

History of NMR: Request for Information and Anecdotes	Becker, E. D.	2
Electronic Substituent Effects on $^2\Delta$ Isotope Shifts of 4-Substituted Benzanilides	Morales-Ríos, M. S., Zepeda, L. G., and Joseph-Nathan, P.	5
^{13}C Chemical Shift Tensors from Carbon-13 Depleted Compounds	Maliniak, A., Greenbaum, G., Poupko, R., Zimmermann, H., and Luz, Z.	9
25th Southeastern Magnetic Resonance Conference	Brey, W. S.	10
AM-DISCO	Janssen, J. W. G., and van Os, J. W. M.	13
Conformational Interchange in Dicyclohexanodiethyleneglycol by ^{13}C NMR	Buchanan, G. W.	14
An Unusual Velocity Calibration Device	Altobelli, S., and Fukushima, E.	17
Nitrogen Chemical Shift Assignments for D-Fructose Oxime	Snyder, J. R.	18
Applications of Pulsed Field Gradients in High-Resolution NMR Spectroscopy	Reid, D. G., MacLachlan, L. K., Hubbard, J. A., Doddrell, D. M., and Brereton, I. M.	21
Adiabatic Pulses on a GBS2	Hajek, M., and Herynek, V.	25
Equipment Available	Bigler, B.	26
Structural Assignment of a Regioisomer of C_{60}H_4	Henderson, C. C., Cahill, P. A., and Assink, R. A.	29
Proton Exchange - Selective Pulses	Krawanja, L., and Sterk, H.	33
Flat Baselines in 1D and Baseplanes in 2D Spectra	Brownlee, B., and Sadek, M.	34

Continued on page 52

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 open 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.

TABLE 1 DEUTERATED SOLVENTS

Cat. No.	Description	Formula	Min. Isot. (% D)	Density (g/ml)	MP (°C)	BP (°C)	$-X_v \times 10^5$ @ (°C)
D-11	Acetone-d ₆	CD ₃ COCD ₃	99.8%	1.12	7	56	0.551 (32)
D-120	Acetone-d ₆ + 1% TMS		99.8%				
D-13	Acetone-d ₆	CD ₃ COCD ₃	99.8%	0.81	54	56	0.460 (20)
D-121	Acetone-d ₆ + 1% TMS		99.8%				
D-129	Need Deuterated Solvents? WILMAD HAS YOUR SOLUTION!						
D-14	Cost-conscious quality NMR solvents offered by Wilmad, such as CDCl ₃ , are frequently priced lower than more traditional sources. Included in this offering are the most common solvents, like Acetone-d ₆ , Benzene-d ₆ , D ₂ O, and DMSO-d ₆ , as well as some of the most unusual solvents for specialty applications, like 1,1,2,2-Tetrachloroethane-d ₂ , Octane-d ₈ , and Trifluoroacetic Acid-d.						0.543 (20)
D-21							0.611
D-122							
D-130							
D-28	Chloroform-d	CDCl ₃	99.8%	1.50	-64	62	0.740 (20)
D-31	Chloroform-d + 1% TMS		99.8%				

VARIAN
BOX/500 SHEETS

Cat. No.	Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	XL-100, HA-100, HA-100D	Gridded-Two Color
WCV-60 (S-60C)	11" X 26"	A-60, A-60C, A-60D	Gridded-Two Color
WCV-XL (X-100)	11" X 26"	XL-100, XL-100C, XL-100D	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100, XL-100C, XL-100D	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
Use Plotter Paper and Pens? WILMAD HAS YOUR CHART!			
We provide the largest variety of paper and pens for NMR recorders or plotters available anywhere. Included in these listing are the newest spectrometers from Varian, Bruker, (and IBM), General Electric and JEOL, as well as the latest models, such as the Hewlett Packard 7475A, 7550A, and Thinkjet 2225A, Zeta 8 or 8A, and Western Graphtec 4730 plotters and printers.			
WCV-20 (CFT-20)	11" X 16 3/4"	CFT-20, FT-80, FT-80A	Gridded-Two Color
WCV-11" (CFT-11)	11" X 17"	All Models 11"	Blank

Searching for the Unusual Requirement?
WILMAD HAS YOUR ANSWER!

The most comprehensive offering of "widgets, gadgets and specials" for NMR spectroscopy, including:

Spatula for 5mm NMR Tubes
Three types of Valve NMR Tubes
(including the new J. Young Valve Tube)
Solvent Jet NMR Tube Cleaners
pH Electrode for 5mm NMR Tubes

Taperlok® NMR Tubes
A multitude of Coaxial Inserts
Alumina NMR Tube for Si-29 Studies
Ultra-thin wall NMR Tubes
Throwaway "THRIFT" and "ECONOMY" NMR Tubes

Serving the Spectroscopic Aftermarket



WILMAD GLASS COMPANY
 Route 40 and Oak Road • Buena, NJ 08310 U.S.A.
 609-697-3000 • TWX 510-687-8911
 FAX 609-697-0536

TEXAS A&M NMR NEWSLETTER**NO. 420, SEPTEMBER 1993****AUTHOR INDEX**

Altobelli, S. 17	Doddrell, D. M. 21	Landucci, L. 37	Ralph, J. 37, 38
Assink, R. A. 29	Fukushima, E. 17	Landucci, W. 37	Ralph, S. 37
Becker, E. D. 2	Greenbaum, G. 9	Laukien, F. H. 41	Reid, D. G. 21
Bigler, B. 26	Hajek, M. 25	Luz, Z. 9	Sadek, M. 34
Brereton, I. M. 21	Henderson, C. C. 29	Maas, W. E. 41	Schaefer, T. 51
Brey, W. S. 10	Herynek, V. 25	MacLachlan, L. K. 21	Snyder, J. R. 18
Brownlee, B. 34	Hubbard, J. A. 21	Maliniak, A. 9	Sterk, H. 33
Buchanan, G. W. 14	Janssen, J. W. G. 13	Morales-Ríos, M. S. 5	Zepeda, L. G. 5
Cahill, P. A. 29	Joseph-Nathan, P. 5	van Os, J. W. M. 13	Zimmermann, H. 9
Cory, D. G. 41	Krawanja, I. 33	Poupko, R. 9	

TEXAS A&M NMR NEWSLETTER**NO. 420, SEPTEMBER 1993****ADVERTISER INDEX**

Acorn NMR. 19	JEOL outside back cover
American Microwave Technology. 15	Polyflon Company 39
Bruker Instruments, Inc. 11	Precision Electronic Glass, Inc. 47
Cambridge Isotope Laboratories, Inc. inside back cover	Tecmag, Inc. 35
Chemagnetics 7	TRIPOS Associates, Inc. 27
Hitachi Instruments, Inc. 23	Varian 3
Isotec, Inc. 31	Wilmad Glass Company, Inc. inside front cover
International Equipment Trading, Ltd. 45	

SPONSORS OF THE TAMU NMR NEWSLETTER

Abbott Laboratories	Merck Research Laboratories
American Microwave Technology	Millipore Corporation, Waters Chromatography Division
ATI Instruments	The Monsanto Company
Bruker Instruments, Inc.	Nalorac Cryogenics Corporation
Burroughs Wellcome Co.	Norell, Inc.
Cambridge Isotope Laboratories, Inc.	Oxford Instruments
Chemagnetics	Petroleum Recovery Institute
Cryomagnet Systems, Inc.	The Procter & Gamble Company, Miami Valley Labs
The Dow Chemical Company	Programmed Test Sources, Inc.
The Du Pont Merck Pharmaceutical Company	Shell Development Company
Eastman Kodak Company	Tecmag
E. I. du Pont de Nemours & Company	TRIPOS Associates/New Methods Research, Inc.
Hitachi Instruments, Inc.	Unilever Research
Isotec, Inc.	Union Carbide Corporation
JEOL (U.S.A.) Inc., Analytical Instruments Division	The Upjohn Company
The Lilly Research Laboratories, Eli Lilly & Company	Varian, Analytical Instrument Division

FORTHCOMING NMR MEETINGS

- 1993 FACSS Meeting**, Detroit, Michigan, **October 17-22, 1993**; Contact: H. N. Cheng, Hercules, Inc., Research Center, 500 Hercules Road, Wilmington, DE 19808; Phone: (302) 995-3505; Fax: (302) 995-4117. See TAMU NMR Newsletter 411, 10.
- 1993 Pacific Conference on Chemistry and Spectroscopy**, (Joint Meeting with the American Chemical Society's Western Regional Meeting), Pasadena, California, **October 19-23, 1993**; Contact: Ms. B. Belmont, Pacific Conf., 14934 S. Figueroa St., Gardena, CA 90248; Phone: (310) 538-9709.
- 1993 CABM Fall Symposium on "Macromolecular Recognition"**, Piscataway, NJ, **October 21-22, 1993**; Program Organizer: G. T. Montelione; Contact: Linda Van Derveer, Symposium Coordinator, Center for Advanced Biotechnology and Medicine, 679 Hoes Lane, Piscataway, NJ 08854; Phone: (908) 235-5309; Fax: (908) 235-4850. See Newsletter 419, 30.
- 35th ENC (Experimental NMR Conference)**, Asilomar Conference Center, Pacific Grove, California, **April 10 - 15, 1994**; Contact: ENC, 815 Don Gaspar, Santa Fe, NM 87501; (505) 989-4573; Fax: (505) 989-1073.
- 12th International Meeting on NMR Spectroscopy**, Sponsored by the Royal Society of Chemistry, Manchester, England, **July 2 - 7, 1995** [sic]; Contact: Dr. J. F. Gibson or Ms. G. B. Howlett - See TAMU NMR Newsletter 415, 5; Phone: (44-71) 437-8656; Fax: (44-71) 437-8883.
- ISMAR 1995**, Sydney, NSW, Australia, **July 16-21, 1995** [sic]; Contact: Dr. Wm. A. Bubbs, Secretary, Univ. of Sydney, Dept. of Biochemistry, Sydney, NSW 2006, Australia. See TAMU NMR Newsletter 419, 26.

Additional listings of meetings, etc., are invited.

ENCYCLOPEDIA OF NUCLEAR MAGNETIC RESONANCE

Reply to: Building 5, Room 124
National Institutes of Health
Bethesda, MD 20892

Phone: 301 496-1024
Fax: 301 496-0825
Internet: tbecker@sunder.niddk.nih.gov

Editors-in-Chief
D M Grant
R K Harris

Section Editors
E D Becker
S I Chan
J W Emsley
T C Farrar
B C Gerstein
I R Young

*International
Advisory Board*
I Ando
E R Andrew
A Bax
R Blinc
R R Ernst
J Frahm
R Freeman
M Goldman
H Günther
H S Gutowsky
P C Lauterbur
A Pines
G K Radda
J D Roberts
J N Shoolery
C P Slichter
H W Spiess
J S Waugh
B Wruckmeyer
K Wüthrich

July 26, 1993
(received 8/2/93)

Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dear Barry:

History of NMR: Request for Information and Anecdotes

Readers of the *Newsletter* may be interested to know that an *Encyclopedia of NMR* is being developed for publication in 1995, the 50th anniversary of the first successful observations of NMR in bulk materials. Although the *Encyclopedia* will consist predominantly of technical articles arranged in typical alphabetical fashion, one volume will be devoted to the history of NMR. We plan to include a number of short, largely autobiographical sketches from leading figures in NMR and an overall account of the development of NMR, from its "pre-history" in molecular beams to contemporary topics.

As editor of this historical volume, I am concerned that we tell the story of NMR as objectively and completely as possible. I would be interested in receiving suggestions of topics that might not occur to the Editorial Board and anecdotes that will spice up the historical account. Since space will be limited, I cannot promise that any specific item will be included, but I surely would like to have as much information as possible to draw from.

Please contact me with any interesting items. My address, phone and fax numbers are given above.

Sincerely,



Edwin D. Becker
Section Editor



JOHN WILEY
& SONS

- Chichester
- New York
- Brisbane
- Toronto
- Singapore

Acquire Solid State Triple Resonance Results



These solid state triple resonance REDOR results on isotopically enriched glycine were obtained on a three channel 400 MHz system equipped with a REDOR probe. The spectra show an increasing number of rotor periods from left to right. The results with and without nitrogen pulses are interleaved.

Ease-of-use in the most advanced experiments

Now the UNITYplus™ NMR platform provides a unique level of hardware and software flexibility. You can obtain excellent results in the most advanced experiments, such as REDOR, without system modification. The software includes the required tools for precise, flexible control of the UNITYplus NMR system; and the clean, stable rf provides the highest caliber data.

Varian offers solid state triple resonance capabilities with a REDOR probe and a three channel UNITY® or UNITYplus NMR system. For more information, contact the Varian office near you.

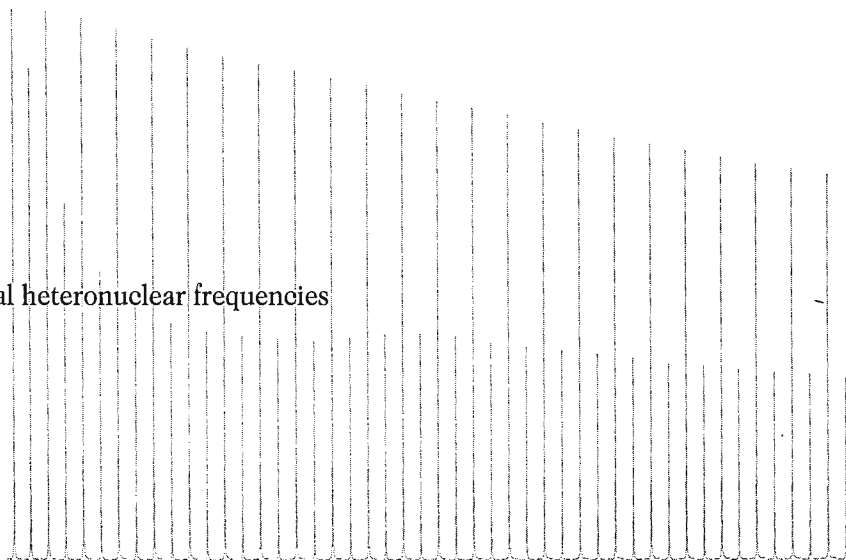
Varian Associates 3120 Hansen Way, Bldg. 4, Palo Alto, CA 94304-1030, U.S.A. Tel: 1-800-356-4437 • Varian International AG Kollerstrasse 38, CH-6303, Zug, Switzerland Tel: (42) 44 88 44 • Varian GmbH Alsfelderstrasse 6, D-6100 Darmstadt, Germany Tel: (0 61 51) 70 30 • Varian Instruments Ltd. 3rd Matsuda Bldg., 2-2-6 Ohkubo-Shinjuku, Tokyo, Japan Tel: (3) 3204-1211

varian 

Solid State Triple Resonance Experiments with Standard Hardware

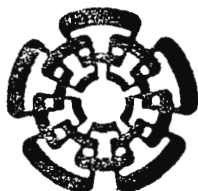
Probes

- Excellent sensitivity
- Variety of field strengths available
- Triple tuned for proton and two additional heteronuclear frequencies
- Robust high-power decoupling



Spectrometer

- Choose frequency and power ranges suitable for your applications on each rf channel
- Match rf pulse timing to spinning speed with rotor synchronization
- Switch between liquids, solids, and microimaging applications in minutes
- Move modules between UNITY*plus* spectrometers as needed



CENTRO DE INVESTIGACION Y DE ESTUDIOS AVANZADOS DEL I.P.N.

APARTADO POSTAL 14-740

MEXICO 07000, D.F.

July 20, 1993

(received 7/26/93)

Dr. Bernard L. Shapiro
 Editor/Publisher
 TAMU NMR Newsletter
 966 Elsinore Ct.
 Palo Alto, CA 94303 USA

Electronic substituent effects on $^2\Delta$ isotope shifts of
 4-substituted benzanilides

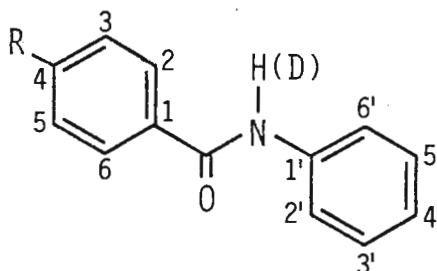
Dear Professor Shapiro:

We have been studying a series of para-substituted benzanilides in terms of theoretical interest concerning the substituent influence on isotope shifts transmitted across the D-labelled nitrogen. In these series of compounds a trend towards larger $^2\Delta$ isotope shift with increase of the electron-attracting character of the substituent is clearly evident at the carbonyl carbon $^2\Delta_{C=O(ND)}$ and at C-1' of the anilinio ring $^2\Delta_{C-1'(ND)}$ (see Table 1). This behavior differs from the finding in acetanilides substituted at the para position in the anilinio ring¹, in which the size of the two $^2\Delta$ isotope shifts owing to the neighboring carbons to the NH(D) site vary in opposite direction. This means that an acceptor substituent induces larger $^2\Delta$ shifts at C_{Ar} and smaller shifts at C=O. The change in the response of the two two-bond isotope shifts in these systems can be related to the corresponding changes in the contribution of mesomeric structures induced by the electronic effects of the substituent. In fact, an electron-withdrawing substituent at the para position in the benzoyl ring of benzanilides is expected to cause an increase of the mesomeric interaction between the nitrogen atom lone pair and the carbonyl group, with the consequent increase of the C-N amide bond order, whereas the same electron-withdrawing group at the para position in the anilinio ring, such as in acetanilides¹, is expected to cause a competitive delocalization of the nitrogen lone pair in the anilinio ring, which in turn reduces the C-N amide bond order. This reasoning can explain why for an amide carbonyl group the large $^2\Delta_{C=O(ND)}$ isotope effect is not always related to a large $^2\Delta_{C_{Ar}(ND)}$ ².

From the above qualitative discussion it might be expected that the two $^2\Delta$ isotope shifts in benzanilides are mutually interdependent. This interdependence is expected to occur via a cross-conjugated pathway arising from the D-labelled nitrogen, and thus, the observed magnitudes of the $^2\Delta$ shifts at the C=O and C-1' could be the result of a balance between them, rather than a proportionality. Consequently, the influence of the substituent on the two two-bond isotope shifts observed at the carbons sharing a D-labelled nitrogen of the type $C(sp^2)-NH(D)-C(sp^2)$ could be better described by a single correlation embracing both $^2\Delta$ isotope shifts. In fact, linear relationships exist between the average values of $^2\Delta_{C=O}$ and $^2\Delta_{C-1'}$ with either $\delta^1H(NH)$ (Eq. 1, $r = 0.993$) or with ^{15}N SCS (Eq. 2, $r = 0.998$). This behavior suggests that $^2\Delta$ isotope shifts could be used as an independent tool to prove the substituent perturbation of the π -electron structure in conjugated systems.

Equation	y	m	x	b	r
1	${}^2\Delta(\text{C=O} + \text{C-1}')/2\text{ppm}$	12.15	$\delta^1\text{H}(\text{NH})\text{ppm}$	-34.53	0.993
2	${}^2\Delta(\text{C=O} + \text{C-1}')/2\text{ppm}$	1.38	${}^{15}\text{N SCS ppm}$	90.60	0.998

Table 1. Deuterium isotope shifts (in ppb)^a and NMR parameters (in ppm) of 4-R-benzanilides.



R	${}^2\Delta(\text{C=O})$	${}^2\Delta(\text{C-1}')$	$\delta^1\text{H}(\text{NH})^b$	${}^{15}\text{N SCS}^c$
NO_2	88.1	99.4	10.59	2.3
CO_2Me	85.3	100.1	10.48	-
Br	84.8	98.1	10.35	-
Cl	84.4	98.9	10.35	0.7
F	83.1	98.6	10.29	-
H	82.4	98.3	10.29	0.0
Me	81.4	97.7	10.20	-0.9
OMe	80.0	96.5	10.11	-1.8
NMe_2	76.9	94.1	9.90	-3.6

^a0.6 M in DMSO-d_6 ; negative sign: deshielding; ^b0.6 M in DMSO-d_6 ; ^cIn DMSO-d_6 ; values calculated from Ref. 3; a positive value indicates a downfield shift from R=H.


REFERENCES

1. M.S. Morales-Ríos and P. Joseph-Nathan, *Magn. Reson. Chem.* **29**, 49 (1991); M.S. Morales-Ríos, P. Joseph-Nathan, B. Wrackmeyer and Ě. Kupčė, *Magn. Reson. Chem.* **31**, 238 (1993).
2. P.E. Hansen, A. Kolonicny and A. Lycka, *Magn. Reson. Chem.* **30**, 786 (1992).
3. H. Suezawa, T. Yuzuri, M. Hirota, Y. Ito and Y. Hamada, *Bull. Chem. Soc. Jpn.* **63**, 328 (1990).

