TEXAS A&M UNIVERSITY

NMR NEWSLETTER

No. 422

November 1993

Temperature Mapping and Chemical Shift Imaging . **Briguet, A., Fenet, B.,** and **Zaim-Wadghiri, Y.** 2

High Resolution, High Field, Rodent Cardiac Imaging with Flow Artifact Suppression . . . . . **Rose, S., Zelaya, F., Crozier, S., Wilson, S.,** and **Doddrell, D. M.** 5

35th ENC (Experimental NMR Conference), Pacific Grove, California, April 10-15, 1994 . **Blümich, B.** 9

Computer Program for Analyzing Protein Primary Sequence by Spin Systems . . . . . **Ingalls, C., Omecinsky, D., Reily, M. D.,** and **Thanabal, V.** 13

Dependence of CP and INEPT Transfer Efficiencies on RF Inhomogeneity: A Computer Simulation . . . . . **Majumdar, A.,** and **Zuiderweg, E. R. P.** 17

Differentiation of Intra- from Intermolecular ^{15}N-Filtered NOEs . . . . . **Emerson, S. D.** 21

Uncommon Sugars in Polysaccharides by NMR Spectroscopy . . . **Reddy, G. P.,** and **Bush, C. A.** 25

Fixing 2D Baselines with Felix . . . . . **Ellena, J.,** and **Rule, G.** 26

Determination of Coupling Constant Signs in Halothane . . . . . **Ribeiro, A. A.** 29

Anisotropic Solvent Shifts in Anisotropic Media . . . . . **Diehl, P.** 30

Accurate Measurement of T_1 Relaxation Time in the Presence of Chemical Exchange . . . . . **Horská, A., Horský, J.,** and **Spencer, R. G. S.** 33

Indirect Detection wth Tecmag System on a GN-300 . **Berghmans, K., Gasparovic, C.,** and **Theisen, L.** 34

Vile Vials and Crappy Caps . . . . . **Miner, V. W.,** and **Conover, W. C.** 37

Continued on page 50

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. Iso. (Deuterium)	Density (g/cc)	MP (°C)	BP (°C)	$-\chi_v \times 10^6$ @ (°C)
D-11	Acetone-d_6	CD_3COCD_3	99.5%	1.12	-97	56	0.551 (32)
D-120	Acetone-d_6 + 1% TMS						
D-13	Acetone-d_6	CD_3COCD_3	99.8%	0.87	-94	57	0.460 (20)
D-121	Aceonte-d_6 + 1% TMS		99.8%				
D-129							
D-14							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%				

Need Deuterated Solvents?
WILMAD HAS YOUR SOLUTION!

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_2O, 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 Trifluroacetic Acid-d.

VARIAN

BOX/500 SHEETS

Cat. No.	Size	Instrument Model	Type
WCV-100 (S-100A)	11" X 26"	A-100, HA-100, HA-100D	Gridded-Two Color
WCV-60 (S-60C)	11" X 26"	A-60, A-60A, A-60D	Gridded-Two Color
WCV-XL (XL-100)	11" X 26"	XL-100	Gridded-Two Color
WCV-XLFT-100	11" X 26"	XL-100 (FT and FT)	Gridded-Two Color
WCV-XL-200	11" X 26"	XL-200, XL-300, XL-400	Gridded-Two Color
WCV-20 (CFT-20)	11" X 16 3/4" X 17"	CFT-20, FT-80, FT-80A All Models	Gridded-Two Color Blank

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.

