Texas A & M University N - M - R Newsletter

No. 207

December, 1975

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DEADLINE DATES: No. 208: 5 January 1976
No. 209: 2 February 1976

All Newsletter Correspondence, Etc. Should Be Addressed To:

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

AUTHOR INDEX - TAMU NMR NEWSLETTER NO. 208

COMPAGNIE D'APPLICATIONS MÉCANIQUES CAMECA A L'ELECTRONIQUE AU CINÉMA ET A L'ATOMISTIQUE
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Professor B.L. SHAPIRO
Department of Chemistry
Texas A & University
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Ref : GH/IT. 85 846/75

SELECTIVE PROTON DECOUPLING IN ^{13}C OBSERVATION

Courbevoie, November 5th, 1975

Dear Professor Shapiro,

The CAMECA RMN 250 spectrometer is now equipped with a variable temperature double nuclei probe, operating at 62,86 MHz for carbon 13 and at 250 MHz for proton. To change from one frequency to the other one, only one commutation is needed. Consequently, ^{1}H and ^{13}C spectra are recorded with the same sample tube, which eliminates all concentration, solvent, geometry and temperature differentials effects.

Proton line position are exactly measured, independantly of the deuterium lock frequency. Selective continuous wave proton spin decoupling while recording ^{13}C spectra can be easily and quickly set up. After recording the ^{1}H spectrum and choosing the line to be irradiated, one can set the corresponding frequency on the decoupler and, for example, record the homonuclear proton decoupled spectrum. Without retouching this frequency, by switching to ^{13}C, one can record ^{13}C selectively proton decoupled spectrum with same line being irradiated. Figure B shows the ^{13}C ethylbenzene partially decoupled spectrum obtained by irradiation of methyl protons at a constant power ($\gamma H_2 / 2\pi = 440$ Hz). This irradiation leads to complete cancellation of CH_3 group splitting, residual coupling of CH_2 group ($J_R = 83$ Hz) and no effect on the aromatic carbon signals.

The method has proved to be useful in the ^{13}C resonances assignment, but can also be utilized as a tool in the analysis of complex proton spectra (Yu. N. Luzikov et al. J. Magn. Reosnance 18, 406, 1975)

As a consequence of the good sensitivity in proton observation (≈ 200) since the proton coil is tightly coupled to the sample, the noise power necessary to decouple the protons on a 3000 Hz range is very low (2 to 3 watts) ; the heating of the sample is easily cancelled by an ambiant temperature air flow.

This probe can be used for observation of different nuclei by changing the field. At 250 MHz we observe proton (5.87 T) and fluorine (6.24 T). At 62.86 we observe ^{13}C (5.87 T), ^{11}B (4.62 T), ^{23}Na (5.58 T), ^{31}P (3.64 T) and many other nuclei. For example, to switch from ^{1}H to ^{19}F, it takes 20 minutes, stabilisation of the field included. The magnet, permanently connected to the power supply, can be used almost like a frequency synthesizer.

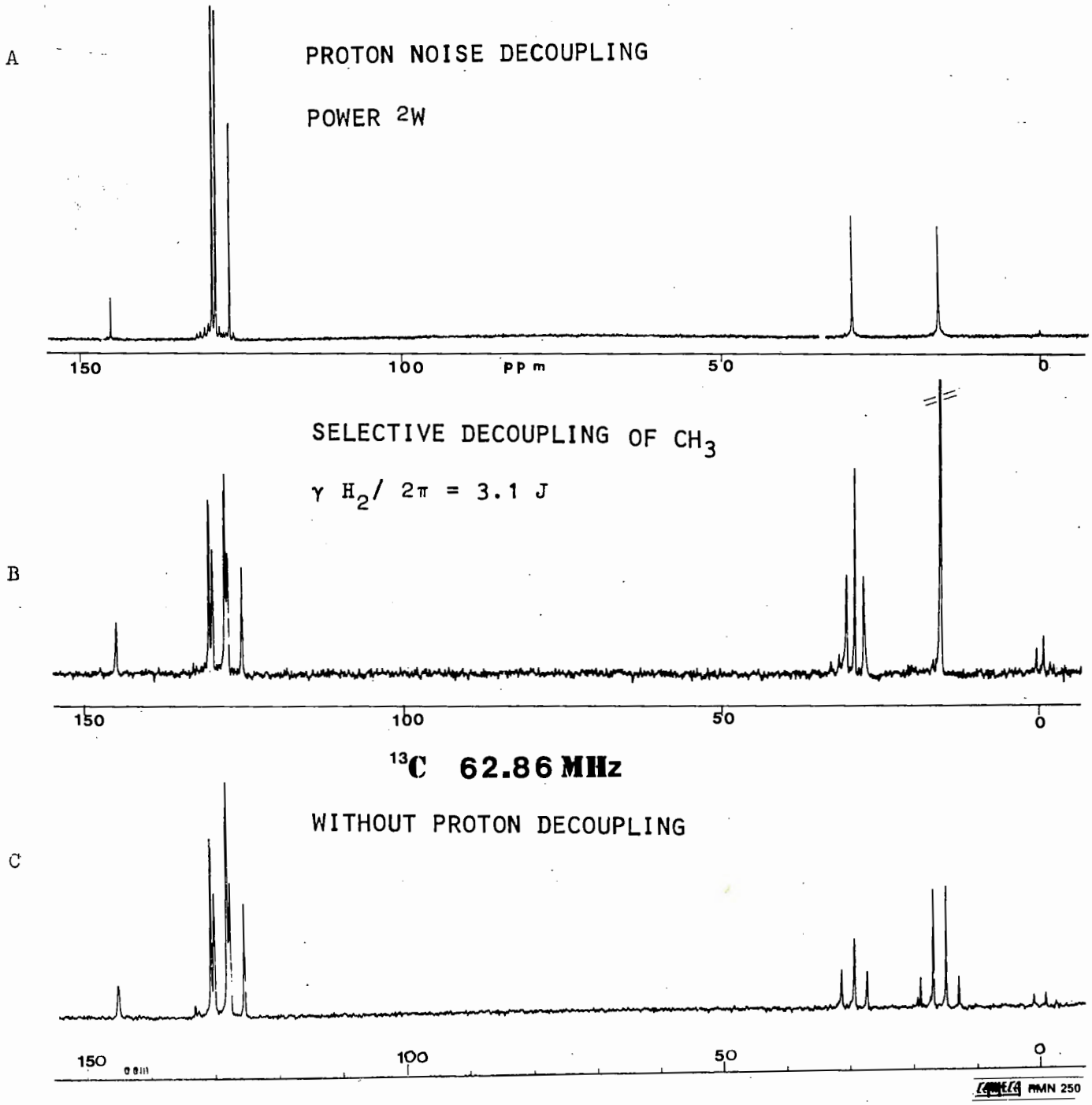
Yours Sincerely,

G. Hajdukovic

Dr GORDANA HAJDUKOVIĆ
NMR Applications Laboratory

CAMECA Suite à notre lettre du 5.11.75 à Pr SHAPIRO N° 85 846

ETHYLBENZENE

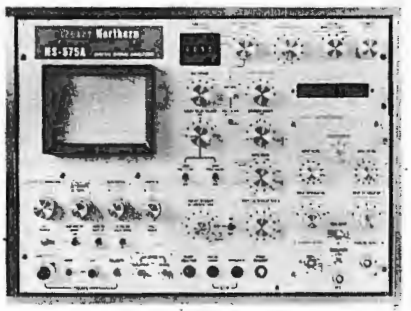


^{13}C natural abundance spectra of ethylbenzene (98 %) + TMS.

8 mm sample tube. Spectrum width 12500 Hz/8K

Multi-budget systems for multi-discipline applications...Tracor Northern signal averagers.

Data once thought to be unobtainable due to its fringe position is now easily captured by Tracor Northern's signal averagers. The adaptability and versatility of our powerful systems is proven by their daily use in fields like spectroscopy, electronics, physics, medicine, mechanical vibration studies and analytical chemistry to name a few.



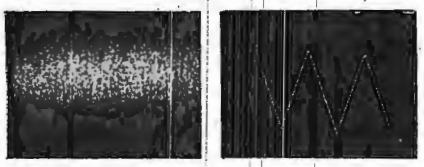
NS-575A Digital Signal Analyzer. The most advanced fixed program signal averaging system.

