

Texas
A &
M
University
N - M - R
Newsletter

No. 206

November, 1975

B. L. Shapiro	
Editorial Matters.	1
C. H. DePuy	
A Long Range Deuterium Isotope Effect on a Europium Chemical Shift	3
S. N. Rosenthal and E. J. Fendler	
¹³ C NMR of Solubilizates in Dodecylammonium Propionate-CDCl ₃ Solutions	7
L. Müller, A. Kumar and R. R. Ernst	
2D Resolved ¹³ C-Spectroscopy; HR 220 For Sale	10
J. A. den Hollander	
CIDNP Evidence for "Hot Radicals"?	13
R. Nunnally and D. P. Hollis	
How to Save Your V 2708 Power Supply	16
D. L. Dalrymple and B. A. Jezl	
RACES C-13 Spectral Analysis Program	20
F. H. Köhler	
¹ H NMR Improvement for Paramagnetic Compounds	21
G.-J. Béné	
(1) Travaux Récents en Magnétisme Nucléaire: Déplacements de Fréquence dans des Systèmes de Spins Nucléaires; Polarisation Dynamique en Champ Magnétique Faible.	
(2) XIXeme Congrès du Groupement Ampère, Heidelberg, Germany, 27 September to 1 October 1976.	23

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

These restrictions apply equally to both the actual Newsletter participant-recipients and to all others who are allowed access to the Newsletter issues. Strict adherence to this policy is considered essential to the successful continuation of the Newsletter as an informal medium of exchange of NMR information.

WILMAD[®]

NMR CHART PAPER

WE STOCK ALL THE RECORDING CHARTS YOU NEED ...

- THE WORLD'S FINEST NMR SAMPLE TUBES • EQUIPMENT, GLASSWARE, AND SUPPLIES FOR SPECTROSCOPIC RESEARCH
- EVERYTHING BUT THE SPECTROMETER • SEND FOR OUR NEW FREE 100-PAGE GIANT CATALOG 875. • IF YOU HAVE ANY QUESTIONS, PLEASE CALL.

CHART PAPER

Finest grade NMR Chart Paper made to be used in every model spectrometer. All charts have been updated to coincide with the newest instrument techniques . . . Fourier Transformation, Hetero-Decoupling, and Time Averaging.

NOTE: All charts packaged 500 sheets to a box except roll charts or as otherwise noted.

CATALOG NUMBER	INSTRUMENT	TYPE	PRICE PER BOX			
			1-10 boxes	11-24 boxes	25-49 boxes	50 & over
VARIAN						
WCV-100 (S-100A)	HA-100, HA-100A, and D	Cal.	\$35.00	\$34.50	\$34.00	\$33.50
WCV-60 (S-60C)	A-60, A-60A and D	Cal.	35.00	34.50	34.00	33.50
WCV-60EL	HA-60EL and IL	Cal.	37.50	37.00	36.50	36.00
WCV-XL (XL-100)	XL-100 (Standard)	Cal.	35.00	34.50	34.00	33.50
WCV-XL-100FT	XL-100 (Fourier)	Cal.	37.50	37.00	36.50	36.00
WCV-220 (S-220)	HR-220	Cal.	37.50	37.00	36.50	36.00
WCV-56 (S-56A)	A-56/60	Cal.	37.50	37.00	36.50	36.00
WCV-360-K-10	EM-360	Cal.	28.00	27.50	27.00	26.50
WCV-20 (CFT-20)	CFT-20	Cal.	30.00	29.50	29.00	28.50
WCV-EM-300R (3E)	EM-300 (6 rolls/box)	Cal.	14.40	13.80	13.80	13.80
WCV-EM-300F (300X10)	EM-300 (flatbed)	Cal.	28.00	27.50	27.00	26.50
WCV-60T (S-60T)	T-60 (two color)	Cal.	20.00	19.50	19.00	18.50
WCV-60B	T-60 (one color)	Cal.	17.50	17.00	16.50	16.00
WCV-60U (S-60U)	T-60 (multi-nuclei)	Cal.	20.00	19.50	19.00	18.50
WCV-60TS	T-60 (no grid, delta)	Blank	16.00	15.50	15.00	14.50
WCV-BL	11" x 26"	Blank	16.00	15.50	15.00	14.50
WCV-60T-BL	8-1/2" x 11"	Blank	10.00	9.50	9.00	8.50
WCV-CFT-20K-11	11" x 16"	Blank	13.00	12.50	12.00	11.50
WCV-360BL	11" x 16"	Blank	13.00	12.50	12.00	11.50
JEOL						
WCJ-4HA	C-60H, 4H-100, (9.0 ppm)	Cal.	37.50	37.00	36.50	36.00
WCJ-4HB	C-60HL, MH-100, (9.0 ppm)	Cal.	37.50	37.00	36.50	36.00
WCJ-4HC	MH-100, PS-100 (10.8 ppm)	Cal.	37.50	37.00	36.50	36.00
WCJ-4HD	MH-100, PS-100 (9.0 ppm)	Cal.	37.50	37.00	36.50	36.00
WCJ-4HE	PFT-100 (standard)	Cal.	37.50	37.00	36.50	36.00
WCJ-PFT-100	PFT-100 (Fourier)	Cal.	37.50	37.00	36.50	36.00
WCJ-FX-60(FX-2)	FX-60	Cal.	37.50	37.00	36.50	36.00
WCJ-FX-60-BL	FX-60 (para only)	Blank	19.00	18.50	18.00	17.50
WCJ-PFT-100BL	11" x 17" (para only)	Blank	19.00	18.50	18.00	17.50
WCJ-BL	11" x 17"	Blank	15.00	14.50	14.00	13.50
BRUKER						
WCB-"UC"	HX-90, HFX-10	Cal.	40.00	39.50	39.00	38.50
WCB-BL	12-1/2" x 29"	Blank	17.00	16.50	16.00	15.50
WCB-WH-90	WH-90	Cal.	35.00	34.50	34.00	33.50
WCB-BX-FT	HX-270	Cal.	40.00	39.50	39.00	38.50
PERKIN-ELMER						
WCPE-2018	R-20, R-20A	Cal.	37.50	37.00	36.50	36.00
WCPE-2021 (WCPE-60)	R-20B	Cal.	37.50	37.00	36.50	36.00
WCPE-462-1075	R-12, R-12A (6 rolls/box)	Cal.	35.00	34.50	34.00	33.50
WCPE-435-0065	R-24 (roll) (6 rolls/box)	Cal.	35.00	34.50	34.00	33.50
WCPE-435-7204	R-24A (rect.) (100 sh./box)	Cal.	7.00	6.50	6.00	5.50
WCPE-441-1580	R-22 (rect.) (100 sh./box)	Cal.	8.00	7.50	7.00	6.50
WCPE-R-32	R-32 (roll) (6 rolls/box)	Cal.	35.00	34.50	34.00	33.50
WCPE-2018BL	11" x 22"	Blank	15.00	14.50	14.00	13.50
WCPE-2021BL	11" x 19"	Blank	15.00	14.50	14.00	13.50
WCPE-526-1102	R-26 (rect.) (100 sh./box)	Cal.	7.00	6.80	6.60	6.40
WCPE-435-9020BL	R-24A (rect.) (100 sh./box)	Blank	4.50	4.25	4.00	3.75
WCPE-441-1580BL	R-22 (rect.) (100 sh./box)	Blank	4.50	4.25	4.00	3.75
NMR SPECIALTIES						
WCN-60/100	A-60, HA-100, A-56/60	Cal.	35.00	34.50	34.00	33.50



WILMAD GLASS COMPANY, INC.