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

Berghmans, K. . . 34
Berlin, K. D. . . 48
Blümich, B. . . . 9
Briguet, A. . . . 2
Brown, D. E. . . 49
Bush, C. A. . . . 25
Conover, W. C. . . 37
Crozier, S. . . . 5
Diehl, P. . . . 30
Doddrell, D. M. . . 5
Ellena, J. . . . 26
Emerson, S. D. . . 21
Ezell, E.. . . . 41
Fenet, B. 2
Gasparovic, C. . . 34
Griffith, G. L. . . 42
Holbrook, R. . . 47
Horská, A. . . . 33
Horský, J. . . . 33
Ingalls, C. . . . 13
Majumdar, A. . . 17
Matson, G. . . . 47
McCabe, M. A. . . 42
Miner, V. W. . . 37
Omecinsky, D. . . 13
Quast, M. . . . 41
Ray, B. D. . . . 42
Reddy, G. P.. . . 25
Reily, M. D.. . . 13
Ribeiro, A. A. . . 29
Rose, S.. . . . 5
Rule, G. . . . 26
Spencer, R. G. S . . 33
Thanabal, V. . . 13
Theisen, L. . . . 34
Wassall, S. R. . . 42
Weber, P. . . . 45
Wilson, S. . . . 5
Wilson, D. M. . . 48
Zaim-Wadghiri, Y. . 2
Zelaya, F. . . . 5
Zuiderweg, E. R. P. . 17

American Microwave Technology 7
Bruker Instruments, Inc. 3, 31
Cambridge Isotope Laboratories, Inc. . . . . inside back cover
Chemagnetics 15
Hitachi Instruments, Inc. 19
Isotec, Inc. 39
International Equipment Trading, Ltd. 43
JEOL . . . . . outside back cover
Oxford Instruments Ltd. 23
Shigemi, Inc. 35
Varian 11, 27
Wilmad Glass Company, Inc. . . . . inside front cover

SPONSORS OF THE TAMU NMR NEWSLETTER

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

FORTHCOMING NMR MEETINGS

International Symposium on Biological NMR, On the Occasion of Professor Oleg Jardetzky's 65th Birthday, Stanford California, **March 24 - 26, 1994**; Contact: Ms. Robin Holbrook, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, California 94305-5055; Fax: (415) 723-2253; See TAMU NMR Newsletter 422, 47.

Symposium on *In Vivo* Magnetic Resonance Spectroscopy VII, Monterey, California, **April 9 - 10, 1994**; Contact: Radiology Postgraduate Education; Room C-324, University of California School of Medicine, San Francisco, CA 94143-0628; Phone: (415) 476-5731; Fax: (415) 476-9213; For registration, call (415) 476-5808; Fax: (415) 476-0318 See TAMU NMR Newsletter 422, 47.

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 See TAMU NMR Newsletter 422, 9.

8th International Symposium on Molecular Recognition and Inclusion, Ottawa, Ontario, Canada, **July 31 - August 5, 1994**; Contact: H. Morin-Dumais, Steacie Institute for Molecular Sciences, National Research Copuncil of Canada, 100 Sussex Drive, Ottawa, ON K1A 0R6, Canada; (613) 993-1212; Fax: (613) 954-5242 See TAMU NMR Newsletter 419 34

Gordon Conference on Order/Disorder in Solids, New London, New Hampshire, **August 7 - 12, 1994**; Contact: Prof. M. A. White, Dept. of Chemistry, Dalhousie University, Halifax, Nova Scotia, Canada B3H 4J3; Tel. (902) 484-3894; Fax: (902) 494-1310. See TAMU NMR Newsletter 421, 44.

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. Bubb, Secretary, Univ. of Sydney, Dept. of Biochemistry, Sydney, NSW 2006, Australia. See TAMU NMR Newsletter 419, 26.

Additional listings of meetings, etc., are invited.