Sincerely yours,


Martha S. Morales-Ríos


L. Gerardo Zepeda


Pedro Joseph-Nathan

What's the difference between the new CMX spectrometer and the competition?



We took the complicated and made it simple.

Computer System

- Sun™ SPARCstation™ 2GX workstations
- X-Windows™ software on the spectrometer
- High speed communications for "real time" display

Advanced Console Design

- Full *channel modularity*
- Full channel expandability
- Easy customer access

RF Design

- High IF design provides clean RF
- Ultra fast amplitude, phase, and frequency control
- Direct digital synthesis

Digital Design

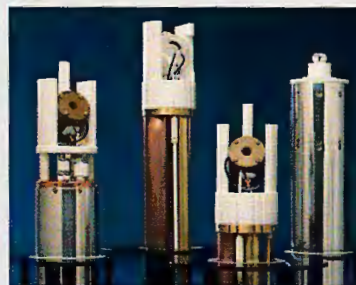
- High-speed 100 MHz pulse programmer on each channel
- Complete pulse program flexibility
- Multiple expansion capability

Solids Probes System

- PENCIL™ spinning system for high stability MAS
- Constant spinning speed from - 150°C to + 250°C
- COAX design affords superior tuning and RF capability for multiple resonance experiments
- High-stability, microprocessor-controlled, speed controller

Ergonomics

- Superior comfort, functionality and increased work space for optimum productivity



Dedicated to Design Excellence

Chemagnetics



United States

2555 Midpoint Drive
Fort Collins, Colorado 80525
(303) 484-0428
1 (800) 4 OTSUKA

France

"Le Copernic"
Parc d'Innovation
67400 Strasbourg-Illkirch
(33) 88 66 82 00
FAX (33) 88 66 82 04

United Kingdom

Claro Court Business Centre
Claro Road
Harrogate, HG1 4BA United Kingdom
0423 531 645
FAX 0423 531 647

Sun, the Sun logo, and SunWindows are trademarks or registered trademarks of Sun Microsystems, Inc.

PENCIL is a trademark of Chemagnetics, Inc.

X-Windows System is a trademark of the Massachusetts Institute of Technology

All SPARC trademarks, including the SCD compliant logo, are trademarks or registered trademarks of SPARC International, Inc. SPARCstation is licensed exclusively to Sun Microsystems, Inc.

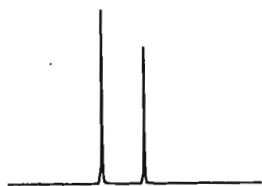
Variable Amplitude Cross Polarization - The Latest Technique from Chemagnetics

The introduction of the Variable Amplitude Cross Polarization (VACP) technique by Professor Steve Smith at the Yale University at the 1993 ENC may completely change the approach to optimization of solid state NMR experiments. This technique produces high quality solid state CP/MAS data, even on samples where the match condition may be impossible to optimize, such as weakly coupled systems. This opens up great potential for effective quantitation and comparison of data from different samples.

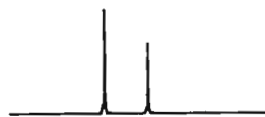
The results shown below obtained at Chemagnetics combine the unique design of the high-speed CMX linear amplitude modulator, amplifier technology, and the ease-of-use, high-speed double resonance Pencil™ spinning probe to demonstrate the power of this technique.

This power to implement new and future techniques lies at your fingertips when you invest in the CMX design philosophy. While others attempt to emulate its flexibility and modularity, the CMX continues to evolve into the most effective, versatile NMR spectrometer available.

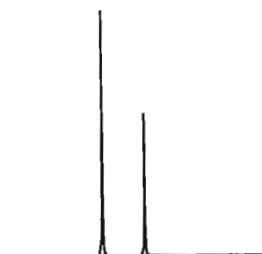
Call to find out more about state of the art performance with the Chemagnetics CMX, the revolutionary and evolutionary NMR system.



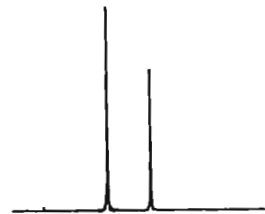
Variable amplitude cross polarization (VACP)
under -2.6 KHz mismatch condition



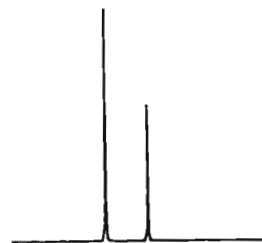
Deliberate "mismatch" of -1.9 KHz



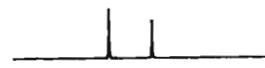
VACP under normal match condition



Normal "ideal" matched conditions



VACP under +2.0 KHz mismatch condition



Deliberate "mismatch" of +1.4 KHz

Chemagnetics would like to thank Professor Steve Smith for suggestion of this work and useful discussions during its implementation.



מחלקה לפיזיקה כימית
Chemical Physics Department

מכון ויצמן
למדע

76100 רחובות
08 34 טלפון
08 466966 פקס המכון
344123 פקס המחלקה
381300 טלקס
@WEIZMANN ביטנט

Weizmann Institute
Of Science

Rehovot 76100, Israel
Phone 972 8 34
Institute Fax 972 8 466966
Direct to Dept. Fax 972 8 344123
Telex 381300
Bitnet @WEIZMANN

August 3, 1993 (received 8/11/93)

Dr. Bernard L. Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303.

Carbon-13 Chemical Shift Tensors from Carbon-13 Depleted Compounds

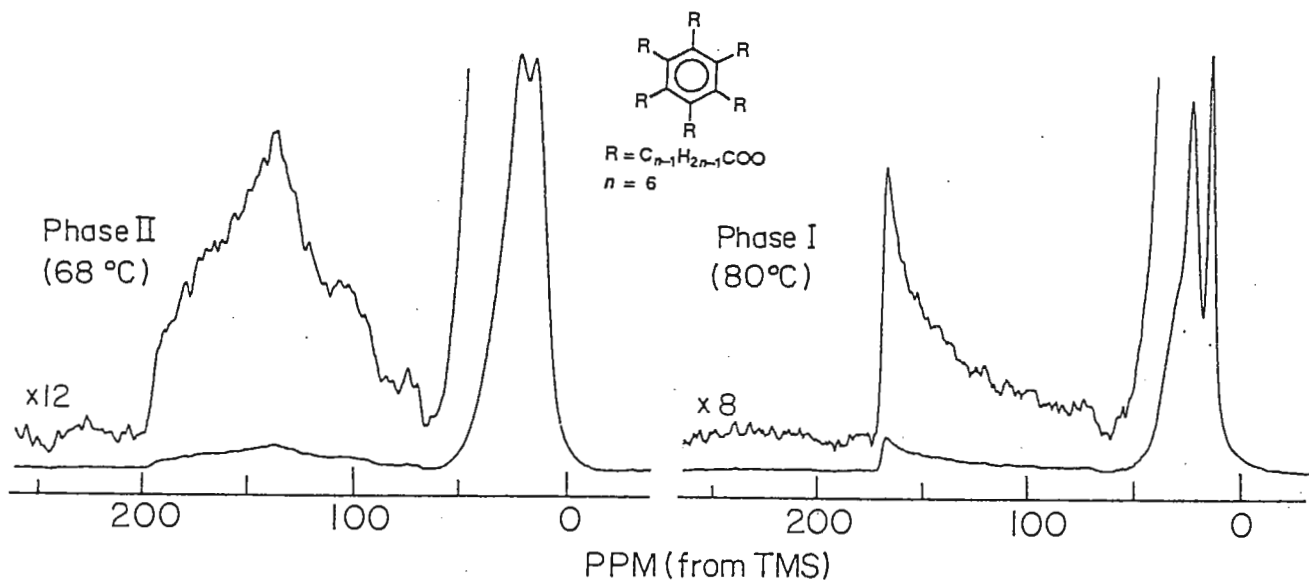
Dear Barry,

Carbon-13 chemical shift tensors provide information on the dynamic state of the molecules in the solid state. Aromatic sites are usually difficult to label by chemical methods and one then needs to record the signal using compounds of natural isotopic abundance. Beside the long averaging time that such experiments require there is often a difficulty due to overlap between signals from other carbons having similar chemical shifts values. Specific examples are the hexaalkanoyloxy derivatives of benzene, BHAn, which in the solid state exhibit several different phases.¹ To check whether these solid-solid transitions involve the setting-in of whole-molecule reorientation we were interested to record the aromatic carbon-13 signal from the benzene ring in the various solid phases. This signal falls however in the same range as that of the carboxylic carbon. To eliminate the signal of the latter we prepared the homologue BHA6 with carbon depleted from the 13-isotope using CO₂ with more than 99.9% carbon-12 that we happened to have in stock. The carbon-13 NMR spectra obtained from this compound at two temperatures corresponding to solid I and solid II are shown in the figure.² In solid II a spectrum corresponding to a biaxial tensor was obtained with $\sigma_{11}=197.0$, $\sigma_{22}=139.2$, $\sigma_{33}=68.9$ ppm (relative to TMS), while in solid I the spectrum was that of a uniaxial tensor with $\sigma_{11}=\sigma_{22}=168.9$, $\sigma_{33}=67.4$ ppm. The results clearly indicate that in solid II the benzene moiety is static while in solid I it rapidly reorients about the σ_{33} direction (normal to the benzene plane).

Sincerely yours,

A. Maliniak G. Greenbaum R. Poupko H. Zimmermann Z. Luz

1. M. Sorai, K. Tsuji, H. Suga, H. Seki, Molec. Cryst. Liq. Cryst. 59, 33 (1980).
2. A. Maliniak, S. Greenbaum, R. Poupko, H. Zimmermann, Z. Luz, J. Phys. Chem. 97, 4832 (1993).



Carbon-13 NMR spectra at 75 MHz of a powder sample of BHA6 in which the carboxylic carbon is depleted to less than 0.1% ^{13}C . Delay time between pulses, 90s; total accumulation time, 3 days for each spectrum.

August 16, 1993

Dear Barry:

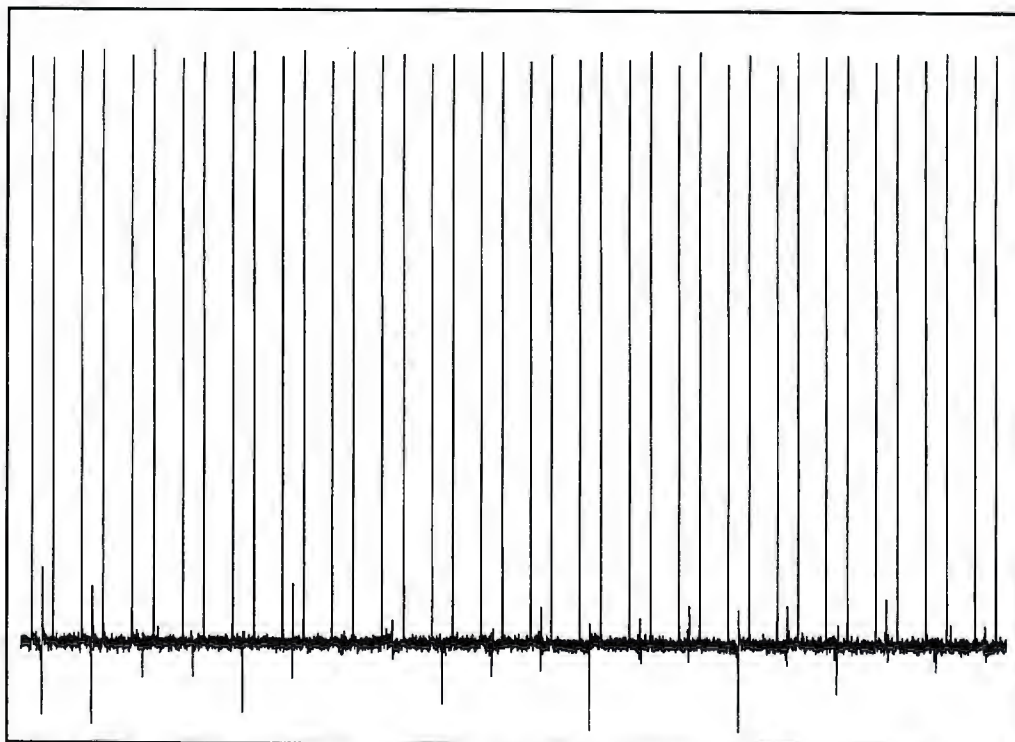
The *Twenty-Fifth Southeastern Magnetic Resonance Conference* will be hosted by the Departments of Chemistry, Biochemistry, and Physics of the University of Florida on October 29-30, 1993. The program will include invited lectures, contributed papers, posters and exhibits. Topics will include NMR of proteins; solid-state NMR; magnetic resonance imaging; NMR spectroscopy in vivo; magnet and coil design; ion cyclotron resonance; EPR spectroscopy; NMR at very low temperatures. Keynote talks will be given by Alex Pines, Charles Johnson, Stan Opella, Alan Marshall, Raymond Andrew, and Angela Gronenborn. Jack Crow of the National High Magnetic Field Lab will give an overview of developments in the Laboratory and Denis Markiewicz will discuss design of high-field magnets.

Rooms for Thursday through Saturday nights have been reserved at the Holiday Inn, University Center, Gainesville, FL, and those planning to attend the conference may call the hotel at 904-376-1661. A registration reception at the Holiday Inn is planned for Thursday evening, and the sessions will continue until mid-afternoon, Saturday. Gainesville is served by Delta and U.S. Air. Further information and registration materials may be obtained by writing or calling Wallace S. Brey, Chemistry Department, University of Florida, Gainesville, Florida, 32611-2046; phone 904-392-0520; FAX 904-392-8758.

SPECTROMETER STABILITY AND THE DIGITAL LOCK

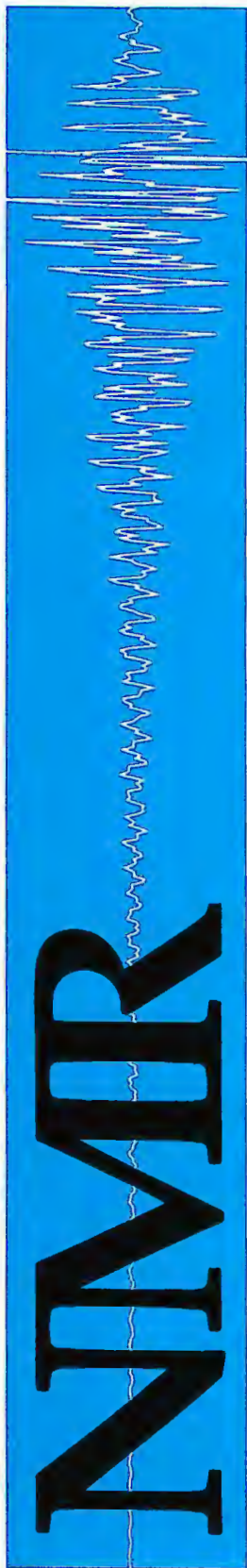
The AVANCE™ DMX series of spectrometers features the innovative Digital Lock™ technology, which is also available as an option on AMX and ARX series spectrometers. The advantages of the Digital Lock in difficult environments, such as in the vicinity of subways, elevators, compressors, or other magnetic fluctuations found in most major facilities, has been extensively documented.

However, the Digital Lock fundamentally improves the stability of the AVANCE DMX spectrometer, even under favorable and near-ideal environmental conditions. In order to prove this point, we recorded the most demanding version of a Spin-Echo Difference (SED) experiment on a well shimmed sample of 3% chloroform. We used the absolute minimum of 2 scans (not 8-16 scans), no line broadening, and the Digital Lock. The 600 MHz system was located in a good environment without known magnetic and other disturbances.



20 Spin-Echo Difference experiments; 3% CHCl₃, NS=2, LB=0 (Digital Lock, good environment)

NMR



We found in 20 out of 20 experiments, the peak-to-peak height of the suppressed centerband never exceeded 50% of the height of the satellites. With a traditional analog lock system in a similar environment, it was more typical to quote the following specification: "in 7 out of 10 experiments the centerband peak-to-peak height will not exceed two times the height of the satellites".

Thus, under fairly good conditions the Digital Lock still provides an improvement of a factor of 4, and on 20 out of 20 experiments! The very stringent requirements for stability of modern 2D/3D/4D NMR experiments, especially those involving cancellations and inverse detection, hardly require any discussion. Suffice it to point out, that the new generation of gradient versions of these experiments are even less forgiving, because there is no phase-cycling which would attenuate any imperfections and instabilities.

The most impressive example, comparing the conventional analog system with the digital lock under unfavorable conditions, was provided from an AMX 600 in Professor Richard Ernst's lab at the ETH next to the Zürich street cars. In the presence of magnetic perturbations the Digital Lock gives an improvement of approximately a factor of 10-25 over traditional analog lock technology in spin-echo difference suppression experiments. (TAMU NMR Newsletter No. 408, 9/92, pages 12-14)

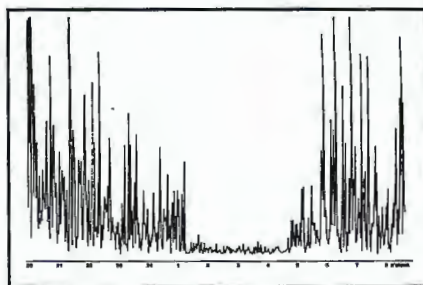


Figure 2

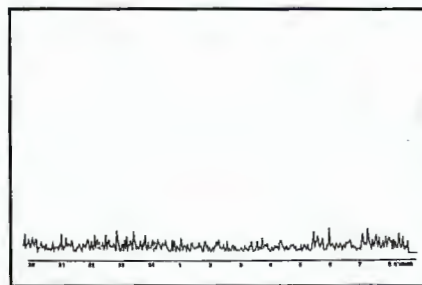


Figure 3

ANALOG → DIGITAL

Both figures show an inverse spectrum of chloroform. The center line is plotted every 3 minutes, during a 24 hour period. Figure 2 is using the **analog lock**, figure 3 is using the **digital lock**. Both are run under conditions of external field disturbances from the Zürich street cars.



NSR Center

Nijmegen SON Research Center for
Molecular Structure, Design and Synthesis

SON National HF-NMR Facility
Toernooiveld, 6525 ED Nijmegen
The Netherlands

A joint Institute of the University of Nijmegen and the Netherlands Foundation for Chemical Research

Nijmegen, July 29, 1993 (received 8/2/93)

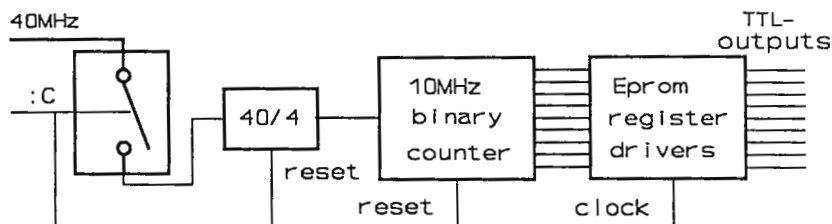
AM-DISCO

Dear Professor Shapiro,

In recent years a number of novel experiments for multinuclear NMR have been developed that cannot be performed on standard AM- or older type spectrometers. A case in point is a set of triple resonance experiments developed by A. Bax for assigning $^{13}\text{C}/^{15}\text{N}$ -enriched protein spectra in particular. In this set there are two, the HCACO and the HCA(CO)N experiments, that require, besides powerswitching, two ^{13}C frequency sources with independent phase-cycling. To get these experiments smoothly running on an AM-type spectrometer we have built a double digital synthesizer. When one wants to exploit all of its possibilities, one ends up with ten different functions just for its phase- and frequency control. So we ran into trouble because of the limited number of TTL-outputs on our Bruker AM-spectrometers. These spectrometers "only" have a total of eight to ten freely programmable TTL-outputs available.

To solve this problem now and to avoid more of this kind in the future, we have built a device, that extends the possibilities of TTL-control of any type of spectrometer by a factor of almost infinity. Triggered by the number of monitoring LED's on its front we called this device "AM-DISCO" and thought that the abbreviation might stand for "Decoder for Innovative Spectrometer Control".

