Sayer, I. and Davies, D.B.
Static and Dynamic Molecules in Disordered Solids

Smith, R.L. and Hunt, C.T.
P-31 NMR of Adsorbed DFP on Ion Exchange Resins

MacKenzie, N.E.
Specific Deuteration in Proteins.

Stibbs, P.
Less Sample, More Signal.

Amides Revisited: Cis/Trans Isomerism in BzC-Aminoacids

James, T.L., Gonzalez-Mendez, R., de Olivares, J., and Litt, L.
Tailored Excitation Using Fourier Transform Pairs: A Frequency-Space Notch Filter and One-Pulse Multislice Imaging Using Shaped Radiofrequency Pulses

Moskau, D. and Gunther, H.
COSY Experiments with Quadrupolar Nuclei: 7Li and 2H

Schraml, J.
How to Present 20 COSY Spectra

Borer, P.N. and Levy, G.C.
Natural Abundance 13C NMR of Small Double-Stranded DNA Molecules

Frenkel, G.F. and Chow, A.
Platinum Hydride Lineshape Analysis; HITNET: H5P Budget in Jeopardy

Tschufin, R. and Miyama, Y.
"Free Running" of Nicotol Explorer Interface

Rae, I.A.
Spin-Spin Coupling Mechanism

Havens, J., Reiser, J., and Amundson, K.
Solid-State NMR Characterization of Poly(aryletherketones)

Graveron, D., Briguet, A., and Lahr, H.
Modification de la Sequence d'Echos Stimules et Mouvements Liquidiens

Boyko, W.J.
HETCOR of Ortoriclyl Bromide

Buchanan, G.K.
Stereochemical Dynamics of a Dicyclohexano-14-C-4

Clem On, E. and Nettel, D.A.
In Search Of

Jansson, R., Tijihj, E., and Veeman, W.
High Temperature 23Na NMR of Zeolite MA

Ngo, J.T. and Morris, P.G.
Selective Excitation: Something for Nothing

Fontaine, E.A.
Performance of 1H Decoupling on a Bruker AM-400 'H-{1} Probe Head

Chazin, W., Rance, M., Dalvit, C. and Wright, P.E.
Discrimination of Couplings in Triple Quantum Filtered COSY Spectra

Eaton, G.R. and Eaton, S.S.
Spectral-Spatial 2-Dimensional EPR Imaging

Turner, C.J.
Spin Echo NMR of Intact Lenses

de Ropp, J., Moonen, C., and Unger, S.
Equipment for Sale

Medley, J.M.
Effect of Salt Form of C-13 Spectra of Antibiotics

Farmer, B.T. II and Brown, L.R.
Sensitivity in the ROESY Experiment; Positions Available
INEXPENSIVE • RELIABLE
PRACTICAL • GUARANTEED

THE BEST POSSIBLE ANSWER FOR LOWER-END NMR SPECTROSCOPY NEEDS.

COMPARE THESE OUTSTANDING FEATURES
OF OUR NEW WG-5mm-THRIFT-7 TUBE:

• 100% Wilmad inspection of structural parameters.
• Spinning Reliability. Passes our stringent Spinner Bearing Test which determines straightness and spinning stability using a Precision Bore Bearing just 0.002" larger than the tube O.D. No halted data accumulations or scratched tubes. Send for your free 5mm Spinner Bearing today.
• Round bottom is standard. Flat bottom ... no extra charge.
• Non-Pyrex compatible borosilicate glass.
• Packaged individually for maximum protection.
• Standard length is 7". Available in 8" and 9" lengths. (Add 15c per inch.)
• Immediate shipment from available stock.

The Wilmad 5mm THRIFT tube is one of the most important recent developments in NMR tube manufacture. It is not recommended for use in high resolution spectrometers, but it provides exceptional performance in lower-end spectroscopic investigations and, at the same time, retains its low cost. It is manufactured from Non-Pyrex compatible borosilicate drawn tubing and is carefully selected for size and structure. At 95¢ each, this tube is really a bargain. In fact, it is an infinitely better bargain in both price and quality than the following Norell tubes: 508-UP ($4.50 each); 507-HP ($3.50 each); 506-P ($2.50 each); 505-P ($1.80 each); XR-55 ($2.00 each); and 502 (80¢ each). Our examination of numerous Norell tubes has shown that the only significant difference between them is price. They consistently fail to meet even the most liberal structural standards used to define them.

Even with our less expensive 5mm THRIFT tubes, you'll soon learn why IT PAYS TO STANDARDIZE ON WILMAD!

WILMAD GLASS COMPANY, INC.
Serving the Spectroscopic Aftermarket
Route 40 and Oak Road, Buena, NJ 08310, U.S.A.
Phone: (609) 697-3000 • TWX 510-687-8911
Professor B. L. Shapiro
Department of Chemistry
Texas A&M University
College Station
Texas 77843.

STATIC AND DYNAMIC MOLECULES IN DISORDERED SOLIDS

Dear Professor Shapiro;

We are studying the solid state NMR properties of sandwich molecules of the type \([\{(C_6H_4X_2)Fe(C_5H_5)\}_2\text{EF}_6\}]^+\) \((X = \mathrm{H}, \mathrm{P}, \text{As}, \text{Sb})\). The 75 MHz \(^{13}\text{C}\) CP-MAS NMR spectrum of compound I \((X = \mathrm{H}, \text{E} = \text{As}; \text{Fig. 1})\) consists of a pair of equally intense benzene signals \((\text{Bz}; 87 \text{ and } 88 \text{ ppm})\) and a pair of equally intense cyclopentadienyl signals \((\text{Cp}; 76 \text{ and } 77 \text{ ppm})\). Although the width of the \(^{13}\text{C}\) resonance lines depends on the level of proton decoupling \(\Delta\nu_1\), the downfield signals of each pair are always relatively broad with \(\Delta\nu_1 \approx 50\text{Hz}\) \((\text{at } J \text{DP} = 70\text{kHz})\) compared to the upfield signals where \(\Delta\nu_1^2 \approx 15\text{Hz}\).

Previous studies\(^1\) have shown that the monoclinic phase \((265\text{K to } 305\text{K})\) of I contains two different molecular sites \((\text{Fig.1})\). The molecule in site 1 is disordered through rapid rotation \((\tau_c \approx 10^{-9} \text{ sec. at } 290\text{K})\) whilst the molecule in site 2 is relatively static. Intuitively one would assign the broad and narrow components of the spectrum to the static and dynamic molecules respectively, and this assignment was confirmed by introducing a 2 millisecond dipolar dephasing delay between the end of the CP contact and the start of the FID acquisition. The effect is to remove the broad components of the spectrum and hence to assign the broad signals to the more strongly coupled static molecules of the unit cell; the result is summarised in fig.1. The motional characteristics of the monoclinic and cubic phases of compound I will be published in the near future.

Yours sincerely

Ian Sayer
David B Davies

REFERENCE

Fig. 1. Carbon-13 CP-MAS NMR spectra of compound I in the monoclinic phase. A dipolar dephasing delay TAU is introduced into the CP sequence causing dephasing of the downfield broad components of each pair of signals. Hence, the downfield component of the Cp and Bz signals can be assigned to the static molecules of the unit cell (Bz-1/Cp-1) and the narrow upfield resonances can be assigned to the rotating molecules (Bz-2/Cp-2).
P-31 NMR of Adsorbed DFP on Ion Exchange Resins

Dear Barry,

Recently we have become interested in monitoring the decomposition of diisopropyl fluorophosphate, DFP, on a variety of chemically modified ion exchange resins. DFP is a cholinesterase inhibitor and is highly toxic to the central nervous system. The ion exchange resins are commercially available styrene/divinyl benzene macro-reticular beads with and without strong acid sites. The normal method of studying these systems involves extraction techniques which are time consuming and often ambiguous.

Initially we started to characterize these adsorbed molecules using solid state NMR. We currently have a Doty Scientific broadband, double-tuned CPMAS probe for our Varian XL-200. We were quite surprised to find that the DFP is highly mobile on the surface of the ion exchange resin and looks essentially "liquid-like" on the NMR timescale. In fact, we were able to obtain reasonable spectra using our Varian solution probe by placing the dry beads in 10 mm tubes and acquiring data using a one pulse experiment. The linewidths were approximately 200 Hz without spinning or proton decoupling. As shown in the figure, we start with essentially pure DFP on the surface of the ion exchange resin (B) and over time it decomposes by hydrolysis (C,D,E). It is interesting to note that when DFP is loaded onto activated carbon it appears to become immobilized on the surface as evidenced by a 20-fold increase in the P-31 NMR linewidth to 5 kHz (F).

Further characterization of these samples is in progress using F-19 and solid state NMR.