U.S. Route 40 & Oak Road • Buena, N.J. 08310, U.S.A. • (609) 697-3000 • TWX 510-687-8911

TAMU NMR NEWSLETTER - ADVERTISERS

Bruker Scientific, Inc. - see p. 9
 Electronic Navigation Industries - see p. 19
 JEOL Analytical Instruments, Inc. - see outside back cover and (i)
 Merck, Sharp & Dohme - see p. 15
 Nicolet Instrument Corporation - see pp. 5 and 6
 Varian Instrument Division - see inside back cover
 Wilmad Glass Co., Inc. - see inside front cover

TAMU NMR NEWSLETTER - SPONSORS

Abbott Laboratories
 Bruker Scientific, Inc.
 JEOL, Analytical Instruments, Inc.
 Dr. R. Kosfeld, Abt. Kernres.,
 Inst. f. Phys. Chem., TH Aachen (Germany)
 The Lilly Research Laboratories, Eli Lilly and Co.
 The Monsanto Company
 Nicolet Technology Corp., Palo Alto, CA
 (formerly Transform Technology, Inc.)
 Unilever Research
 Varian, Analytical Instrument Division

TAMU NMR NEWSLETTER - CONTRIBUTORS

The British Petroleum Company, Ltd. (England)
 Eastman Kodak Company
 E. I. DuPont DeNemours & Company
 The Perkin-Elmer Company
 Pfizer, Inc.
 The Procter & Gamble Co., Miami Valley Labs
 Shell Development Company
 Union Carbide Corporation

Deadline Dates: No. 207: 1 December 1975
 No. 208: 5 January 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. 207

Bene, G. J.23	Jezl, B. A.20
Dalrymple, D. L.20	Kohler, F. H.21
DePuy, C. H.	3	Kumar, A.10
Ernst, R. R.10	Muller, L.10
Fendler, E. J.	7	Nunnally, R.16
den Hollander, J. A.13	Rosenthal, S. N.	7
Hollis, D. P.16	Shapiro, B. L.	1

TEXAS A&M UNIVERSITY

COLLEGE OF SCIENCE

COLLEGE STATION, TEXAS 77843

Department of
CHEMISTRYEDITORIAL MATTERS

I would like to bring four matters, three of them connected with our constant economic worries, to the attention of all TAMU NMR Newsletter recipients:

1. Advertising Revenue: The Newsletter financial picture remains bleak indeed, at least in part because: a) no new advertising revenue has been generated, and b) because we have lost one of our monthly advertisers. Once again it seems appropriate that I ask all Newsletter recipients to explore with their own companies or with other companies with whom you do business, if there is any way we can engender some additional advertising revenue with them to keep the Newsletter alive.

Also, please let me request most earnestly once again that you find some way to let our advertisers know that their efforts are appreciated. While no one (at least not me!) expects you to buy a new costly spectrometer system every month or so, it would seem reasonable that you at least solicit literature and quotes, etc., from those vendors who support the Newsletter via advertising, Sponsorship and Contributorship. The company which terminated its advertising with the Newsletter did so not because of economic considerations, but understandably because they got little feedback from the public that their ads were being read. Your cooperation in this area is most important, indeed essential, in order to keep the Newsletter going.

2. Space Economies: Again, one of my periodic calls for scrupulous attention to the three-pages-per-contribution limit, and for space economies in general in the preparation of your Newsletter contributions.

In particular, the following policy with regard to Positions Available, Positions Wanted, Equipment for Sale, Meeting Notices, etc., will be in effect: all such notices (as well as any other, similar types) should be submitted in the briefest possible format, on plain paper (not letterhead) in order that such notices can be collected on one Newsletter page (if necessary, by retyping in our office). It is felt that significant savings of space can be effected in this way. In a given issue, if there are too few such notices to fill a page, we will put them at the bottom of other, partially filled, pages. As always, the above types of notices are "no credit" contributions.

3. Postage Rates and Mailing Class: Our already beleaguered finances will suffer another severe blow at the end of 1975 when the U.S. postal rates go up substantially again. This will make it even more difficult for us to return to First Class mailing rather than the present, much slower, Third Class mail, a change we would very much like to make. To return to the subject of section 1, above: if more advertising revenue could be raised, we would be able to more seriously consider providing the Newsletter recipients with the First Class mailing service that is so desirable. If any present or other financial benefactor of the Newsletter would like to step forward to aid in this return to better mailing service, it would be greatly appreciated and we will see that such help is appropriately acknowledged.

As a poor substitute (poor because it entails much more work for us), anyone who wishes to pay extra for First Class or Air Mail service is invited to send some extra funds directly to me. We will send your Newsletter copies by surface First Class mail or Air Mail, whichever you specify, until your little fund is used up, at which point we will notify you for replenishing, etc. For U.S. and Canadian subscribers, calculations indicate that the change from Third Class to First Class surface mail will cost approximately \$5.00 extra per year per subscription. For European and other overseas subscribers there are two potential options to the present Third Class mail: (i) First Class surface mail, estimated *extra* cost of approximately \$8.00 per year, although this amount may vary from one country to another; (ii) Air Mail/Printed Matter (really the best way, although very expensive), for which the estimated average *extra* cost would be \$15.00 per year. While we are not anxious to take on additional work of this kind, we recognize the desirability for you to receive the Newsletter within a reasonable period of time. Anyone who wishes to exercise this custom mailing service should only indicate your wishes to us and send a check made out and mailed to "Dr. B. L. Shapiro, Editor, TAMU NMR Newsletter", Department of Chemistry, Texas A&M University, College Station, TX 77843 U.S.A. Please note that checks should be in U.S. funds, and must accompany your request for First Class or Air Mail posting of your Newsletter issues.

4. Figures, Graphs, Spectral Traces, Etc., Appearing in the Newsletter: We are still suffering from several cases of more-and-less visible figures, graphs and spectral drawings in the Newsletter, arising from the poor quality of the originals sent to us. There are limits to the amount of improvement which even our talented printer can make when he is sent "originals" which are poor quality Xerox or other photocopies. Sometimes these copies are themselves made from spectral drawings where the contrast was poor, thus compounding the problem. Please! send us decent pictures, graphs, drawings, etc. - by "decent" we refer to contrast and not the artistic or aesthetic qualities. If necessary, please go over your spectral lines, etc., with a good dark black ink, or even a dark pencil. Contributions with drawings which do not meet minimal quality will be returned for improvement, no matter how close to the deadline date for a particular issue the contribution is received. Your cooperation will be gratefully appreciated.

Suggestions re any of the above, or indeed any other aspect of the Newsletter, are always appreciated.

B. L. Shapiro

B. L. Shapiro
(5 November 1975)

UNIVERSITY OF COLORADO

BOULDER, COLORADO 80302, U.S.A.

October 9, 1975

DEPARTMENT OF CHEMISTRY

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

Dear Barry:

We would like to point out a remarkable long-range deuterium isotope effect on a lanthanum induced chemical shift. In connection with some of our cyclopropane work we had occasion to prepare the four diastereomeric methyl ethers IT, IE, IIT and IIE (Fig. 1). The first pair of compounds are threo ethers and differ from one another only in the relative stereochemistry of the hydrogen and deuterium. The second pair are the erythro ethers and differ from one another in the same way. We found that not only does the shift reagent $\text{Eu}(\text{fod})_3$ separate the methoxyl methyl signal of the threo ether from that of the erythro ether, as expected, but that within each ether there is separation depending upon the stereochemistry of the deuterium atom!

If you make a space filling model of each of these diastereomers you see that in a favorable conformation the oxygen of the ether is in close proximity to the deuterium atom when the latter is threo(T) and farther away when it is erythro(E). Our tentative explanation for the observation is a steric one. It is well known that a C-D bond acts as if it were shorter than a C-H bond because of its smaller vibrational excursions. This could lead to a slightly larger complexation constant with the shift reagent for IT and IIT than for IE and IIE, and hence to a slightly larger chemical shift for the protons on the methoxyl group. We know the effect is not inductive because threo and erythro 3-deuterio-2-butyl methyl ether do not show separate methoxyl signals in the presence of $\text{Eu}(\text{fod})_3$, and in I and II the dideutereo analogs absorb at the same position as the T isomers and the undeuterated at the same place as the E isomers.

We are carrying out additional experiments to test this theory and to see if similar effects are found in the CMR spectra. As an alternative explanation we have been wondering if there is any way the effect could be due to some magnetic property of the deuterium,

perhaps transmitted via the europium.
 There is no effect on the methoxyl
 protons when we decouple the deuterium.
 Anyone have a suggestion?