Over 400 signal averaging installations provide the background and expertise in designing the 575A. Implementation of the most sophisticated production techniques enables us to offer these features as standard:

* 1024, 2048 or 4096 memory with 2^{20}-1 counts per address.
* 4 digit thumbwheel time base control. 10 uSec. to 10 Sec. per address.
* Pre and post analysis delay.
* Continuous or real time display.
* Digital display of address, data, and number of sweeps performed.
* Built in oscilloscope.
* 7 data processing modes.

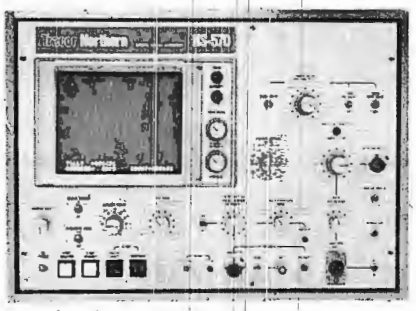
To provide users with extensive flexibility and versatility, over 20 plug-in modules are available for:

* High resolution (9 or 12 bit) averaging of 1 to 8 signals at speeds up to 10 uSec. per address.



Left: NS-575A with Biomation plug-in module examines severe noise situation. Right: 100KHz triangular waveform is detected after 1024 sweeps (real time = 8 sec.).

* 10 nSec. per address averaging with Biomation Transient Recorder.
* PHA/MCS analysis.
* Auto and cross correlation analysis.
* Parametric sweep and pre-synchronization signal averaging.
* Time interval trend/distribution analysis.
* Signal frequency analysis.
* Voltage distribution analysis.
* High resolution waveform digitizing.



NS-570. A unique low cost signal averaging system.

Years of design experience have gone into Tracor Northern's NS-570 to produce a system ideal for dedicated averaging and MCS applications. No other system available today can match the 570's low cost and state of the art features. And no other system comes close to the 570's ease of operation.

Built into this unique system are 6.5" Tektronix oscilloscope, data processor and continuous display. The solid state memory is available in 1024 or 2048 with 2^{20}-1 counts per address.

To make sure our system operates within yours, we offer options like character generator display and NMR synchronizer with easy

Left: NS-570 in a direct averaging application examines a severe noise environment. Right: Sine wave is detected after 1024 sweeps (real time = 20 sec.).

to operate NMR controls. Like the main system, options are low cost.

Both the 575A and 570 are compatible with Tracor Northern accessories which aid data manipulation . . . readout/readin via teletype, high speed paper tape punch and reader, reel to reel and cassette magnetic tape and EIA compatible devices. And interfaces are available for DEC-11, H-P 9800 and Nova series computers.

If you're not sure about your application for signal averaging, give us a call. Our applications assistance department will be able to advise you based on years of experience and over 400 installations. We undoubtedly have worked in a situation similar to yours so we can talk results before you decide. Would you take anything less from the leader?

Computer based Signal Averaging Systems also available. Call or write for details.

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Carr Laboratory, Department of Chemistry
MOUNT HOLYOKE COLLEGE
South Hadley, Massachusetts 01075
Telephone 413 538-2214

24 October 1975

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

Dear Barry,

Magnetic Tape Storage of Diagnostic Programs

We have suffered innumberable instances of frustration when attempting to run diagnostic programs for our HA-100-Digilab NMR-3 FT system. Three fundamental reasons for the frustration are: an extremely tempermental paper tape reader; physical deterioration of the paper tapes; and the length of the more useful diagnostic programs. Any combination of these three circumstances would often result in either failure to load the program or an excessive amount of time spent to accomplish a successful loading. The availability of a magnetic tape assembly suggested, to us, the possibility of storing the diagnostic programs on magnetic tape with the consequent elimination (at worst, amelioration) of the three problems.

We have now devised a procedure for storing the diagnostics on magnetic tape, thus permitting quick and easy loading of the core memory. An additional advantage, because of limited magnetic disk space, is the capability of storing, with rapid access, the Assembler and Editor programs and any user originated program up to a length of approximately 3900_{10} data words (core memory is 4096_{10}). Compared to the normal loading of our diagnostics which requires a functional teletype and computer, our new procedure requires a functional magnetic tape assembly and computer but has the option of the normal routine if the tape assembly is disabled. A big benefit is the decrease in time consumed while loading since the entire core memory is loaded in approximately 2 seconds.

The procedure requires the addition of metallic sensing strips to a blank tape for the purpose of separating program files and incorporates part of the magnetic tape bootstrap program provided by Digilab. Although the present procedure software is specific for Digilab diagnostics, Nova computer, and Kennedy tape assembly, the basic steps are, probably, easily adapted to other systems. A detailed description, along with a copy of our present procedure software, is available upon request.

A good description of the operation is "crude but effective".

Sincerely yours,

Curt P. Beeman
Postdoctoral Fellow

Kenneth L. Williamson
Professor of Chemistry

DEPARTMENT OF BIOCHEMISTRY

THE UNIVERSITY OF ALBERTA
EDMONTON, CANADA T6G 2E1

November 6, 1975

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

Dear Barry:

Title: "270 MHz Cryospectrometer without liq. He in the Frozen North"

I hope our mail strike has not delayed this contribution beyond the end of your colored notices. We are completely up and running with our Bruker HX-270 Spectrometer, after our move to Edmonton, and Grayson Snyder and I have been continuing our investigations of the bovine pancreatic trypsin inhibitor[1,2]. This protein has a unique spectrum with resolvable NH protons in the low field region which do not exchange with D_2O over periods of months to years. A portion of dinitro-BPTI spectrum is shown in the enclosed Figure. We are presently measuring T_1's and T_2's at high fields and the Figure shows a portion of one such T_1 experiment. Unfortunately the pulse lengths were not set to exact 90° & 180° lengths in this experiment but the results are clear. Each of this spectra are several thousand scans (given protein concentration of the order of 1-2 mM) and the whole experiment took a couple of days (given the longer T_1's ~ 1-2 seconds at the high fields). We used a phase alternated version of the normal 180-τ-90 experiment so that phase anomalies at short τ values from resonances with long T_2^*'s were cancelled out. We are now trying to puzzle out exactly what is relaxing these internally H-bonded NH protons.