Sincerely,

Rebecca L. Smith
Catherine T. Hunt

P.S. Please credit this contribution to the Rohm and Haas subscription of Ed Greer.
A. DFP, Neat

B. DFP on Strong Acid Resin, Fresh

C. DFP on Strong Acid Resin, Aged

D. DFP on Unmodified Resin, Aged

E. H₃PO₄ Reference

F. DFP on Activated Carbon
Specific Deuteration in Proteins

As an aid in the assignment of resonances in the complex spectra obtained from proteins in the 20 kDalton range, we are studying molecules enriched in 13C, 15N and 2H. An example of the type of information to be gleaned in this manner is shown in Figure 1. This depicts a series of 400 MHz 1H NMR spectra of the imidazole ring C2 protons of the three histidine residues in pituitary bovine somatotropin (MW ~ 22 kDaltons). These protons slowly exchange with deuterium at rates dependent on the solvent accessibility of the residues. The fastest exchanging proton is obviously 3, assigned to HIS-170 (in a separate experiment) with a first-order rate constant k of 1.9 x 10^5 s^-1.

Yours sincerely,

Neil E. MacKenzie, Ph.D.
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 ±10.00 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:
- Measure Residual Fields
- Analyze Magnetic Circuits and Components
- Classify Magnets
- Measure Absolute & Differential Fields
- Plot Field Uniformity
- Measure Stay & Leakage Fields

FEATURES:
- Positive and/or negative peak reading
- 3½ Digit ±0.05% Bipolar Display
- DC & AC Fields, ±100 milligauss to ±19.99 KG with 1X probes
- Range Extendable to 150.0 KG with Select Probes
- Wide Selection of Precalibrated Probes: 1X, 10X & 100X
- True RMS Readings to 20 KHz
- Operates with either AC or Battery; Fully Portable
- Analog Outputs For External Monitoring
- No field decay in Peak Mode
- High Impact Plastic Case with Carrying Handle
- One Year Warranty

ACCESSORIES:
- Precalibrated Hall Probes
  A wide selection of 1X, 10X and 100X precalibrated Hall probes is available to meet most applications.
- Zero Gauss Chamber (Model ZG-1)
  A mu-metal shield used to shunt the earth's field around the Hall element in order to more accurately zero the gaussmeter when precise low field measurements are required.
- Reference Magnets
  Transverse and axial precision reference magnets are available when precise instrument calibration at a particular field is desirable.
MG-5DP Peak Reading Portable Laboratory Gaussmeter

**SPECIFICATIONS:**

- **Distance:** 10% ± 0.2% (600 mm ± 0.4 mm)
- **Temperature Range:** 0°C to 40°C
- **Humidity:** 10% to 90% non-condensing, input 10 V ± 0.5 V

**Range:**
- **100X** (0.5 mm, ±0.005)
- **10X** (1 mm, ±0.005)
- **1X** (2 mm, ±0.005)

**Accuracy:**
- ±0.1°/°C
- ±0.008 ΔT

**Resolutions:**
- 1°
- 0.008°
- 0.0008°

**Functional Range:**
- 50-60 Hz
- AC-100-125 V
- AC-200-240 V

**Output:**
- RMS
- DC

**Specifications:**
- **Power Requirements:**
  - AC-100-125 V 50-60 Hz
  - AC-200-240 V 50-60 Hz

**Portable Laboratory Gaussmeter:**
- **Range Setting:**
  - 100G (3 Hz to 20 KHz)
  - 10G (3 Hz to 20 KHz)

**Peak Reader Resolution:**
- 10 Gauss

**Portable Probe:**
- **Input:** 1.000 KG and 1.000 milligauss
- **Output:** 1.000 counts

**Shell Weight:**
- 4.75 lbs.
- 2.2 kg

**Shipping Weight:**
- 6.0 lbs.
- 2.7 kg

**HALL PROBES:**

<table>
<thead>
<tr>
<th>Model</th>
<th>% Linearity of Reading</th>
<th>Temperature Coefficient °/°C</th>
<th>Temperature Range °C</th>
<th>L</th>
<th>S</th>
<th>W</th>
<th>T</th>
<th>Active Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP-145</td>
<td>±1% to 20 KG</td>
<td>-0.1</td>
<td>-50 to +100</td>
<td>V-1X</td>
<td>0.5° (12.70 mm)</td>
<td>0.25° (6.35 mm)</td>
<td>0.25° (6.35 mm)</td>
<td>0.4° (10.25 mm)</td>
</tr>
<tr>
<td>HP-145F</td>
<td>±0.25% to 20 KG</td>
<td>-0.25</td>
<td>-50 to +100</td>
<td>V-1X</td>
<td>1.0° (25.40 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.8° (20.32 mm)</td>
</tr>
<tr>
<td>HP-145S</td>
<td>±0.25% to 20 KG</td>
<td>-0.25</td>
<td>-50 to +100</td>
<td>V-1X</td>
<td>1.0° (25.40 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.8° (20.32 mm)</td>
</tr>
<tr>
<td>HP-145R</td>
<td>±1% to 30 KG</td>
<td>±0.005</td>
<td>-50 to +100</td>
<td>V-1X</td>
<td>1.0° (25.40 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.8° (20.32 mm)</td>
</tr>
<tr>
<td>HP-145</td>
<td>±1% to 20 KG</td>
<td>-0.1</td>
<td>-50 to +100</td>
<td>V-1X</td>
<td>0.5° (12.70 mm)</td>
<td>0.25° (6.35 mm)</td>
<td>0.25° (6.35 mm)</td>
<td>0.4° (10.25 mm)</td>
</tr>
<tr>
<td>HP-145F</td>
<td>±0.25% to 20 KG</td>
<td>-0.25</td>
<td>-50 to +100</td>
<td>V-1X</td>
<td>1.0° (25.40 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.8° (20.32 mm)</td>
</tr>
<tr>
<td>HP-145S</td>
<td>±0.25% to 20 KG</td>
<td>-0.25</td>
<td>-50 to +100</td>
<td>V-1X</td>
<td>1.0° (25.40 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.5° (12.70 mm)</td>
<td>0.8° (20.32 mm)</td>
</tr>
</tbody>
</table>

**Note:** Thickness of standard probe applies to 1/2 from tip only.

**Resources, Inc.**

P.O. Box 642 • 262 Lakeshore Drive • Ashburnham, Massachusetts 01430
Professor Peter Stilbs
Physical Chemistry
Royal Institute of Technology
S-100 44 Stockholm, Sweden
Tel: +46 8 7878201

Uppsala 5 September 1986
(Received 15 September 1986)

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

Re: Less sample - MORE signal. Change of address.

Dear Barry:

Thank you for the reminder. Please note the address change. The chemistry department at KTH will be equipped with a new AM-400 and an HLS-90. I will continue to use the FX-100 and XL-300 in Uppsala. We have successfully made FT-PGSE measurements on the XL, using a custom-built Varian 5mm switchable probe with integral anti-Helmholtz gradient coils. The first results can be found in my review on FT-PGSE that will appear shortly in Progress in NMR Spectroscopy.

As a contribution I would like to comment on the prevalent opinion that more sample will always give a stronger NMR signal. Varian, in recent newsletters for example, recommend dilution of the sample (in the case of limited amounts of solute) to fill up the whole active sample volume on supercon systems. While this may usually be good practice, it is not a general rule. In a recent 13-C study on 25% w/w Lithium dodecylsulphate micelles in water (P. Stilbs, O. Söderman and H. Valderhaug, J. Magn. Resonance, in press) we made the initially puzzling observation that the signal was far weaker than expected. The experiments were made on the FX-100 in the tuneable 10 mm multinuclear probe, using about 4 cm sample height in thin-wall 10 mm tubes. It was also observed that probe tuning was not behaving normally, requiring quite different settings as compared to a "normal" sample. Reducing sample volume to about 1.5 cm and adding a vortex plug removed all those problems. Sensitivity went up by at least a factor of 5. Evidently the presence of a large volume of an electrically conductive aqueous solution in the probe did reduce its Q-value to a very significant extent. It is reasonable to assume that similar problems may arise in many studies on biological or biochemical samples, and one should consequently check for effects of sample volume. The effect on sensitivity could be very significant.

Yours Sincerely

Peter Stilbs

/Peter Stilbs/
29 August 1986 (Received 8 September 1986)

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

Amides Revisited: Cis/Trans Isomerism in Boc-Aminoacids

Roche Products Limited · PO Box 8 · Welwyn Garden City · Hertfordshire AL7 3AY
Telephone Welwyn Garden 328128
Telex 262098 ROCHEW

Dear Barry

One of the consequences of introducing high field nmr spectrometers for organic chemists is the regular appearance in their spectra of unexpected and usually unwanted peaks, mostly due to solvent or other impurities, but occasionally to dynamic effects. For years we have been asked to run simple "Boc" and "Z" amino acid derivatives (Boc.AA = BuO.CO.NHCH(R)COOH, Z.AA = PhCH2O.CO.NHCH(R)COOH) at 100 MHz rarely seeing anything out of the ordinary. At 300 and 400 MHz we were therefore startled to see two unequal resonances for the NH proton in Boc-Glu (R = CH2CH2COOH) in d6-dmso and in several mono- and diesters and amides. Under the fond illusion that monosubstituted amides existed entirely in the trans conformation in peptide derivatives, we started to hypothesise cyclic hydrogen bonded structures involving the glutamic acid & carbonyl. When checking other Boc- and Z-amino acids however, just about all of them showed two sets of peaks in CDCl3 or dmso-d6. A typical spectrum of Boc-Glycine in dmso-d6 is shown in the Figure. Coalescence of the NH resonances at raised temperatures gave a $\Delta H^\circ$ value of ca. 16 Kcals/mole. Therefore this phenomenon is certainly due to cis/trans isomerism about the amide bond in these urethanes.