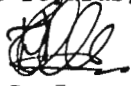
The basic idea of this apparatus is to use just one TTL-output to control one entire device or external RF-channel, regardless of the number of functions it has. To get this done, we use a pulsetime-decoder. Depending upon the length of a TTL-pulse (a ":C"-pulse in our case) we activate a series of TTL-outputs to control the external devices. The principles of operation are explained with the help of the simplified blockdiagram below.

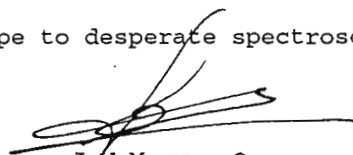


The blockdiagram shows a binary counter with a timebase of 100ns that is synchronised to the main clock (40MHz) of the timing device (process controller) of the actual spectrometer. When the input pulse (":C") is "high", for instance for 1.4μs, the counter will reach its binary value fourteen. This number now acts as an address for the EPROM. The contents of this memorylocation is the desired combination of active and non-active TTL-outputs. Both the number of addresses and the wordlength of this EPROM can be virtually infinite. This means that the possibilities of the device are only limited by the decoding time it takes to activate the different output combinations. (100ns. per combination).

May this contribution give new hope to desperate spectroscopists.

Best regards,


J.W.G. Janssen
(haja@nmr.kun.nl)


J.W.M. van Os
(jaos@nmr.kun.nl)

P.S. Please contribute this letter to E. de Boer's account.



Carleton
UNIVERSITY

Department of Chemistry

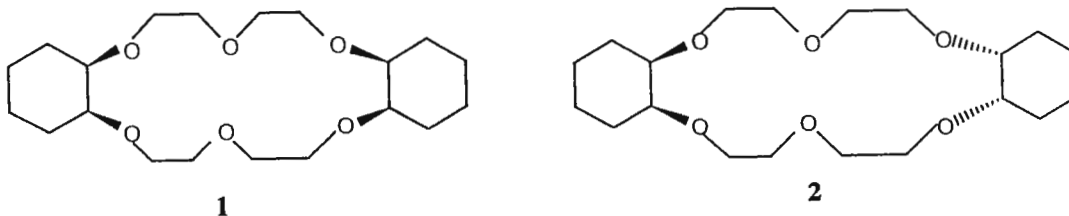
203 Steacie Building
1125 Colonel By Drive
Ottawa, Canada K1S 5B6
Tel: (613) 788-3841
Fax: (613) 788-3749

Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto California, 94303 USA

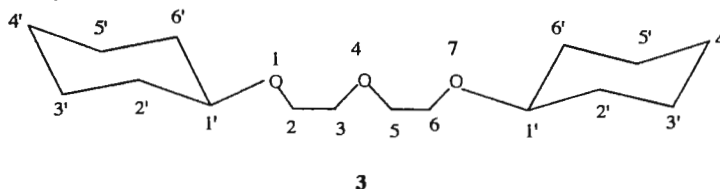
July 6, 1993
(received 7/31/93)

Title: Conformational Interchange In Dicyclohexanodiethyleneglycol As Studied By ^{13}C NMR

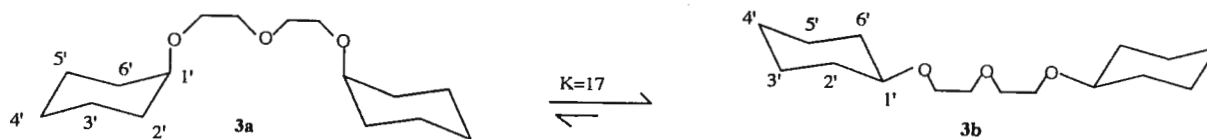
Conformational equilibria in cis-syn-cis and cis-anti-cis dicyclohexano-18-crown-6 ethers (**1** and **2** below) are amenable to examination via ^{13}C NMR at low temperature. Generally, the detected processes are cyclohexane ring inversions and conformational free energy differences are small ($< 1.2 \text{ kJ mol}^{-1}$), due to the cis-1,2-disposition of the oxygenated substituents.



The 1,7-dicyclohexanodiethyleneglycol molecule (**3**, below) is a structural constituent of **1** and **2** and it is of interest to determine if it possesses any conformational equilibria detectable by NMR at low temperature.



The 125 MHz ^{13}C NMR spectrum of **3** shows 6 lines at 275K, indicating that all conformational interconversions are rapid on the NMR timescale. At 205K in CD_2Cl_2 solution, resonances for $\text{C}1'$, $\text{C}2'$, $6'$ and $\text{C}3'$, $5'$ and $4'$ appear as doublets with relative intensities in each case of ca. 17:1. Of course the usual precautions regarding nOe differences and T_1 variations must be taken, but it appears from these results that we have an interconversion between **3a** and **3b** (below) in which the conformational free energy difference is ca. 5 kJ mol^{-1} .



We are currently exploring the influence of alkali metal cations on this and related equilibria.

G.W. Buchanan
Professor of Chemistry,
Director, Ottawa-Carleton Chemistry Institute

AMT Delivers More For Less!

10–90 MHz, 1000 Watt RF power amplifiers for less than \$10,000

Key Features:

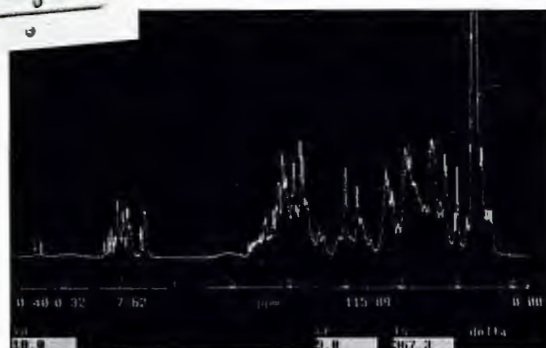
- Full 10–90 MHz frequency range
- 1000 watt power rating
- Linearity ± 1 dB
- Noise blanking in less than $2 \mu\text{s}$
- Less than 5% pulse droop
- Dual mode, Pulse/CW operation
- Digital power meter
- Dual range power limits



Model 3426

The AMT NMR/NMRI product family

SERIES	FREQUENCY	POWER RATINGS
3130	200–500 MHz	50 to 300 W
3200	6–220 MHz	150 to 1000 W
3304	30–310 MHz	400 W
3420	10–90 MHz	1000 to 2000 W



Call Jim Lukes at AMT, or your local distributor NOW
for a price that will flip your spins!





Model 3426

10–90 MHz, pulsed,
solid-state, RF power
amplifier system

Electrical specifications:

Frequency range	10–90 MHz
Pulse power (min.) into 50 ohms	1000 W
CW power (max.) into 50 ohms	100 W
Linearity (± 1 dB)	0–900 W
Gain (typ.)	65 dB
Gain flatness	± 2 dB
Input/Output impedance	50 ohms
Input VSWR	$< 2:1$
Pulse width	20 ms
Duty cycle	Up to 10%
Amplitude rise/fall time	250 ns typ.
Amplitude droop	5% to 20 ms typ.
Phase change/output power	10° to rated power, typ.
Phase error overpulse	4° to 20 ms duration, typ.
Noise figure	11 dB typ.
Output noise (blanked)	< 20 dB over thermal
Blanking delay	$< 2 \mu\text{s}$ on/off, TTL signal
Protection	1. VSWR: infinite VSWR 2. Input overdrive: up to 10 dB 3. Over duty cycle/pulse width 4. Over temperature

Supplemental characteristics:

Connectors, rear panel	1. RF input: BNC (F) 2. RF output: Type N (F) 3. Noise blanking: BNC (F) 4. Interface: 25 pin D(F), EMI filtered
Indicators, front panel	1. AC power on 2. Peak power meter 3. Over pulse width 4. Over duty cycle 5. Over temperature 6. Over drive 7. CW mode
System monitors	1. Forward/Reflected RF power 2. Over pulse width/duty cycle 3. DC power supply fault 4. Thermal fault
Front panel controls	1. AC power 2. Pulse width 3. Duty cycle
Cooling	Internal forced air
Operating temperature	+10 to 40°C
AC line voltage	208/230 VAC, $\pm 10\%$, 50–60 Hz
AC power requirements	2000 watts
Package	Rack mount
Size (HWD, inches)	8.75 \times 19 \times 20.25
Net weight	100 lbs.

03/92

LOVELACE MEDICAL FOUNDATION

Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

July 23, 1993
(received 7/28/93)

An Unusual Velocity Calibration Device

Dear Dr. Shapiro,

We recently measured the velocity of water flowing in a curved duct of rectangular cross-section, which we installed in special slots cut into an 80.3 MHz birdcage rf probe. Figure 1 shows the geometry. Three orthogonal velocity components were measured in a slice perpendicular to the duct axis at the site where the duct had curved 90°. We used a "phase contrast" velocity measurement technique to encode velocity. Phase contrast velocimetry is a difference method that relies on phase shifts induced by motion along a field gradient. The velocity sensitivity of a gradient waveform is proportional to the "first moment" of the gradient

waveform (e.g., $\int_0^{TE} t \cdot G_x dt$ for the x-direction, with TE = echo time). 2-D images with and without velocity sensitivity are used to compute velocity. In our view, it is necessary to calibrate imaging sequences used for this type of measurement, and therein lies the utility of the device shown in figure 2, which was assembled from pieces supplied by Greg, Lauren and Peter Icenogle. This material is non-magnetic, non-conductive, easily assembled, and inexpensive. The shaft extending to the right was rotated via a remote motor with the device positioned so that a cross-section of the water bottle could be imaged. The motor speed was adjusted to produce 15 rpm rotation of the bottle, and after a few hundred seconds (several times R^2/v) velocity images were acquired. The water rotated as a rigid body: velocity components were $v_x = 1.6 \cdot y$ and $v_y = -1.6 \cdot x$ cm/s. This provided calibration for the transverse, or secondary, velocity components in the duct cross-section. Because access to the imaging volume is limited it is difficult to produce controlled motions in these directions without a device such as this. The axial, or streamwise, component was calibrated by timed collection with the square duct *in situ*.

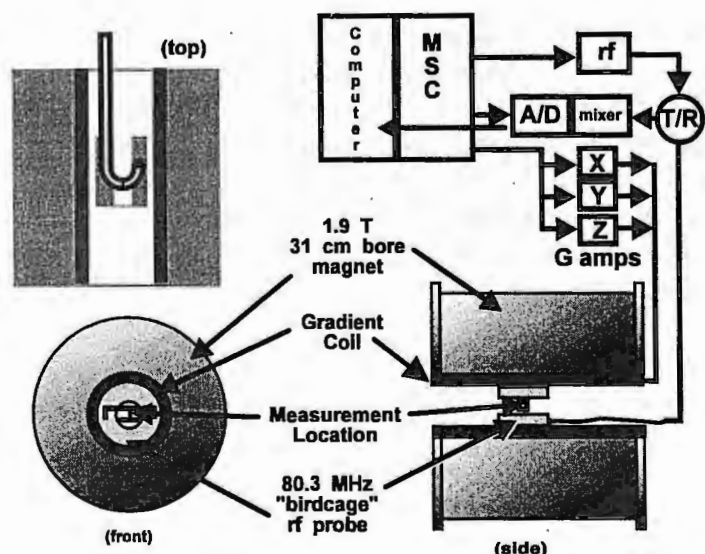


Figure 1. Three cross-sections of the layout of duct installed in the modified birdcage.

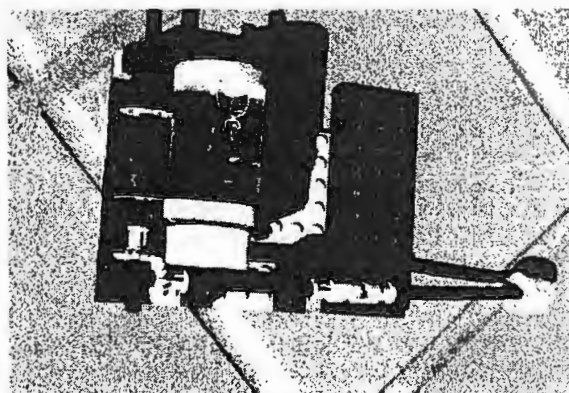


Figure 2. LEGO™ right angle turn velocity calibrator.

Sincerely,

Steve Altobelli

Steve Altobelli

Eiichi Fukushima

Eiichi Fukushima

ZENECA

Dr. B.L. Shapiro, Editor
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

(received 8/13/93)

ZENECA Ag Products

Western Research Center
1200 S. 47th Street
Box 4023
Richmond
California 94804-0023

Telephone (510) 231-1000
Fax (510) 231-1368
Telex 172197

Subject: Nitrogen Chemical Shift Assignments of D-Fructose Oxime

Dear Barry:

Keto-monosaccharides exist in many different tautomeric forms in aqueous solution. The linear "keto" tautomeric form is normally the least populated. Oxime derivatives of monosaccharides lock the sugar into predominantly the linear form and may serve as a good first approximation of the underivatized linear form's conformation.

D-(2-¹⁵N)Fructose oxime was synthesized using N-15 enriched hydroxyl amine hydrochloride, for a mass spectrometry experiment. Since the material was available, it was of interest to measure the nitrogen chemical shifts of D-fructose oxime. The spectrum was recorded at ambient temperature and two resonances appear at 345.1 and 341.4 ppm with similar intensities (referenced to external formamide @ 112.4 ppm).

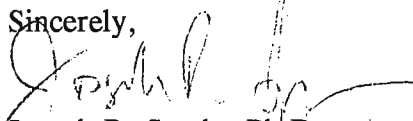
It has been shown that ¹⁵N resonances of Z isomers of acyclic ketoximes appear upfield of the resonances of the corresponding E isomers¹. To verify whether this trend was consistent with carbohydrate oximes a long-range ¹⁵N-¹H 2D HETCOR experiment was run. Indeed the results are consistent with this trend. The resonance at 345.1 ppm correlates with H-3E and H-1E. The upfield resonance correlates with H-3Z, H-1Z and H-4Z.

One bond carbon-nitrogen coupling constants were measured from the ¹³C NMR spectrum. The results indicate that ¹J(C-N)E=(-)2.98 Hz, whereas ¹J(C-N)Z=(-)3.15 Hz. The values are expected to be negative based upon earlier work.²

1)R.E. Botto, P.W. Westerman and J.D. Roberts, *Organic Mag. Res.*, **11**, 550, 1978.

2)G.W. Buchanan and B.A. Dawson, *Can J. Chem.*, **56**, 2200, 1978.

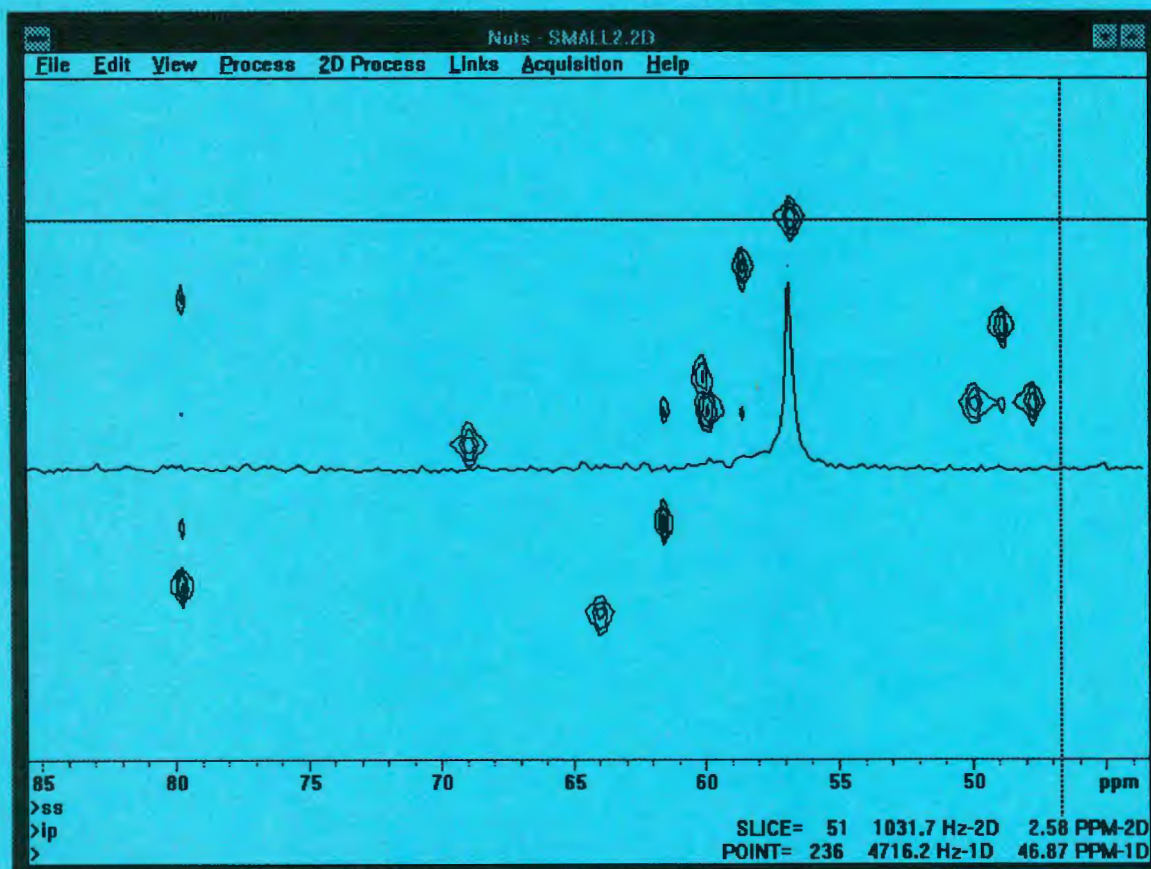
Sincerely,


Joseph R. Snyder Ph.D.
Senior Research Chemist



A business unit of ZENECA Inc.,
a member of the ICI Group.

2D Processing on Your PC !



2D contour plot with real-time display of a slice at the cursor location. Coordinates of the crosshair cursor are displayed in real time in the lower right corner of the screen.

NUTS-2D features include:

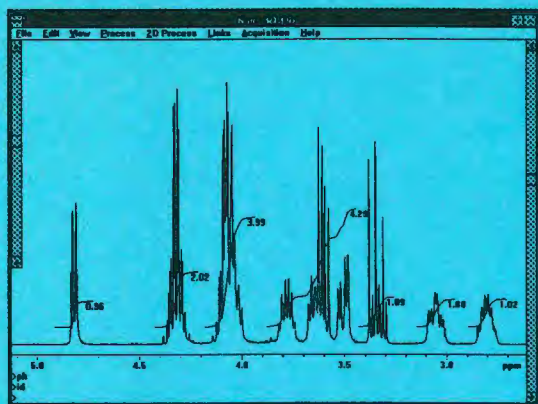
- Powerful, flexible and inexpensive
- Quick intensity map display or contour plots
- User control of contour level spacing and colors
- Real-time readout of cursor coordinates in both dimensions
- Real-time display of slices overlaid on contour plot
- Sample processing procedures supplied

Plus all the features of the 1D version.



Acorn NMR

46560 Fremont Blvd., #418
Fremont CA 94538-6482
Telephone: (415) 683-8595
FAX: (415) 683-6784



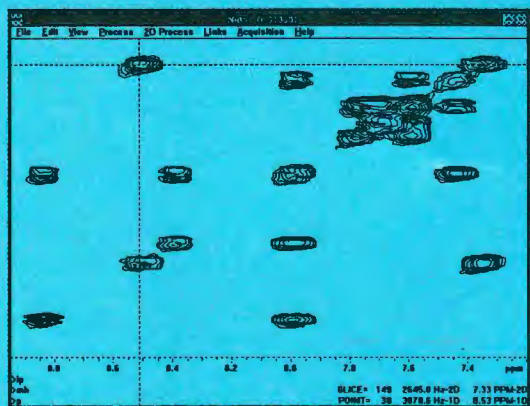
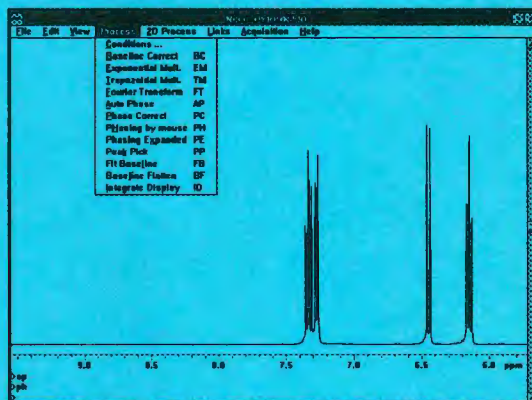
Data Translation from most spectrometer models

- Bruker Aspect 3000 data files
- Varian XL, Gemini and older VXR files
- Nicolet Dexter and TMON files
- GE Omega and TMON files
- JEOL *.GXD/*.GXP data files
- ATI ASCII and binary files

Other data file translations will be added on request if file format information can be supplied.