Yours sincerely,

Brian

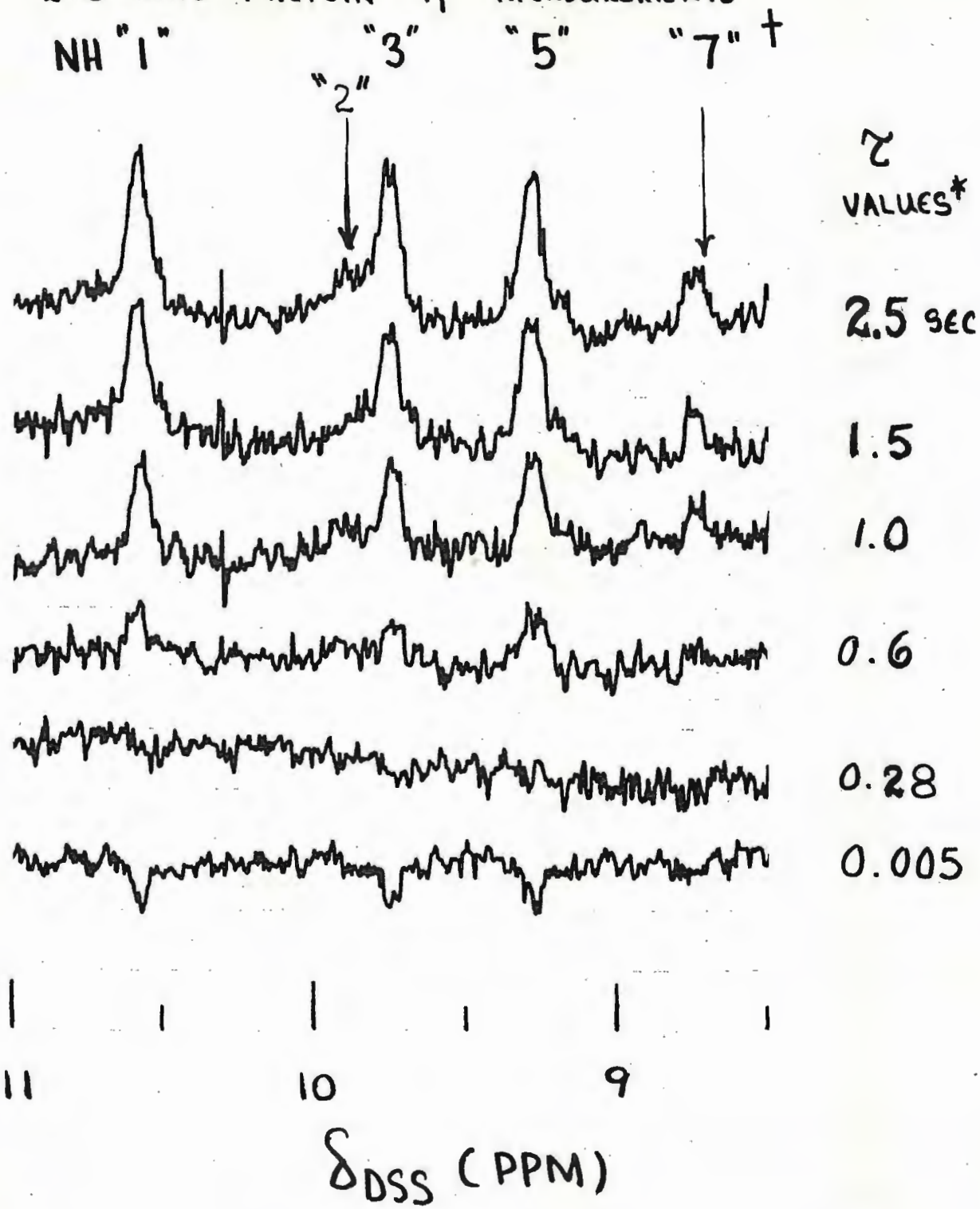
Brian D. Sykes
Associate Professor

Grayson

BDS/jkd

1. Biochem. 12, 1323 (1973)
2. Biochem. 14, 3765 (1975)

270 MHz Protein T_1 Measurements



* 90° & 180° PULSE LENGTHS WERE MISCALIBRATED RESULTING IN EFFECTIVE INVERSION OF LESS THAN 100%.

† NUMBERING OF NH'S FROM REF. #1

UNIVERSITÄT DES SAARLANDES
Fachbereich 14 - Organische Chemie

Professor Dr. H. Dürr

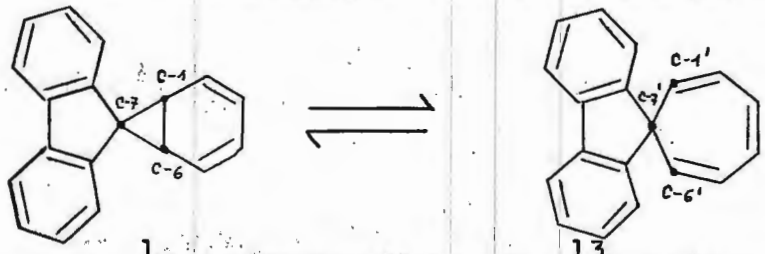
66 Saarbrücken, den 10.11.75 Dü/Wi
Telefon: (0681) 302.3409

Professor
Dr. B. L. SHAPIRO
Department of Chemistry
Texas A and M University
College Station
T e x a s 77843
U. S. A.

Dear Professor Shapiro,

Line shape analysis with the aid of C^{13}-NMR

Bei [2.3]-Spirenen sind die genauen Aktivierungsparameter der Valenzisomerisierung Cycloheptatrien ⇌ Norcaradien im Gegensatz zu anderen Verbindungen dieses Typs noch nicht bekannt.[1.2] Dies ist vor allem auf die Komplexität der ^{1}H-NMR-Spektren dieses Verbindungstyps zurückzuführen, die sich nur durch eine quantenmechanische Behandlung der Linienformanalyse auswerten lassen.



1 1'

Nimmt man jedoch die ^{1}H-breitbandentkoppelten ^{13}C-Spektren dieser Verbindungen auf, so erhält man für alle Kohlenstoffatome Singuletts, auf die sich eine Linienformanalyse mit Hilfe der modifizierten Bloch'schen Gleichungen anwenden lässt. Die Messungen und Berechnungen wurden am Beispiel 1 ⇌ 1' durchgeführt.

Die für die Linienformanalyse notwendigen Prozentgehalte an Cycloheptatrien beziehungsweise Norcaradien wurden oberhalb des Koaleszenzpunktes mit Hilfe der Gleichung (1) bestimmt:

$$K = \frac{\tau_{AB} - \tau_{B}}{\tau_{A} - \tau_{AB}} = \frac{C_A}{C_B} \quad (1)$$

Im Bereich der Koaleszenz und unterhalb des Koaleszenzpunktes wurden die Molenbrüche durch Extrapolation mit Hilfe einer Auftragung lg K = f (1/T) bestimmt.

Alle Messungen wurden mit einem Bruker HX-90-Gerät aufgenommen. Die Konzentration der Lösungen war 20%ig in $CDCl_3$. Das D-Signal des Lösungsmittels wurde als Lock benützt. Die Zahl der Akkumulationen war bei allen Spektren 8192. Durch exponentielle Multiplikation des Interferogramms wurde das Signal-Rausch-Verhältnis zusätzlich verbessert. Die hierdurch hervorgerufene Linienverbreiterung wurde bei der Berechnung der Spektren berücksichtigt. Die Einstellung der Messtemperatur geschah direkt mit Hilfe der Bruker-Temperatur-Kontrolleinheit, die unter Anwendung eines Thermoelementes geeicht worden war.

Die Linienformanalyse wurde sowohl für die Resonanzen der C-1.6 als auch z.T. der C-7-Kohlenstoffatome durchgeführt.

Die chemischen Verschiebungen sind für C-7 δ = 16.00 ppm, C-7' δ = 56.00 ppm, C-1.6 δ = 38.6 ppm und C-1.6' δ = 129.2 ppm. Es ergaben sich keine wesentlichen Unterschiede in den τ -Werten der beiden Messreihen.

Als Aktivierungsparameter für die Reaktion $\underline{\underline{1}} \rightleftharpoons \underline{\underline{1}}'$ ergeben sich folgende Werte: E_A=10.8 ± 0.4 kcal/Mol; $\Delta H^{\neq}$ = 10.4 ± 0.4 kcal/Mol; $\Delta G^{\neq}$ = 10.6 ± 0.6 kcal/Mol; $\Delta S^{\neq}$ = -1 ± 2 cal/Mol . Grad.

Für die Rückreaktion $\underline{\underline{1}}' \rightleftharpoons \underline{\underline{1}}$ erhält man : E_A = 9.7 ± 0.4 kcal/Mol; $\Delta H^{\neq}$ = 9.2 ± 0.4 kcal/Mol; $\Delta G^{\neq}$ = 10.4 ± 0.5 kcal/Mol; $\Delta S^{\neq}$ = -4 ± 2 cal/Mol Grad.

Mit freundlichen Grüssen

(H. Dürr) (M. Kausch)

References:

1.) M. Görlitz und H. Günther, Tetrahedron 25, 4467 (1969)

2.) N. J. Reich, E. Ciganek und J. D. Roberts, J. Amer. Chem. Soc. 92, 5166 (1970)

Check the Nicolet NIC-1180 Applications

The NIC-1180 currently offers software packages for these applications:

APPLICATION: *General Use*

CAPABILITIES: This includes: an assembler-editor program, both for paper tape and disk-based systems, for user programming in machine language; diagnostics for machine maintenance; a floating point package consisting of subroutines for most arithmetic functions; and a disk monitor program for both the floppy and cartridge disk systems.

APPLICATION: *NMR Spectroscopy*

CAPABILITIES: Four programs are currently offered in the NMR applications software package:

1) FT-NMR-1180. This package includes the capability to perform Fourier transforms on time domain data blocks from 256 data points to 64K data points. A 4K transform takes 2½ seconds; a 32K transform takes 24 seconds. Routines are included for real-time phase correction, zoom display, peak picking, inverse FFT, 9 point smooth (per Savitsky-Golay)[1] exponential or trapezoidal windowing functions, constant speed plotting while acquiring new data, integration with baseline and slope correction, and addition and subtraction of spectra.