Sincerely yours,

C. H. DePuy
 C. H. DePuy
 Professor of Chemistry

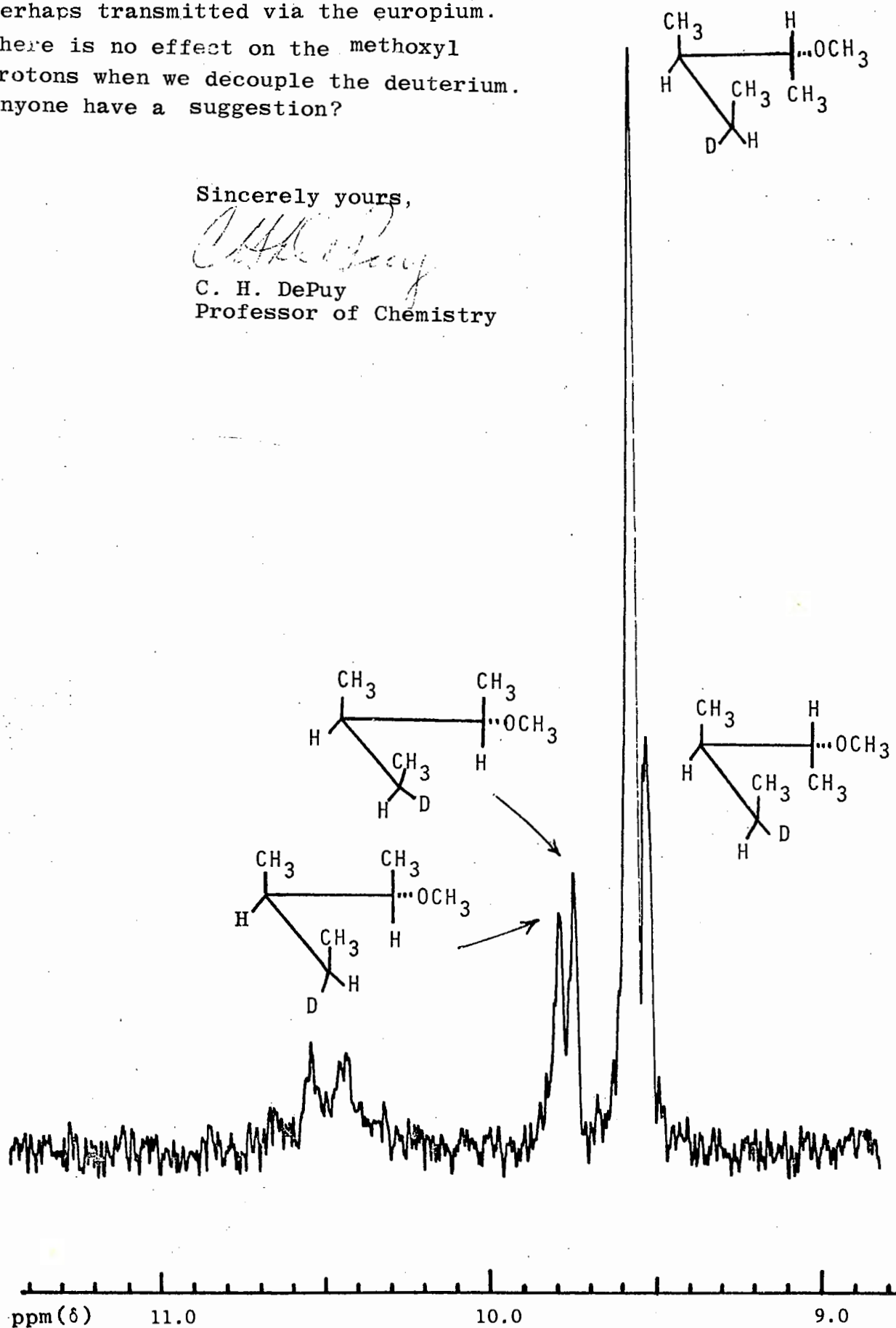


Fig. 1

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.

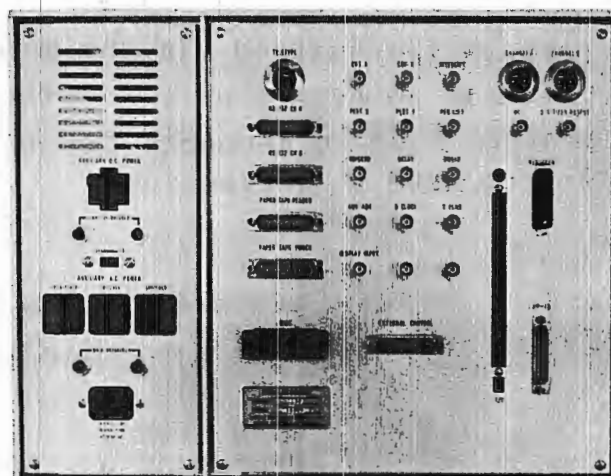
APPLICATION: NMR Spectroscopy

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-By³ 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-Leigh⁴ 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.

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

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

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

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

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

NIC



Check the New Nicolet 1180 laboratory data system

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.

For more details or to discuss your proposed NIC-1180 application please telephone or write.

**NICOLET
INSTRUMENT
CORPORATION**

5225 Verona Road
Madison, Wisconsin 53711
Telephone: 608/271-3333

NICOLET INSTRUMENT GmbH, Goerdeler Strasse 48,
D-605 Offenbach am Main, West Germany, 0611/852028,
Telex: 841/4185411

ALLAN CRAWFORD ASSOCIATES, LTD/LTEE Toronto,
416/678-1500; Ottawa, 613/829-9651; Calgary, 403/276-9658;
Halifax, 902/469-7865; Montreal, 514/670-1212; Vancouver,
604/980-4831

TEXAS A&M UNIVERSITY

COLLEGE OF SCIENCE

COLLEGE STATION, TEXAS 77843

October 24, 1975

Department of
CHEMISTRYDr. Bernard L. Shapiro
Department of Chemistry
Texas A&M University
College Station, Texas 77843Title: ^{13}C Nmr of Solubilizates in Dodecylammonium Propionate- CDCl_3 Solutions

Dear Barry:

We are continuing our investigations of surfactants in nonpolar media using ^1H and ^{13}C nmr. In an effort to verify and establish the validity of some of our earlier ^1H work on reversed micellar systems,¹ we decided to investigate solubilization behavior in dodecylammonium propionate (DAP)-deuteriochloroform solutions using ^{13}C nmr spectroscopy. Solubilize ^{13}C chemical shifts, at constant concentration, for dimethyl sulphoxide, imidazole, methanol, pyrazole and 2-pyridone have been determined as functions of DAP concentrations in CDCl_3 . Equilibrium or binding constants between solubilizates and micellar DAP have been obtained from these chemical shifts using the following equation:

$$\frac{1}{\delta - \delta_o} = \frac{1}{K_{\text{MS}} (\delta_{\text{MS}} - \delta_o)} + \frac{1}{[\text{M}]}$$

where δ is the observed chemical shift of the magnetically discrete solubilize carbons at different DAP concentrations, δ_o is the chemical shift of the uncomplexed or "free" substrate in the bulk solvent, δ_{MS} is the chemical shift for the DAP-"complexed" solubilize, K_{MS} is the solubilize binding constant and $[\text{M}]$ is the micelle concentration. Binding constants determined from ^{13}C nmr data are in fairly good agreement with the values previously determined using ^1H nmr. The results are summarized in the Table. In addition to using CDCl_3 as a solvent for these studies, we have begun to investigate solubilize behavior in DAP-benzene systems. These results, although not complete, also agree with earlier ^1H nmr studies.¹ Furthermore, we are in the process of examining the effect of varying the concentration of solubilizates on the chemical shifts of DAP at constant concentration. These results will be reported at a later time.


¹ O. A. Seoud, E. J. Fendler, and J. H. Fendler, J. C. S. Faraday Trans. 1, 70, 459 (1974).


Association of Solubilizates with DAP in Deuteriochloroform

Solubilizate ^a	Position	$K_{MS}, \text{ dm}^3 \text{ mol}^{-1} (\text{M}^{-1})$	
		^1H	^{13}C
DMSO	-CH ₃		1.0 ₀
Imidazole	4,5	28 ± 1	36.8
	2	17 ± 1	
Methanol	-CH ₃	13.4 ± 0.5	8.8 ₀
Pyrazole	4	34.5 ± 0.5	20.0
	3,5	34 ± 2	16.3
2-Pyridone	2		27.8
	3	15.5 ± 1	4.7 ₃
	4		
	5	12.5 ± 0.5	
	6		1.5 ₂

^a Concentration of solubilizates = 0.10 M.