We tentatively assign the major form in all cases as the cis form, based on the chemical shift of the amino acid $\alpha$-protons, which are probably at high field in the cis isomer, from previous work with thiocarbamate derivatives.

Changing the amino acid hardly affects the cis/trans ratio, but changing the solvent does: e.g. in Boc-Glycine trans:cis :: 1:7.7 in dmso, 1:1.5 in CDCl3. Searching the Cambridge crystal data file, the majority of Boc-amino acids and peptides crystallise with the urethane amide bond

Registered office 46 Broadwater Road Welwyn Garden City Hertfordshire Registered number 100614 London
trans, but there are a few exceptions e.g. Boc-Phe, which are cis oriented. A literature search, which is what we should have done first, unearthed a paper describing this isomerism in Boc-Alanine and Boc-Ala peptides. The reason why these dynamic effects were not obvious at 100 MHz is probably that the probes in electromagnets operate at least 15°C higher than in superconducting magnets.

We hope this contribution cancels out this years’ pink ultimatum.

Yours sincerely

WA Thomas

W A Whitcombe

H S Simmonite


Figure

The 300 MHz $^1$H nmr spectrum of Boc-Glycine in dmso-d$_6$: "c" and "t" mark the tentatively assigned cis and trans isomers respectively.
Tailored Excitation Using Fourier Transform Pairs: A Frequency-Space Notch Filter and One-Pulse Multislice Imaging Using Shaped Radiofrequency Pulses

Dear Barry:

FT NMR has employed only a few of the characteristics of FT pairs. The use of shaped rf pulses for selective excitation has been limited to: the gaussian pulse, whose Fourier transform in the frequency domain is another gaussian; the sinc function, whose Fourier transform is a square wave; and the sech function, whose Fourier transform is another sech function. The two former shapes have been used primarily for slice selection in NMR imaging, although their use for selective $\pi/2$ pulses has been suggested. The latter shape has been shown to be a selective $\pi$ pulse. We have begun to explore a well-known relationship, known as the Modulation Theorem, that exists between the two members of an FT pair, $f(t)$ and $F(t)$:

$$\int [F(t) \cos(\omega t)] e^{-2\pi ivt} dt = \left(\frac{1}{2}\right)f(v-\omega/2\pi) + \left(\frac{1}{2}\right)f(v+\omega/2\pi).$$

We have chosen $F(t)$ to be $\cos(t)$, so that $f(\omega)$, the other member of the FT pair, is a square wave function. The resulting frequency-space function on the right side of the equation is two square waves separated by a frequency gap. In principle, it should be possible to position this gap to act as a square wave "frequency-space notch filter" in the excitation profile.

We have demonstrated that this theorem can be implemented on a 4.7 Tesla, 33 cm bore NMR system equipped with a Nalorac Quest 4300 Spectrometer, which incorporates a VAX 11/730 computer system and an array processor. Figure 1 illustrates the Modulation Theorem in the context of NMR imaging. Figure 1A shows a slice selected along the z-axis using a sinc-shaped pulse. Figures 1A through 1F show the effect of modulation by $\sin(\omega t)$ when $\omega$ is increased from $\pi/3$ to $2\pi$. Note that the signal intensities in Figures 1B through 1F are each approximately 40% of the intensity of the slice in Figure 1A, which is close to the value of 50% predicted by theory. Figure 2 shows that when the frequency offset of the $\sin(\omega t)$-modulated sinc pulse is changed by $\pm 4000$ Hz, both slices move in the same direction demonstrating that they are not quadrature images. This shows that it is possible to do multislice imaging in one pulse. Spectra with an excitation profile similar to that of the one-dimensional projections have also been obtained using a Hahn spin-echo technique. We have also used our "frequency space notch filter" for suppression of the water proton resonance and have shown that it is comparable to the 1931 pulse sequence for suppression of the water proton resonance, but it gives level excitation to the rest of the spectrum.
unlike the binomial series pulse sequences. The use of the FT pair that consists of the convolution of the shah function with sinc(t) has been used as a further extension of the one-pulse multislice imaging technique.

References:

Dear Barry,

our interest in COSY spectroscopy of quadrupolar nuclei continues. After completion of the \textsuperscript{6}Li,\textsuperscript{6}Li experiment (THL 27, 2251 [1986]) we used the same cluster prepared with \textsuperscript{7}Li. As expected, the experiments proved to be more difficult because of faster Li relaxation ($Q = -4.5 \times 10^{-2}$, while for \textsuperscript{6}Li $Q = -8 \times 10^{-4}$). Therefore, a shorter $t_1$ had to be used (Fig. 1).

\textsuperscript{2}H,\textsuperscript{2}H COSY is again less problematic and is the method of choice for the analysis of \textsuperscript{2}H spectra of perdeuterated compounds (Fig. 2). Needless to say that in both cases scalar coupling is not resolved in the 1D spectrum.

With kind regards,

Yours sincerely

\[\text{D. Moskau} \quad \text{H. Günther}\]
Now, with the new GN Series high resolution NMR spectrometers, GE brings you the greatest versatility in multinuclear liquids and solids research... and backs it up with GE's unequalled quality, reliability, and continuous support.

GE is committed to providing you with the highest performing NMR systems today and in the future. With the GN Series, available at various field strengths and bore sizes, you can perform simple one-pulse analysis, or complex state of the art experiments like triple quantum correlation and various selective excitation experiments through the system's automated hardware features which include:

- A comprehensive observe and decoupling phase shifter for < 90° phase shifts.
- Complete computer gain control of lock observe and proton/x-nucleus decoupler channels.
- A new, super-sensitive deuterium lock.

The Spectrometer Control Processor is easily controlled by GEM, the latest generation of NMR software. GEM-users can direct the GN system to perform a complete series of predetermined experiments for total sample analysis — or take control of individual components and develop their own custom NMR analysis.

With a variety of accessories including array processor, x-nucleus decoupler, liquids probes and six different solids probes with unique capabilities, and a choice of data storage devices, your GN spectrometer can give you an ultimate advantage!

Step into the future with GE. We're ready to assist you with expanding support through our toll-free 800 customer service number. To receive a comprehensive new GN Series brochure or arrange for a demonstration, call (415) 490-8310, or write General Electric Company, NMR Instruments, 255 Fourier Ave., Fremont, CA 94539.
More and more labs are looking into the automated QE-300 NMR system for superior results at a price of just $160,000.

For powerful analytical insight... you need fast, accurate NMR results that you can interpret at a glance. With the General Electric QE-300 system you will not have to accept a compromise system that almost does the job. For the lab, a 300 MHz system can now be your minimum acceptable field strength with the best price/performance ratio.

Better dispersion, faster analysis, and easier interpretation result directly from a 300 MHz superconducting magnet under the control of our custom analysis software... software that adapts with a few keystrokes to fit your specific analytical needs.

With the QE-300 you obtain exceptional high-resolution proton and carbon spectra from the same sample in just minutes. Plus, you have the option to observe a wide variety of other nuclei ranging from phosphorus through nitrogen. Our hardware/software options allow you to customize the system to fit your lab.

To find out what more there is to see in the QE-300 system, call (415) 490-8310 or write General Electric, NMR Instruments, 255 Fourier Avenue, Fremont, CA 94539.

See what you’ve been missing.
re: How to present 2D COSY spectra?

Dear Barry,

recently we had an argument with a referee who requested that the scales in a COSY spectrum should be given in ppm units. Since his view was supported by the editor we would like to solicit the opinion of others.

The argument held by the referee was essentially based on common experience and use of ppm units. We object the rule for two reasons: (i) if the axis presents broad-band decoupled spectrum (as f_2 axis in heteronuclear COSY) with chemical shift information only, ppm units are preferable but, if the axis represents coupled spectrum, Hz units are at place, (ii) since the author is not always in a position to re-plot the spectrum any presentation rules should be made publicly known before being enforced.

Undoubtedly, the rate under which 2D spectra are published wears off as the method becomes more and more routine. This desirable trend will be accelerated by journal formal presentation rules. However, there will be always cases which will require presentation of a 2D spectrum. In such cases should be the axes in ppm or in Hz units? (Irrespective of IUPAC rules which call for Hz in diagrams).

Sincerely yours

[Signature]

Jan Schraml
Dear Barry:

Natural abundance $^{13}$C-nmr of small double-stranded DNA molecules can provide detailed structural information, not readily available from other solution techniques. $^{13}$C-spectra of the nucleobases in DNA are sensitive to hydrogen bonding, especially for several of the carbons immediately bonded to heteroatoms acting as H-bond acceptors or donors. Thymine C4, guanine C2 and C6 are strongly deshielded (about 1 ppm) upon Watson-Crick base-pair formation, and cytosine C5, adenine C6, and the thymine methyl carbon are deshielded to a lesser extent. Shielding increases would be expected for these carbons due to ring current and steric effects in the absence of hydrogen bonding, so deshielding at these sites can be used to distinguish bases involved in Watson-Crick base pairs from unpaired bases. These conclusions are based on strand dissociation of three different DNA oligonucleotide duplexes. Figure 1 shows the bases and the hydrogen bonding sites and Fig. 2 gives $\delta$ vs. T profiles for the guanine carbons in these duplexes; the negatively sloping profiles are for resonances which are most sensitive to H-bonding.