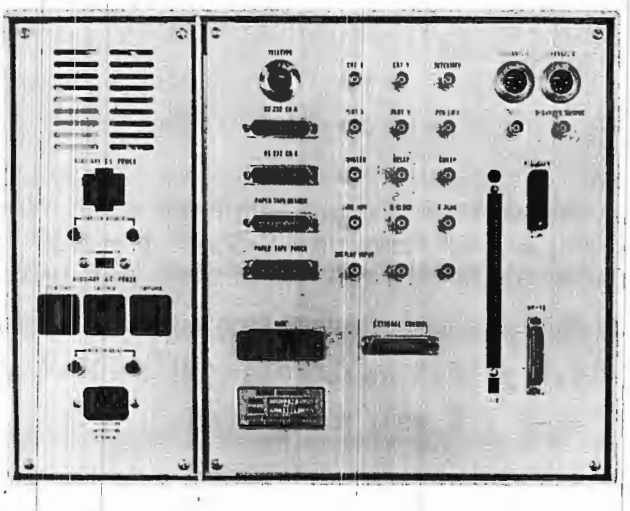
2) Quadrature FT-NMR. This package includes the above plus the ability to acquire and transform quadrature data (per the technique of Shaefer/Stejskal).[2]

3) ITRCAL. This program for the analysis of complex-coupled NMR spectra uses the method of Castellano and Bothner-By[3] to calculate a theoretical spectrum which is then iterated for a best fit with a set of experimentally observed lines.

4) T1/1180. This program is for the measurement of the spin-lattice relaxation time, T1, using either inversion recovery, progressive saturation or McDonald-Leigh[4] pulse sequences.

APPLICATION: *Laser Raman Spectroscopy*

CAPABILITIES: The Laser Raman package for the NIC-1180 uses the Multi-Channel Scaling acquisition mode to collect data from the spectrometer discriminator. The spectrometer is driven by pulses from the data processor which also monitors the progress of the spectrometer's stepper motor to provide reproducible signal averaging. Processing software includes a routine to divide spectra (for depolarization ratios), a full scale expansion routine which allows spectra of different intensities to be compared to each other, and calibration commands.



APPLICATION: *EPR Spectroscopy*

CAPABILITIES: This two-part package includes subroutines for data collection and processing (to fit specific EPR needs) and for EPRCAL, a simulation program for nitroxide spin labels. EPRCAL can simulate both conventional EPR spectra and saturation transfer spectra, explicitly accounting for variations in magnetic tensors, relaxation times, correlation time, diffusional model, microwave power, and modulation frequency.[5]

APPLICATION: *FT-IR Applications*

CAPABILITIES: Routines for signal averaging and Fourier transformation of infrared interferograms into main frame memory (up to 64K data points) or onto disk memory (up to 512K data points) are provided. The Fourier transformation of 512K time domain points into 256K real and imaginary frequency domain points takes 28 minutes.

[1] A. Savitsky and M.J.E. Golay, *Anal. Chem. 36*, 1627 (1964).

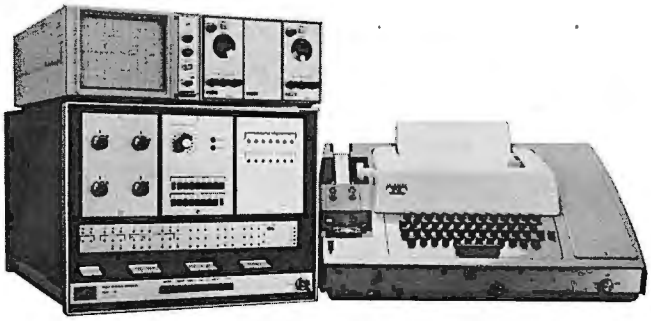
[2] E.O. Stejskal and J. Schaefer, *J. Mag. Res. 14*, 160 (1974).

[3] S. Castellano and A.A. Bothner-By, *J. Chem. Phys.* 41, 3863 (1964).

[4] G.G. McDonald and John S. Leigh, Jr., *J. Mag. Res.* 9, 358 (1973)

[5] P. Coffey, B.H. Robinson, and L.R. Dalton, *Chem. Phys. Lett.*, to be published.

NIC



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FEATURE: *The NIC-1180 is a complete "turnkey" system with a wide range of software.*

BENEFIT: The system is delivered and installed with software and training provided at time of installation so system can be put to use immediately. NMR, FT-IR, EPR and Laser Raman applications software, in addition to general laboratory data collection and processing software are currently offered.

FEATURE: *Field-expandable memory is 20-bit word, MOS solid state.*

BENEFIT: This word length is optimal for signal averaging and high-dynamic-range frequency analysis applications. It offers a more efficient instruction set, minimizes the requirements for double precision arithmetic, and permits up to 80K of memory to be easily addressed.

FEATURE: *Optional analog-to-digital converters (ADC's) are offered.*

BENEFIT: The choice of standard 12-bit, 333 kHz ADC or optional 15-bit, 48 or 90 kHz; 8-bit, 5 MHz ADC's; or 15-bit pulse counting buffer permits the user to choose the most appropriate bandwidth or resolution.

FEATURE: *Direct memory access is provided through two concurrent DMA ports.*

BENEFIT: Operating at rates up to one million transfers to memory per second, DMA permits, for example, concurrent memory accessing by the ADC and the disk memory.

FEATURE: *A seven-level, vectored priority interrupt scheme is provided.*

BENEFIT: This feature supports a multi-task environment. For instance, acquisition, processing and plotting can be conducted simultaneously.

FEATURE: *A hardware Read Only Memory (ROM) program loader is included.*

BENEFIT: Containing programs for tape readers and disk memory systems the ROM eliminates time-consuming "bootstrap" program loading through the switch register.

FEATURE: *Many arithmetic and logic functions required for high speed data processing are hardwired.*

BENEFIT: Fixed point and floating point operations are executed quickly. Divide, signed multiply, bit inversion and 60-bit shift are hardwired and allow a 4K FFT computation in 2.5 seconds.

FEATURE: *Four front panel parameter knobs provide user interaction in CRT display routines.*

BENEFIT: These controls permit determination of the start point and width of the displayed data and entry of phase correction constants, integration, baseline and slope correction factors and other data manipulation operations.

FEATURE: *Real-time alphanumeric displays are provided on the CRT.*

BENEFIT: A 64-character ASCII character generator provides up to 30 characters per line to display number of sweeps made while acquiring time domain data or scale factors, peak positions, and intensities during frequency domain measurements.

FEATURE: *Many peripheral interfaces are built into the NIC-1180 main frame.*

BENEFIT: These include two channels of RS-232C of which one can be used for a Teletype. All common baud rates from 110 to 38,400 bauds, and one channel each for high speed reader and high speed punch are built in. Provisions have also been made for the new Hewlett-Packard Interface Bus (HPIB).

FEATURE: *A complete list of optional peripherals includes every conceivable need.*

BENEFIT: The list of peripherals includes CRT terminals, tape readers, tape punches, cartridge and "floppy" disk memories, X-Y plotters, line printers, magnetic tape recorders, a signal conditioner, and a pulse programmer.

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CENTRAL RESEARCH & DEVELOPMENT DEPARTMENT
EXPERIMENTAL STATION
Spectroscopy Division

November 7, 1975

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

Dear Barry:

Hexamethyl Phosphoramide: A Possible Carcinogen

We wish to draw the attention of nmr spectroscopists who may use hexamethyl phosphoramide (HMPA) as a solvent, to a recent letter by J. A. Zapp, Jr., which appeared in Science, 190, p.422 (1975). This describes experiments in which a rare form of cancer (squamous cell nasal carcinoma) has been produced in rats by inhalation exposure to HMPA in a concentration as low as 400 ppb in a period of about 8 months and urges everyone using HMPA to handle it with the precautions appropriate to a potential carcinogen.

Sincerely,

Derick W. Ovenall

Derick W. Ovenall

DWO/dew
5

ABBOTT

Scientific Division

Abbott Laboratories
North Chicago, Illinois 60064

November 11, 1975

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

C-H and C-F Coupling Constants

Dear Barry,

We recently had two fluorinated hydrocarbons pass through our lab. Before they evaporated we measured their ^{13}C-NMR spectra and present here an analysis of both their chemical shifts and coupling constants measured from 80% solutions in acetone-d_6.

Ethrane

CHF_2-O-CF_2-CHFCl
1 2 3

C_1 = 114.3 ppm
$J_{\underline{C}-F}$ = 261.8 Hz
$J_{\underline{C}-O-C-F}$ = 6.0
$J_{\underline{C}-H}$ = 227.4

C_2 = 119.1 ppm
$J_{\underline{C}-F}$ = 274.4 Hz
$J_{\underline{C}-C-F}$ = 26.9
$J_{\underline{C}-O-C-F}$ = 4.2
$J_{\underline{C}-C-H}$ = 25
$J_{\underline{C}-O-C-H}$ = 3

C_3 = 95.7 ppm
$J_{\underline{C}-F}$ = 250.0 Hz
$J_{\underline{C}-C-F}$ = 40.5
$J_{\underline{C}-H}$ = 189.9

Forane

CHF_2-O-CHCl-CF_3
1 2 3

C_1 = 117.1 ppm
$J_{\underline{C}-F}$ = 265.4 Hz
$J_{\underline{C}-H}$ = 229.2
$J_{\underline{C}-O-C-H}$ = 4.6

C_2 = 81.4 ppm
$J_{\underline{C}-C-F}$ = 40.9
$J_{\underline{C}-O-C-F}$ = 5.5
$J_{\underline{C}-H}$ = 176.5
$J_{\underline{C}-O-C-H}$ = 5

C_3 = 122.0 ppm
$J_{\underline{C}-F}$ = 278.3
$J_{\underline{C}-C-H}$ = 1.7

No attempt was made to determine the relative signs of the couplings. We have recently reported the ^{1}H and ^{19}F chemical shifts and coupling constants for ethrane (TAMNMR 175) and now need only to measure ^{17}O and ^{35}Cl to finish the job.