Sincerely yours,

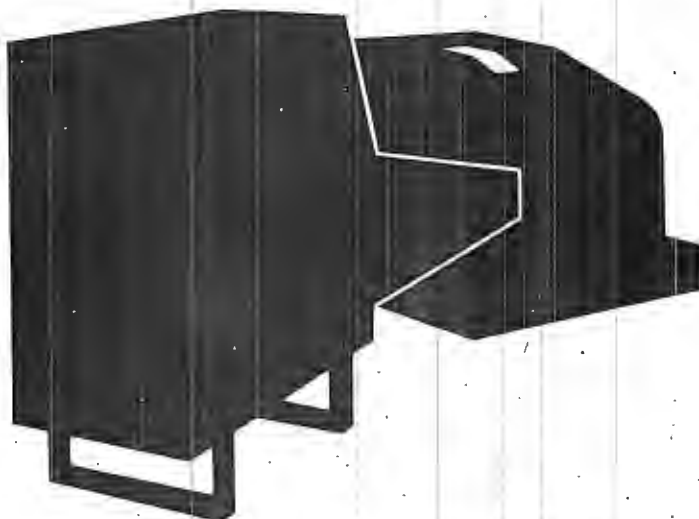

 Steven N. Rosenthal


 Eleanor J. Fendler



WFP 60

The ultimate in low-cost
FT NMR Spectroscopy...



- Full multinuclear capability
- High resolution magnet for proton FT
- 10 mm variable temp for C¹³
- Superior sensitivity

FOR DETAILS, PLEASE CONTACT YOUR NEAREST BRUKER REPRESENTATIVE.

USA

Bruker Scientific Inc.
One Westchester Plaza
Elmsford, N. Y. 10523
Tel. (914) 592-5470
Tlx. 13-1524

Bruker Magnetics Inc.
1 Vine Brook Park
Burlington, Mass. 01803
Tel. (617) 272-9250
Tlx. 94-9493

Bruker Research
1548 Page Mill Road
Palo Alto, Calif. 94305
Tel. (415) 493-3173
Tlx. 34-5533

CANADA

Bruker Spectrospin Ltd.
84 Orchard View Blvd., Suite 101
Toronto, Canada
Tel. (416) 486-7907
Tlx. 02-2771



EIDG. TECHNISCHE HOCHSCHULE
ZÜRICH

Laboratorium
für Physikalische Chemie

Prof. Dr. R. R. Ernst

CH-8006 Zürich, 18 October 1975
Universitätstrasse 22
Tel. (01) 32 62 11

Prof. B. L. Shapiro
Department of Chemistry
Texas A+M University

2D RESOLVED ^{13}C - SPECTROSCOPY

Dear Barry,

More and more ^{13}C - fans try to utilize nowadays the additional information contained in undecoupled spectra, very often with bad success due to the numerous overlapping multiplets for medium size or larger molecules. Off-resonance decoupling is one way out but not without considerable loss of information.

We would like to present here an intriguing idea to resolve overlapping multiplets: Apply a 90° pulse to your carbons (Fig.1), wait a bit, turn on the decoupler and record at the same time the FID. Repeat this experiment several times but with different waiting times. Perform a 2D-Fourier transformation on the obtained data set and plot the 2D set of Fourier coefficients in a suitable manner. The result will look like Fig. 2 (if you have done it properly). The 2D spectrum displays all multiplets resolved in two dimensions and the interpretation of the undecoupled spectrum is a matter of seconds. One can see that the decoupled spectrum is just the projection of the 2D plot onto the ω_2 -axis and the undecoupled spectrum is the projection onto the ω_1 -axis. Resolution is in the moment limited by the available computer memory.

Numerous modifications of the basic experiment are possible, utilizing the Overhauser effect, selecting one specific carbon and mapping its multiplet structure and many more. The computer requirements are not trivial, but instrument manufacturers will be happy to supply you with more memory or with a disk. We are convinced that this technique will find its application in the routine NMR laboratory whenever there is a need for undecoupled

Eidg. Technische Hochschule Zürich
Laboratorium für Physikalische Chemie

^{13}C spectra. The complete story will be published shortly.

Sincerely yours

Luciano Müller

Luciano Müller

Anil Kumar

Anil Kumar

Richard R. Ernst

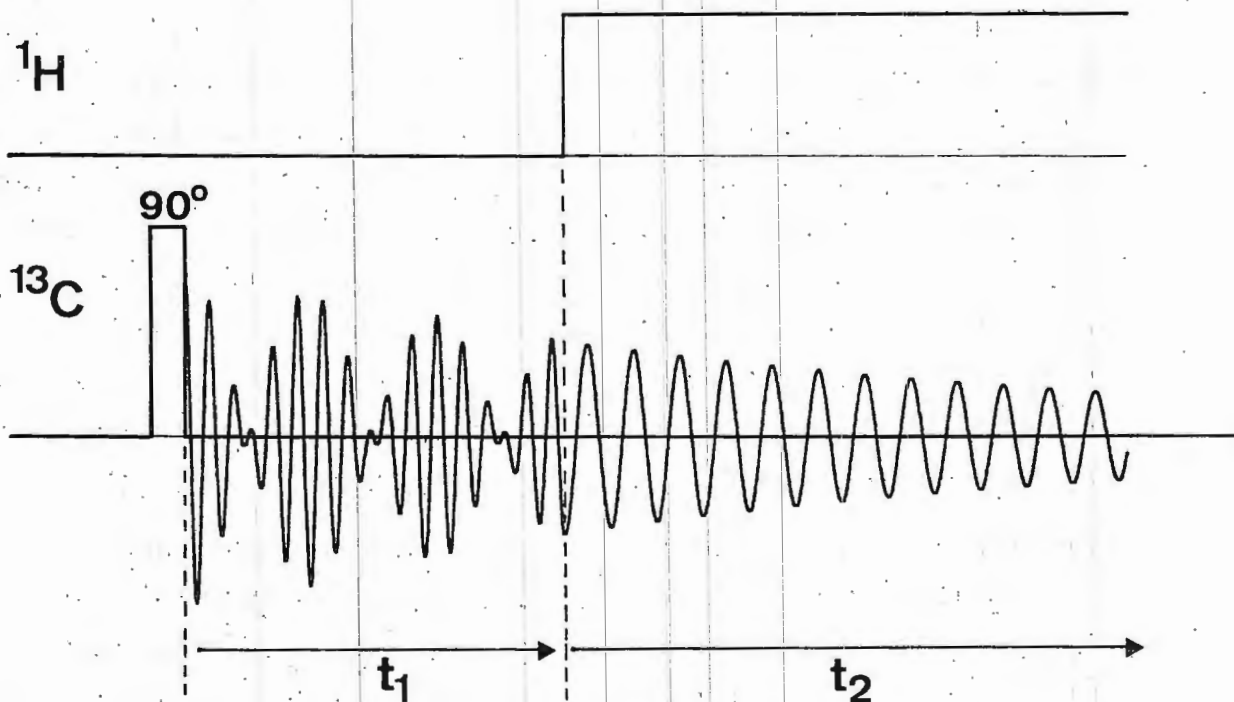
Richard R. Ernst

HR 220 FOR SALE

I would also like to draw the attention of your readers to the Varian HR 220 spectrometer which we have presently for sale. We will replace it by a new high performance spectrometer to fulfil the needs of the molecular biochemists. The performance of the HR 220 (purchase date 1969) is still within specification. It is a proton-only spectrometer without lock nor FT.