Easy to learn and use Windows format

- On-line Help saves searching through manuals
- Menus and command line interface active at all times for maximum flexibility
- Command sequences automated with customizable quick keys
- Copy spectra and paste into reports



Other Features

- 16K complex FT in 1.5 sec on 486 PC
- Automatic and real-time phasing
- Multiple baseline correction options
- Integrals labelled on the screen and plots
- Intelligent peak-picking
- Acquisition parameters translated & printed on plots
- Configuration file lets you customize NUTS for your preferences

NUTS 1D version 1.5 for IBM PC (math coprocessor required) on 3.5" HD floppy\$ 499.00
 NUTS 2D version 1.0 for IBM PC (math coprocessor required) on 3.5" HD floppy\$ 750.00



Acorn NMR

46560 Fremont Blvd., #418
 Fremont CA 94538-6482
 Telephone: (415) 683-8595
 FAX: (415) 683-6784

Dr B L Shapiro
 TAMU NMR Newsletter
 966 Elsinore Court
 Palo Alto
 California 94303
 United States of America

(received 8/2/93)

Dear Barry,

Applications of pulsed field gradients in high-resolution NMR spectroscopy.

Last year we acquired a pre-emphasis and Z₀ compensation unit for our AMX500 spectrometer and have finally managed to obtain a working configuration (after a new analogue router board and other miscellaneous but essential modifications - beware retrofitting one of these to an AMX!) Currently we are testing it out on the standard experiments, before moving on to more advanced things.

We have been exploring the use of gradient pulses to discriminate between protons bound to an isotopically enriched nucleus (eg. ¹⁵N) and those bound to the abundant but not interesting isotope (¹⁴N). In the figure, the bottom spectrum is a ¹H spectrum of the peptide VTHRLAGLLSRSGGVVKNNFVPTNVGSKAF, where all valine, leucine, alanine, glycine and phenylalanine residues have been selectively labelled with ¹⁵N. The middle spectrum is ¹⁵N edited, ie. displaying protons bound to ¹⁵N only, acquired with a standard Bruker AMX pulse sequence. The top spectrum is ¹⁵N-suppressed, ie. displaying protons not bound to ¹⁵N. The pulse sequence used was:

¹H: - 90 - 1/4J(XH) - 180 - 1/4J(XH) - 90 - - 90 - 1/4J(XH) - 180 - 1/4J(XH) -
 X: - 180 - - 90 - 180 -
 Gz: _|---|_

¹H: - 90 - 90 - ACQ.
 X: - 90 -

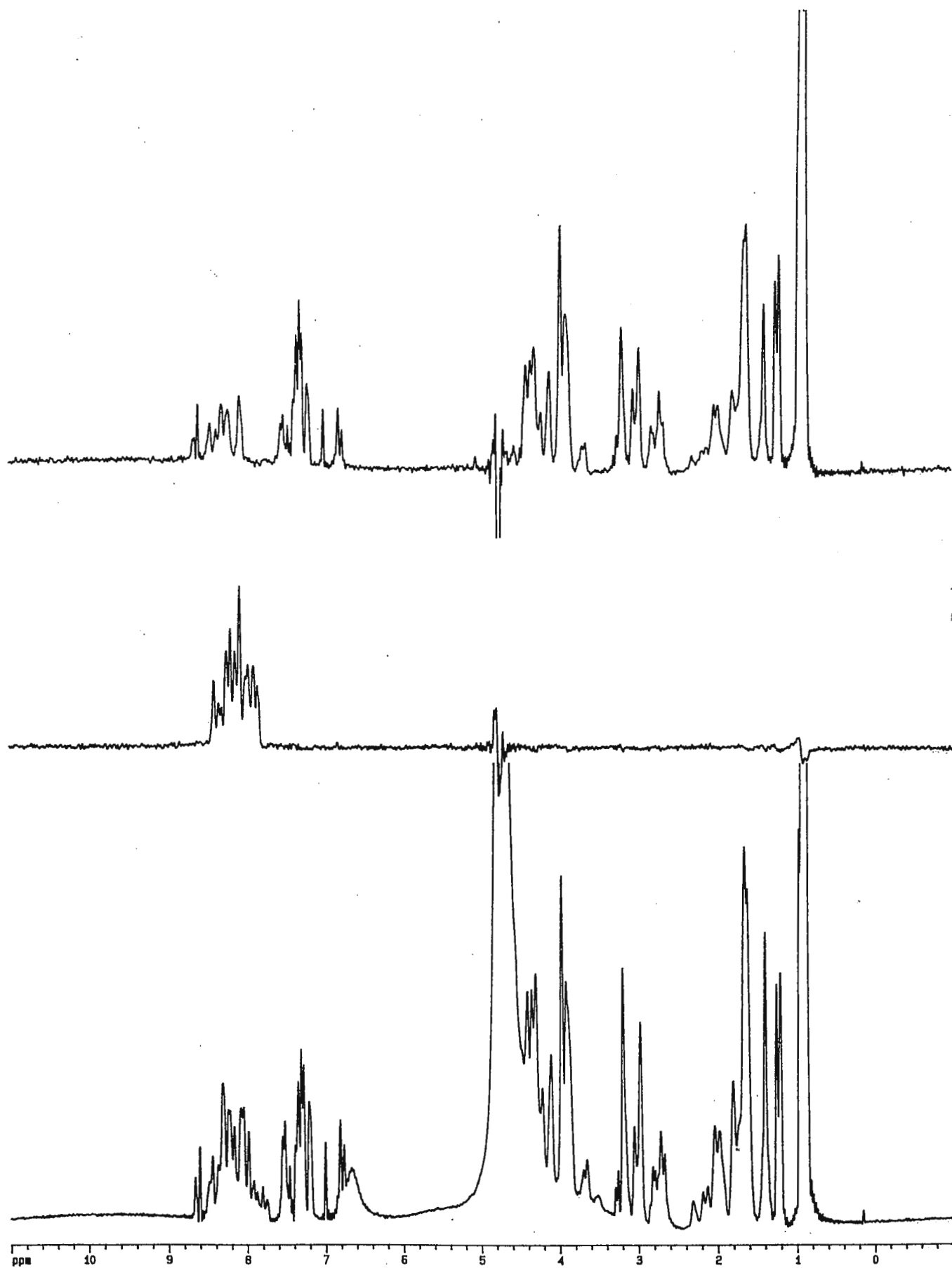
This filter may also be used as an input for various nD experiments, to give X-edited or X-suppressed spectra. We are currently applying this method to the study of protein-ligand interactions.

Yours sincerely,

David Reid Lesley MacLachlan Julia Hubbard

David G Reid Lesley K MacLachlan Julia A Hubbard David M Doddrell

Ian M Brereton

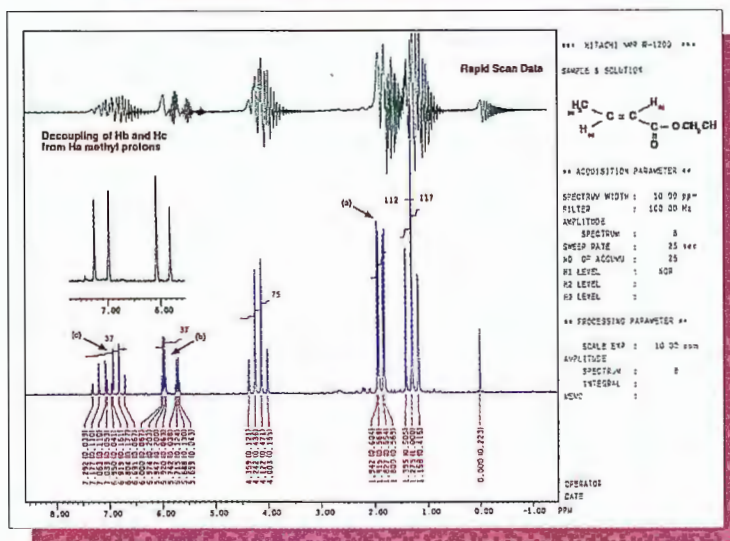


Now, A Superior 60 MHz Spectrum Acquired in as Little as 10 Seconds.



Auto Resolution in less than 1 minute

Compact Size & Low Maintenance of a Permanent Magnet



Ringings is Removed by Cross-Correlation

Multiple Spectra Accumulation via Field Cure

The R-1200 Rapid Scan cw Spectrometer Redefines the Standard for 60 MHz NMR.

The R-1200 is the only microprocessor controlled 60 MHz, rapid-scanning cw spectrometer. It acquires a quality, ringing free spectrum in as little as 10 seconds. Our Field Cure™ technique makes corrections for field shifts during multiple spectra accumulations. As many as 256 spectra can be collected, increasing your signal-to-noise ratio in the same way as more expensive, higher field instruments.

Maximum resolution is set with a single keystroke through an auto-resolution feature, eliminating the tedious shimming process. Also, software correction of spectral phasing and integral balance is made in seconds prior to output to the four-pen digital plotter. All spectral information, including chemical shifts, integration, with integration numbers, and peak heights are digitally calculated and printed directly on the spectrum. The spectrometer can save up to three spectra. In addition, all spectral information can be output in an ASCII format to a PC for storage and post-run processing. Double Resonance is a standard feature; triple resonance is available as an option.

For complete information, call toll free **800-548-9001**.
Hitachi Instruments, Inc. 3100 North First Street, San Jose, CA 95134

HITACHI

VÍDEŇSKÁ 800
140 00 PRAGUE 4
Czech Republic



INSTITUTE FOR CLINICAL AND EXPERIMENTAL MEDICINE
DEPARTMENT OF DIAGNOSTIC AND INTERVENTIONAL RADIOLOGY
HEAD: JAN PEREGRIN, MD, PhD

June 22, 1993
(received 8/2/93)

Dr. B. L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, California 94303

Dear Dr. Shapiro,

ADIABATIC PULSE ON GBS2

We are concerning in phosphorus in vivo spectroscopy using surface coil with large diameter. Because our old Siemens GBS 2 system is not too flexible to generate new pulses, we developed new pulse generator on IBM/PC, which extends feasibilities of our system.

First pulse, which we tested, was adiabatic pulse BIR-1 [1]. It is modulated by functions cosinus in amplitude and sinus in phase. For testing we have been using phosphorus fantoms. The different concentration of Na^+ , H^+ , PO_4^{3-} determined the pH and consequently chemical shift of the samples⁴.


The first comparison of the pulses has been performed using the simplest excitation sequence. For sinc and BIR-1 pulses we have measured signal intensity dependence on the transmitter voltage. According to published works the adiabatic pulse needs much more higher transmitter voltage than sinc one. The shape of the dependence and maximum intensity obtained for both pulses shows no significant differences.

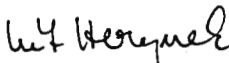
Practically same results we have obtained with sequence ISIS, we have been using rectangular respective adiabatic $\pi/2$ -pulses for the excitation and hypersecant π -pulse for the inversion. Slightly higher signal intensity in the case of the adiabatic pulse hardly balances the need of the considerably higher voltage, which can make impossible to use it in clinical in vivo practice.

Significant improvement of the signal/noise ratio has been brought by the adiabatic pulse used for excitation in the presence of the magnetic field gradient (2mT/m). Gradient has influenced the spectra obtained with adiabatic pulse only slightly, while spectra obtained with sinc pulses (used in the absolutely same sequence) were practically disturbed by the noise (Figs. 1-3).

In the view of our results we can expect much more homogeneous excitation in the presence of magnetic gradient using adiabatic pulses designed by our new pulse generator in comparison with by now used pulses.

Yours sincerely,


Milan Hájek


Vít Herynek

1. Ugurbil K., Garwood M., Bendall M. R.: J. Magn. Reson. 72, 177-185 (1987)

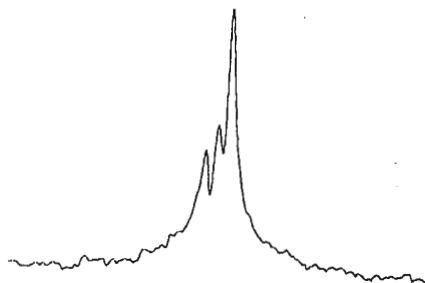


Fig. 1:
Excitation by SINC pulse
without gradient

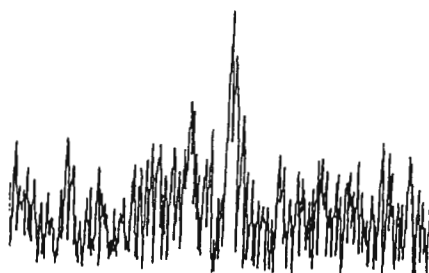


Fig. 2:
Excitation by SINC pulse
gradient = 2mT/m

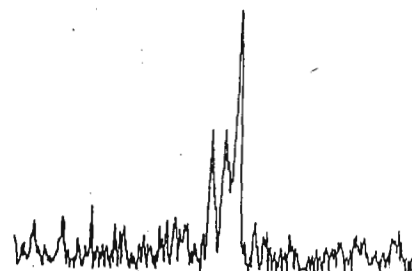


Fig. 3:
Excitation by pulse BIR-1
gradient = 2mT/m



Universität Bern

Institut für organische Chemie

CH-3012 Bern, Freiestrasse 3
Telefon 031 65 43 11

PD Dr. P. Bigler
Institute of Organic Chemistry
University of Berne
Freiestrasse 3
3012 Bern
Switzerland

Tel.: (041 031) 631 39 48
Fax: (041 031) 631 44 59
e-mail: bigler@ioc.unibe.ch

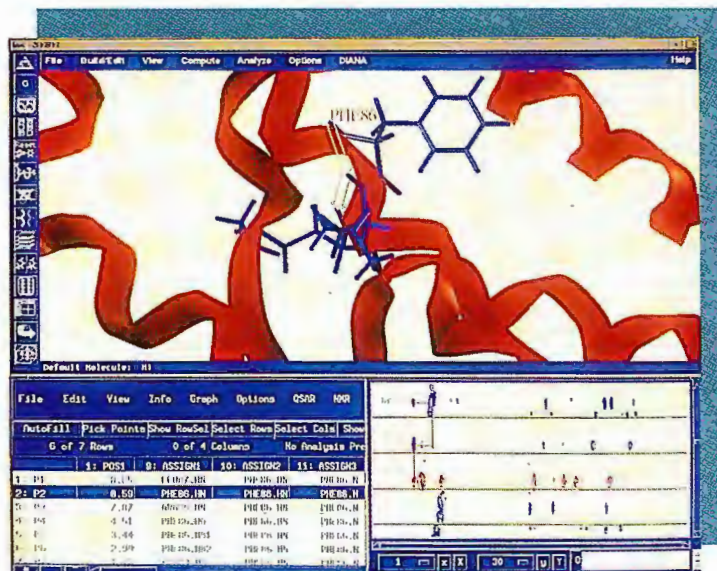
The NMR facility of the department of organic chemistry at the university of Berne has **BRUKER AM 400 accessories** for sale. This includes

- a **selective excitation unit** (SEU),
- **5 mm probeheads** for a 9.3T wide-bore magnet ($^1\text{H}/\text{BB}$ reverse, $^1\text{H}/^{13}\text{C}$ dual, $^1\text{H}/^{19}\text{F}$ dual, ^1H selective),
- a **10 mm probehead** ($^1\text{H}/\text{BB}$),
- **computer boards** and
- **various other equipment.**

All the accessories work fine in the moment and will be available to strongly reduced prices this fall. Please contact me if you are interested via e-mail, Fax or phone (see above).

**"For the first time it is possible to go from
NMR data to refined NMR structure in one single
user-friendly environment. TRIAD is extremely useful."**

Leading Pharmaceutical Researcher



TRIAD NMR Advanced

Spectra, Spreadsheet, and Structures

All-in-one!

TRIAD NMR Advanced software delivers a new vision to biomolecular research by bringing together NMR spectral processing and powerful molecular modeling in one program. This unification makes TRIAD NMR Advanced the total solution from NMR data processing to structure analysis.

- ▲ worry-free data importing from virtually all spectrometers
- ▲ centralized information and control with the powerful Molecular Spreadsheet
- ▲ AUTOMATIC and INTEGRATED tools for peak assignment and analysis
- ▲ complete flexibility for easy customization

Call today to find out how TRIAD NMR Advanced can accelerate your research.



TRIPOS
Associates, Inc.

1699 S. Hanley, Suite 303
St. Louis, MO 63144-2913
800-323-2960
Phone: +1 314-647-1099
Fax: +1 314-647-9241

•Munich, Germany
•Palaiseau Z.I., France
•Bracknell, UK
•Rigong International, Inc (Korea)
•Sumisho Electronics Co., Ltd. (Japan)
•M & Vactek Corp. (Taiwan)
•3-Link Systems PTE LTD (Singapore)
•3-Link Systems PTE LTD (Beijing)

Phone:
+49 (89) 4510300
+33 (1) 69-191670
+44 (344) 300144
+82 (2) 516-2636
+81 (3) 5228-5525
+886 (2) 7883836
+65 2278671
+86 (1) 2563388

Fax:
+49 (89) 45103039
+33 (1) 69-320947
+44 (344) 360638
+82 (2) 516-5362
+81 (3) 5228-5526
+886 (2) 7836255
+65 2278679
+86 (1) 2568640

Sandia National Laboratories

Albuquerque, New Mexico 87185

Departments 1811 and 1812

July 22, 1993

(received 8/4/93)

Dr. Barry L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

Dear Dr. Shapiro,

Structural Assignment of a Regioisomer of $C_{60}H_4$

We are currently investigating the chemistry of buckminsterfullerene, C_{60} , particularly the hydrogenation of C_{60} to yield a single regioisomer of $C_{60}H_2$ and several regioisomers of $C_{60}H_4$. During this work, we have found 1H NMR to be extremely valuable in the characterization of the $C_{60}H_4$ isomers.

Treatment of C_{60} with borane-THF complex in toluene followed by protonolysis with water yields 1,2- $C_{60}H_2$ (addition of hydrogen across a 6,6 ring fusion) as the major hydrogenated product.¹ The reaction mixture also yields three isomers of $C_{60}H_4$ separable by preparative HPLC (less than 2% total yield based on the uncorrected peak heights). The 400 MHz 1H NMR of one of these regioisomers is shown in Figure 1. This isomer of $C_{60}H_4$ (Figure 1a) has a distinctive ABB'A' type of coupling pattern centered at 5.03 ppm in toluene- d_8 . This type of coupling pattern is only possible for the 1,2,3,4 isomer of $C_{60}H_4$ (see Figure 2), assuming hydrogenation only occurs across 6,6-ring fusions.

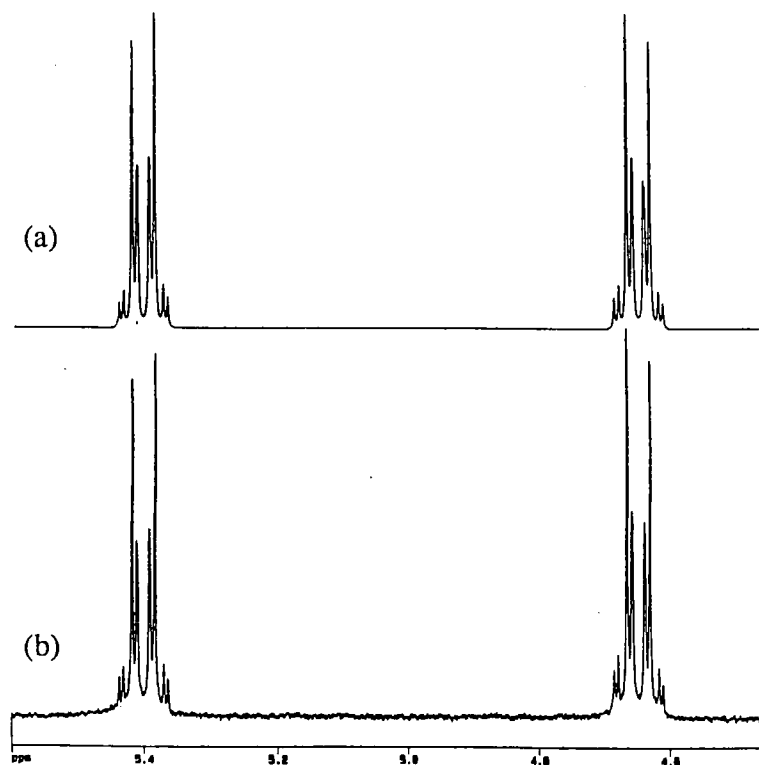


Figure 1: Simulated (a) and experimental (b) 400 1H NMR spectra of 1,2,3,4- $C_{60}H_4$ in toluene- d_8 .