Netropsin binds to the central AT region of the octanucleotide duplex, $[d(G-G-T-A-T-A-C-C)]_2$, and is thought to displace water specifically bound in the minor groove. In the netropsin-octanucleotide complex, +1.4 and -0.6 ppm changes in $^{13}$C chemical shift are noted for one thymine C2 and one adenine C4 resonance, which are atomic neighbors of putative hydrogen bonding sites for water or netropsin. These and other observations provide direct support for the existence in solution of a “spine of hydration” in AT segments of DNA that is displaced upon netropsin binding. $^{13}$C chemical shifts show promise as a method for the determination of hydrogen-bonded sites in complexes of nucleic acids with themselves and other ligands.

Sincerely,

Philip N. Borer
Research Associate Professor
and Associate Director

George C. Levy
Professor
and Director
Figure 1. Base pairing schemes in (a) Cyd-Guo and (b) Thd-Ado, netropsin and water hydrogen bond to ThdO2 and AdoN3.

Figure 2. Chemical shift vs. temperature profiles for the guanine carbons of three oligonucleotide duplexes; shielding increases toward the bottom of each panel.
Dear Barry,

In response to the pink note, here are some recent results and general remarks.

**Platinum hydride NMR**

From time to time molecules are found where dynamic effects allow determination of relative signs of coupling constants. Such is the case for the hydride resonance of several diphosphinosilylplatinum hydrides, eq. 1. We have been analyzing the hydride lineshapes in collaboration with Mark Hampton-Smith and H. C. Clark at Guelph.

![Diagram](https://example.com/diagram.png)

Hydride is coupled to both phosphorus as well as $^{195}$Pt. So the hydride spectrum (toluene-$d_8$ solution) consists of one quartet for $\ce{Cy3P}_2$ with non-magnetic platinum flanked by the two quartets coming from $1-195$Pt. The effect of intramolecular exchange of silicon for hydride is to average the cis and trans hydride phosphorus couplings. At slow exchange rates this has the effect of broadening the outside lines of each quartet whereas the inside lines always remain sharp.

One such unaffected transition is $\ce{aaa <--- aab}$ the order of spins being $\ce{P1, P2}$ and $\ce{H}$. From the density matrix equations one can see that the separation between the inside lines is $J_{\text{cis}} + J_{\text{trans}}$ (both P, H couplings) whereas the gap between the two outside broadened lines is $J_{\text{trans}} - J_{\text{cis}}$, hence these two $3^1\text{P}, 3^1\text{H}$ coupling constants must have opposite signs.

Above 25° all hydride resonances broaden as a result of fast reversible dissociation. We have incorporated both exchange processes into the density

$$(R_2\text{P})_2\text{PtHSiO}_3 \Leftrightarrow (R_2\text{P})_2\text{Pt} + \text{HSiO}_3$$
matrix equations using our Pi method and hence fished out activation parameters for both processes, to be duly published.

NMRNET

Now a word about communications: To facilitate interaction among Newsletter people I propose that all your contributors who have BITNET addresses include them on future contribution to the Newsletter, as I have. BITNET is an electronic mail system which interconnects many universities and research institutes throughout the free world. TAMU-MR could then put together a directory of BITNET addresses. The obvious advantages of BITNET is speed and it's free. One can send a manuscript (without the figures) half way round the world in a minute.

Further information on BITNET is available at your local computer center.

NSF Budget in Jeopardy

Readers may be aware that the NSF budget which so far sailed through Congress without a scratch has just suffered a serious cut of $145.7M at the hands of the House Appropriations Subcommittee on HUD Independent Agencies. This puts the NSF budget $145.7M below the Administration's request. One can only hope that the Senate Appropriations Subcommittee on HUD-Independent Agencies will put some or all of this back, then, that the Conference Committee will at least agree to the original request. Hopes are more likely to be realized if lots of scientists write to Jack Garn, Chairman of the above Senate Subcommittee and explain why the NSF needs the money, what will happen to their programs if the NSF budget is cut, what sort of future the country will have with defunct science.

Directly or otherwise NSF funding is critical to all Newsletter writers. A one $trillion budget is currently being carved up. Whether or not there will be something left in it for us depends on how active scientists are politically, what we say to Congressmen. If we don't speak up now we shall only have ourselves to blame when the funding we hoped would be for science goes somewhere else.

Senator Garn's address is US Senate, Washington, D.C. 20510, Tel. #202-224-5444, subcommittee at 202-224-3471.

Best wishes.

Gideon F. Fraenkel
Professor of Chemistry


September 4, 1986 (Received 8 September 1986)

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

Dear Professor Shapiro:

"Free Running" of Nicolet Explorer Interface

We've had an intermittent problem with the interface between our Nicolet 2090 Explorer scope and 1280 computer for the past 18 months. When first installed, the scope would occasionally start to transfer data repeatedly (cycle time less than 50 ms), regardless of the delay time setting, without accumulating signals. Recently we've been able to make the system fail consistently, enabling us to locate the source of the problem.

The problem was caused by a glitch picked up on the 'Record' line. When the 1280 told the 2090 to arm itself for the next trigger, the 'ARM' pulse caused a glitch on the 'RECORD' line high enough to propagate through IC20 and IC19, by which time it was a clean 100 ns pulse resetting IC4. This caused a 'POST RECORD' signal to appear, telling the Computer and Explorer to transfer data again, and again.... The problem was solved by connecting a .03µF capacitor from IC20 pin 1 to IC20 pin 7 (GND) to suppress any glitch.

Sincerely yours,

Rolf Tschudin  
Laboratory of Chemical Physics  
NIADDK  

Yukio Hiyama  
Bone Research Branch  
NIH  

Please credit this to  
Dennis Torchia's account
New Advanced Function Series FTNMR from IBM Instruments

Automation makes it easy to use... Standard "extras" make it easier on the budget

New automation features and a new bit-slice multiprocessor combined with proven electronics make this FTNMR extraordinarily easy to use. And many features usually regarded as extras have been made standard equipment... so high performance capability doesn’t have to mean high price.

Single-knob control
A single knob controls magnet and lock functions and a digital readout panel displays settings. Entering commands on the alphanumeric keyboard is simple and quick. Key functions such as shim, lock and receiver gain are automated. The Advanced Function FTNMR will perform complex experiments unattended for long periods.

Multiprocessor data system
The fast, micro-programmed, bit-slice CPU is supported by specialized processors that handle Fourier Transform, instrument control, data acquisition and output devices. This gives the instrument exceptional power and versatility, including multitasking capability.

Extras are standard
Features frequently costing extra, such as a broadband transmitter, digital plotter, diskette drive, hard disk drive and color display, are standard. And because of the economies that standardization brings, you get powerful performance at a modest price.

Let us tell you more
To learn more about the new Advanced Function Series FTNMR Spectrometers from IBM Instruments, send the attached reply card. Or call 800-243-7054. In Connecticut, 800-945-1073. Or write IBM Instruments, Inc., Orchard Park, PO Box 332, Danbury, CT 06810. 

Integrated solutions for Science and Industry
How to improve magnetic field measurement and control

Hall effect field regulator
ER 031M

Hall effect magnetometer
ER 031Z

Hall effect field regulator
BH 15

NMR magnetometer
EE 035M

You can get a new standard of performance by upgrading your field regulation and measurement equipment. The superiority of advanced design field regulators and magnetometers from IBM Instruments can be seen clearly in the performance curves shown at the right. Regulator accuracy is 200mG from -50G to +23kG.

Microprocessors in each unit provide ease of operation and complete flexibility of application. Most units also can be programmed through RS 232 or IEEE 488 (IEC 625) interfaces. Other outstanding features include:

ER 031M Hall effect regulator—Low noise, 0.1mG rms in 1Hz bandwidth. Excellent long-term stability, 2 ppm/degree.


BH 15 Hall effect magnetometer—combines features of ER 031M and ER 031Z plus sweep capability, digital and analog outputs and homogeneity plots (x-y) with resolution of 1mG.

ER 035M—Extremely accurate NMR magnetometer, 5mG from 450G to 20kG. Tracking rate, 1kG/3 sec. Optional EPR in-cavity probe.

Let us tell you more

To get more information on these IBM Instruments products, just send the attached reply card or call 800-243-7854. In Connecticut, 800-952-1075. Or write IBM Instruments, Inc., Orchard Park, PO Box 332, Danbury, CT 06810. Outside the U.S.A., get in touch with your nearest Bruker-Spectrospin sales representative.

Integrated solutions for Science and Industry
Since we 'met' in the lonely hearts section of TAMU NMR Newsletter in 1982, Professor Ruben Contreras and I have been collaborating on an investigation of mechanisms by which spin-spin couplings are transmitted. Theoretical work is done in Buenos Aires, molecular design is a shared activity, synthesis and spectroscopy done here in Melbourne. Initially our progress was slow but we are now making good progress and enjoying by mail some Southern Hemisphere camaraderie.