Richard R. Ernst

Fig. 1



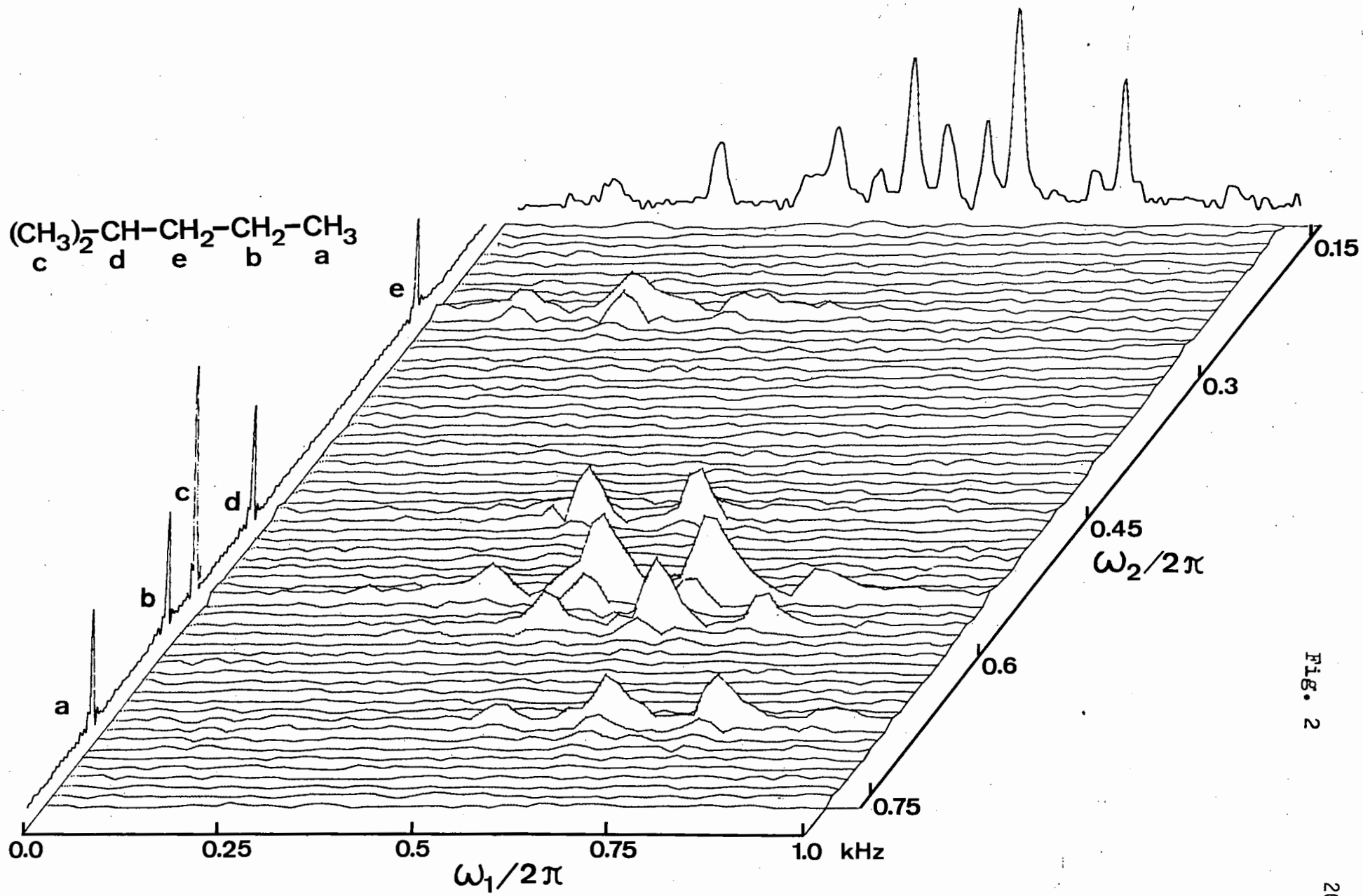


Fig. 2

Wassenaarseweg 76

Postbus 75

Telefoon 48333

toestel:

Afdeling:

Onderwerp:

LEIDEN, October 27, 1975

Prof. B.L. Shapiro

Department of Chemistry

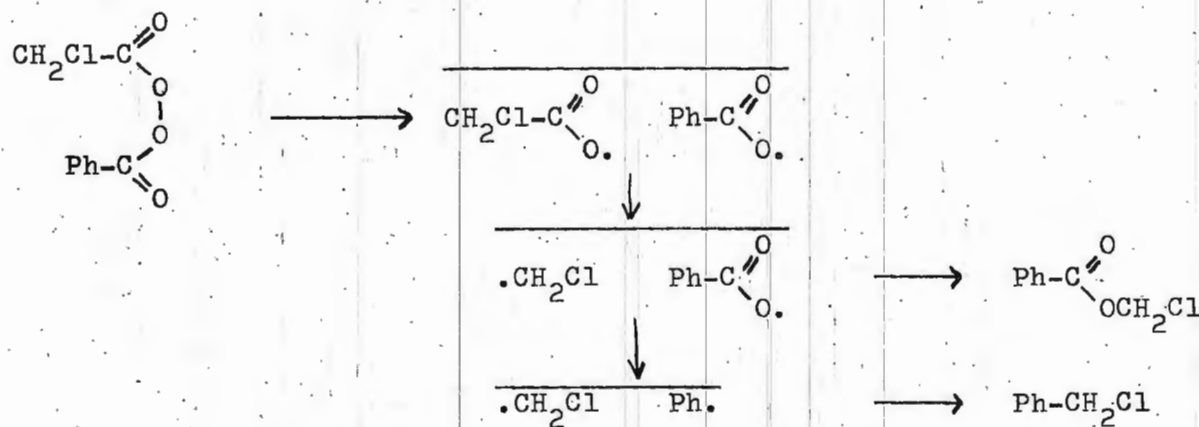
Texas A and M University

College Station, Texas 77843

Dear Dr. Shapiro,

CIDNP evidence for "hot radicals"?

During the thermal decomposition of chloroacetylbenzoylperoxide in solution several CIDNP signals are observed (Scheme).



SCHEME

Apart from the solvent dependent scavenging products the recombination products chloromethylbenzoate and benzylchloride both show CIDNP. For the methylene protons of the ester we observe a large emission signal, for the corresponding protons of benzylchloride a small emission. These observations can be explained similarly to the case of acetylbenzoylperoxide^{1,2}. Since the chloroacetoxyl radical lives too short to affect CIDNP in any of the products the emission observed for the ester is readily accounted for by considering recombination for a singlet generated chloromethyl/benzoyloxy radical pair (pair I): the g-factor of the benzoyloxy radical is larger than that of the chloromethyl radical, the hyperfine interactions of the protons of chloromethyl are negative.

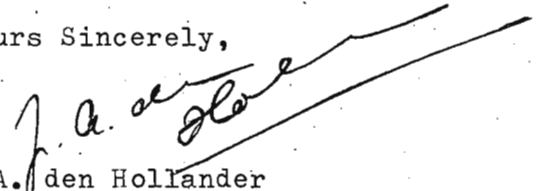
The emission for the methylene protons of benzylchloride is more tricky to explain, since the g-factor of the chloromethyl radical is considerable larger than that of the phenylradical: we would expect enhanced absorption for the

recombination product of a singlet generated chloromethyl/phenyl radical pair (pair II). The emission can only be explained by noting that the "memory effects" due to pair I on the recombination products of pair II overshadow the effect of S-T⁰ evolution in pair II itself^{3,4}. In any case, the observation of emission for the methylprotons of benzylchloride in the present system unambiguously demonstrates the intermediacy of the benzoyloxyradical.

The direct photolysis of this peroxide in solution gives a quite different result: again the emission for the methylene protons of the ester is observed, but for the benzylchloride we observe an enhanced absorption, much larger than the emission signal for the ester. In our opinion this result shows that at least for an important fraction pair I is bypassed and that the photolysis either leads in part directly to pair II or that the benzoyloxyradical lives much shorter in the photolysis experiment compared with the thermal decomposition ("hot radical"⁵). Unfortunately CIDNP is not able to tell something about the events on a very short timescale, and other experiments are needed to discriminate the two possibilities.

In order to preclude possible uncertainties with respect to precursor multiplicities we also performed a sensitized decomposition using benzophenone. As expected both the ester and the benzylchloride signals reverse compared with direct photolysis⁶; furthermore the ratio of the two signals changed somewhat. Apparently the benzoyloxyradical has now more chance to survive the initial photochemical processes, due to less excess energy compared with direct photolysis.

Yours Sincerely,


J.A. den Hollander

References

- 1) A.V. Kessenikh, A.V. Ignatenko, S.V. Rykov, and A.Ya. Shteinshneider, Org. Magn. Res., 5, 537(1973)
- 2) J.A. den Hollander, Chem. Comm., 352(1975)
- 3) R.E. Schwerzel, R.G. Lawler and G.T. Evans, Chem. Phys. Letters 29, 106(1974)
- 4) J.A. den Hollander, Chem. Phys., 10, 167(1975)
- 5) J.G. Calvert and J.N. Pitts, "Photochemistry", John Wiley, New York, 1966
- 6) R. Kaptein, J.A. den Hollander, D. Antheunis, and L.J. Oosterhoff, Chem. Comm., 1687(1970)

206-T5

574

MERCK ISOTOPES

We deliver

Stable Isotopes.
Deuterium.
Carbon-13.
Nitrogen-15.

We list over 500 compounds labelled with stable isotopes, and can custom synthesize hundreds more. WE DELIVER. Please request information.

U.S.A.

Merck & Co., Inc./Isotopes,
4545 Oleatha Ave., St. Louis,
Mo. 63166.