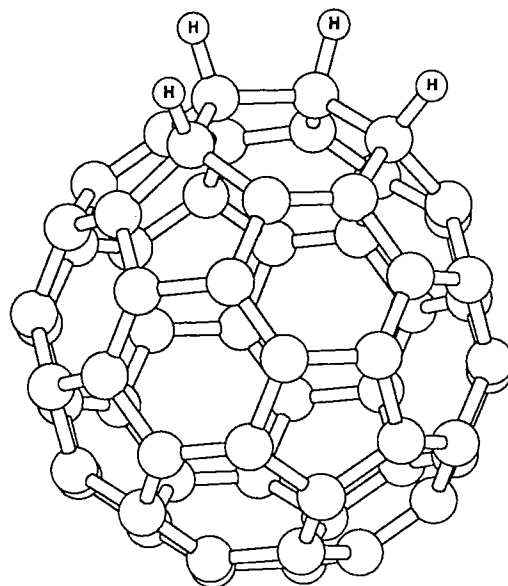


Figure 2: 1,2,3,4 isomer of $C_{60}H_4$.

We verified this structural assignment by simulating the ^1H NMR spectrum of 1,2,3,4- C_{60}H_4 using the NMRSIM program for the GE 1280 data station. The following parameters were used to generate the spectrum in Figure 1b: $\Delta\delta = 300$ Hz, $^3J_{\text{AB}} = 14.1 \pm 0.5$ Hz, $^3J_{\text{BB}'} = 9.8 \pm 0.5$ Hz, $^4J_{\text{AB}'} = -0.2 \pm 0.5$ Hz, and $^5J_{\text{AA}'} = 1.7 \pm 0.2$ Hz (see Figure 3). Line broadenings of 1.0 Hz and 0.2 Hz were used in the simulated and experimental spectra respectively. The agreement between the experimental and calculated spectra is excellent -- even the fine splittings and relative intensities are reproduced. Changing the parameters slightly results in significant differences between the experimental and simulated spectra.

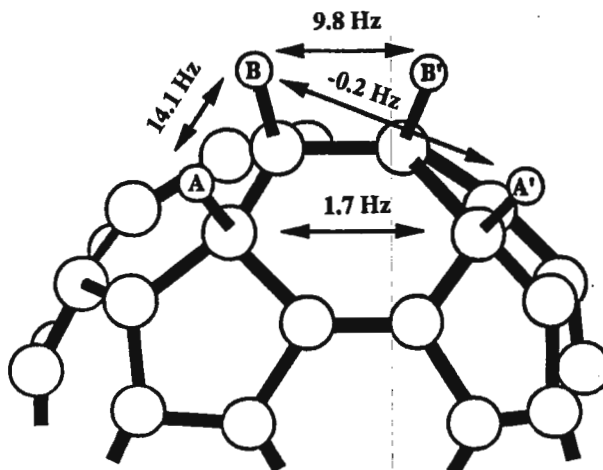


Figure 3: Coupling constants of 1,2,3,4- C_{60}H_4 .

The AB (=A'B') coupling of 14.1 Hz is in agreement with the value of 15.7 Hz observed for the vicinal hydrogens of C_{60}H_2 .¹ Although smaller in magnitude, the value of 9.8 Hz for the BB' coupling is also reasonable for cis, vicinal hydrogens. The AB' coupling is almost zero, consistent with the geometry between the two C-H bonds, and the AA' coupling is 1.7 Hz due to small coupling through the double bond in the six-membered ring. Overall, the observed coupling pattern is clearly unique to the 1,2,3,4 isomer of C_{60}H_4 .

The formation of 1,2,3,4- C_{60}H_4 as the major tetrahydro isomer is intriguing because this isomer is calculated by Matsuzawa, et al.,² to be the second *highest* energy isomer of the eight possible isomers resulting from hydrogenation across the 6,6-ring fusions of C_{60} . This may be a result of the reaction kinetics or a limitation of semi-empirical calculation theory which predicts all eight isomers to lie within 3.5 kcal/mole of each other. Further investigations into this question of preferred regiochemistry will certainly involve the use of high field ^1H NMR to identify the isomers of hydrogenated fullerenes.

Best Regards,

Craig C. Henderson

Paul A. Cahill

Roger A. Assink

¹ Henderson, C. C. ; Cahill, P. A. , *Science* **1993**, 259, 1885.

² Matsuzawa, N.; Fukunaga, T.; Dixon, D. A., *J. Phys. Chem.* **1992**, 96, 10747.

ISOTEC INC. = STABLE ISOTOPES!!

ISOTEC INC. offers a wide variety of N-t-BOC, F-MOC and CBZ protected amino acids. Below is a partial list of the protected amino acids ISOTEC now has available:

82-12243-3	L-Alanine-3,3-d ₃ , N-t-BOC Derivative	99 atom%
83-12358-8	L-Alanine-1- ¹³ C, F-MOC Derivative	99 atom%
83-12331-5	L-Alanine-1- ¹³ C, N-t-BOC Derivative	99 atom%
83-12364-6	L-Alanine-2- ¹³ C, N-t-BOC Derivative	99 atom%
83-12320-8	L-Alanine-3- ¹³ C, N-t-BOC Derivative	99 atom%
83-12327-3	L-Alanine- ¹³ C ₃ , N-t-BOC Derivative	99 atom%
85-12266-1	L-Alanine- ¹⁵ N, F-MOC Derivative	99 atom%
85-12241-4	L-Alanine- ¹⁵ N, N-t-BOC Derivative	99 atom%
83-12353-9	L-Aspartic-3- ¹³ C Acid, N-t-BOC Derivative	99 atom%
83-12354-7	L-Aspartic-4- ¹³ C Acid, N-t-BOC Derivative	99 atom%
85-12259-6	L-Aspartic- ¹⁵ N Acid, F-MOC Derivative	99 atom%
85-12244-8	L-Aspartic- ¹⁵ N Acid, N-t-BOC Derivative	99 atom%
83-12322-4	Glycine-1- ¹³ C, N-t-BOC Derivative	99 atom%
83-12365-3	Glycine-2- ¹³ C, F-MOC Derivative	99 atom%
83-12326-5	Glycine-2- ¹³ C, N-t-BOC Derivative	99 atom%
83-12339-8	Glycine- ¹³ C ₂ , N-t-BOC Derivative	99 atom%
85-12260-4	L-Glutamic- ¹⁵ N Acid, F-MOC Derivative	99 atom%
85-12261-2	L-Glutamic- ¹⁵ N Acid, N-t-BOC Derivative	99 atom%
85-12267-9	L-Glutamine- ¹⁵ N ₂ , α-N-t-BOC Derivative	99 atom%
85-12263-8	L-4-Hydroxyphenylalanine- ¹⁵ N, N-t-BOC Deriv. (Tyrosine)	99 atom%
83-12359-6	L-Leucine-1- ¹³ C, F-MOC Derivative	99 atom%
83-12323-2	L-Leucine-1- ¹³ C, N-t-BOC Derivative	99 atom%
85-12245-5	L-Leucine- ¹⁵ N, N-t-BOC Derivative	99 atom%
81-12258-2	L-Leucine-2- ¹³ C, ¹⁵ N, N-t-BOC Derivative	99 atom% ¹³ C, ¹⁵ N
83-12324-0	L-Methionine-1- ¹³ C, N-t-BOC Derivative	99 atom%
83-12337-2	L-Methionine- ¹³ C ₁ (methyl- ¹³ C), N-t-BOC Deriv.	99 atom%
83-12357-0	L-Phenylalanine, N-t-BOC- ¹³ C ₁ Derivative (carbonyl- ¹³ C)	99 atom%
83-12356-2	L-Phenylalanine-1- ¹³ C, N-t-BOC Derivative	99 atom%
83-12372-9	L-Phenylalanine-2- ¹³ C, F-MOC Derivative	99 atom%
83-12351-3	L-Phenylalanine-3- ¹³ C, N-t-BOC Derivative	99 atom%
83-12350-5	L-Phenly-1- ¹³ C-alanine, N-t-BOC Derivative	99 atom%
85-12243-0	L-Phenylalanine- ¹⁵ N, N-t-BOC Derivative	99 atom%
83-12325-7	L-Valine-1- ¹³ C, N-t-BOC Derivative	99 atom%
85-12265-3	L-Valine- ¹⁵ N, F-MOC Derivative	99 atom%
85-12256-2	L-Valine- ¹⁵ N, N-t-BOC Derivative	99 atom%

We look forward to providing you with the highest quality stable isotope labelled compounds for your research needs!!

For further Information:

ISOTEC INC.
A Matheson, USA Company

3858 Benner Road
Miamisburg, Ohio 45342

Tel. 800-448-9760

FAX 513-859-4878

Our NEW 1993 catalog will be mailed soon...

Here are some more labelled compounds recently synthesized in-house by Isotec's highly experienced staff of research chemists:

81-12248-9	L-Arginine- $^{15}\text{N}_2$ HCl (Guanidineimino- $^{15}\text{N}_2$)	99 atom%
81-12800-1	L-Cysteine-2- ^{13}C , ^{15}N	99 atom% ^{13}C , ^{15}N
81-12259-0	Cytosine-2,4- $^{13}\text{C}_2$, $^{15}\text{N}_3$	99 atom% ^{13}C , ^{15}N
82-00815-2	DL-1,4-Dithiothreitol- d_{10} (Cleland's Reagent)	98 atom%
85-30200-8	D-Glucosamine- ^{15}N HCl	98 atom%
83-42050-5	Hexadecanoic Acid-1,2,3,4- $^{13}\text{C}_4$ (Palmitic Acid)	99 atom%
81-61002-4	ISOGRO TM - ^{13}C , ^{15}N Powder- Growth Medium	99 atom% ^{13}C , ^{15}N
83-30005-3	D-Lactose-1- ^{13}C · H_2O (Glucose-1- ^{13}C)	98 atom%
81-12211-1	L-Lysine-6- ^{13}C , 6- ^{15}N HCl	99 atom% ^{13}C , ^{15}N
83-00804-5	2-Mercaptoethanol-1- ^{13}C	99 atom%
83-42052-1	Octanoic-5,6,7,8- $^{13}\text{C}_4$ Acid	99 atom%
83-12362-0	DL-Selenomethionine- $^{13}\text{C}_1$ (methyl- ^{13}C)	99 atom%
82-00804-6	Sodium Dodecyl- d_{25} Sulfate	98 atom%
83-02536-1	Sodium Trifluoroacetate-1- ^{13}C	99 atom%

" ISOTEC'S capabilities in the custom synthesis of specific labelled compounds and high-purity NMR solvents are unique! "

To receive a copy of our new catalog, technical information, price quotations, or to place an order please call:

ISOTEC INC.
A Matheson[®], USA Company

3858 Benner Road
Miamisburg, Ohio 45342

Tel. 800-448-9760

FAX 513-859-4878

KARL-FRANZENS-UNIVERSITÄT GRAZ
Institut für Organische Chemie

Dr. Heinz Sterk

A-8010 Graz, 28.7.93
 Heinrichstraße 28
 Tel. (0316) 380 —

(received 8/17/93)

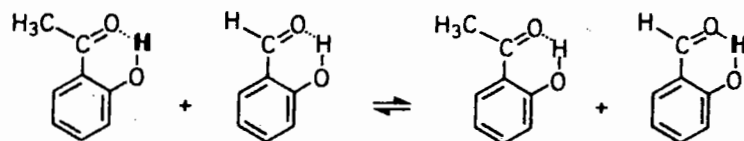
Unser Zeichen:

Dr. Bernhard Shapiro
 TAMU NMR Newsletter
 966 Elsinore Court
 Palo Alto, CA 94303

Proton exchange - selective pulses

Dear Dr. Shapiro :

Proton exchange between e.g. OH, and NH groups has been studied widely in the past, mostly by saturation transfer, solvent saturation or other techniques. Within the context of some investigations concerned on peptides we were interested in proton exchange rates between NH groups and the solvent (H_2O). To unravel overlapping NH protons the selective HOHAHA technique proposed by the group of Bodenhausen came into use. To check the mathematics and the usefulness of a selective inversion instead of a presaturation, a selective inversion (G3) pulse and selective detection pulse, were applied onto a mixture of hydroxy-acetophenone and salicylic-aldehyde where the following exchange can occur.



The measured OH intensities of the salicylic aldehyde at different variable delays (0.001 - 30 s) were used to calculate the lifetime, of the hydroxylic group of the hydroxy-acetophenone and vice versa. The lifetimes, obtained from the fit procedure, are 9s for the hydroxy-acetophenone and 30s for the hydroxyl proton of the salicylic-aldehyde which is in good agreement with the data obtained by Forsen and coworkers. That shows that as long as pulse duration of a selective pulse is negligible compared to the relaxation time, selective techniques can be successfully applied.

Yours sincerely


 I. Krawanja


 H. Sterk

Dr. Barry Shapiro
TAMU Newsletter
966 Elsinor Court
Palo Alto, CA 94303
USA

LA TROBE UNIVERSITY CHEMISTRY DEPARTMENT

PS 3 Room 130
La Trobe University
BUNDOORA
Victoria 3083 Australia

PHONE: (03) 479 2582
FAX: (03) 479 1399
International: 61-3
E-mail: brownlee@latrobe.edu.au



Dear Barry:

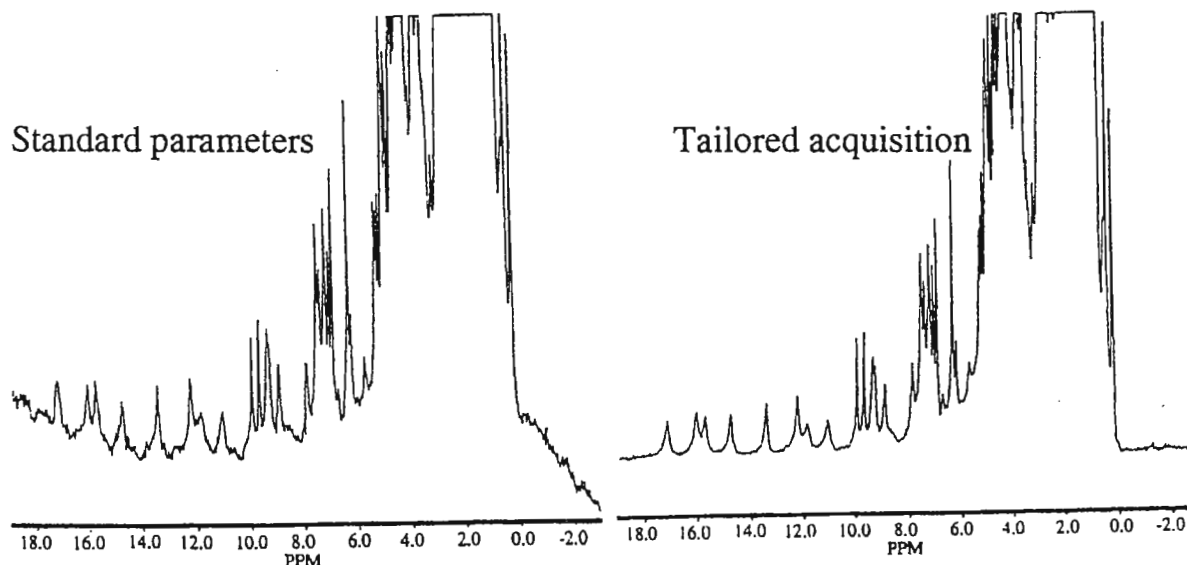
(received 8/5/93)

Flat baselines in 1D and Baseplanes in 2-D Spectra.

Although the standard default acquisition parameters in modern spectrometers are suitable for most samples there are a number of situations in which attention to detail can result in significant improvement of the experimental data resulting in the ability of detecting otherwise unseen peaks.

Our group is involved with the assignments of the contact shifted protons in paramagnetic proteins. Some of these peaks can be seen in the spectra of ferredoxin shown below at shifts from 10-18 ppm. The contact shifted peaks have short relaxation times and hence broad linewidths (100Hz) resulting in peaks of about 2% in height of the resonances from the diamagnetic protons. In 2-D spectra, the identification of cross peaks from these signals depends critically on the quality of data.

Part of the rolling baseline in 1-D spectra results from the distortion of the acquired signal by the filters in the spectrometer, and part is the result of using a real Fourier transform in our Bruker AM system required by the sequential acquisition mode. These problems can be overcome by a) resetting the receiver phase to zero prior to acquisition so that the spectrum requires no zero order phasing, and b) by choosing the dead time and filter width so that the spectrum requires no first order phasing and the spectrum has no baseline roll. In a 2-D spectrum any residual baseline slope can be corrected after F2 transformation.



The acquisition of data in this manner (tailored acquisition) requires considerable patience and care, but the results are worth the effort as can be seen from a comparison of the spectra shown below acquired without (standard parameters) and with (tailored acquisition) this procedure. It is to be hoped that a new generation of spectrometers will soon be available with better digitizers and distortionless filters.

Please credit this contribution to David Kellys subscription.

With best wishes,

Bob Brownlee

Bob

Maruse Sadek

ZODIAC SERIES

Multi-Purpose NMR Data Acquisition Systems

The **ZODIAC™** Series has been designed as a Real Time Data Acquisition System for Nuclear Magnetic Resonance (NMR) Applications. Each Zodiac unit integrates in the same chassis a pulse programmer, a signal averager, a general purpose Input/Output interface board, a spectrometer module and the exclusive **MacNMR™** software to control the spectrometer.

The Zodiac Series can be used for two different types of applications:

Scientists designing their own equipment can use the LIBRA or the ARIES in various configurations. Whether the experiment is Imaging, Liquid Spectroscopy or Solid State, there is a Zodiac Configuration tailored for that experiment.

Existing spectrometers can be upgraded easily, thanks to interfaces designed specifically to replace the original equipment. Upgrading with a Zodiac station can be usually accomplished within a few hours. Because of the Zodiac series design, the capability of switching back and forth between the old and the new configuration is maintained. This can become handy when old data need to be retrieved.

The Zodiac Series is based around Apple Macintosh (Centris or Quadra) computers, and as such, comes with Ethernet interface.



6006 Bellaire Blvd.
HOUSTON, TX 77081
Tel.: (713) 667-1507
Fax: (713) 667-3180

The **ZODIAC™** Series has been designed to offer an alternative to costly NMR computer equipment. This powerful data acquisition system with its numerous and versatile options can acquire virtually any NMR experiment, for example:

Solids	CP/MAS	CRAMPS	Liquids	NOESY	TOCSY
	wideline	DAS		COSY	HETCOR
	DOR	REDOR		inverse detection	
	solids imaging (nD)			triple resonance	
				4D or 4 channel experiments	
				Gradient Enhanced Spectroscopy	
Imaging (nD)	Multi-slice	Multi-echo			
	Grass	Flash			
	EPI	CSI			
	ISIS	Steam			

Both the **NMRkit-II™**, a multi-nuclei observe unit and the **DECKit-IV™**, a multi-nuclei decoupler unit can be added to a Zodiac unit to form part of a powerful NMR console.

Although not being an integral part of the system, the Macintosh computer can be purchased through Tecmag or directly from other vendors. All Zodiac systems can communicate to other systems via AppleTalk, Ethernet or through other LAN's.

The Zodiac series provides high speed flexible pulse programmers, fast computation (16 K complex floating point FFT in less than 700 ms), reliable high capacity hard disk, optical drives, laser printers, high resolution color printers and easy to use graphical NMR software. All the original functions necessary to control the spectrometer remain available. Each upgrade comes with a special "interface" window for all the commands unique to the spectrometer. All upgrades provide multi-dimension NMR capabilities.