Sincerely,

Rich

Richard S. Egan

Ruth

Ruth S. Stanaszek

msg

November 18, 1975

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

Dear Barry:

^{13}C Chemical Shift Changes Induced in Organic Compounds by Silver Complexation or Protonation. Peak Intensity Calculation on the CFT-20

We have been interested in the effects of metal complexation on the ^{13}C spectra of organic compounds. In the course of the work we have found that complexation with one equivalent of Ag^+ shifts the resonance of the thiocarbonyl carbon of tetramethylthiourea upfield by 8.3 ppm. Similar, but smaller, upfield shifts have been reported for the amide carbonyl carbons upon complexation to aluminum ions through oxygen.[1]

Although the direction of these shifts is opposite to that which might naively be expected for coordination of a neutral ligand to a positive metal ion, the results may be explained in terms of a greater contribution in the metal complex from resonance structures containing doubly-bonded nitrogen.

Oximes, at least, absorb around 155 ppm from TMS, and it seems likely that carbons doubly bonded to nitrogen absorb generally in that region, far upfield from the absorptions of either carbonyl or thiocarbonyl carbons.

It is somewhat disconcerting, however, that carboxylate anions also show upfield shifts upon protonation even though the resonance argument, at least as phrased above, is inapplicable since only carbon double bonds to oxygen are possible. On the other hand, the carbonyl carbons of ketones and esters do show downfield shifts upon protonation of the parent compounds in sulfuric acid,[2] as might be explained in terms of charge density changes alone. Furthermore, several quick experiments have shown that the carbonyl carbon of dimethylformamide absorbs only 0.6 ppm further downfield in sulfuric acid than in chloroform relative

to an uncorrected capillary. The small difference could be a result of a balance between the resonance effect and charge density changes.

There are various other examples in the literature in which multiply-bonded carbons in negatively charged species resonate substantially downfield from corresponding neutral compounds.[3] The factors responsible for the shifts may (or may not) be related to the results for the metal complexes.

CN^-	168.6 ppm	HCN	110.9 ppm	CH_3CN	117.2	CH_3NC	158.7
NCS^-	134.4	CH_3NCS	128.7				
NCO^-	129.8	CH_3NCO	121.5				

The overall results suggest that care should be taken in the formulation of explanations for chemical shift differences even between related species. In the cases above, a balance between several different factors, perhaps including changes in the average excitation energies of various compounds, may be important. Differences in the excitation energies of carbonyl and thiocarbonyl compounds have already been invoked to explain the downfield position of thiocarbonyl carbons relative to corresponding carbonyl carbons.[4]

With regard to a different matter, the program changes suggested by Lincoln and Wray[5] are indeed needed if accurate line positions are to be obtained with the CFT-20. The program as written not only did not interpolate peak positions correctly, however, but did not interpolate peak intensities at all, merely giving the intensity of the largest computer channel. The proposed patch does not change this. The programming changes needed to correct the problem would be tedious but might be worthwhile for some applications. The patch may be placed with slight modification in locations 33647 to 33666 (right before the Bodenhausen, Turner and Freeman modification)[6], if it is desired not to destroy the Sykes routines. More information will be sent on request.

Sincerely yours,

Mark Henrichs

PMH:nc

Paul Mark Henrichs
Chemistry Division
Research Laboratories

1 D. Canet, J.-J. Delpuech, M. R. Khaddar, and P. Rubini, J. Magnetic Res., 15, 325 (1974).
2 G. E. Maciel and J. J. Natterstad, J. Chem. Phys., 42, 2752 (1965).
3 J. B. Stother, "Carbon-13 NMR Spectroscopy," pp. 307-308.
4a. G. E. Maciel and D. A. Beatty, J. Phys. Chem., 69, 3920 (1965).
b. H.-O. Kalinowski and H. Kessler, Ang. Chem., 86, 43 (1974).
5 D. N. Lincoln and V. Wray, NMR Newsletter, No. 204, 49 (September 1975).
6 G. Bodenhausen, D. Turner and R. Freeman, NMR Newsletter, No. 201, 9 (June 1975).

207-15

Department of Chemistry

The Florida State University
Tallahassee, Florida 32306

November 24, 1975

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

Dear Barry,

Leo Mandelkern and I have at least two openings for postdoctoral research fellows in the areas of nmr studies of peptide conformations and synthetic polymer dynamics. These studies will include ^{13}C (some ^{15}N nmr) and also high field ^{1}H nmr work on our Bruker HX-270 and HFX-90 spectrometers.

The peptides research area positions can either be taken as joint fellowships spanning the two research groups or as individual positions within either group, depending on the scientific interests of the applicant. The synthetic polymer dynamics researcher will be a member of both research groups.

I also have a continuing need for a good physical chemistry oriented nmr spectroscopy Ph.D. to do research in ^{13}C and ^{15}N relaxation of "small molecules". This person will also help in the construction of a new design wide-bore other nuclei nmr spectrometer.

Some of these positions are available immediately; they should be filled by next summer if possible. The positions are for 1 year with renewal possible upon mutual agreement; starting salary will range from $8,000-9,500 per year depending on experience.

Interested persons should write to me as soon as possible, enclosing a short vita. Applicants are also requested to have two letters of recommendation forwarded to me.

Best Yuletide regards,

George C. Levy
Associate Professor

GCL/lck

REMINDER

The 17th ENC will be in Pittsburgh; Apr. 25-29th. Submit Posters to:

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November 20, 1975
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Professor B.L. Shapiro
Department of Chemistry
Texas A & M University
College Station
Texas 77843
U.S.A.

Dear Professor Shapiro:

In a collaboration with groups of Prof.Lippmaa in Tallinn (Dr.Mägi) and Prof. Calas in Bordeaux (Drs.Dunogues and Bourgeois) we have investigated ^{13}C and ^{29}Si NMR spectra of several compounds with sterically crowded trimethylsilyl groups. The experimental results for some of these compounds are given bellow together with the data for similar compounds where no crowding can be expected.

Compound	Chemical shift in $(CH_3)_3Si$ group (relative to TMS, downfield shift is positive) $\delta(^{29}Si)$	$\delta(^{13}C)$
para-bis(trimethylsilyl)benzene	-4.7	-0.9
ortho-bis(trimethylsilyl)benzene	-4.1	2.0
trans-bis(trimethylsilyl)ethene	-8.9	-1.7
cis-bis(trimethylsilyl)ethene	- 11.8	-0.7

If the sign of the chemical shift difference between crowded and not crowded molecules can be identified with the sign of the crowding effect then in the benzene derivatives crowding leads to deshielding of silicon (similarly as it deshields tert.carbon in tert.butylbenzenes[1]) but in ethylene derivatives it increases the shielding (as usually assumed for carbon in analogous compound[2] but not found in tert.butyl ethylenes[3]).

With the best regards,

Sincerely yours,

Jan Schraml

1.Motell E.L.,Lauer D.,Maciel G.E.:J.Phys.Chem.77,1865(1973).
2.Levy G.C.,Nelson G.L.:Carbon-13 Nuclear Magnetic Resonance for Organic Chemists, p.24.
3.Garratt D.G.,Tidwell T.T.:Org.Magn.Res.6,87(1974).