Tel: 314-353-7000
TWX: 910-761-0437

CANADA

Merck Sharp & Dohme Canada
Limited/Isotopes
P.O. Box 899, Pointe
Claire/Dorval, Quebec,
Canada H9R-4P7
Tel: 514-697-2823
TELEX: 05-821-533
TWX: 610-421-3617

MERCK & CO., Inc.



ISOTOPES

MERCK & CO., Inc./Isotopes
4545 Oleatha Ave., St. Louis, MO. 63166
Please send me your literature. I am particularly interested in:

Name _____
Title _____
Company _____

THE JOHNS HOPKINS UNIVERSITY
SCHOOL OF MEDICINE
725 N. WOLFE STREET · BALTIMORE, MARYLAND 21205

DEPARTMENT OF PHYSIOLOGICAL CHEMISTRY

TELEPHONE 955-5000
AREA CODE 301

HOW TO SAVE YOUR V 2708 POWER SUPPLY

The windings of the Varian V 2708 power supply transformer can be damaged over a very short span of time by arcing of worn carbon brushes in the moving brush carriage commutators. This can occur even when routine maintenance and examinations have been made. The six year old power supply in this laboratory incurred such a failure six months ago. One of the poles of the variable transformer was severely pitted to a depth of 2-3 mm. on six adjacent windings (figure 1) and to a lesser depth on several other windings in the same region. A second pole had some slight pitting and the third was undamaged.

All information available and obtainable from the manufacturer indicates that the pole windings are not to be sanded or dressed in any manner. It is also clear that any damage to the windings is considered to be irreparable. The following repair was attempted since there appeared to be nothing to lose and a substantial gain to be made (a new power supply costs approximately \$11,000.00).

Disconnect the 480 V power line to the power supply. A rubber sanding block (Sand-Pak Sanding Block-3M Company) was used in all sanding and dressing steps. This sanding block has a width to match the width of the primary windings without the need of any alteration. Course aluminum oxide sandpaper (60 grit) is used to smooth the deeply pitted areas until no black arcing marks remain. The important factor is to sand in such a fashion to form a gently sloping, dished-out surface on the transformer pole. (This is also termed "feathering out"). A sufficient overlap of unaffected windings must be included to provide a very gradual sloping (figure 2).

The next step requires the use of 200 grit aluminum oxide paper to smooth the winding surfaces and remove any roughness made by the course paper. This step should be carried out until all winding surfaces look and feel smooth and flat. Finally, extensive dressing of the windings is achieved with 400 grit aluminum oxide paper. The result should be a highly polished smooth surface.

During the various sanding steps care should be taken to minimize the spread of copper filings and dust to the front part of the power supply. When all surfaces are prepared, the rear of the power supply and the poles are carefully cleaned with a brush and vacuum cleaner to remove all traces of copper. It is now necessary to undercut the insulation material between the windings that have been sanded down. A small cutting tool can be made from square bar stock steel (3/8" square) using a grinder to form a cutting edge (figure 3). It is possible that other suitable tools may serve equally well without having to go to this trouble. A caveat; it is very easy to slip from the insulator onto the winding surface while trying to undercut it. This will necessitate a re-dressing of the surface. The insulator is undercut to a depth of ~2mm. below the adjacent winding surfaces. The rear of the power supply is again cleaned carefully with a brush and a vacuum to remove the insulator material.

In some areas of the windings where the pitting was severe, the heat generated was sufficient to damage the insulator material. If during the undercutting step large amounts of insulator crumble off, a good quality epoxy can effect a patch. The epoxy should be applied with precautions to limit any of the cement getting onto the winding surfaces. Sand off after curing if any overlap is suspected. This can also be undercut after the epoxy has cured.

New carbon brushes are installed in the commutators and prepared according to the method outlined in the V 2708 manual. Next, a careful check of the electronics section of the unit is made.

In our unit a capacitor (C101-A, 120 μ F) was found to have leaked extensively around its base and all three capacitors of the C101 series (C101-A, B, C) were replaced.

Since the above repairs, the V 2708 power supply reported herein has been in continuous operation for six months. No further problems have occurred and no extra-ordinary wear of the brushes has been observed on the armature which was severely damaged. This armature has shown a tendency to develop a fine carbon coating over a period of several months in the area that was originally damaged. This is easily removed with a few passes of 400 grit aluminum oxide sandpaper over the affected area.

Sincerely,

Ray Nunnally

Ray D. Nunnally

Donald P. Hollis

Don Hollis

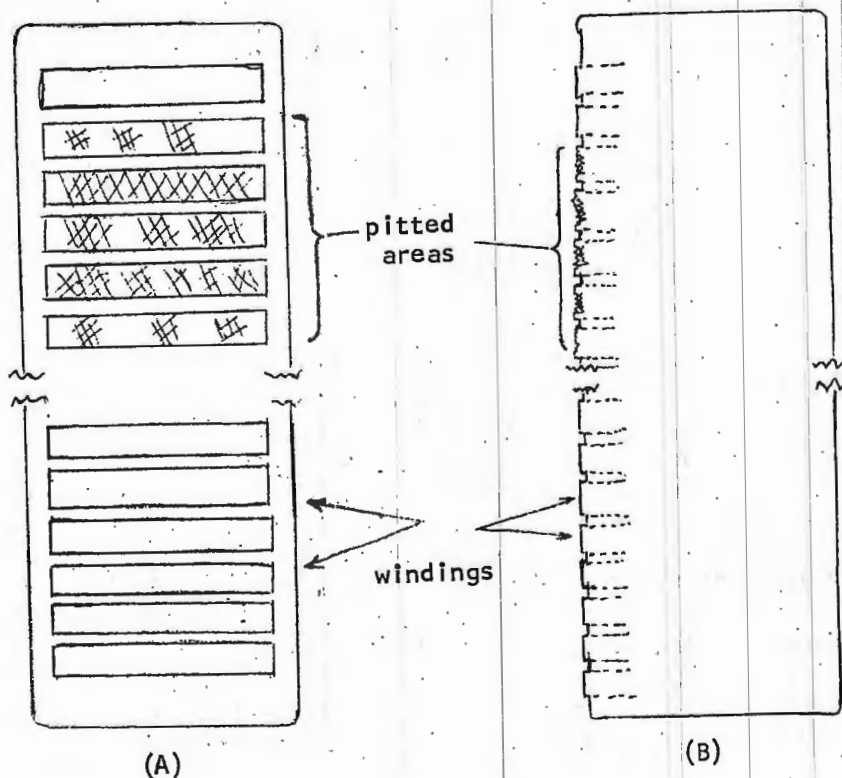


Figure 1. Diagram showing front (A) and side (B) views of damaged V 2708 transformer pole.

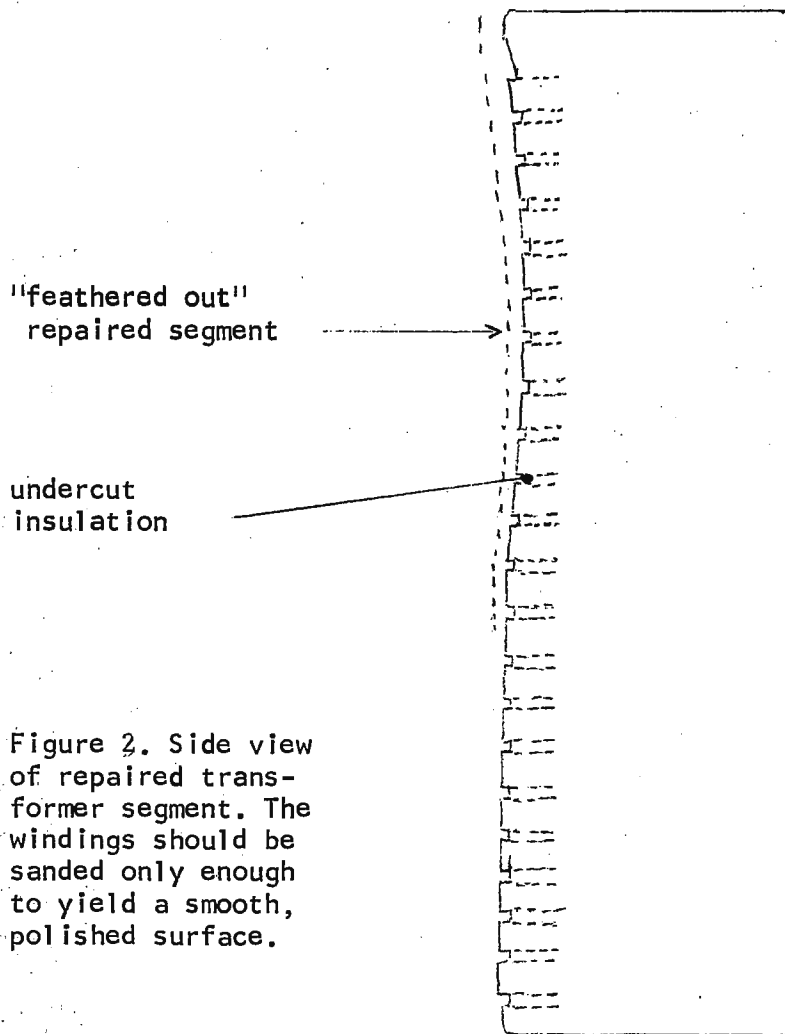


Figure 2. Side view of repaired transformer segment. The windings should be sanded only enough to yield a smooth, polished surface.