SPECIFICATIONS	ARIES, TAURUS, LEO, miniMacSpect	LIBRA, SCORPIO, MacSpect
Minimum Pulse Width:	200 ns	100 ns
Maximum Pulse Width:	430 s	430 s
Time Resolution:	100 ns	100 ns (10ns with FPUL)
Loop Counters:	5	5 (10 with LXT)
No. of Steps:	2048	2048
No. of Control Lines:	20	76 (204 with LXT)
ADC Resolution:	12 bits	12 bits (16 bits option)
No. of Channels:	2 (simultaneous sampling)	2 (simultaneous sampling)
Min. Sampling Time:	5.0 μ s (1.0 μ s option)	up to 100 ns /channel
Max. Bandwidth:	200 kHz (1MHz option)	up to 10 MHz
Memory Size:	32 k-Word x 32 bits	128 k-Word x 32 bits
Minimum Macintosh Configuration		
Macintosh Computer with floating point unit (FPU) and at least 1 NuBus Slot (2 for GE/Nicolet)		
8 Mbyte CPU Memory or more. Operating System Software 7.0.1 or greater.		
12" monochrome, 14" color Monitor or better.		
80 Mbyte Hard Disk Drive. Any Macintosh compatible printer.		
Pen plotters require additional software such as MacPlot Pro or PLOTTERgeist.		



United States
Department of
Agriculture

Agricultural
Research
Service

Midwest Area
U.S. Dairy Forage
Research Center

1925 Linden Drive West
University of Wisconsin
Madison, WI 53706-1108

(608) 264-5407
E-Mail RALPHJ@MACC.WISC.EDU
August 4, 1993

(received 8/7/93)

Dr. B.L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303

Dear Dr. Shapiro,

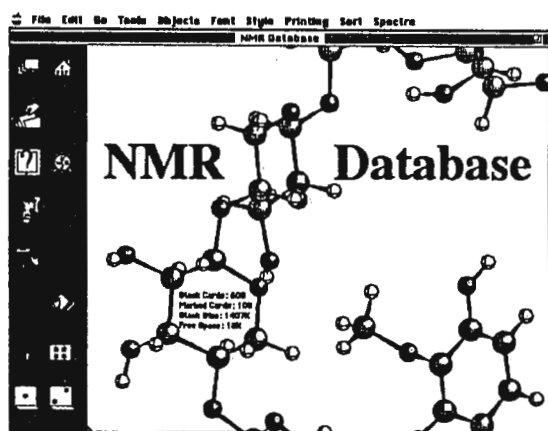
Release of a Plant Cell Wall Compound NMR Database

About 5 years ago, personnel at the US Dairy Forage Research Center and the US Forest Products Laboratory decided that an NMR Database of plant cell wall model compounds, with particular emphasis on lignin model compounds, would be created. This has been a need for quite some time and has been suggested by a number of groups at meetings for the last 15 years. Finally, we could wait no longer for someone else to do it.

Because of the agreement between chemical shifts of carbons in good model compounds and in the polymer itself, assignments in the complex lignin polymer have been substantially made from model compound data. However, lignin spectra are run in a variety of solvents, including DMSO and 9:1 acetone:water for underivatized lignins, and CDCl_3 and acetone for acetylated lignins. For that reason, and because ^{13}C chemical shifts can vary substantially with solvent, it was decided from the outset that all compounds would be run in the three solvents, acetone- d_6 , CDCl_3 , and DMSO- d_6 , and under a fixed set of conditions. Most spectra have been run routinely and assigned by reference to the literature and by comparison with related compounds in this database but we couldn't resist fully authenticating some of the more interesting models by the usual complement of COSY, long-range COSY, (perhaps TOCSY), HMQC, and HMBC experiments — it is noted in the database when fully unambiguous authentication has been performed. The result is that the database currently has some 200 compounds, and a rather large number simply entered from literature data. Some of these compounds and data are in advance of the literature; for example, the latest cell-wall cross-linking structures that have been synthesized in these laboratories are already included.

The database format itself underwent considerable development. We wanted a computer-based interactive system with considerable flexibility and some chemical intelligence. At the time, databases were considerably more boring than some of the emerging ones, and our choice at the time was to write it in Apple's HyperCard, to which anyone with a Mac has access. That turned out to be an excellent choice from the point of view of flexibility and customizability. We now retain that as our favored format but have ported the data to Claris FileMaker Pro, a cross-platform application for both MS Windows and Mac OS.

Anyway, we are about to release the Database to the general public. Until we add a little more functionality to the FileMaker Pro version, and address a few of the cross-platform issues, hopefully by the end of this year, it will be available only in HyperCard format and on the Internet, but the price is right — it is free! Users wishing to access our machine via Gopher or FTP on the internet should E-Mail me at RALPHJ@MACC.WISC.EDU so that I can give them access to our server. Documentation is provided in a format which does not require the creating application to view, print, and even steal from (i.e. selection of text and graphics for copying to other applications is supported). In the future we will be able to provide copies on diskette for either platform, and will provide hard copy versions too as required, but there may be a cost associated with these distribution methods. The Internet method will provide access to the continually changing database and is the preferred distribution. There will also be instructions for researchers to add compounds to their own database and to submit those entries (via an export feature) to us for inclusion in the official database. Data in this database can be cited in publications — this is a repository for data that will be available in the long term. We expect this database to grow substantially and to evolve into a complete tool for researchers in the plant cell wall chemistry area.



There are various viewing modes depending on whether you want to see intensities or not, proton data as well as ^{13}C data, or a line graph of the spectra. Being in HyperCard, the interaction possibilities with other applications (e.g. molecular modeling, spreadsheets) are endless. Obviously the database features the ability to search and sort by a variety of means, including directly from the structure itself; the data can be exported in a variety of forms; there is a direct

too many features to mention, from Top Card

card number

structure box

flag
("Marked" or blank)

compound names

compound description

ring type
(specific for Lignins)

NMR Database

101

Chemical structure of Guaiacylglycerol- β -guaiacyl ether. The structure shows a central glycerol backbone with two guaiacyl groups attached via ether linkages. The rings are labeled A and B. The structure is enclosed in a box with a flag icon.

Guaiacylglycerol- β -guaiacyl ether

1-(4-hydroxy-3-methoxyphenyl)-2-(2-methoxyphenoxy)propan-1,3-diol

Dimer	Ar OH	Ar OH	β OAr	C3
Ring A	B			
Ring B	B			
Ring C	...			

Ring A B

Ring B B

Ring C ...

J. Ralph JRB 178.8
30mg
J. Ralph Holzforschung
42(1968) p275-5

Assign	CDC13	Acetone	DMSO
OMe	55.86	56.20	55.35
OMe	55.94	56.27	55.52
Y	60.75	61.81	60.04
X	72.78	73.82	71.60
B	87.02	86.72	83.67
A2	108.81	111.38	111.35
B2	112.19	112.50	112.60
A3	114.31	115.12	114.48
A6	119.07	119.70	115.86
B5	120.57	120.45	119.43
B6	121.59	121.85	120.56
B1	124.00	123.32	120.89
A1	131.95	134.23	133.15
A4	145.09	146.61	145.35
A3	146.64	147.93	146.86
B4	146.89	149.04	148.00
B3	151.40	151.96	149.68

erythro

Chemical structure 100 Chemical structure 100

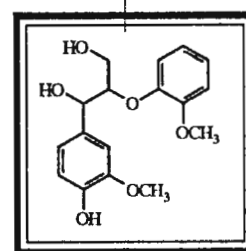
reference and notes

push-button stick spectra generation

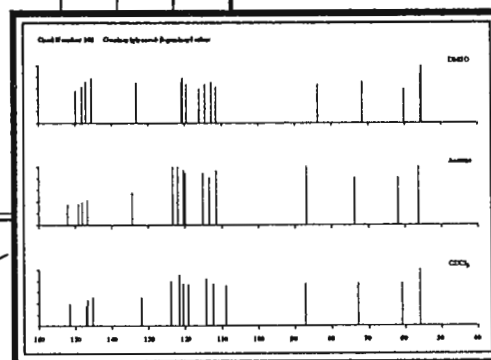
assignments

chemical shifts in three
different solvents

interactive structure
drawing (via commercial
packages)



isomer



connection with Chemistry drawing software; and there are many more features than can be described here. It should be quite easy to master, although a manual is provided and perhaps required for some of the less obvious features.

Sincerely,

John Ralph, William Landucci, Sally Ralph, and Lawrence Landucci.

POSITION AVAILABLE

I have further funding available for a good Ph.D. student with strengths or interests in Organic Synthesis, Plant Cell Wall Chemistry and, of course, NMR to do an exciting project in the area of plant cell wall cross-linking mechanisms. Please contact me at (608) 264-5407, FAX (608) 264-5275, or E-Mail at RALPHJ@MACC.WISC.EDU if you have an interest, want lots of easy-access NMR time, and enjoy cold winters. The US Dairy Forage Research Center is an equal opportunity employer.

Application Profile

Electrical



Description/Application

Radio-frequency coils for magnetic resonance imaging (MRI) and image-directed spectroscopy (IDS) equipment rely on TEFLON® PTFE fluoropolymer for support and insulation of conductors. MRI and IDS are valuable noninvasive techniques for showing soft tissue contrast in medical and biological examinations.

The coil's functions are twofold: to transmit pulses of radio-frequency energy to sample tissue and to pick up faint signals emitted by the tissue in response. It does not come into contact with the tissue.

The unique feature of the coil shown is the application of conductors by electroplating rather than adhesive bonding or lamination. The coil operates in the 100 to 600 MHz range with very high radio-frequency voltage, up to 2 kV. Current sizes range from about 3.50 to 5.25 in. (89 to 133 mm) high with flange diameters from 2.25 to 3.50 in. (57 to 89 mm).

Manufacturer

Polyflon Co., Div, of Crane Co., New Rochelle, NY
Phone: (914) 636-7222. Fax: (914) 633-3664.

Co-developer

Centre for Magnetic Resonance, University of Queensland
Brisbane, Australia. Phone: (61) (7) 365-4100. Fax: (61) (7) 365-3833.

Benefits Gained from this Development

Higher quality images. Compared with adhesive lamination, use of plated TEFLON reduces the potential for gaps and eliminates the need for adhesives, both of which would decrease reception sensitivity and increase background noise, according to Polyflon.

Faster imaging. Improved image quality reduces needs for image enhancement computations, saving time in examining patients.

Lower production costs. Plating and etching technology is less costly than alternative methods, Polyflon reports.

Material Chosen and Why

The excellent dielectric properties of TEFLON PTFE at radio frequencies and its nonmagnetic nature are critical to image quality.

**Start
with
DuPont**

H-51652



TAKING ADVANTAGE OF PURE PTFE'S QUALITIES IN RF & MICROWAVE APPLICATIONS

PTFE Provides High Performance

Electroplated PTFE meets the most demanding mechanical and electrical design requirements. Polyflon's unique electroplating process ensures a powerful molecular bond between the PTFE dielectric and conductive metal layer. Without voids or entrapments, this bond ensures products with no corona at the interface, for superb high-voltage performance.

Polyflon's CuFlon® Pure PTFE microwave substrates electroplated with copper, match the peel strength of copper clad laminates at 8 lb/sq. in. (min.). Custom applications utilizing this process typically have peel strengths exceeding 15 lb/sq. in.

Electroplated PTFE withstands continuous temperatures of 175°C and 225°C short term without damage to the plated material, dielectric, or the bond between them. This makes electroplated PTFE ideal for solder reflow and surface mount circuit processing, along with other high-temperature processing techniques.

Electroplated PTFE also performs well at low temperatures, even under cryogenic conditions. While most substrate materials become brittle below -196°C, PTFE remains ductile at -268°C — nearly the temperature of liquid helium. As a result, elec-

troplated PTFE is suitable for many superconductive designs, as well as circuits and systems in harsh environments, including avionics and space-related systems.

Why Pure PTFE?

Pure PTFE exhibits extremely low loss, with a dielectric constant of 2.06. This value does not change with temperature or frequency, permitting the design of stable broadband circuits with high performance over wide temperature ranges.

Pure PTFE features high surface resistivity, more than 10^{16} ohms/sq, and high volume resistivity, greater than 10^{18} ohm-cm. Its dissipation factor is less than 0.00045 from 1 to 3 GHz and 0.0003 up to and beyond 18 GHz. These characteristics account for pure PTFE's low leakage current and negligible power loss, even in high voltage applications.

The combination of pure PTFE and Polyflon's proprietary electroplating process produces materials that handle a wide range of applications, from specially shaped antennas and tiny, non-magnetic high voltage trimmer capacitors to medical imaging coils for NMR/MRI systems. Whatever the requirement, Polyflon's electroplated PTFE can fit your needs for today's applications and tomorrow's opportunities.

CRANE®

POLYFLON

Polyflon Company · 35 River Street · New Rochelle, N.Y. 10801 · Tel: (914) 636-7222 · FAX: (914) 633-3664

The technical data contained herein are guides to the use of DuPont resins. The advice contained herein is based upon tests and information believed to be reliable, but users should not rely upon it absolutely for specific applications since resin and adhesion properties will vary with processing conditions. It is given and accepted at user's risk and confirmation of its validity and suitability in particular cases should be obtained independently. The DuPont Company makes no guarantee of results and assumes no obligation or liability in connection with its advice. This publication is not to be taken as a license to operate under, or recommendation to infringe, any patents.

CAUTION: Do not use in medical applications involving permanent implantation in the human body. For other medical applications, see DuPont Medical Caution Statement," H-50102.



**Bruker
Instruments,
Inc.**

Fortune Drive
Manning Park
Billerica, MA 01821
(508) 667-9580
Fax: (508) 667-3954



Dr. B.L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303

(received 8/11/93)
Billerica, August 6, 1993

RF-GRASPTM: Gradient Spectroscopy with B_1 - Fields

Dear Barry,

We have recently developed an NMR probe which has in addition to a homogeneous RF coil, a coil which creates a gradient RF field. We are interested in using B_1 - gradients for coherence pathway selection in high resolution NMR spectroscopy. This interest is a reflection of the demonstrated power and versatility of B_0 - gradient methods, and the perceived promise of B_1 - gradient methods. B_1 - gradients share many of the capabilities of B_0 - gradients, and have some advantages including: (1) the gradient is **frequency selective** either for a given spectral line, or for a given nuclear spin, (2) B_1 - gradients have **no influence on the line shape**, (3) B_1 - gradients **do not disturb the lock channel**, (4) **fast switching** ($< 10 \mu s$), (5) more degrees of freedom, variables include the amplitude, length, phase and frequency of the gradient pulse, (6) **B_1 - gradients are non-secular**, so they do not commute with internal Hamiltonians. Internal Hamiltonians can be selectively suppressed by B_1 - gradients.

In addition, the hardware necessary for RF-GRASPTM experiments is quite modest, requiring only the RF-GRASPTM probe and coil switching electronics (see Figure 1). No additional amplifiers, and no modifications to the console are required.

In the design of an RF-gradient probe, one has to choose the symmetry of the B_1 - gradient field. The easiest spin response is obtained from a planar gradient field (see Figure 2), in which only the amplitude of the RF field is spatially dependent. In terms of RF efficiency a better solution is a quadrupolar coil, which creates a radial gradient field (Figure 2) and which has a higher filling factor. A quadrupolar RF gradient has some definite advantages over a planar gradient. The dephasing due to a planar gradient is in a plane perpendicular to the rotating frame axis along which the gradient RF field is applied. In the case of a quadrupolar RF gradient the dephasing is the result of both an amplitude and phase distribution, scattering the magnetization over the surface of a sphere rather than in a plane. Also due to the radial phase distribution the gradient phase does not have to be adjusted to the phase of the homogeneous coil. Finally, a radial RF gradient field, or any non-planar RF gradient field, can be converted into a planar spin response via multiple pulse cycles.

One of the earliest applications of B_0 -gradients was to select either the echo or anti-echo diagonal in COSY spectra¹. This is readily accomplished by introducing a spatially dependent phase shift prior to the mixing pulse so that the mixing pulse transforms both x and y components of the magnetization. A B_1 - version of this experiment² can be implemented by combining the B_1 - gradient into a composite z-pulse, which then influences the spin system exactly as would a B_0 - gradient. The gradient field generated in the RF-GRASP probe has

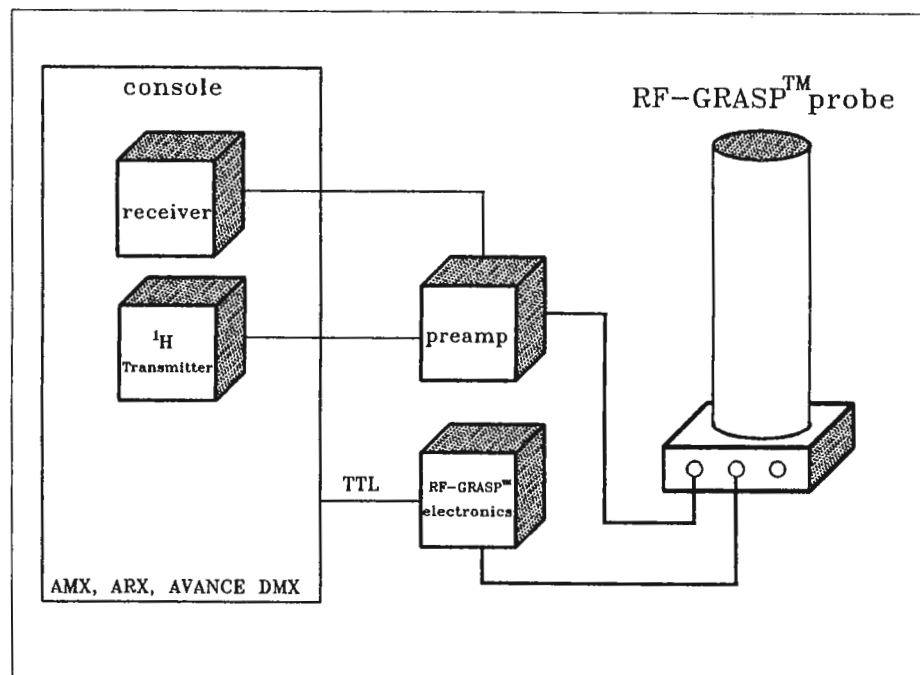
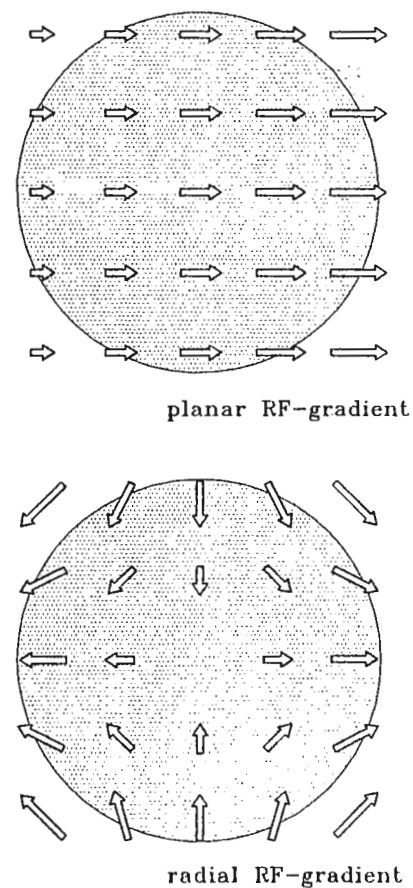
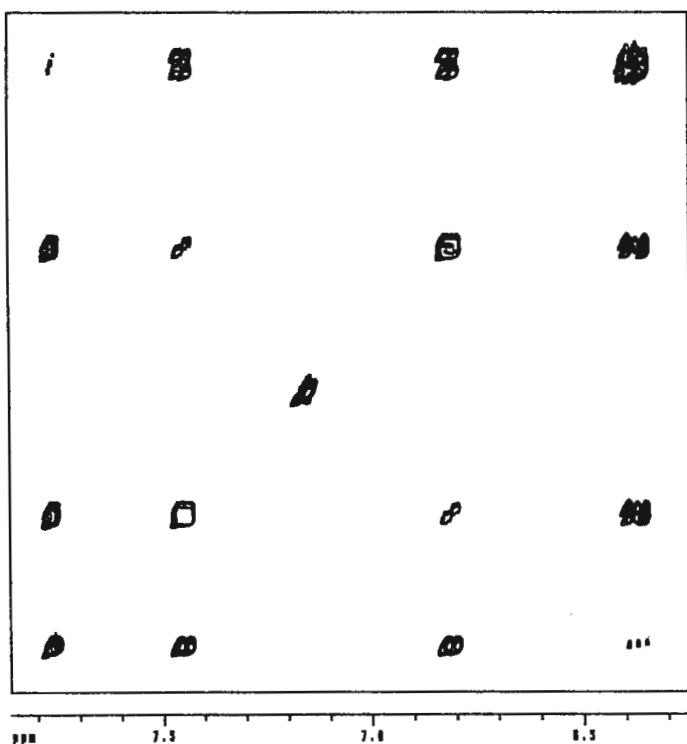
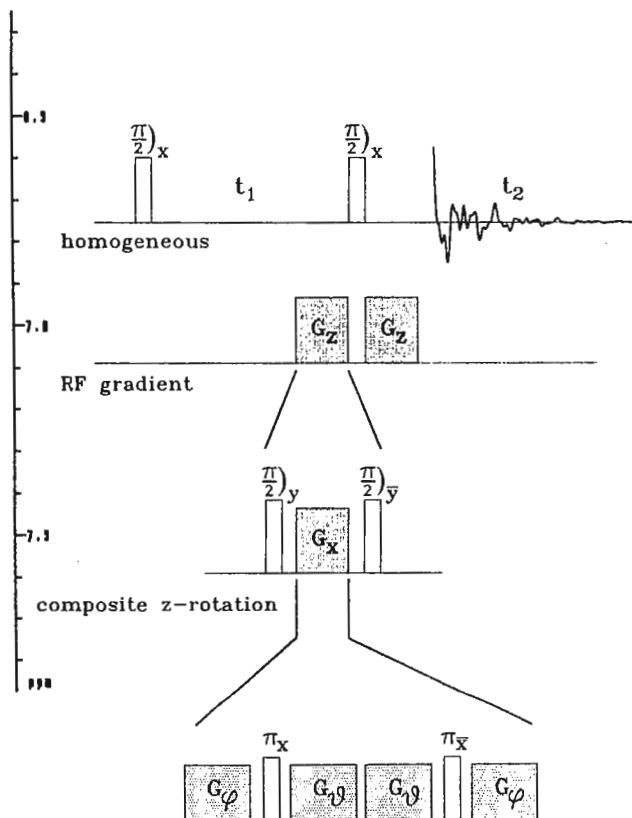
Figure 1: RF-GRASPTM set-up