The PPP method used by the Contreras group predicts that coupling between carbon and a heteroatom X is enhanced when a C-H bond occupies the space between C and X. Our first success (1) was in finding a predicted $3J_{CF}$ of 3 Hz in o-fluoroacetophenone and also a $4J_{CF}$ to the methyl group of the predicted 7 Hz, which had been overlooked by earlier workers.

The C-H bond in this case is not in the main plane of the molecule and the calculations suggested a large increase in J if coplanarity could be achieved. It is, too - in the 8-fluoronaphthaldehyde shown below, for which $4J_{CF}$ is 26.2 Hz. This is not the calculated value (77 Hz) but then our molecule probably does not have the simple geometry shown and the calculated values are extremely sensitive to the hydrogen-fluorine separation. We await the crystal structure.

We hope that we can synthesize naphthalenes of this kind with other heteroatoms in place of fluorine, so further tests of theory are in store.

I'm sorry to conclude this letter with the news that Stan Johns, who worked at a nearby CSIRO laboratory and shared David Kelly's TAMU subscription, died last month after a long struggle with multiple myeloma. He'll be missed.

Ian D. Rae

Solid-State NMR Characterization of Poly(aryletherketone)'s

Dear Professor Shapiro:

We are interested in using solid-state NMR to characterize the effects of processing on poly(aryletherketone)'s. These materials enjoy good mechanical properties in conjunction with high thermal and chemical stability, which make them attractive for many specialty plastics applications. To date, we have concentrated on PEK, represented by

\[ \begin{align*} &\text{-(O-O-C)-} \\ &\text{O} \end{align*} \]

The degree of crystallinity, the orientation of crystalline and noncrystalline chains, and the presence of any low molecular weight species remaining after the workup constitute our current focus.

The accompanying table presents $^1H$ spin-lattice relaxation times in both laboratory and rotating frames for PEK subjected to different processing histories. All samples were sealed under nitrogen in glass tubes. The $^1H$ $T_1$'s (at 180 MHz) are sufficiently long to permit spin diffusion measurements as a means of characterizing the morphology in the tens of nanometers regime. They also indicate a considerable difference among the samples in relaxation efficiency.

The $^1H$ $T_1$ decays have been decomposed into sums of exponentials by nonlinear least squares fits. The short component (< 5 msec) is probably due to mobile noncrystalline material; the intermediate component (10-20 msec) to crystalline regions; and the long component (≥ 100 msec) to residual bound water. This last assignment is made by observing the proton lineshape following various periods of spin locking. The long $T_{1p}$ component has a relatively narrow lineshape, which suggests a mobile, proton-bearing species. Attempts to assign the chemical shift of this component by using magic-angle spinning to average the remaining dipolar coupling and chemical shift anisotropy have not yet been successful because of a residual proton signal in the spinning probe. If the assignment of the slowly relaxing component to residual water is correct, extended drying of the material well above its glass transition temperature (165°C) does not completely remove adsorbed water from the polymer. Efforts to improve the accuracy of the quantitative results by using multiple-pulse relaxation are currently underway.
Raychem

This work was performed in Jeff Reimer's laboratory at the Chemical Engineering Department of U.C. Berkeley.

John Ravens
Raychem Corporation

Jeff Reimer
U.C. Berkeley

Karl Amundson
U.C. Berkeley

Spin-Lattice Relaxation Results for PEEK

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>$T_1$ (sec)</th>
<th>SPIN-LOCK $T_{1p}$ AT 5.1 GAUSS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amorphous sheet before annealing</td>
<td>1.3</td>
<td>2.8 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>130 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>130 msec</td>
</tr>
<tr>
<td>Amorphous sheet after annealing for 6 hours at 250°C</td>
<td>2.0</td>
<td>3.7 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>18 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>18 msec</td>
</tr>
<tr>
<td>Powder</td>
<td>4.7</td>
<td>2.8 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>11 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 msec</td>
</tr>
<tr>
<td>Spun fiber (5.2 x melt draw) oriented $\mathbb{E}_0$</td>
<td>13</td>
<td>2.6 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>13 msec</td>
</tr>
<tr>
<td></td>
<td></td>
<td>240 msec</td>
</tr>
</tbody>
</table>
Titre: MODIFICATION DE LA SEQUENCE D’ECHOS STIMULES ET MOUVEMENTS LIQUIDIENS

Cher Professeur Shapiro,

L’emploi des échos stimulés en imagerie RMN prend de plus en plus d’importance car cette technique s’applique à de nombreuses situations : imagerie avec pondération par le temps de relaxation spin-milieu, imagerie multicoupe, imagerie spectroscopique, imagerie rapide et imagerie de débits (1).

Les échos stimulés s’utilisent également pour la localisation spatiale des observations spectroscopiques (2).

En présence de mouvements liquidiens on peut tirer profit des échos stimulés, à condition d’introduire une modification de la séquence de base. En effet si une impulsion de 180° est placée au milieu de l’intervalle de temps $t_2$ séparant la seconde et la troisième impulsion de 90° (Figure 1), la refocalisation des aimantations transversales observée habituellement à $t_3 = t_2$ et $t_1 = t_2 ± t_1$ (t_2 > t_1) a lieu à la date de l’écho stimulé ($t_3 = t_1$). Ce dernier est dû aux aimantations longitudinales qui sont "gelées" le long de l’axe Oz pendant l’intervalle $t_2$. Le choix de la phase de l’impulsion de 180° permet soit l’addition, soit la soustraction des échos transverses et longitudinaux pour $t_3 = t_1$. Si, pendant $t_2$, un gradient bipolaire est appliqué dans la direction d’un écoulement, l’intensité de l’écho global est modifiée en raison du déphasage $\phi = \gamma G v t (t + t')$ acquis par l’aimantation transversale. Dans tous les cas cette intensité tend, lorsque $\phi$ est élevé, vers une valeur limite $I_{lim}$ indépendante de la vitesse $v$. Lorsque les échos transversal et longitudinal sont opposés, les signaux en provenance d’éléments fixes de l’échantillon ont donc une intensité plus faible que $I_{lim}$. On modifie ainsi le contraste d’une image en faveur des éléments en mouvement. Une illustration est donnée sur la Figure 2.

Un des intérêts de cette méthode est qu’elle permet d’incorporer avec souplesse les différentes étapes de préparation du signal (codage de phase, conversion vitesse/phase puis phase/amplitude) dans une séquence relativement simple. Une autre utilisation de cette séquence d’échos stimulés modifiée, peut s’envisager pour l’étude de la diffusion.

Recevez, cher Professeur Shapiro, nos meilleures salutations.

Daniele GRAVERON
André BRIGUET
Hana LAHRECH

(Contribution à mettre au crédit du Laboratoire de Résonance Magnétique Nucléaire - Professeur Jean DELMAU)
Figure 1 : Séquence d'écho stimulé modifiée par une impulsion de 180° placée au milieu de l'intervalle de temps $t_2$. $G_s$ = gradient de sélection $G_v$ = gradient de codage de la vitesse $G_{ph}$ = gradient de codage de phase $G_{lec}$ = gradient de lecture

(Cas d'une image pondérée par les vitesses perpendiculaires au plan de coupe)

Figure 2 : Contraste observé entre deux tubes (1,2 cm diamètre) contenant de l'eau en mouvement (3 cm/s, à gauche) et immobile (à droite). Le profil correspondant est également donné au dessus de l'image.

Références :
(2) G. Mc KINNON, 5th Meeting SMRM Montréal (Aug. 86)
(3) J. FRAHM et al, 5th Meeting SMRM Montréal (Aug. 86)
August 27, 1986

Dear Professor Shapiro:

HETCOR of NORTRICYCLYL BROMIDE.

We have recently been interested in using the $^1H$ and $^{13}C$ spectra of nortricyclyl bromide (A) to assist in the interpretation of the complex spectra of isomer mixtures from the reaction of the new halonium transfer reagents with bicycloalkenes. A literature search has revealed little information on the spectral assignments for A.

It is fairly easy to assign $H_3$, $H_4$, $H_7$-sym, $C_3$ and $C_4$ in the $^1$D-NMR spectra, while DEPT indicates which carbon resonances are methylenes. In addition, the 18.2 ppm resonance can be assigned to $C_2$ since it is 8.3 ppm downfield from the parent hydrocarbon shift. Further assignments from the $^1$D-NMR spectra are difficult, especially for the $^1H$ 1.0-1.5 ppm region.

Heteronuclear correlation (HETCOR) 2D-NMR has proven valuable for completing the assignments, especially for unravelling the crowded proton region. Figure 1 shows a HETCOR run on a Varian XL-200. The 31.0 ppm resonance correlates with two well separated $^1H$ resonances where the $H_7$-sym resonance is shifted 1.3 ppm downfield from $H_7$-anti due to the effect of the 3-exo-bromine. The $C_5$ resonance correlates with the $H_5$ methylene protons which are expected to give a narrow proton pattern with a shift similar to $H_7$-anti. $H_4$ is expected to be shifted downfield from $H_4$ since it is closer to the bromine. On the other hand, the bromine causes greater steric compression on $C_1$ than on $C_6$. It is expected that the $C_1$ resonance will come upfield of the $C_6$ resonance. Figure 1 nicely demonstrates this correlation. We are presently attempting to confirm this last point by means of a carbon-carbon connectivity experiment.