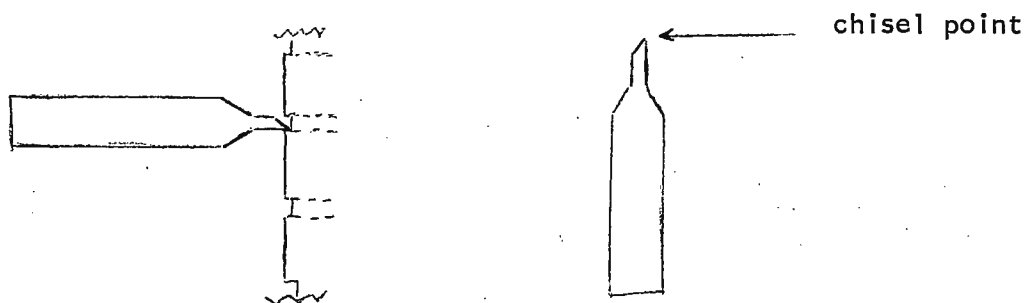


Figure 3. Diagram shows cutting tool and the method for undercutting insulation between sanded down windings. The tool is carefully drawn across the insulation in a horizontal direction.

ENI

The world's leader in solid state rf power amplifiers

Once upon a time if you wanted broadband RF power, you had to settle for bulky tube-type power amplifiers. No more. Because ENI has developed a full line of all-solid-state

Class A power amplifiers, covering the frequency spectrum of 10 kHz to 560 MHz, with power outputs ranging from 300 milliwatts to over 1000 watts. And there's more to come.

Driven by any signal generator, frequency synthesizer or sweeper, ENI's compact portable amplifiers, like the ones shown below, are versatile sources of power for general laboratory work, RFI/EMI testing, signal distribution, RF transmission, laser modulation, data transmission, NMR, ENDOR, ultrasonics and more.

Completely broadband and untuned, our highly linear units will amplify inputs of AM, FM, SSB, TV and pulse modulations with minimum

distortion. Although all power amplifiers deliver their rated power output to a matched load, only ENI power amplifiers will deliver their rated power to any load regardless of match.

We also designed our amplifiers to be unconditionally stable and fail-safe—you need never fear damage or oscillation due to severe load conditions (including open or short circuit loads).

ENI instrumentation amplifiers come complete with an integral AC power supply and an RF output meter. Ruggedized amplifiers capable of operating under severe environmental conditions are available.

For a **complete** catalog of power amplifiers and multicouplers, write: ENI 3000, Winton Road South, Rochester, New York 14623. Call 716-473-6900. TELEX 97-8283 ENI-ROC.



40 WATT/ MODEL 240L

- 20KHz to 10MHz coverage
- More than 40w linear power output
- Up to 150w CW & pulse output
- Works into any load impedance
- Metered output

Extraordinary performance in a wide range of transducer drive applications. Deliver up to 150w into any load regardless of its impedance. Compatible with all signal and function generators, the 240L is a high quality laboratory instrument for ultrasonics, biological research & electro-optic modulation.

100 WATT/ MODEL 3100L

- 250 KHz to 105MHz coverage
- More than 100w linear output
- Up to 180w CW & pulse
- Works into any load
- Unconditionally stable

Designed to replace bulkier and less efficient tube type amplifiers, the Model 3100L will provide reliable and maintenance free operation. NMR, ENDOR, ultrasonics and laser modulation are just a few of the applications for this versatile source of RF energy.

20 WATT/ MODEL 420L

- 150KHz to 250MHz coverage
- 20 Watts power output
- Low noise figure
- 45dB \pm 1.5dB gain
- Class A linearity

The widest band solid state power amplifier available at its 20w power level, the ENI 420L is a truly state-of-the-art instrument. As a drive source for high resolution acousto-optic modulators and deflectors the Model 420L is invaluable. Its Class A linearity will amplify AM, FM, TV and pulse signals with minimum distortion.

.3 WATT/ MODEL 500L

- Flat 27dB gain 2MHz to 500 MHz
- 1.7MHz to 560MHz usable coverage
- Thin film construction
- 8dB noise figure
- Failsafe

This compact unit can deliver more than 300 milliwatts from 1.7MHz to 560MHz at low distortion. A thin film microelectronic circuit is the heart of this general utility laboratory amplifier. Extremely wide band response at a very modest price.

UNIVERSITY OF DELAWARE
NEWARK, DELAWARE
19711

DEPARTMENT OF CHEMISTRY

October 14, 1975

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

Dear Professor Shapiro:

Title: RACES C-13 Spectral Analysis Program

Further development and testing of our computer program for Retrieval and Assignment of Chemical Environments and Shifts ((Anal. Chem., 47, 203 (1975)) have led us to the point of being able to offer it to others for use. The program at present is only compatible with disk-equipped Nicolet 1080 and related processors, and considering other efforts in this area, we have shelved temporarily the task of developing a Fortran version of the program. Anyone interested in the system is invited to write one of us (D.L.D.).

One feature of the system which we would commend to anyone developing such a program is the direct substructure-chemical shift correlation used. Such correlations are familiar to all spectroscopists, and with the explosive growth of ^{13}C literature, computerization of this method of spectral interpretation becomes at once more practical and more necessary. Even with only slightly over 2000 spectra as a data base, we find the program quite powerful in painlessly assigning spectra. With the ongoing expansion of the size and breadth of the spectral files, this ability is certain to increase.

Sincerely,



David L. Dalrymple
University of Delaware

Barbara A. Jezl
University of Cincinnati

ag

P.S. Please credit this contribution to the account of Joe Noggle.

ANORGANISCH-CHEMISCHES INSTITUT
DER
TECHNISCHEN UNIVERSITÄT MÜNCHEN

8 MÜNCHEN 2, den 24. 10 1975

Arcisstraße 21, Postfach-Nr. 20 24 20
Ruf-Nr. (089) 2105/330/331/332 (Prof. Fischer)
333 (Prof. Fritz)
308 (Prof. Schmidbaur)

Prof. B.L. Shapiro
Department of Chemistry
Texas A & M University
College Station, Texas 77 843
U.S.A.

^1H nmr improvement for paramagnetic compounds.

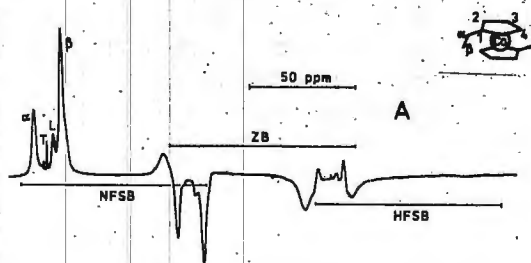
Dear Professor Shapiro,

modern nmr spectrometers have certainly not been designed in close contact with people doing nmr work on paramagnetic molecules; several of their requirements are not met.

One big problem is the interference of at least three spectra. This arises from the fact that signals are usually received by means of sideband stabilisation and that high rf power is needed to detect broad paramagnetic signals. For a consequence a non optimum modulation index produces not only the desired low field sideband spectrum (NFSB in fig. A) but also a centreband (ZB) and high field (HFSB) spectrum. In the case of bisethylated cobaltocene presented in fig. A protons 2-5 in NFSB are severely disturbed by protons α and β in ZB.