Figure 2: Symmetry of RF-Gradient fields

Figure 4: 400MHz RF-GRASPTM N-type COSY spectrum of 1-chloro 3-nitrobenzene in deuterated benzeneFigure 3: RF-GRASPTM N-type COSY

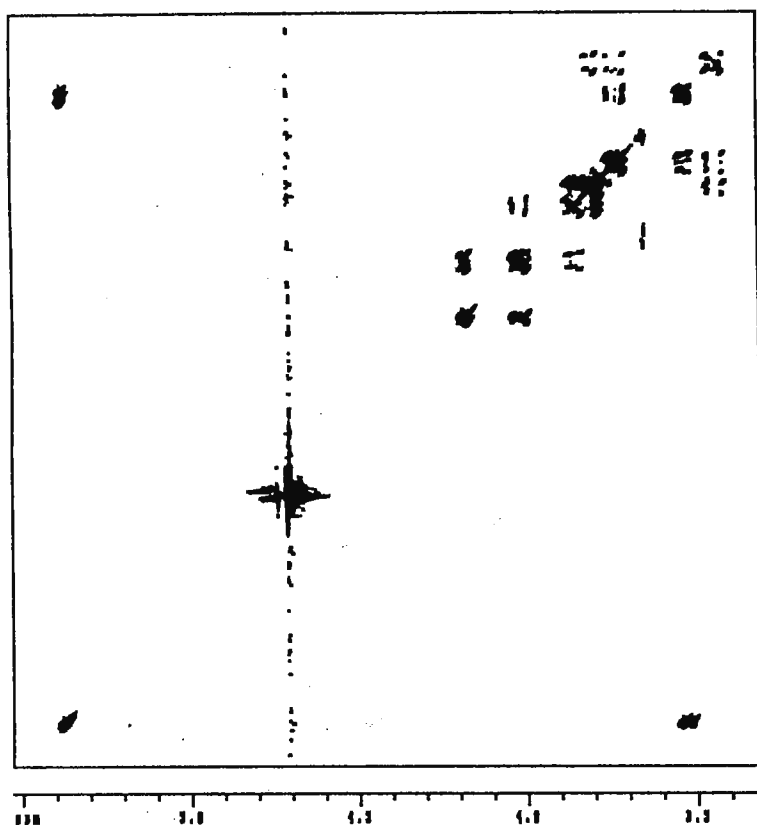


Figure 5: 400 MHz RF-GRASP DQF-COSY of sucrose in water.

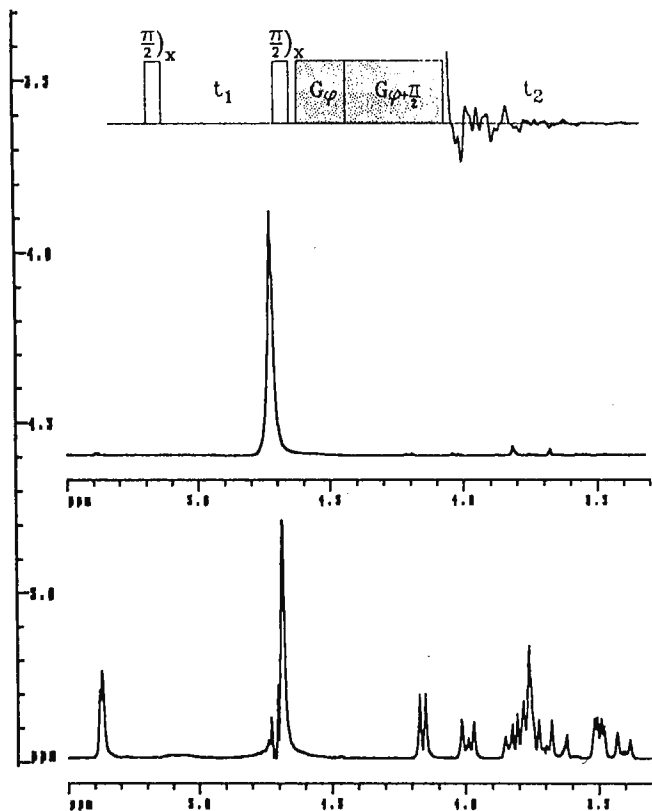


Figure 6: Pulse sequence of the RF-GRASP DQF-COSY experiment and the spectrum of sucrose in water compared to a projection of the DQF-COSY experiment.

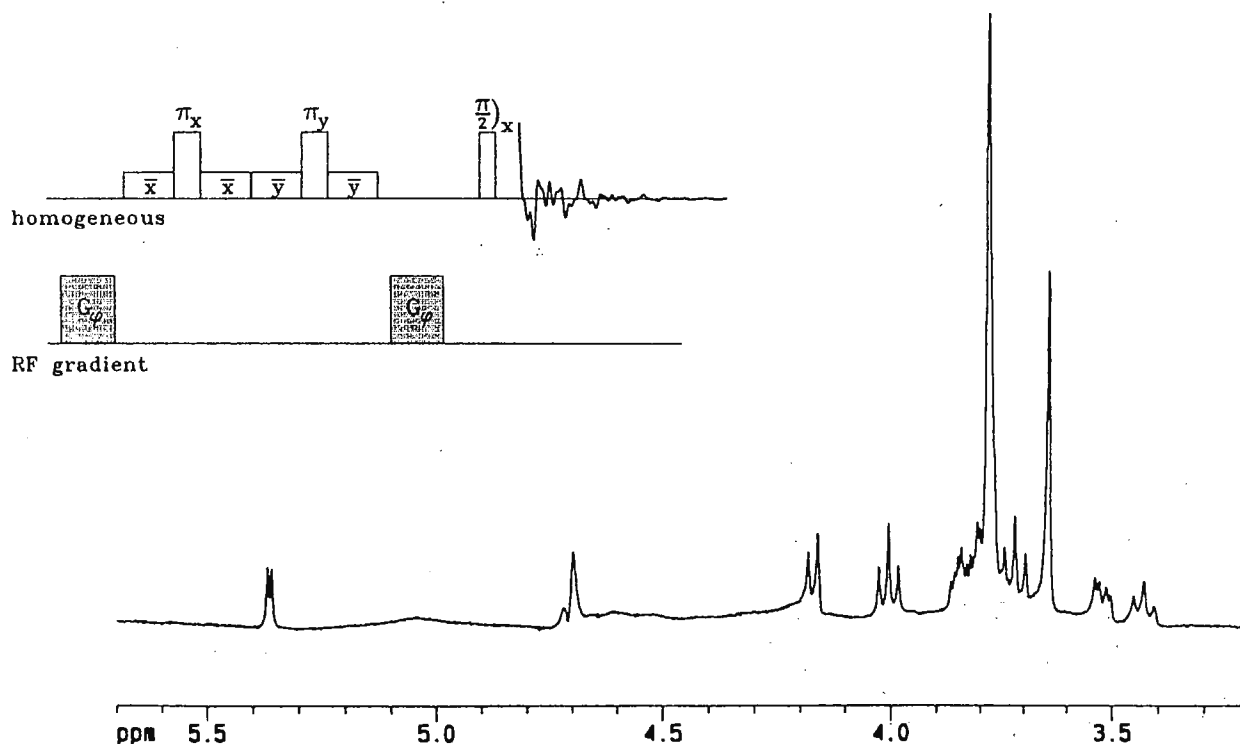


Figure 7: 400 MHz single scan proton spectrum of sucrose in water, obtained with the RF-GRASP water suppression sequence shown above.

a radial symmetry, and so the composite z-pulse is also used to convert this radial field into a planar field. Figure 3 shows the pulse sequence for the P- and N-type selected COSY experiment and the N-type 400 MHz proton cosy spectrum of 1-chloro 3-nitrobenzene in deuterated benzene is depicted in Figure 4. A single scan per t_1 increment is acquired. The RF gradient strength is 15 G/cm, and the total duration of the composite z-pulse is 4 ms.

One of the most exciting applications of GRASP has been in multiple quantum filtering of which an important homo-nuclear example is the double quantum filtered COSY experiment. An RF-GRASP DQF COSY experiment was introduced by Canet and coworkers³ for use with a planar RF gradient. In the case of a radial RF gradient field a small modification of the sequence is used⁴. In Figure 5 a phase-sensitive RF-GRASP DQF COSY spectrum is shown below for a sample of sucrose in water. The spectrum is acquired with a single scan per t_1 increment. The radial RF gradient pulse duration is 700 μ s, at a gradient strength of 15 G/cm. The degree of water suppression is best seen by comparing the one-pulse spectrum with a projection from the 2D dataset (Figure 6).

Recently a successful solvent suppression method was introduced, which combines pulsed field gradients with selective excitation⁵. We have adapted this so-called Watergate sequence to allow the use of B_1 -gradients⁶. The sequence consists of an RF gradient spin echo sequence in which the formation of an echo from the solvent resonance is prevented by applying a selective π -pulse in addition to a hard π -pulse. The suppression sequence can be performed with either planar B_1 -gradients or with radial B_1 -gradients (or indeed with any non-planar RF gradient). In the general case two orthogonal inversions are needed to recover the full echo intensity. The magnetization is refocussed along the z-axis and can be observed after a read-out pulse; alternatively this sequence can be incorporated in multi-dimensional experiments such as during the mixing time of a NOESY experiment. Figure 7 shows the suppression pulse sequence and a single scan 400 MHz proton NMR spectrum of sucrose in water. The RF gradient pulse is 500 μ s at a gradient strength of 15 G/cm. The selective $\pi/2$ durations are 2.8 ms.

These are a few examples of what we have accomplished with B_1 -gradients. We hope to report to you soon on new homonuclear RF-GRASPTM experiments, and also on the implementation of B_1 -gradients in heteronuclear spectroscopy.

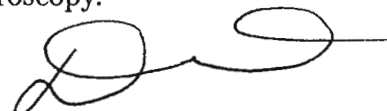
Best Wishes,



Werner E. Maas



Frank H. Laukien



David G. Cory
Dept. of Nuclear Engineering
Massachusetts Institute of Technology

¹ P. Barker and R. Freeman, *J. Magn. Reson.*, **64**, 334, 1985

² W.E. Maas, F.H. Laukien and D.G. Cory, *J. Magn. Reson.*, **A103**, 115, 1993

³ J. Brondeau, D. Boudot, P. Mutzenhardt and D. Canet, *J. Magn. Reson.* **100**, 611, 1992

⁴ D.G. Cory, F.H. Laukien and W.E. Maas, *J. Magn. Reson.* in press

⁵ M. Piotto, V. Saudek and V. Sklenár, *J. Biomol. NMR*, **2**, 661, 1992

⁶ W.E. Maas and D.G. Cory, submitted to *J. Magn. Reson.*



EXPLORE THE ALTERNATE ROUTE...!

QUALITY REFURBISHED NMRs

BRUKER . JEOL . VARIAN

PERMANENT MAGNETS . ELECTRO MAGNETS . CRYO MAGNETS

CURRENTLY IN STOCK: BRUKER NR-80, AM-300, AM-400

**VARIAN EM-360A, EM-360L,
EM-390, XL-200, VXR-400**

JEOL FX-90Q, GX-270

FOR FURTHER DETAILS CALL: (708) 913-0777

**International Equipment Trading Ltd.
960 Woodlands Parkway, Vernon Hills, IL 60061
FAX: (708) 913-0785**

Call Now for the Best Value!

New and Reconditioned
Analytical Instruments

INVENTORY UPDATE FALL 1993

X-RAY:

ARL 8680 Fluorescence
Cordell MXR-14 X-ray film processor
Stoe, Bragg-Brentano diffractometer
Tracor TX-5000

GAS CHROMATOGRAPHS:

Carlo Erba FID/NPD + A/S
HNU 311 PID
HP 5880 FID/TCD + 7672 A/S
HP 5880 TCD/FPD
HP 5880 dual FID
HP 3393 integrator
Hitachi D2000 Integrator
O.I. 4460A purge and trap
PE 8500 FID/NPD/ECD
PE 8320 FID
Shimadzu C-R3A integrator
Spectra Physics 4270 integrator
Tekmar LSC-II purge and trap
w/ ALS-10 autosampler
Varian 3400 FID/NPD
Varian 3700 dual FID capillary
Varian 3700 ECD/FID capillary
Varian Vista 6000 PID/FID w/ P&T
Varian Vista 6000 dual ECD
Varian 8035 autosampler

H.P.L.C.:

Beckman 112 solvent delivery w/ 421
gradient pump & controller, 340 organizer
Dionex 2010I Ion chromatograph
HP 1040M diode array detector
HP 1090M HPLC w/ Pascal workstation, A/S
& diode array detector
Waters 600 multi solvent system
Waters 710B Wisp autosampler
Waters 712B Wisp autosampler
Waters 510 pump
Waters 501 pump
Waters 490 multiwavelength detector
Waters Delta-prep 3000
Waters 481 uv-visible detector
Waters ILC-I Ion chromatograph
Waters Auto-500 Prep-LC

MASS SPECTROMETERS:

Finnigan 5100 GC/MS
Finnigan TSQ-70B triple quad
Finnigan Incos-50B
Finnigan TSQ-700 triple quad
Finnigan 806A Ion Trap w/Varian 3400 GC

HP 5970B w/ HP 5890A GC
HP 5971 w/ DOS, UNIX, jet separator, 7673 A/S
HP 5987 GC/MS w/ particle beam
HP 5988 w/ 7673 A/S
VG 70-250
VG ZAB 1F system

UV-VIS:

Beckman DU-50 w/Kinetics
Beckman DU-7 w/ A/S
Beckman DU-8B w/ Gel Scanner
Varian 2290

ATOMIC ABSORPTION:

ARL 3510 ICP
Baird PS-4 ICP
Baird PSX-18 ICP w/ IBM PS/2
IL Video 22
Jobin Yvon JY-70 Plus ICP
Leeman Labs PS-950 ICP
Leeman Labs PS-1 ICP
PE 603 w/ HGA-2100 + EDL
PE 2380
PE 5000 w/ HGA-500 & AS-40
PE Zeeman 5000
PE Zeeman 3030
PE Zeeman 4100ZL
PE HGA-400
PE HGA-500 w/ AS-40 A/S
PE HGA-600 w/ AS-60 A/S
PE AS-51 autosampler
PE 6500 ICP
PE Plasma 2000
PE Plasma 400
Spectro FMA-05 ICP
Varian Spectra 40 w/ GTA-96 & PSC-56
Varian Spectra 30
Varian 975 w/ GTA-95 & A/S
Varian 475 w/GF & A/S

DISPERSIVE IR:

PE 683
PE 781
PE 710B
PE 1421
PE 983G

FT-IR:

Bio-Rad FTS-60 GC
Mattson Cygnus 100 w/ IR plan microscope
Nicolet 20SX-B
Nicolet 740
PE1850 w/ IR plan microscope

RESONANCE SPECTROMETERS:

Bruker NR-80 NMR
Bruker AM-300 NMR
Bruker AM-400 NMR
JEOL FX-90Q NMR
JEOL GX-270 NMR
Varian EM-390 NMR
Varian EM-360L NMR
Varian VXR-400 NMR
Varian XL-200 NMR
Varian XL-400 NMR

ELECTRON MICROSCOPES:

Cambridge 120 SteroscanSEM
Hitachi S-520 SEM w/ Kevex
Hitachi S-2300 SEM w/ Kevex
ISI SS 40 SEM
Hitachi 600 AB TEM w/ Kevex
JEOL 1200 EX TEM w/ Kevex-Delta
Phillips CM-12 TEM w/ EDAX-9900

BIOTECHNOLOGY:

Applied Biosystems (ABI) Protein Sequencer 477A
w/120A PTH
ABI DNA Synthesizer 380B
ABI Peptide Synthesizer 430A
ABI Separation system 130A
Beckman L8-80 ultra centrifuge
Coulter EPICS C Flow Cytometer
Sorvall RC-2B
Sorvall RC-3B
Sorvall RC-5B
Sorvall OTD-65B ultra centrifuge

ELEMENTAL ANALYZERS:

Antek 707C nitrogen analyzer
Dohrmann DC-80 TOC analyzer
Dohrmann DX-20A TOX analyzer
Leco CS-244
Leco SC-132 auto sulfur determinator
Leco DH-103 hydrogen analyzer
PE 240C CHN

MISCELLANEOUS:

A.O. model 820 microtome
Fisher 490 coal analyzer
Kjeldahl 24-position apparatus
NELSON Lab automation system
Nova Analytical ISE analyzer
PE LS-3 fluorometer
PE DSC-4
Rheometrics RDS-7700 II
Technicon Auto Analyzer II



Reconditioned equipment carries a 90-day warranty for parts and labor unless
otherwise noted. The availability of all equipment is subject to prior sale.

International Equipment Trading, Ltd.

960 Woodlands Parkway • Vernon Hills, Illinois 60061

(708) 913-0777 Fax (708) 913-0785

SERVING THE
SCIENTIFIC COMMUNITY
FOR 13 YEARS
1980-1993

PEG

Precision Electronic Glass

ULTRA-PRECISION NMR SAMPLE TUBES



ACE GLASS *INCORPORATED*

Vineland NJ (Box 688) 08360 • (609) 692-3333 • Louisville KY (Box 996) 40201 • (502) 584-8144

FAX: 1-800-543-6752

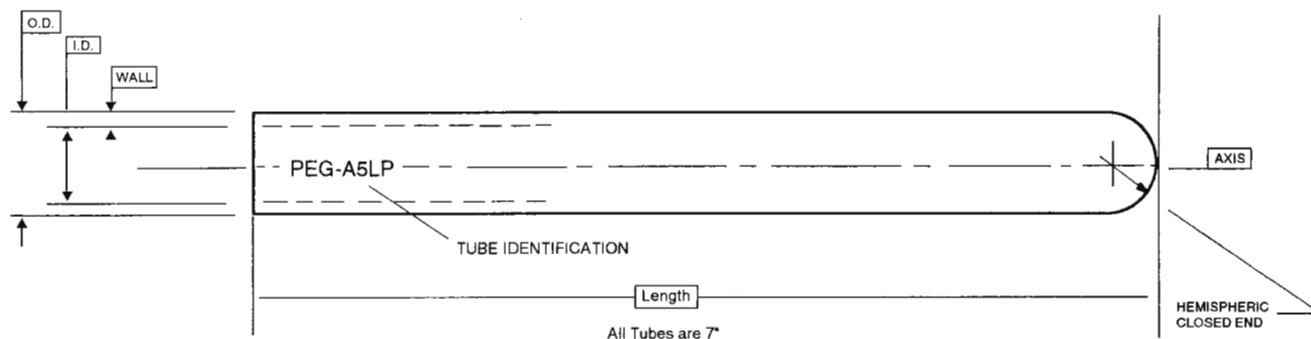
FAX: 1-800-635-4698

TOLL-FREE: 1-800-223-4524

TOLL FREE: 1-800-626-5381

TUBE SPECIFICATIONS

5 and 10mm x 7" length



ACE Cat. No.	Megahertz (MHz) Inst. Frequency	O.D. inches	O.D. Tolerance	I.D. inches	I.D. Tolerance	Concentricity I.D. to O.D.	Camber (Over 7" Lgth.)
16XXA5LP-07	>500	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0005"	≤ .00025"
160XA5LP-07	360	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0010"	≤ .0005"
1600A5LP-07	200	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0015"	≤ .0010"
1600B5LP-07	150	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0020"	≤ .0015"
1600C5LP-07	100	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0020"	≤ .0020"
16XA10LP-07	360	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0015"	≤ .0005"
160A10LP-07	150	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0020"	≤ .0010"
160B10LP-07	80	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0030"	≤ .0015"
160C10LP-07	60	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0050"	≤ .0020"
DISPOSABLE NMR TUBES							
1600A5LS-07	80	.1955	+ .0000"/- .0005"	.165	± .005	≤ .003	≤ .002
1600A5RS-07	60	.196	± .003	.165	± .005	≤ .005	≤ .002

All Tubes are 7" long. Other sizes are available, phone for information.