207-18
THE ROYAL INSTITUTE OF TECHNOLOGY
DIVISION OF PHYSICAL CHEMISTRY
S-100 44 STOCKHOLM 70
SWEDEN

Cable address: Technology

Stockholm, November 24, 1975

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

^{14}N RELAXATION IN AQUEOUS MICELLAR SOLUTIONS

Dear Barry:

We have used ^{14}N relaxation to study the molecular dynamics of aqueous solution of some cationic surfactants containing quarternary nitrogen. Fig. 1 shows the concentration dependence of ^{14}N T_1 and T_2 for n-hexadecyltrimethylammoniumbromide (CTAB) and chloride (CTAC). It is seen that the relaxation behaviour is quite different for the bromide and the chloride. From other observations, it is known that CTAC forms spherical micelles while CTAB at higher concentrations forms large rodlike aggregates (1). Following Berendsen and Edzes (2) and Wennerström et al. (3) we have interpreted the relaxation data in terms of a rapid local anisotropic motion of the surfactant molecule which partially averages the quadrupole Hamiltonian and a slower motion over distances of the order of the dimensions of the micellar aggregates. The fast motion is characterized by a correlation time $\sim 7 \cdot 10^{-10}$ s in CTAB micelles and it is approximately twice as fast in CTAC micelles. The correlation time for the slow motion in CTAB micelles increases with concentration. From comparisons with models for the motion of rodlike aggregates it appears that the larger micelles are flexible.

Sincerely

Lars Ödberg

Ulf Henriksson

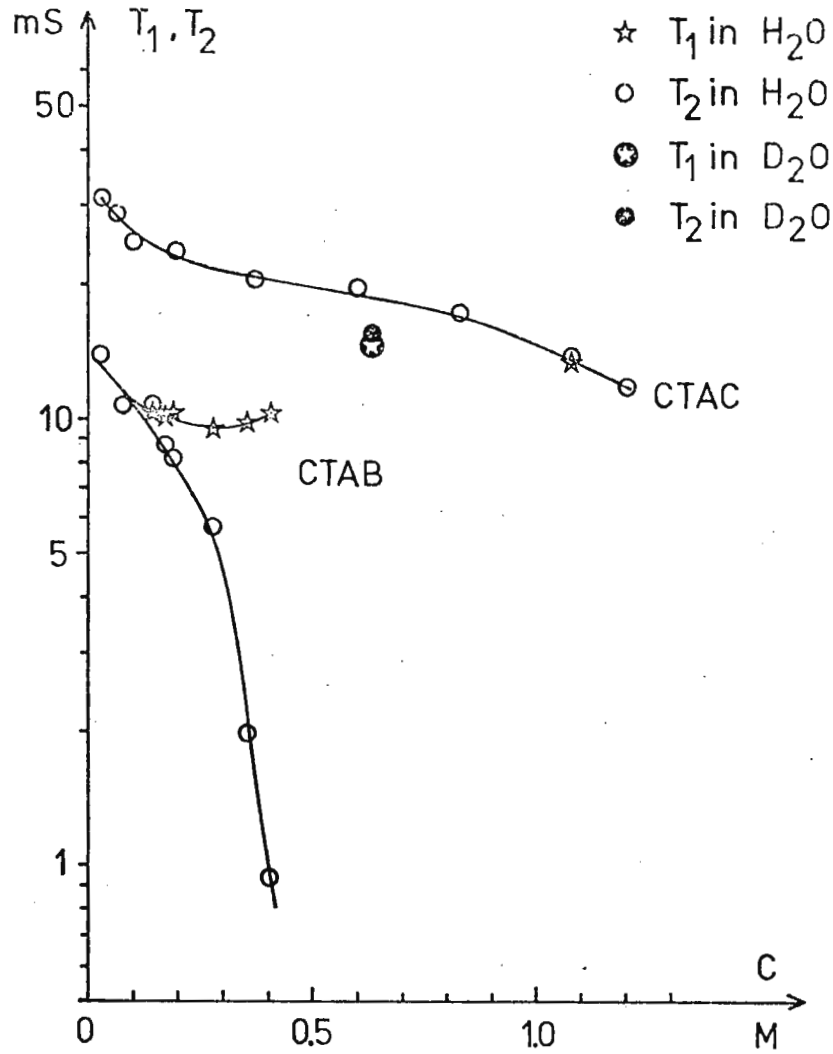


Fig. 1 ^{14}N relaxation times in aqueous solutions of CTAB and CTAC at 28.5 °C.

References

1. Reiss Husson, F. and Luzzati, V., J. Phys. Chem. 68, 3504 (1964).
2. Berendsen, H.J.C. and Edzes, H.T., Ann. N.Y. Acad. Sci. 204, 459 (1973).
3. Wennerström, H., Lindblom, G. and Lindman, B., Chemica Scripta 6, 97 (1974).

Professeur PIERRE LASZLO

Institut de Chimie
Université de Liège
Sart-Tilman par 4000 Liège 1, Belgique

November 25, 1975

Structures of complexes between Na^+ and oxygenated solvents.

Dear Barry,

We have investigated solvent exchange in complexes between the sodium cation and oxygenated solvents, monitoring the sodium-23 chemical shift as a function of composition in binary mixtures. The data point to tetrahedral geometries in which each corner is occupied by an oxygen : coordination numbers are 2 for bidentate ligands such as ethylene glycol tetrahydrofurfuryl alcohol or the glymes, and 4 with monodentate ligands such as THF. The solvent exchange proceeds through intermediates, also of tetrahedral geometry :

$$\text{species A} \underset{}{\overset{K_1}{\rightleftarrows}} \text{(intermediate)} \overset{K_2}{\rightleftarrows} \text{species B}$$

The geometry of this intermediate is as shown (see on the next page).

All these results and others are consistent with description of solvent exchange as an associative process, with a trigonal bipyramidal transition state. Only diglyme and triglyme can occupy diequatorial sites. The other bidentate ligands are constrained to span apical and equatorial positions.

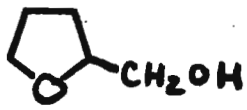
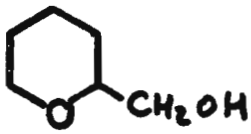
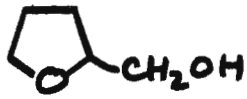
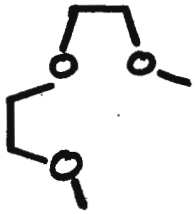
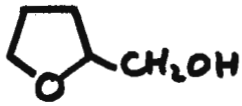
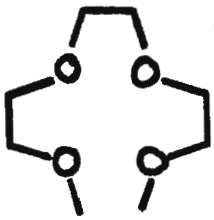
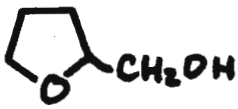
This is the ground work for a study of the sodium-sugar interactions, which we are now completing.

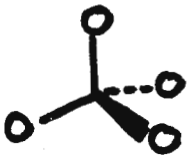
With best regards,

Sincerely,

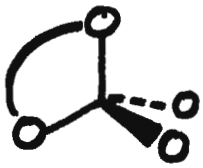
Christian DETELLIER

Pierre LASZLO

solvent A	solvent B	intermediate	K_1/K_2 : calc	exp
		OPEN	36	36
		OPEN	36	30
		MONOCYCLIC OR BICYCLIC	4-16	9
		BICYCLIC	4	4
		BICYCLIC	4	4



OPEN



MONOCYCLIC

BICYCLIC

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Prof.Dr.H.Günther

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DURCHWAHL: 470 4102

Nov. 20, 1975

New Adress!

Prof.Dr.B.L.Shapiro
Dept. of Chemistry
Texas A + M University
College of Science
College Station, Texas 77843
USA

^{13}C-NMR Data of Cyclopropenes

Dear Barry:

We have determined the - to our knowledge - hitherto unknown nmr data of cyclopropene and two methyl derivatives. Methyl-cyclopropane was measured for comparison. The results are given in the Table below, a communication has been submitted for publication to the J. Amer. Chem. Soc.

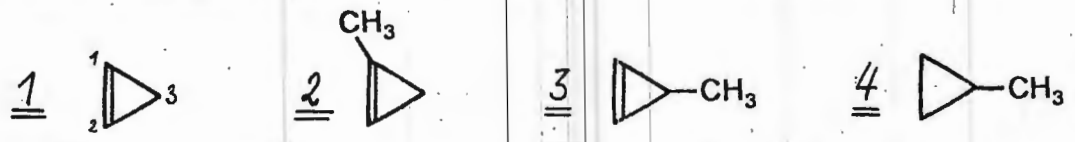


Table I. Chemical Shifts δ_{TMS} (ppm) and One Bond ^{13}C,^{1}H Coupling Constants (Hz, in brackets) for Cyclopropenes and Methyl-cyclopropane (4)

	C-1	C-2	C-3	CH_3
1	108.9 (228.2[a])	---	2.3 (167.0)	---
2	116.5 ---	98.8 (224.7)	6.2 (169.0[a])	12.5 (129.0)
3	117.6 (224.0)	---	10.1 (162.4)	23.6 (124.5)
4	5.7 (161.1)	6.1 (159.9)	---	19.9 (126.1)

[a] These values differ from those obtained earlier from the ^{13}C satellites in the ^{1}H NMR spectra: 226 Hz[6] and 172 Hz[7], respectively. For the first case, the agreement with the calculated value of 229 Hz[8] is now much better.