Please credit this to the account of Dr. Amos J. Leffler.

Sincerely,

Walter J. Boyko, Ph.D.
NMR Laboratory Directory

(continued on page 33)
How to use solid-state NMR to get results in all these applications areas:

- Chemical Structure and Composition
- Solid-State Reactions
- Catalyst Studies
- Polymer Morphology
- Molecular Dynamics

Varian wideline solids systems are available now.

This NMR powder spectrum of NaNO₃ showing detailed crystallite distribution within the sample.
Solid-State Results from Varian NMR

**Si MAS – Zeolites**

Silicon is the major nucleus of interest in silicates. MAS reveals information about the types of Si present in a sample. The upper spectrum is of Na-X, which has a lower Si/Al ratio than does Na-Y (lower spectrum).

The resonances correspond to Si[4Al] (lowfield) through to Si[0Al] (highfield). Neither of these Si spectra have been run with cross-polarization, as there are few, if any, framework protons present. However, CP/MAS experiments are used whenever investigating adsorption of small molecules onto zeolites.

Varian’s CP/MAS Accessory allows the user to obtain a wealth of chemical information from many different nuclei at different sample temperatures. Applications range from characterization of polymer dynamics and composition, using "C CP/MAS, through catalyst investigations (using "Si, "Al, "Pt or 23Na MAS) to organic chemistry problems, e.g., "Co-MAS.

**Poly (ethylene-d4)**

The sample shown here was mainly crystalline (which gives the doublet spectrum), with some amorphous material (showing as a central hump). The crystalline T, relaxation is long, demanding a 10-second wait between acquisitions. Techniques such as this have been used to investigate the chain orientations of stressed or drawn samples. Molecular order may be correlated to sample morphology using spectra such as this.

Varian’s Wideline Solids Accessory opens new possibilities for the study of paramagnetic and quadrupolar nuclei. Information can be obtained about molecular dynamics, ordering the morphology of deuterated polymers ("H NMR), microbiological systems ("H, "C, "Cd, "P), and solid-state chemical reactions ("Si, "Al, "Na,T2, etc.).

The spectra above represent two examples of the performance you can achieve on a Varian NMR Solids System. Our solids capabilities cover a broad range of applications, including the following:

- Chemical structure and composition
- Solid-state reactions
- Catalyst studies
- Polymer morphology
- Molecular dynamics

Get all the facts today

Call or write Varian today to learn how you can achieve these results, or to receive a copy of our Solids NMR Spectra Catalog.

In the U.S., call 800-231-5772. In Canada, call 415-457-4130. In Europe, call 2ug, Switzerland, at (042) 44-88-44; Darmstadt, Germany, at (06151) 7030. In Japan, call 3-204-1211.

Or write: Varian Instrument Group, 220 Humboldt Court, Sunnyvale, CA 94089.

- In Canada: 332 Guelph Street, Georgetown, Ontario L7G 4B5.
- In Europe: Steinhauserstrasse, CH-6300 Zug, Switzerland.
(continued from page 30)

Figure 1

![Chemical Structure and 2D NMR Spectrum](image-url)
Dear Professor Shapiro,

Title: "Stereochemical Dynamics of a Dicyclohexano-14-C-4"

Recently, Bob Kirby, as part of his B.Sc. Honours Project and now M.Sc. research, has purified one configurational isomer from the catalytic hydrogenation of dibenzo-14-C-4. As yet we do not know if it is cis-syn-cis I, or cis-anti-cis II, but it does have a most interesting 1H NMR behaviour as a function of temperature.

From a simple 5 line spectrum at 298K in CD2Cl2 solvent, the spectrum at 183 K becomes that shown on the subsequent page. There are two temperature dependent phenomena occurring it appears. The first is degenerate cyclohexane ring inversion and the second is non-degenerate ring inversion of the 14-membered macrocycle.

For 14-C-4 itself(1), no coalescence phenomena have been observed, albeit at 15 MHz, in the 13C spectra down to 143K. These authors(1) did suggest, however, based on chemical shift changes in CH2F2 solution, that at least two conformations of 14-C-4 were present.

We are in the process of isolating the second configurational isomer from the hydrogenation product and are using high field spectra to study it as well as 14-C-4. Hopefully our crystals of I and II will be of sufficiently good quality for X-ray analysis.

Department of Chemistry Steacie Building (613) 564-2760
Sincerely,

G.W. Buchanan,
Professor of Chemistry

GWB/cm


P.S. I'd like to thank Bob Fleming at the Univ. of Nottingham for the spectrum shown here, obtained during my recent mini-sabbatical in the U.K.

\[ ^{13}C \text{ NMR of } I \text{ and } II \]

at 103 K

---

The Chemistry Department at the University of Wyoming is searching for either a room or variable temperature \( ^1H \) probe for a Varian EM-360. If you have a probe (or instrument) available for purchase, please contact either Dr. Ed Clennan, Chemistry Department, University of Wyoming, P.O. Box 3838, University Station, Laramie, Wyoming 82071 (Tel. 307-766-6667) or Dr. Daniel A. Netzel, Western Research Institute, P.O. Box 3395, University Station, Laramie, Wyoming 82071 (Tel. 307-721-2370).
Dear Professor Shapiro,

In recent years there has been considerable interest in the NMR of zeolites. Several nuclei in zeolites can be studied, $^{29}$Si, $^{27}$Al, $^1$H, $^{23}$Na to mention a few. Especially magic angle spinning has proven to be very valuable to increase the spectral resolution, in particular for $^{29}$Si. Although MAS can be applied with success as well to quadrupolar nuclei like $^{27}$Al and $^{23}$Na, the gain in resolution for these nuclei is not so great as for spin-1/2 nuclei. The quadrupole interaction for half-integer spins very often gives characteristic spectra even in the absence of magic angle spinning. Also two-dimensional NMR (1,2) can help to increase the resolution.

Without the experimental restrictions imposed by magic angle spinning, it is not difficult to construct a probe for high temperatures. Fig. 1 shows $^{23}$Na NMR spectra of zeolite A obtained with such a high-temperature probe as a function of temperature. To explain these spectra, Fig. 2 shows roomtemperature $^{23}$Na spectra of zeolite A, dried at 300°C for 48 hours at 10⁻² Torr at a 300 (CXP 300) and 500 (AM 500) MHz spectrometer. Clearly, at least two different sites are observed, one with a large quadrupole interaction giving a field dependent splitting, the other with a rather small splitting. By increasing the temperature to 680°C, so far the highest temperature obtained, first the lines corresponding to the broad second-order quadrupole pattern disappear and then the quadrupole line at the center narrows. No doubt the increased motion of the sodium atom at higher temperatures is responsible for this narrowing. A detailed discussion will be presented elsewhere, but the result shows that NMR of zeolites at temperatures where they are used as catalysts, is worthwhile. In the first prototype of the probe with which these spectra are taken, the sample was placed in an oven heated by a DC current. This causes small shifts and therefore in the new design the sample is heated by nitrogen gas, heated outside the probe. The signal denoted by a star is due to a folded-over Cu signal.

Ron Janssen

Best regards,

Ery Tijink

Wibren Veeman

Fig. 1: Temperature dependent $^{23}\text{Na}$-NMR spectra of zeolite A.

Fig. 2: $^{23}\text{Na}$ spectra of zeolite NaA at two different magnetic fields.
SELECTIVE EXCITATION: SOMETHING FOR NOTHING

The technique of selective excitation is central to NMR imaging (1). With the advent of NMR spectrometers equipped with linear RF amplifiers there will also be important applications in high resolution spectroscopy.

We have recently developed a new numerical approach to selective pulse design. Relatively large changes can be made at each step and the method converges very rapidly, typically in a few tens of iterations. We have published a short note on the generation of a self-focusing $\pi/2$ pulse (2). At the end of the pulse, the selected spins are all in phase and do not therefore require a hard $\pi$ pulse or field gradient reversal in order to refocus them.

The algorithm should not be thought of simply as a method for refining selective pulses. It can be used to generate 'novel' pulses without requiring a good starting approximation. Readers may find the following example a convincing demonstration of this 'something-for-nothing property'. Figure 1 illustrates a slice-selective refocusing pulse derived in 30 iterations from a starting approximation which is identically zero. Figure 2 shows the $M_z$ response of the selective pulse. Note that it is a genuine $\pi$ pulse, preserving the $M_x$ magnetization in the slice and reversing the signs of $M_y$ and $M_z$.

A full account of the algorithm and examples of its wider application will be published in due course. This is a contribution from the Cambridge Biochemical NMR Group.

Sincerely yours,

J. Thomas Ngo

Peter G. Morris
Fig. 1

Fig. 2

REFERENCES


Dear Professor Shapiro,

Performance of 

B decoupling on a Bruker 

AM-400 'H-{'B} probe head.