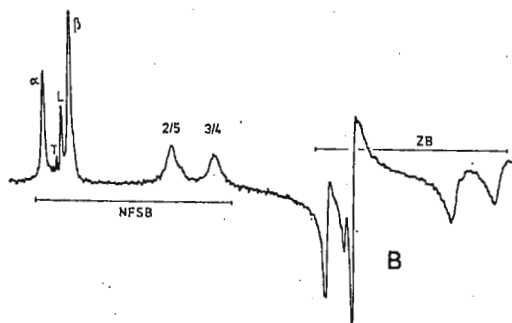
Fig. A. ^1H nmr of
 $(\text{C}_2\text{H}_5\text{C}_5\text{H}_4)_2\text{Co}$.

L = benzene
T = temperature
standard



One way to overcome this problem is numerical spectrum deconvolution as proposed by Pearson and Walter (J. Magn. Res., 16 [1974]348). TAMUNN readers without computing facilities might be interested in the approach we used with our JEOL C 60 HL. The fixed frequency unit (4 kHz) was replaced by a Princeton Applied Research HR 8 amplifier tunable from 1.5 to 1.5×10^5 Hz. The result for $(\text{C}_2\text{H}_5\text{C}_5\text{H}_4)_2\text{Co}$, the spectrum appearing as it should, is shown in fig. B, the modulation frequency being 8 kHz.

Fig. B. As fig. A,
modulation frequency
8 kHz



It is evident that the method is generally applicable and of particular value when recording spectra at different temperatures. Details are given in Z. Naturforsch., 30b[1975]649.

Please credit this contribution to Prof. H.P. Fritz

sincerely yours

(Frank H. Köhler)

CONTINUED FROM P. 23

2. Meetings du Groupement Ampère

Du 27 septembre au 1er octobre 1976, se tiendra à Heidelberg le XIXème Congrès Ampère.

Le programme couvrira les aspects théoriques et expérimentaux de la résonance magnétique ainsi que la spectroscopie de radio-fréquence en Physique, Chimie-Physique et Biophysique.

Avec mes sentiments très cordiaux.

Professeur G.-J. Béné



UNIVERSITÉ DE GENÈVE
SECTION DE PHYSIQUE
TÉLÉPHONE (022) 21 93 55

V/RÉF.

N/RÉF. GB/cc

DÉPARTEMENT DE PHYSIQUE DE LA MATIÈRE CONDENSÉE

32, boulevard d'Yvoy CH-1211 GENÈVE 4

Prof. B.L. SHAPIRO
Dept. of Chemistry
Texas A&M University
College of Science

College Station, Texas 77843
U.S.A.

Genève, le 20 octobre 1975

Cher Professeur Shapiro,

Merci de votre lettre de rappel du 9 octobre dernier.

1. Travaux récents en magnétisme nucléaire

Deux communications à la session d'automne de la Société Suisse de Physique à Aarau (3-4 oct. 1975).

"Déplacements de fréquence dans des systèmes de spins nucléaires"

On examine l'influence d'une excitation RF non résonnante sur un système de noyaux atomiques, lorsque la fréquence d'excitation est de l'ordre de grandeur ou plus petite que la fréquence de Larmor des noyaux étudiés. Les résultats obtenus pour de faibles amplitudes du champ RF ($H_1 \leq 20$ mg) sont en très bon accord avec la solution exacte de l'équation de Schrödinger appliquée au cas du champ tournant : On obtient le déplacement dû au champ alternatif par simple superposition des contributions des deux champs tournants composants. Les effets non linéaires apparaissent pour de plus grandes amplitudes de H_1 (> 50 mg).

"Polarisation dynamique en champ magnétique faible"

La polarisation dynamique protonique obtenue dans les liquides peu visqueux contenant un radical stable nitroxyde dissous est étudiée théoriquement et expérimentalement.

L'effet observé dans différentes solutions organiques prouve l'existence d'un couplage dipolaire entre le proton du solvant et l'électron du radical. Diverses causes influençant l'effet de polarisation, telles que les impuretés paramagnétiques, la largeur des raies électroniques, l'amplitude du champ faible polarisant ou la température sont passées en revue.

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- μ g 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 micro-sample 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.

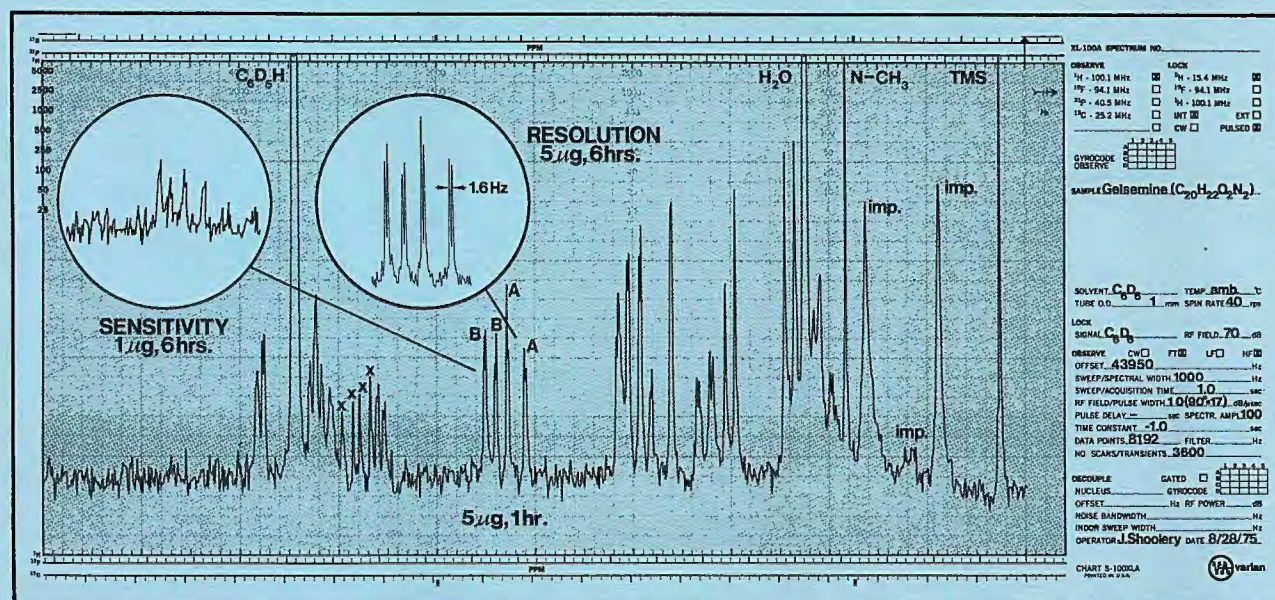
Single-Sideband Filter

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.



We dissolved 5 μ g of gelsemine in 5 μ l of C_6D_6 and pulsed it at one-second intervals for one hour, using a tip angle of 53° (10 μ sec 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- μ g and 1- μ g 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 ¹³C/¹H Dual Frequency Probe

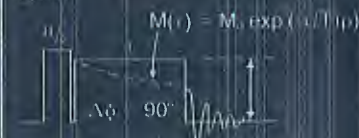
Sample: 10 µg/20 µl solution

Time: 5 minutes

Nucleus: Proton (¹H)

Sample shown is phenacetin

T_1 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.

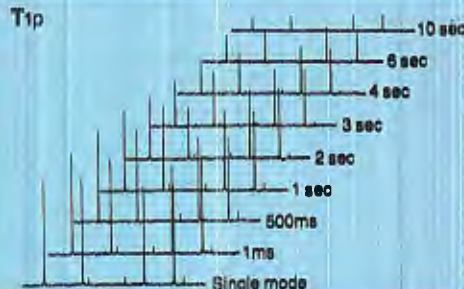
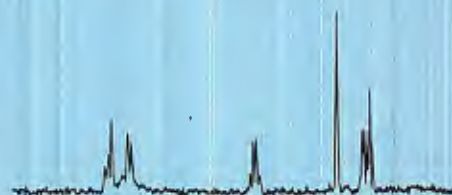
Sample shown is dibromopropionic acid

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)

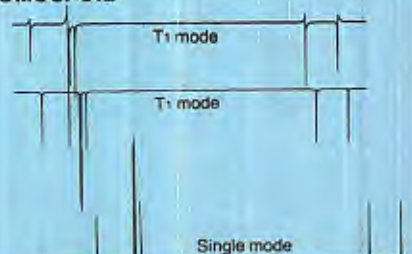
C/H DUAL FREQUENCY MICRO PROBE



SPIN TICKLING



HOMOSPOIL



For further information, call or write . . .

* Patent Pending

JEOL
Analytical Instruments, Inc.

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