(6) J. B. Lambert et al., J. Phys. Chem., 74, 2221 (1970).
(7) G. L. Closs, Advanc. Alicyclic Chem., 1, 53 (1966).
(8) M. D. Newton et al., J. Amer. Chem. Soc., 96, 17 (1974).

Sincerely yours,

H.Günther H.Seel

MONASH UNIVERSITY

TELEGRAMS:
MONASHUNI, MELBOURNE

CLAYTON, VICTORIA. 3168

TELEPHONE:
544 0811

FACULTY OF SCIENCE

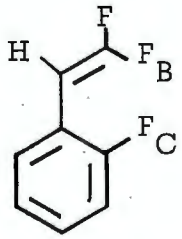
Department of Chemistry

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

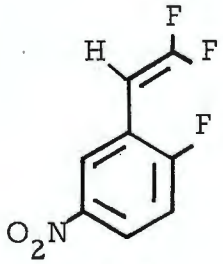
$^5J_{FF}$ in Fluorostyrenes

Dear Barry,

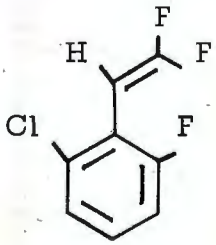
David Burgess and I have prepared a number of fluorostyrenes in the hope of finding some interesting long-range couplings. We got more than we bargained for!



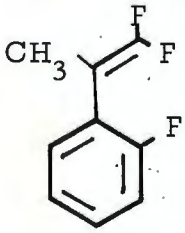
J_{BC} in CCl_4 4.28
in DMF 8.16



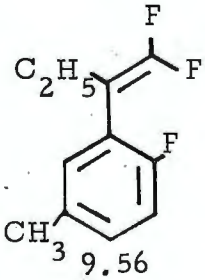
5.51
10.41



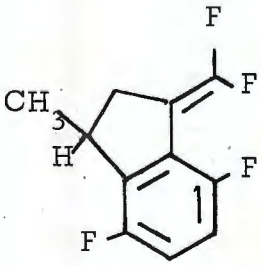
33.34
31.64



11.83
15.07



9.56
11.43



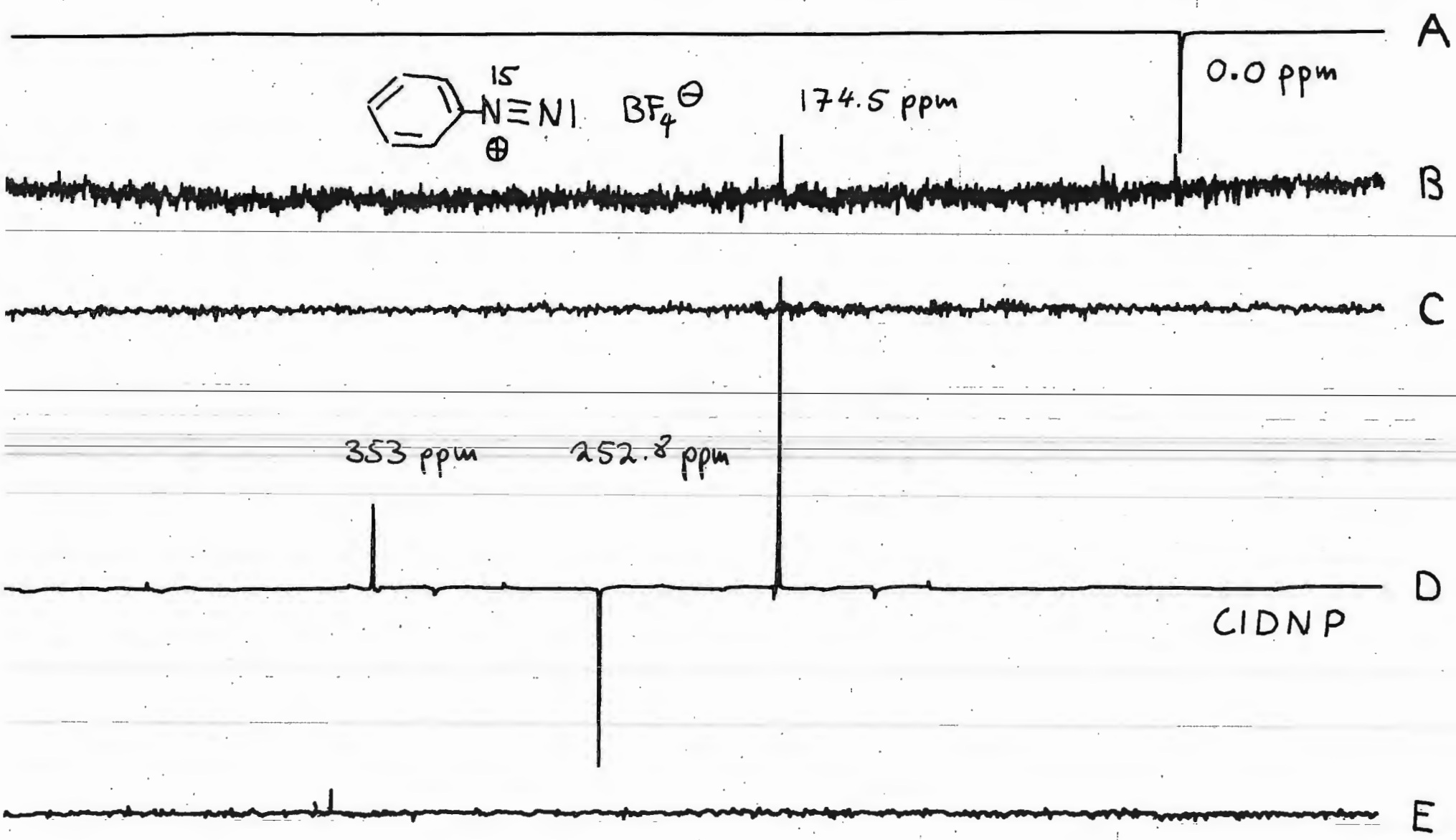
68.39 H_z
69.12 H_z

We believe that the J values are related to preferred conformations of the five articulated molecules. The solvent effect is perhaps also, since it is virtually absent in the rigid compound. We are exercising our imaginations in the preparation of a manuscript for the Australian Journal of Chemistry.

Yours sincerely,

Ian Rae

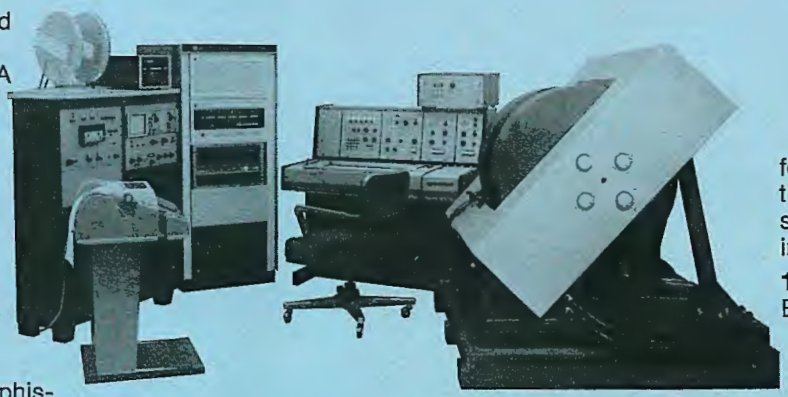
Ian Rae
Senior Lecturer in Chemistry



THIS IS A REPLACEMENT FOR P. 10, ISSUE #205.

New XL-100A Feature:

Single-Sideband Filter Cuts Data Acquisition Time in Half



Each time we introduced a new performance refinement for the XL-100A NMR Spectrometer, it's been a technological milestone in its own right. But when we added the latest — a single-sideband filter which eliminates negative frequency noise — something else happened. It combined with the unshakeable magnet stability and sophisticated interlocking electronics of the XL-100A to create a quantum jump in performance: the ability to obtain outstanding spectra from microgram samples.

We ran a 5-μg sample of gelsemine ($C_{20}H_{22}O_2N_2$) with the result you see here. We think the spectral quality speaks for itself.