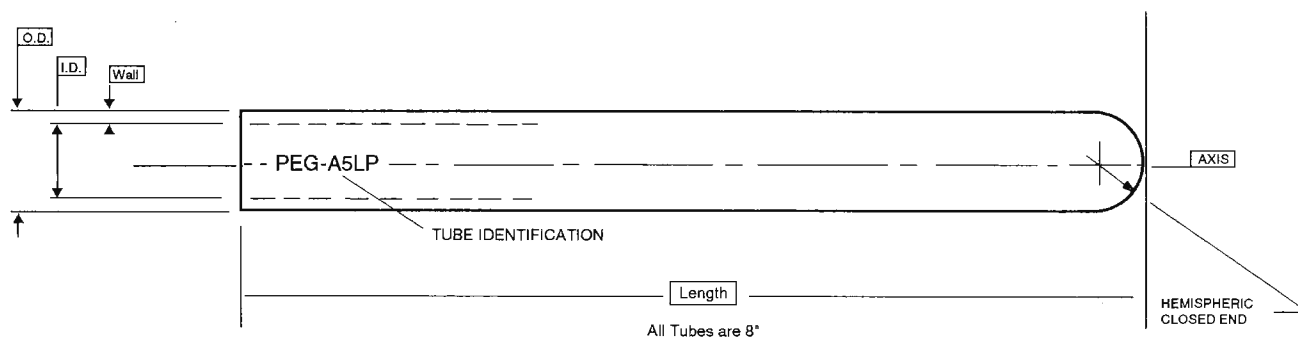
All Tubes supplied in Package Quantity of 5 or 25 pieces.

ACE Catalog No.	Tube Size O.D./Length	Per Pkg./5	Per Pkg./25
16XXA5LP-07	5mm/7"	★58.75	★293.75
160XA5LP-07	5mm/7"	★43.25	★216.25
1600A5LP-07	5mm/7"	★37.00	★185.00
1600B5LP-07	5mm/7"	★27.50	★137.50
1600C5LP-07	5mm/7"	★22.50	★112.50
16XA10LP-07	10mm/7"	★93.75	★468.75
160A10LP-07	10mm/7"	★78.75	★393.75
160B10LP-07	10mm/7"	★68.00	★340.00
160C10LP-07	10mm/7"	★51.25	★256.25
1600A5LS-07	5mm/7"	★17.50	★ 87.50
1600A5RS-07	5mm/7"	★ 8.00	★ 40.00

★ Net

TUBE SPECIFICATIONS

5 and 10mm x 8" length



ACE Cat. No.	Megahertz (MHz) Inst. Frequency	O.D. inches	O.D. Tolerance	I.D. inches	I.D. Tolerance	Concentricity I.D. to O.D.	Camber (Over 8" Lgth.)
16XXA5LP-08	>500	.1955	+.0000"/-.0005"	.1655	+.0005"/-.0000"	≤ .0005"	≤ .00025"
160XA5LP-08	360	.1955	+.0000"/-.0005"	.1655	+.0005"/-.0000"	≤ .0010"	≤ .0005"
1600A5LP-08	200	.1955	+.0000"/-.0005"	.1655	+.0005"/-.0000"	≤ .0015"	≤ .0010"
1600B5LP-08	150	.1955	+.0000"/-.0005"	.1655	+.0005"/-.0000"	≤ .0020"	≤ .0015"
1600C5LP-08	100	.1955	+.0000"/-.0005"	.1655	+.0005"/-.0000"	≤ .0020"	≤ .0020"
16XA10LP-08	360	.3937	+.0000"/-.0005"	.3569	±.0005"	≤ .0015"	≤ .0005"
160A10LP-08	150	.3937	+.0000"/-.0005"	.3569	±.0005"	≤ .0020"	≤ .0010"
160B10LP-08	80	.3937	+.0000"/-.0005"	.3569	±.0005"	≤ .0030"	≤ .0015"
160C10LP-08	60	.3937	+.0000"/-.0005"	.3569	±.0005"	≤ .0050"	≤ .0020"
DISPOSABLE NMR TUBES							
1600A5LS-08	80	.1955	+.0000"/-.0005"	.165	±.005	≤ .003	≤ .002
1600A5RS-08	60	.196	±.003	.165	±.005	≤ .005	≤ .002

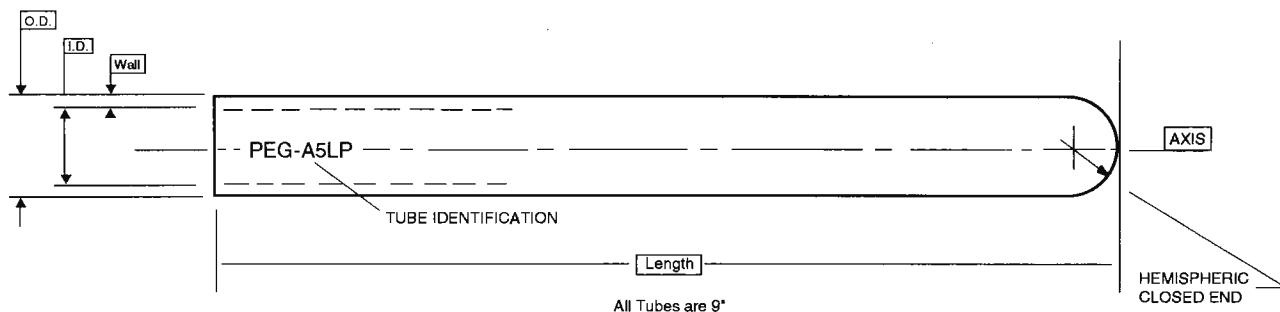
All Tubes are 8" long. Other sizes are available, phone for information.

All Tubes supplied in Package Quantity of 5 or 25 pieces.

ACE Catalog No.	Tube Size O.D./Length	Per Pkg./5	Per Pkg./25
16XXA5LP-08	5mm/8"	★64.75	★323.75
160XA5LP-08	5mm/8"	★47.75	★238.75
1600A5LP-08	5mm/8"	★40.75	★203.75
1600B5LP-08	5mm/8"	★30.25	★151.25
1600C5LP-08	5mm/8"	★24.75	★123.75
16XA10LP-08	10mm/8"	★97.50	★487.50
160A10LP-08	10mm/8"	★83.75	★418.75
160B10LP-08	10mm/8"	★72.25	★361.25
160C10LP-08	10mm/8"	★56.25	★281.25
1600A5LS-08	5mm/8"	★19.25	★ 96.25
1600A5RS-08	5mm/8"	★ 8.50	★ 42.50

TUBE SPECIFICATIONS

5 and 10mm x 9" length



ACE Cat. No.	Megahertz (MHz) Inst. Frequency	O.D. inches	O.D. Tolerance	I.D. inches	I.D. Tolerance	Concentricity I.D. to O.D.	Camber (Over 9" Lgth.)
16XXA5LP-09	>500	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0005"	≤ .00025"
160XA5LP-09	360	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0010"	≤ .0005"
1600A5LP-09	200	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0015"	≤ .0010"
1600B5LP-09	150	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0020"	≤ .0015"
1600C5LP-09	100	.1955	+ .0000"/- .0005"	.1655	+ .0005"/- .0000"	≤ .0020"	≤ .0020"
16XA10LP-09	360	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0015"	≤ .0005"
160A10LP-09	150	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0020"	≤ .0010"
160B10LP-09	80	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0030"	≤ .0015"
160C10LP-09	60	.3937	+ .0000"/- .0005"	.3569	± .0005"	≤ .0050"	≤ .0020"
DISPOSABLE NMR TUBES							
1600A5LS-09	80	.1955	+ .0000"/- .0005"	.165	± .005	≤ .003	≤ .002
1600A5RS-09	60	.196	± .003	.165	± .005	≤ .005	≤ .002

All Tubes are 9" long. Other sizes are available, phone for information. All Tubes supplied in Package Quantity of 5 or 25 pieces.

ACE Catalog No.	Tube Size O.D./Length	Per Pkg./5	Per Pkg./25
16XXA5LP-09	5mm/9"	★ 71.25	★356.25
160XA5LP-09	5mm/9"	★ 52.50	★262.50
1600A5LP-09	5mm/9"	★ 45.00	★225.00
1600B5LP-09	5mm/9"	★ 33.25	★166.25
1600C5LP-09	5mm/9"	★ 27.25	★136.25
16XA10LP-09	10mm/9"	★101.75	★506.25
160A10LP-09	10mm/9"	★ 88.75	★443.75
160B10LP-09	10mm/9"	★ 76.50	★382.50
160C10LP-09	10mm/9"	★ 62.00	★310.00
1600A5LS-09	5mm/9"	★ 22.25	★111.25
1600A5RS-09	5mm/9"	★ 9.00	★ 45.00

★ Net



ACE GLASS INCORPORATED

Vineland NJ (Box 688) 08360 • (609) 692-3333 • Louisville KY (Box 996) 40201 • (502) 584-8144

FAX: 1-800-543-6752

FAX: 1-800-635-4698

TOLL-FREE: 1-800-223-4524

TOLL FREE: 1-800-626-5381



THE UNIVERSITY OF MANITOBA

DEPARTMENT OF CHEMISTRY

Winnipeg, Manitoba
Canada R3T 2N2

August 10, 1993 (received 8/17/93)

Tel: (204) 474-9321
FAX: (204) 275-0905
TELEX: 07-587721Dr. Bernard L. Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303
U.S.A.

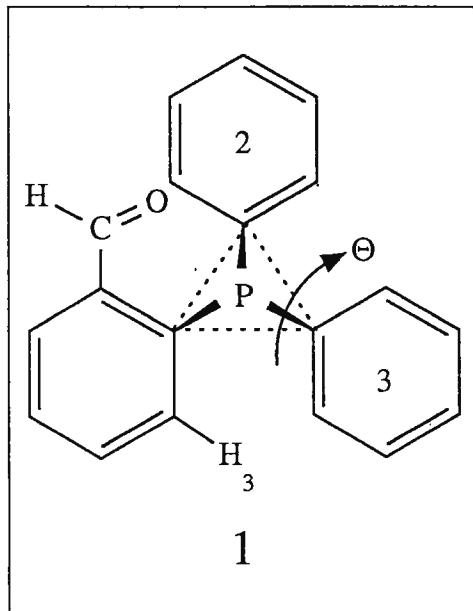
Dear Barry,

Some Properties of Triphenylphosphine (TPP) and 2-(Diphenylphosphino) benzaldehyde (DPPB).

Some time ago the very old news, that TPP and its relatives are ubiquitous as ligands, and so forth, penetrated even my cloister. Some desultory reading implied that a precise analysis of the ^1H nmr spectrum was not available, other than some early ones in benzene solution (ASIS!), and that an intramolecular rotational potential was also unknown or, at least, unpublished for TPP.

Our efforts in this matter led to the following conclusions (Can. J. Chem. 71, 639 (1993): a) the shift range of the phenyl protons is only 1.7 Hz at 300 MHz in $\text{CS}_2/\text{C}_6\text{D}_{12}$ solution at 300 K; b) the rotation about the P-C bonds is hindered by a potential comparable to kT at 300 K; c) the diamagnetic anisotropy of the two phenyl groups causes a negligible shift of the para proton in the third phenyl group; d) the lone-pair on phosphorus will conjugate with a single phenyl group about 30% as well as a lone-pair on nitrogen under optimum conditions, that is, when it lies in a plane perpendicular to the phenyl group e) in TPP the conjugation involves three phenyl groups which are not oriented on average for optimal conjugation - the model and experiment agree as to the observed shifts; f) yes, a reliable analysis is possible if your linewidths are small enough.

Encouraged by these 'successes', Rudy Sebastian managed a full ^1H nmr analysis of DPPB in CS_2 and in acetone solutions and Rob Schurko ran the $^{13}\text{C}\{^1\text{H}\}$ spectra as well. Many relative signs followed from partial decoupling experiments. For each solution one then has 55 chemical shifts and coupling constants, from which a number of useful conclusions can be drawn (see



Can. J. Chem. much later this year). Among them are: a) in the crystal, the C=O bond lies cis to P, whereas in solution the trans conformer predominates and reaction-field theory in first-order cannot account for the relative proportions of cis and trans in the two solvents; b) $^5\text{J}(\text{H}, \text{P})$ can change its sign from ring to ring; c) $^4\text{J}(\text{CHO}, \text{P})$ is -7.1(2) Hz in the trans conformer and $^3\text{J}(\text{CHO}, \text{P})$ is +29.4(1.3)Hz, a result of interest in the computation of couplings via the $\text{O}=\text{C}-\text{H}\cdots\text{l.p. P}$ interaction; d) P is electrically polarized such that the C=O bond is attracted towards a 'positive' region around P, while the C-H bond of the formyl group prefers the lone-pair region of P - all quite sensible in electrostatic terms; e) $^1\text{J}(\text{C}, \text{P})$ in TPP is solvent dependent (10%).

Best wishes from

Ted Schaefer

TS/1

P.S. What is that color of your "Reminder" sheet? It's not pansy-purple, Perkin's violet or madder-violet. Is it, possibly, pontiff-purple?

Ted - The official name of the color is, believe or not, "orchid". I chose the color for the R/U notices because it was the ugliest shade available. I thought this might lower the residence time on the desks of the almost delinquent. Hope this satisfies your insatiable thirst for useless knowledge! With best regards, *Barry*

Table of Contents, cont'd.

Release of a Plant Cell Wall Compound Database	Ralph, J., Landucci, W., Ralph, S., and Landucci, L.	37
Position Available	Ralph, J.	38
RF-GRASP™: Gradient Spectroscopy with B ₁ -Fields	Maas, W. E., Laukien, F. H., and Cory, D. G.	41
Some Properties of Triphenylphosphine and 2-(Diphenylphosphino)-benzaldehyde	Schaefer, T.	51



**All Newsletter correspondence
should be addressed to**

Dr. B. L. Shapiro
966 Elsinore Court
Palo Alto, CA 94303 U.S.A.

(415) 493-5971 - *Please call
only between 8:00 am and
10:00 pm, Pacific Coast time.*

Deadline Dates

No. 422 (November)	22 October 1993
No. 423 (December)	19 November 1993
No. 424 (Jan. 1994)	17 December 1993
No. 425 (February)	21 January 1994



The Newsletter's fiscal viability depends very heavily on the funds provided by our Advertisers and Sponsors. Please do whatever you can to let them know that their support is noted and appreciated.

Mailing Label Adornment: Is Your Dot Red?

If the mailing label on your envelope of this issue is adorned with a large **red dot** or circle: this decoration means that you will not be mailed any more issues until a technical contribution has been received by me.



Page Length Instruction

Attention overseas subscribers: If you must use paper which is longer than 11", *please* take care that all material (including signatures, addresses - everything!) ends no more than 10" from the top of each of your pages. It is costly to make reductions, and henceforth I reserve the right to chop the excess length off any page, no matter what the result. Beware of the dreaded guillotine! Your cooperation in this matter will be greatly appreciated.

The Leader

*in stable isotope
labeled compounds
& research products*



13

C

World's largest isotope separation facility for the production of Carbon-13 and Carbon-12

D

An inventory of more than 6000 products including; the MSD inventory, NMR solvents, biomedical products, specialty gases, and environmental standards

A fully staffed Technical Services Dept. to provide information on custom syntheses and general technical assistance

15

N

CIL

*Please call for product information
and to receive a catalog*

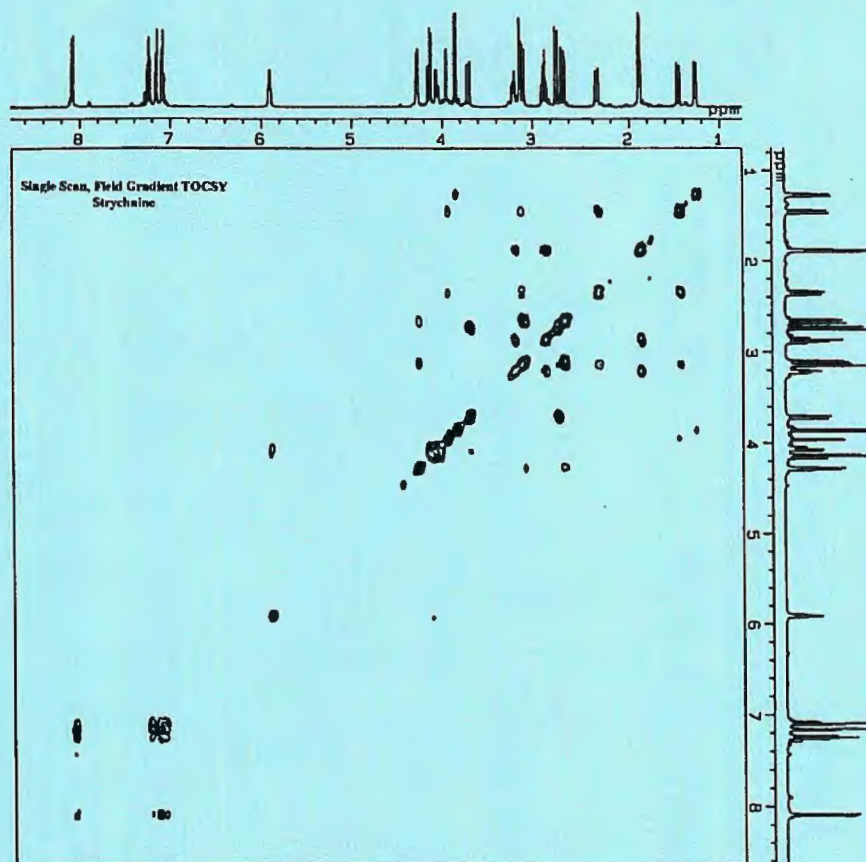
CAMBRIDGE ISOTOPE LABORATORIES
20 Commerce Way, Woburn, Massachusetts 01801

800-322-1174 or 617-938-0067 (USA) 617-932-9721 (Fax) 800-643-7239 (Canada)

α NMR

FROM JEOL USA

Gradient Probes



The above data is a 512 by 512 TOCSY spectrum and was obtained on an Alpha- 500 in less than 5 minutes with JEOL's gradient probe. This same probe is also designed to do reverse and water suppression experiments. Pulse shaping, three channel operation, dynamic Rf control, matrix shims, as well as unsurpassed field reliability are all standard with the Alpha.

ALPHA FIRST IN PERFORMANCE, FIRST IN TECHNOLOGY

JEOL, USA
11 Dearborn Rd.
Peabody, MA 01960
508/535-5900
FAX 508/535-7741

JEOL, NJ
23 E. Brunswick Dr
East Brunswick, NJ
908/254-7028
FAX 908/254-8850

JEOL, CA
3500 West Bayshore Rd
Palo Alto, CA 94303
415/493-2800
FAX 415/493-0581

JEOL, IL
9801W. Higgins Rd Suite 220
Rosemont, IL 60018
708/825-7164
FAX 708/696-2559



Soquelec, Ltd.
5757 Cavendish Blvd
Suite 101
Montreal, Quebec
Canada H4W2W8
514/482-6427
FAX 514/482-1929

JEOL, DE MEXICO
Insurgents Sur #953
Col. Napoles
03810 Mexico D.
525/515-2816
FAX 525/515-3261