LABORATOIRE DE RÉSONANCE MAGNÉTIQUE NUCLÉAIRE
UNIVERSITÉ CLAUDE BERNARD LYON I

Bâtiment 721
43, boulevard du 11 Novembre 1918
69622 Villeurbanne Cedex
Tél. 72.44.82.67 - Secr. 72.44.80.84
Fax (33) 72.44.81.99

Villeurbanne, le 12 Octobre 1993

(received 10/21/93)

Professor B.L. SHAPIRO
Editor Publisher TAMU NMR Newsletter
966 Elsinore Court
PALO ALTO CA 94 303 (USA)

TEMPERATURE MAPPING AND CHEMICAL SHIFT IMAGING

Dear Professor Shapiro,

For some time it has been recognised that ^{59}Co could provide a valuable temperature probe[1,2]. Indeed the chemical shift dependence of ^{59}Co may exceed 1.5 ppm/K and it can be turned into profit to get temperature records. To obtain spatial temperature distribution, the most attractive fashion is to employ the well known spectroscopic imaging approach. We report here one of our first experiments performed on a AM300 Bruker system equipped with a gradient unit. Figure 1 shows the temperature distribution across a 5 mm od, 46 mm height [$K_3(Co(CN)_6)$ / D_20] sample. Data were acquired during continuous excitation at proton frequency.

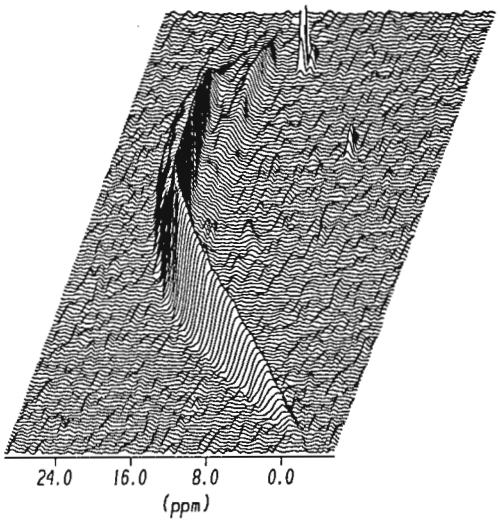


Figure 1.Stacked plot of 2D (1D spatial, 1D spectral) image of ^{59}Co at "room" temperature when CW ^{1}H decoupling(3 Watt) is on. 16 averages 64 (space) x 128 (chemical shift) pixels. Temperature to chemical shift correspondance : 1.34 ppm/K.

This image shows well that the temperature dispersion is almost constant along the sample direction. Notice that other approaches for temperature control have been presented in TAMU Newsletter : 365-34 (B.D. Sykes), 376-16 (M. Hajek and F. Bohm), 419-25 (R. Chapman, H. Gilboa, P.W. Kuchel) if we are right.

Please accept our best regards,

A. BRIGUET B. FENET Y. ZAIM-WADGHIRI

[1] G. C. Levy, J. T. Bailey, D. A. Wright, *J. Magn. Reson.* **37**, 353 (1980)
[2] D. Hoepfel, H.P. Juretschke, S.M.R.M. 7th Annual Meeting, Abstracts p. 1074 (1988).

2.5 mm Microprobe Family GRadient Accelerated SPectroscopy

In the past various attempts have been made in the NMR industry to design probes for milli-, micro- and even high nanogram amounts of sample. However, these probes typically suffer from a number of drawbacks which render them impractical to use. Some designs use solenoidal coils which make it impossible to load/eject the sample at the top of the magnet; instead the probe must be removed from the magnet to change the sample.
Moreover, these probe designs are useful primarily for ^{1}H-only coils, but cannot be readily built as broadband inverse or inverse triple-resonance probes. Finally, lineshapes for older microsample probes typically were unsatisfactory for biological NMR experiments which require water suppression.

BRUKER INTRODUCES a new and unique series of 2.5 mm probes for microsample applications with optional Z-gradient.