Microgram sample capability, of course, is good news if you are involved in metabolite studies, biosynthetic research, flavors and fragrances — any field in which samples come from GLC or TLC, or are scarce for any other reason.

The features that form the backbone of the XL-100A's microsample capability include:

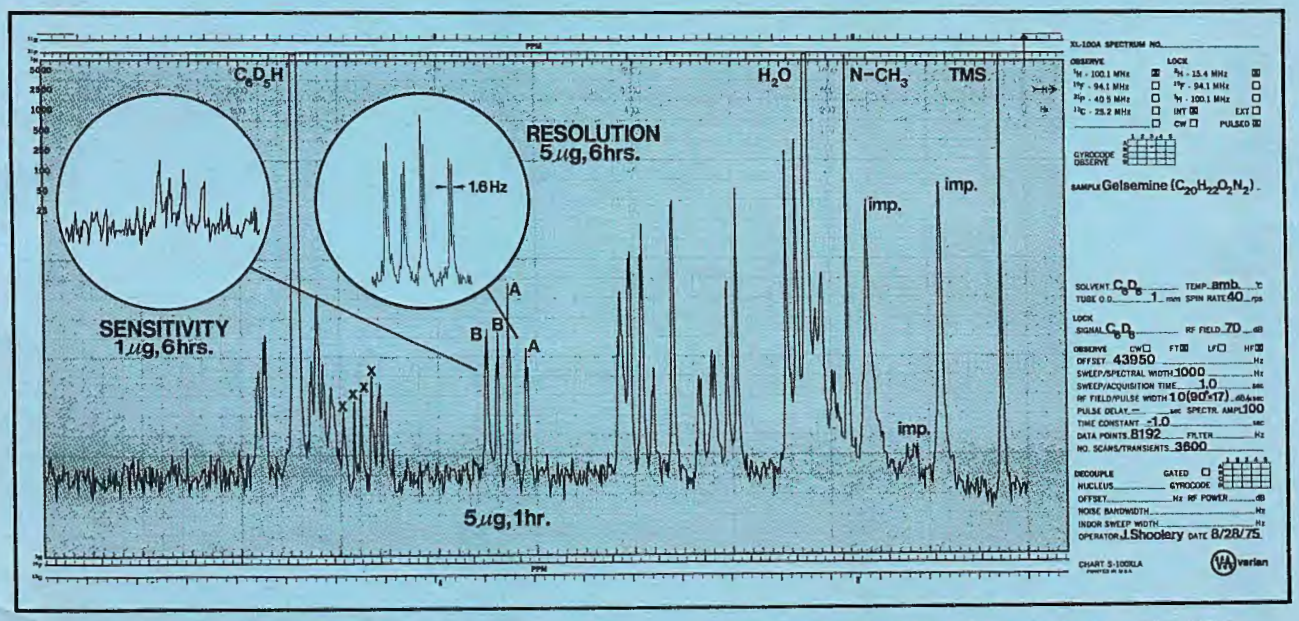
1-mm Microinsert
Ensures best possible signal from small samples by optimizing the rf coupling between the sample and the system's receiver coil.

Single-Sideband Filter
Cuts data acquisition time in half by eliminating the noise which normally folds into an FT spectrum.

FT Disk Accessory
Permits acquisition of high-resolution FT spectra by expanding the maximum data table capacity of the system to 32K words.

For further information write to:
Varian Instruments, 611 Hansen Way,
Box D-070, Palo Alto, CA 94303.

varian



We dissolved 5 μg of gelsemine in 5 μl of C_6D_6 and pulsed it at one-second intervals for one hour, using a tip angle of 53° (10 μsec pulse) and a 2,500-Hz spectral width. Note the clear ABX pattern from the three vinyl protons and the excellent resolution of the 1.6-Hz geminal coupling! The spectral excerpts show two six-hour runs of 5-μg and 1-μg samples to demonstrate how resolution or sensitivity can be further enhanced at the expense of time.

QUADRATURE DETECTION

Is Standard On JEOL's FX100 FT NMR and Built-In Digital* Phase Shifters Permit Precise Alignment

Digital Quadrature Detection (DQD) is the simultaneous detection of an FT NMR signal with phase sensitive detectors whose reference signals are in separate quadrants, digitally shifted 90° in their r.f. phases. This results in an increase in sensitivity by $\sqrt{2}$ (~ 40%), which means sample running times are cut in half on a routine basis over conventional techniques.

Because digital phase control is a basic part of the FX100, Digital Quadrature Detection (resulting in this higher FT NMR performance) is now possible.

Digital* Phase Shifters (DPS) make use of the very rapid rise times of an r.f. digital logic device, thus phase "jitter" is minimized, and phase accuracy and phase stability are optimized. The r.f. output frequency phase can be precisely set at 0°, 90°, 180° or 270° relative to the master oscillator. The result is a system that is virtually **free of imperfect phase angles** or phase drift.

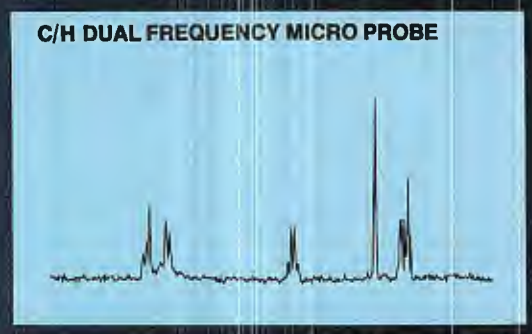
The exclusive JEOL digital phase shifting technique, in contrast to the more commonly used analog method, essentially **eliminates "ghosting"** or false images in the final spectrum — a problem which occurs all too frequently in analog systems.

In addition, we have located the DPS in the IF stage to allow all nuclei to be observed without supplementary phase shifters for each frequency.

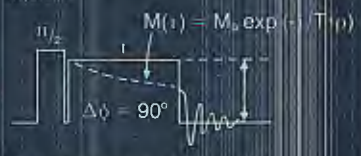
Similar capabilities are available on the FX60 Spectrometer as optional accessories.

Performance Characteristics:

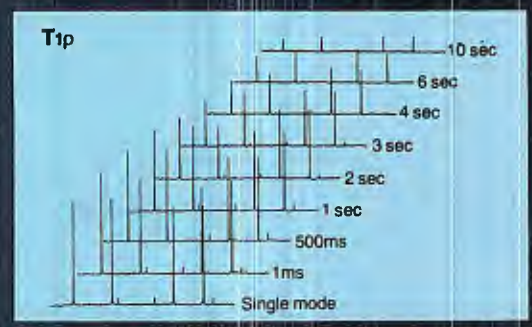
Micro $^{13}C/^{1}H$ Dual Frequency Probe
Sample: 10μg/20μl solution
Time: 5 minutes
Nucleus: Proton (^{1}H)
Sample shown is phenacetin.



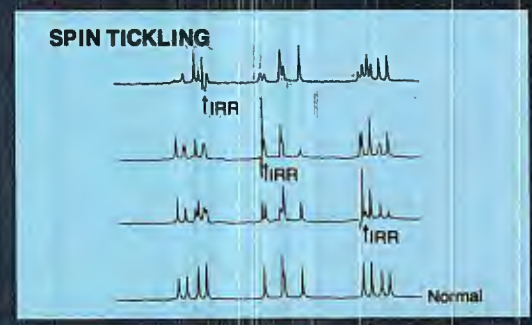
$T_{1\rho}$ is the transverse relaxation time in the rotating frame and is useful for studies of chemical dynamics in liquids.



Sample shown is chlorobutane.

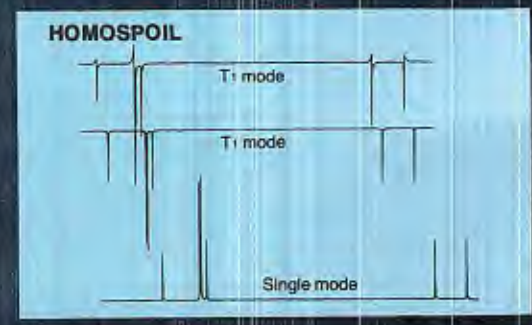


Spin tickling is a double resonance technique used in both homo and hetero nuclear experiments for the selective identification of spin-spin interactions.
Sample shown is dibromopropionic acid.



Homospoil is a technique for eliminating phase and intensity errors which can occur during T_1 measurements.

Sample shown is ethylbenzene (distortions are magnified to illustrate the technique).



For further information, call or write . . .

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

* Patent Pending