day to day. Some kinetic runs were spoiled by temperature drifts of several degrees - temperatures were determined using the temperature dependent OH signal of methanol and checked using calibrated thermistors after rapidly removing the solution from the n.m.r. probe.

As only slight cooling of the probe is required to obtain 25°C we tried ice/water as the coolant. This leads to both more reliable and more convenient thermostating of the n.m.r. probe.

The reactions we are examining are displacements of alkyl halides or esters (RX where X = halogen, sulfonate, phosphonate) by solvent (ROH where R = H, alkyl or tBu alkyl). Such reactions are usually followed kinetically using conductometric or titrimetric techniques. The advantages of n.m.r. are: (i) strongly acidic reaction media, e.g. aqueous sulfuric acid, can be employed; (ii) small amounts of solvent are required - important for fluorinated alcohols as reaction media; (iii) several signals of starting material or products can be monitored readily; (iv) theoretical "infinity" values for disappearance of starting materials can be
obtained easily. The main disadvantage of n.m.r. is that lower precision kinetic
data is obtained because relative signal heights or areas cannot be obtained
precisely and gases provide less precise thermostating than liquids (±0.01°C
or better).

This work is being carried out by S. Jackson and S.J. Morris and we are
grateful for skilled technical assistance from G. Llewellyn and M. Nettle.

Please credit this contribution to the account of Dr. J.M. Williams.

References.

1979, 101, 2486.

Yours sincerely,

[T.W. Bentley]

T.W. Bentley.
August 11, 1981

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

Dear Professor Shapiro,

We have an opening for an NMR spectroscopist to manage the NMR facilities at Columbia. We would very much like to hear from anyone you feel would be a suitable candidate for the job.

The position involves serving as a consultant in NMR problems to our various inorganic and organic research groups as well as carrying out cooperative research with them. We are looking for someone with a thorough grasp of NMR experimental technique and spectral analysis. While a sound understanding of NMR equipment is highly desirable the candidate would not be responsible for more than routine maintenance repairs to the spectrometers. Assistance in running routine spectra would also be available.

Salary depends on experience and qualifications and eventual promotion to the level of Senior Research Associate would be possible.

Applications are to be sent to me and should include a curriculum vita, a list of publications and the names of two referees.

Your help is greatly appreciated.

Sincerely yours,

W. Clark Still
Professor of Chemistry

WCS:jb
August 27, 1981

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

Subject: BRUKER HX-60 SPECTROMETER FOR SALE

Dear Barry,

We have a Bruker HX-60 NMR Spectrometer for sale at a price of $5,000 plus removal and shipping costs (est. $2,500). This spectrometer is a CW instrument but it can be upgraded for FT or solids NMR. The configuration follows:

1. HX-60 console
   - $^2$H lock
   - H$^1$ observe module

2. Magnet
   - Bruker 15" 2.5 cm gap high resolution magnet with power supply

3. Probes
   - 3 each 5 mm H$^1$ insert
   - 2 each 10 mm H$^1$ insert

Any interested parties should write to me at the above address or call (315) 423-4026 or 423-1021.

Yours sincerely,

[Signature]

George C. Levy
Professor

GCL:jrd
A **POSTDOCTORAL POSITION** is available immediately at the Department of Chemistry, University of Missouri, to study water in biopolymers, liquid crystals, and to collaborate with other research groups in research in the biochemical or biophysical areas using pulse NMR techniques. Available facilities include a Nicolet 300 MHz wide-bore spectrometer, a low-field FT spectrometer and a home-built pulse spectrometer. Stipend $14,000/year, available for at least 2 years.

A **NMR SPECTROSCOPIST POSITION** is also available for November, 1981 or shortly after to maintain and operate the above NMR facilities, to advise and train general users and to engage in collaborative or independent research. Salary $18,000-$20,000, dependent on experience and qualifications.

Expertise in NMR in the physical, biophysical or biochemical areas is required in both positions. For inquiry or application (please submit c.v., references, and a list of publications) should be made to:

Professor T.C. Wong  
Department of Chemistry  
University of Missouri  
Columbia, MO 65211

or call: 314-882-7725  
or 314-882-2439
Nicolet Supercon Ff-NMR Spectrometers

Uncompromising performance, limitless adaptability.

Our spectrometer systems have been conceived and designed to provide optimum performance while being fully adaptable to new techniques with minimal cost and difficulty. More than just a collection of instruments, they represent a completely modular approach to FT-NMR instrumentation that allows the user to expand his system as his research needs grow and to easily accommodate new experimental techniques as they develop.

Outstanding Nicolet features include these:

- A full range of superconducting magnets from 4.7T to 11.7T (200MHz to 500MHz proton frequency range), in both wide-bore and narrow-bore configurations.
- Multinuclear observation with a wide variety of fixed-tune and broadband probes.
- Simultaneous acquisition, processing, and plotting for greater sample throughput.
- Simplified control of spectrometer operations and parameters by using easy keyboard commands.
- Advanced Nicolet 1180E Data System with 128K/20-bit memory, 256-step pulse programmer, and the most comprehensive FT-NMR software package available.
- Extended dynamic range performance with 40-bit acquisition and floating-point processing.
- An expandable pulse-sequence library, including T1, T2, Redfield, INEPT, homonuclear and heteronuclear-2D-FT, etc.
- Convenient computer control of field shimming, observe and decoupling frequencies, sample temperature, and probe-tuning.
- Precise digital plotting with full annotation of spectral parameters and flexibility of hardcopy format.

The versatile Nicolet spectrometers provide the user with the ability to easily adapt to the newest techniques and experimental configurations.

Some of these are:

- High resolution studies of solids with Waugh-Pines cross-polarization and magic-angle spinning.
- Automated T1 and T2 measurements.
- High sensitivity wide-bore 13C studies of high molecular weight polymers.
- Chemical dynamics studies.
- Temperature-programmed experiments.
- 31P experiments on living organs.

NICOLET MAGNETICS CORPORATION
A NICOLET INSTRUMENT SUBSIDIARY
145 East Dana
Mountain View, California 94041
TWX: 910-379-6589
Telephone: 415-969-2076
**THE SOLID LEADER in NMR**

With Multi-Nuclear/ Multi-Field Solid State Probes

- **High field** solid sample probe for JEOL's 200 MHz SCM!
- **Tunable heads** — interchangeable plug-in matching units for observation of
  - $^{13}$C (≈50 MHz)
  - $^{31}$P (≈80 MHz)
  - $^{29}$Si (≈40 MHz)
  with one probe!
- Self starting rotor/stator design!
- High speed magic-angle sample spinning (>4.0 KHz)!
- "Magic lift probe" for quick sample change and probe insertion!
- All this, in addition to a full line of dual and broad-band high resolution liquid sample probes!

---

**Low Cost/ Low Field Solids Probe**

Chemagnetics solids probe for JEOL's 60 MHz NMR.

- Solid sample probe for JEOL’s 60 MHz CP/MAS FT-NMR.
- Best results for coal, shale oil & rigid polymers.
- Large sample size (.5cc).
- Probes available for observation of $^{13}$C, $^{31}$P and $^{29}$Si!

---

235 Birchwood Ave., Cranford, NJ 07016
201-272-8820