We have recently been investigating the possibility of 'H-'B-{'B} 

cosy experiments as a viable alternative to 'B-'B-{'H} 

cosy experiments 

in the elucidation of structures in metallaborane and carbaborane 

chemistry and in the assignments of 'H and 'B resonances.\(^1\) 

The 'H-'B-{'B} 

cosy technique has several advantages over the 'B-'B-{'H} 

cosy technique, but at 

present its main drawback is the difficulty to achieve efficient 'B 

broad 

band 

noise 

decoupling over the wide chemical shift range required (often 

more than 100ppm, i.e. 12.8 KHz on an AM-400). Our original 

experiments 

were performed on 5mm samples held in a 10mm 'B-{'H} 

probe head, observing 

'H through the decoupler coil. For other 

reasons, we had on order a 

probe head for 'H observation with tuneable heteronuclear 
decoupling. We 

were, perhaps naively, hoping that this probe head 

would provide us not 

only with significantly improved signal-to-noise ratios but also with a 

more efficient 'B decoupling performance. In the latter respect we have 

been rather disappointed. For an output of ca. 5W of broad band noise 
decoupling (the maximum recommended) we can achieve a range of ca. 100ppm 

for a 'H line-width of < 10 Hz in the 'B-{'H} probe head. However, in 

the 'H-{'X} probe head, the decoupling range is reduced to much less than 

50 ppm, which is inadequate for most compounds in which we are interested. 

This point is perhaps to be borne in mind by anyone else wishing to 

invest in probe heads for 'H-{'X} types of experiment.

Please credit this contribution to the account of Alistair G. Swanson, 

University of Leeds.

Sincerely Yours,

Xavier L.R. Fontaine.

The NMR evolution continues:

Finally—a solids accessory that takes the mystery out of magic angle spinning.

This new high speed CP/MAS accessory, designed for high resolution NMR systems like the Bruker AM Series, is accurate, flexible and above all, easy to use. Pneumatic sample insertion and ejection are simple and routine. A built-in micrometer makes adjustment of the magic angle easy, precise and repeatable.

In fact, every aspect of this accessory reflects the same quality and performance which have placed Bruker's solid-state NMR systems (such as the CXP and MSL Series) in an unparalleled leadership position.

Available for systems up to 500 MHz, it's a powerful yet simple tool that doesn't compromise your materials science analysis, whether it involves polymers, ceramics, zeolites— you name it.

So if you want to take the mystery out of your CP/MAS experiments, get the facts on the Bruker Solids Accessory and the AM Series of NMR Spectrometers.

Bruker Instruments, Inc.
Manning Park, Billerica, MA 01821

In Europe: Bruker Analytische Messtechnik GmbH, Silberstreifen, D-7612 Rheinstetten 4, W. Germany

BRUKER

NMR systems designed to solve problems.
Dear Barry,

In the past few years, Mark Rance, Claudio Dalvit and Peter Wright have put a considerable amount of effort into a detailed examination of the performance (and occasionally, lack thereof) of a variety of multiple-quantum (MQ) and MQ-filtered (MQF) techniques on proteins. One of their standard models for these studies was the blue copper protein, french bean plastocyanin. With all this data in hand, it was high time for the formidable task of obtaining the complete sequence specific assignment of the spectrum for this 99 residue protein. Being smart, they brought in someone else for this little chore.

The assignment of this protein forms a critical foundation for a series of studies to be carried out in Peter's lab on various aspects of protein structure, dynamics, and the basic folding problem. In the next few months, the assignment process will be completed, which will allow us to return to our mounds of data to examine the relationship between various experimental parameters and the nature of the specific amino acid spin systems. As an example, the spectrum shown below demonstrates one of the very powerful aspects of the 3QF-COSY experiment, sign encoding in the multiplet structure. This property came to my rescue one day, when I identified one too many proton resonances in the region of 5.1-5.2 ppm. Upon careful examination of the 3QF-COSY experiment, I noticed the opposite sign of one of the corresponding cross-peaks, as shown in the figure below. This reverse sign of the multiplet pattern is due to the opposite sign of the direct coupling constant, thereby distinguishing this cross-peak (geminal coupling, $J$ negative) from all the others (vicinal coupling, $J$ positive). The pair of spins was subsequently identified as Pro $\delta$ protons with chemical shifts of 3.27 and 5.1 ppm. Proteins never cease to amaze.

Best regards,

Walter Chazin

Mark Rance

Claudio Dalvit

Peter E. Wright

Walter Chazin
Spectral-Spatial 2-Dimensional EPR Imaging

Dear Barry:

In light of the increasing frequency of NMR imaging papers in the Newsletter, we thought that some of the subscribers might be interested to read about related EPR imaging experiments that we are doing.

In the EPR spectra of organic radicals the nuclear hyperfine splittings commonly are greater than the g-value differences and so, unlike NMR, overlapping spectra are the rule rather than the exception. Therefore to image samples containing more than one paramagnetic species it is important to analyze both the spectral and spatial dimensions.

We prepared a test sample composed of two solutions in flat tubes with 0.04 cm path lengths. One tube contained a solution of galvinoxyl radical which gives a ten-line EPR spectrum. The second tube contained a solution of 15N tempone which gives a two-line EPR spectrum. The two tubes were positioned 0.55 cm apart. EPR spectra were obtained as a function of magnetic field gradient for 92 gradients. The first-derivative spectra were integrated. The image in the Figure was constructed using a convoluted back-projection algorithm. The horizontal axis is the magnetic field axis. The shadow of the image along this axis matches with the integral of the first-derivative EPR spectra in the absence of gradient. The vertical axis is the spatial axis. The shadow along this axis accurately reflects the fact that the spin density was localized in two regions of space separated by 0.55 cm. A horizontal slice through the image gives the EPR spectrum at a particular position along the sample. The distinctive spectra from the two samples were clearly resolved.

We feel that this demonstration of the feasibility of spectral-spatial EPR imaging opens up new vistas. Details have been submitted to J. Magn. Reson.

Sincerely,

Gareth R. Eaton
Professor

Sandra S. Eaton
Professor
Dear Barry:

Changes in lens metabolism may precede cataract formation. As a prelude to detecting these changes, Bill Garner of the Department of Ophthalmology and I have been developing methods to establish markers to monitor lens dysfunction. In order to study metabolism in the intact lens, we have been using spin echo NMR.

In this method, the spin echo delay is used as a basis for simplifying otherwise complex proton spectra, and usually limits spectral observation to small metabolites, on the basis of spin-spin relaxation times. The spin echo sequence also helps to improve the degree of solvent suppression. With a delay of 60 msec. the sequence also provides spectral editing on the basis of a nominal $J_{HH}$ of ca 8 Hz. For the first pulse in the echo sequence we use a 150° pulse, as this approximates a $(180^\circ - \text{Ernst})$ angle, and thus improves the data capture rate in these experiments. Examination of lenses in a TC-199 medium, using the $150^\circ - \tau - 180^\circ - \tau$ sequence resulted in several distinctive signals at 3.35, 2.4, 2.15, 1.8, 1.32 and 1.0 ppm. Tentative assignments based on model studies suggest that the major signal at 3.25 may be due to the methylene $\beta$-protons of cysteine in oxidized glutathione (GSSG). The prominent signal at 1.32 corresponds to lactate produced by glycolysis. This latter signal is $180^\circ$ phase shifted because of an eight Hz coupling constant. Signals at 2.55 and 2.15 are associated with the $\gamma$- and $\beta$-glutamyl protons of GSH.

Of course, this work is at a preliminary stage but we hope that comparison of these model studies with human cataract lenses should provide a basis to follow the signals which change with opacification.

Best Wishes

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

September 10, 1986  
(Received 17 September 1986)

Equipment for Sale

The UCD NMR Facility is in the process of obtaining a combined imaging/spectroscopy system. Therefore, we now have for sale our ORS TMR 32/200 spectrometer system. The spectrometer is operating at 1.9T and has a clear bore of 20cm with the profiling coils and 31cm without. The system has outstanding features for spectroscopy on human limbs and on organs of laboratory animals. The homogeneity is good for small volumes (linewidth of 4Hz for a sphere of 3cm diameter). The system includes apart from its standard features:

1) Broadband decoupler under computer control.
2) Profile system to control the size of the homogeneous volume.
3) Honeywell disk cartridge system.

The system is now regularly used an average of 6 days a week. We ask $120,000 for the complete system, but this is of course negotiable. If you are interested please contact the UCD NMR Facility at 916-752-7677.

Jeff de Ropp  
Chris Moonen  
Steve Unger
September 23, 1986 (Received 29 September 1986)

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

Dear Professor Shapiro:

Subject: Effect of Salt Form of C-13 Spectra of Antibiotics

We have been studying the anthracycline antibiotic Aclacinomycin A.HCl by C-13 and proton NMR. The compound was first isolated in 1979 and the C-13 spectrum had been assigned but not the proton. It was therefore decided that the most efficient way to complete the proton assignment was via a proton-carbon 2D experiment. However, our C-13 spectra were characterized by broad lines with poor peak shapes, giving every evidence of gross contamination. The sample was assayed and found to be 98.2% pure, having 1.3% moisture and 0.5% impurity.

After attempting several different solvents and temperatures without result, it was realized that the presence of the HCl salt was causing extensive resonance in the aglycone. After neutralization with aqueous sodium carbonate and deuterium exchange, high resolution C-13 spectra were obtained consistent with the literature spectra. The 2D data was readily obtained and interpreted with the resulting proton assignment shown in Figure 1.

The take-home lesson here is to be aware of the chemistry of the formulation of a pharmaceutical material, something the spectroscopist is prone to forget.

Please credit this to the account of the Bristol-Myers Research Library. Thank you.

Sincerely,

James H. Medley, Ph.D.

cc: J. R. Allison
    E. F. McNiff
Aclacinomycin A neutralized

Aclacinomycin A- HCl

FIGURE 1

Aclacinomycin A
360 MHz H1 NMR assignment (ppm)
Sensitivity in the ROESY Experiment

ROESY, the rotating frame analog of NOESY, offers the advantage that the cross relaxation rate in the rotating frame ($\gamma_{\text{roee}}$) is a positive, monotonically increasing function of the correlation time $\tau_c$ (1,2). In contrast, $\gamma_{\text{noe}}$ in NOESY becomes zero at $\omega_0 \tau_c = \sqrt{2}/2$ for random, isotropic motion (3). Furthermore, the magnitude of $\gamma_{\text{noe}}$ is expected to be at least twice that of $\gamma_{\text{roee}}$ for slow isotropic motion (1,3), suggesting that ROESY will in general be a more sensitive experiment. This limited view of the ROESY experiment may be extremely misleading for the following reasons.

First, $R^\text{roee}_L$, the leakage relaxation rate in the rotating frame (3,4), retains a strong dipolar contribution even for systems undergoing slow motion (4). $R^\text{noe}_L$, however, is essentially independent of the dipolar interaction for an isolated $AXn$ system in this motional regime (3). Since $R^\text{roee}_L > R^\text{noe}_L$ for systems undergoing slow motion, the increased decay rate in ROESY tempers the potential gain in sensitivity. Fig. 1 plots $\xi$, the absolute value of the maximum NOESY cross peak intensity relative to the maximum ROESY cross peak intensity, versus $\tau_c$ for an isolated AX system in which magnetization exchange occurs solely through dipolar coupling. ROESY is clearly expected to be more sensitive than NOESY for this system within the range of $\tau_c$ values typically describing macromolecular rotational diffusion. A similar theoretical plot for an isolated AX$_3$ system (Fig. 2), however, demonstrates that intragroup relaxation may decrease ROE intensities for systems undergoing slow motion. Only for $\tau_c$ very near the NOE cross-over point is ROESY expected to exhibit greater sensitivity than NOESY. Allowing the $X_3$ group to undergo rapid internal rotation ($\omega_0/4D \ll 1$) decreases $\xi$ approximately 4-fold in the limit that $\omega_0 \tau_c \gg 1$ and $r_{XX} > 2r_{AX}$ but does not substantially alter the previous conclusion. Furthermore, as one increases $D_{||} = D_{\perp} = D_{iso}$, $\xi$ first decreases for $\tau_c$ in the slow motion regime, but then begins to increase until the maximum $\xi$ is obtained at $D_{||} = D_{\perp} = D_{iso}$. The effect of intragroup relaxation on the ROE intensity in macromolecules is therefore expected to be less severe for methyl groups, which may undergo rapid internal motion, than for equivalent spins capable of
only restricted internal motion. A more elaborate treatment and analysis of relaxation effects on the relative sensitivity of ROESY compared to NOESY will be presented elsewhere (4).

![Fig. 1](image1)

**Fig. 1:** $|\text{NOE/ROE}|$ relative sensitivity at $\omega_0 = 300$ MHz (---) and $\omega_0 = 500$ MHz (----) for an AX system with $r_{\text{AX}} = 3.0$ Å. The light, dashed line indicates the value for which the ROE and NOE cross peaks exhibit identical maximum intensities.

![Fig. 2](image2)

**Fig. 2:** $|\text{NOE/ROE}|$ relative sensitivity to cross relaxation between a spin A and a methyl group ($X_3$) for several $D_{\|}$ values. The A spin is assumed to lie on the rotation axis of the methyl group. Additional simulation parameters are: $r_{\text{XX}} = 1.80$ Å, $r_{\text{AX}} = 2.96$ Å, $R_{\text{ext}} = 0.2$ Hz, $\omega_0 = 300$ MHz, and $\tau_0 = 1/(6D_{\|})$. $D_{\|}$ is expressed in GHz.

The second aspect of ROESY which diminishes its relative sensitivity is that the sign of the magnetization transferred by dipolar coupling is given by $(-1)^n$ where $n$ is the number of transfers (5). Since direct ($n = 1$) and indirect ($n = 2$) ROE transfers have opposite sign in the spin diffusion regime, their combined presence can lead to a substantial decrease in the maximum intensity of the direct ROE (4). A similar situation manifests itself in NOESY but only in the fast motion regime.
The third factor leading to a loss of sensitivity in the ROESY experiment is the Hartmann-Hahn effect (HHE) \((2,4)\). For dipolar coupling of solely A to M in an AMX system in which only M-X exhibits an HHE, simple considerations \((6,7)\) predict that the maximum A-M ROE intensity will be halved. This is valid only in the limit that \(|q^{mx}/q^{roe}| > 1\) and \(\sin^2(\phi^{mx}) = 1\) \((4)\), where \(q^{mx}\) is the effective homonuclear HHE exchange rate and \(\sin^2(\phi^{mx})\), the maximum efficiency of homonuclear HHE exchange attainable between spins M and X \((6)\). In general for \(|q^{mx}/q^{roe}| > 1\), the maximum A-M ROE intensity is proportional to the factor \(\frac{1}{2}(2 - \sin^2(\phi^{mx}))\). For \(|q^{mx}/q^{roe}| < 1\), there is no effect on the maximum A-M ROE intensity, irrespective of \(\sin^2(\phi^{mx})\). For intermediate values of \(|q^{mx}/q^{roe}|\), the observed ROE will lie between these extremes.

In summary, the experimental \(\xi\) value for a given spin pair is expected to be highly dependent upon local geometry, local dynamics, and global dynamics. For proteins, in which equivalent, dipolar coupled spins generally abound, in which spin pairs are usually surrounded by many non-equivalent, neighboring spins, and in which the suppression of all scalar coupled, Hartmann-Hahn magnetization exchange pathways is virtually unattainable, we estimate that ROESY will offer greater sensitivity than NOESY \((\xi < 1)\) only for motion giving rise to a zero or near zero NOE. This motion, moreover, may arise from internal flexibility rather than the overall tumbling of the macromolecule.

REFERENCES


B.T. Farmer II

L.R. Brown

P.S. - Post-Doctoral Positions: We have finally received our VXR-500 spectrometer to go with the four other supercons. Consequently, I have two positions available to work on structure and dynamics of macromolecules. Airfares to and from Australia will be paid. Interested persons should send a curriculum vitae and addresses for three professional references to me (L.R. Brown) at the above address.
Monsanto Chemical Company has a position available for an NMR spectroscopist at its suburban St. Louis (Creve Coeur) Research Center. The position is in the Physical and Analytical Science Center, a broad range facility that provides support for R&D programs. The successful candidate will be responsible for the operation of a state-of-the-art NMR laboratory and will be involved in the study of a wide variety of products ranging from detergent chemicals to jet engine lubricants. The position offers a mix of long-range studies of new product structure, composition, and performance and shorter term projects related to product improvement, applications, and acceptability. It involves close collaboration with scientists in other disciplines as well as with support group colleagues and provides opportunities for multi-technique approaches to problem solving. The candidate must have a record of technical leadership and be able to interface well with clients, colleagues, and management. The position will involve supervision of the work of technicians.

Interested persons should contact:

Dr. Emmett F. Kaelble
Monsanto Chemical Company, T2B
800 North Lindbergh Blvd.
St. Louis, MO 63167

(314) 694-4262
GE Performance!

\(^1\text{H}, \ ^{13}\text{C}, \text{APT}, \text{and 2D NMR in } \frac{1}{2}\text{ hour} - \text{automatically!}\)

The GE QE-300 does it all—faster than any other NMR spectrometer.

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

With the NMR industry’s most advanced automation.

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

Set-up starts with Autolock.
Lock on as little as 10% CDCl\(_3\) in a 5 mm tube.

Use Comptushim for touching-up spinning shims or complete shimming with both spinning and non-spinning gradients using the lock signal or observe FID.

Autogain optimizes the receiver gain independently for sequential \(^1\text{H}\) and \(^{13}\text{C}\) acquisition.

After data acquisition, Autophaser accurately phases \(^1\text{H}\) and \(^{13}\text{C}\) spectra.

And finally, the analysis is completed with Autointegrate.

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

And the most complete package of hardware accessories.

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

Structural elucidation simplified.

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

High throughput and performance demonstrated.

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

GENERAL ELECTRIC
Why do two experiments when one will do?*

- Simultaneous acquisition of COSY and NOESY

New techniques are easy to implement on a GX Series NMR Spectrometer. Ask today about your application.

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