Our user friendly design offers:

- **sample insertion and ejection without probe removal**
- **single frequency, doubly tuned or broadband decoupling coils**
- **excellent lineshape, water suppression and sensitivity**
- **sample volumes of 80 – 100 microliters**

With optional Z-gradient:

- **GRASP techniques applicable GRadient Accelerated SPectroscopy**
- **gradient strength of 6 G/cm/A**
- **complete suppression of t_1-noise and artifacts for GRASP experiments**
- **significantly boosted throughput due to GRASP techniques**

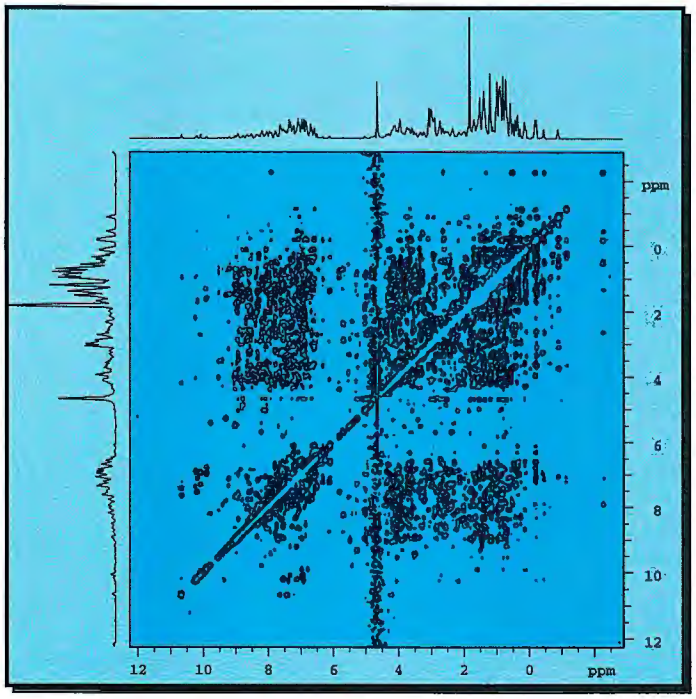


Figure 1: NOESY experiment of 1 mg of Lysozyme in 0.1 ml of 90% H_2O/ 10% D_2O. Mixing time 150 msec, 64 scans were recorded for each of 512 t_1-increments. Experiment time 12h 15 min, 2.5 mm TXI probe.

BRUKER

Comprehensive Support for Innovative Systems.

Due to the very user friendly design of our 2.5 mm Microprobes, sample insertion and ejection is the same as that for all standard 5 mm or 10 mm probes. This unique advantage allows the use of our new Microprobe Family even in completely automatic operation.

2.5 mm Microprobes are available from 200 MHz up to 750 MHz in the following designs:

- selective ^{1}H (SEH)
- broad band inverse (BBI)
- triple resonance inverse (TXI, ^{1}H observe, ^{13}C and ^{15}N dec.)

All above listed types are also available with Z-gradient.

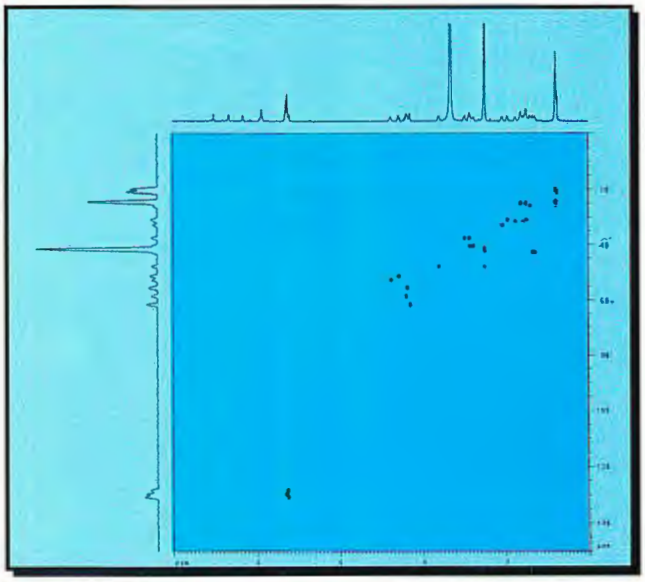


Figure 2: Gradient accelerated ^{1}H, ^{13}C inverse shift correlation experiment HSQC of 400 nanomoles of Gramicidin S in DMSO. 32 transients were recorded for each of 256 t_1-increments. Experiment time 3h, 2.5 mm TXI probe with Z-gradient.

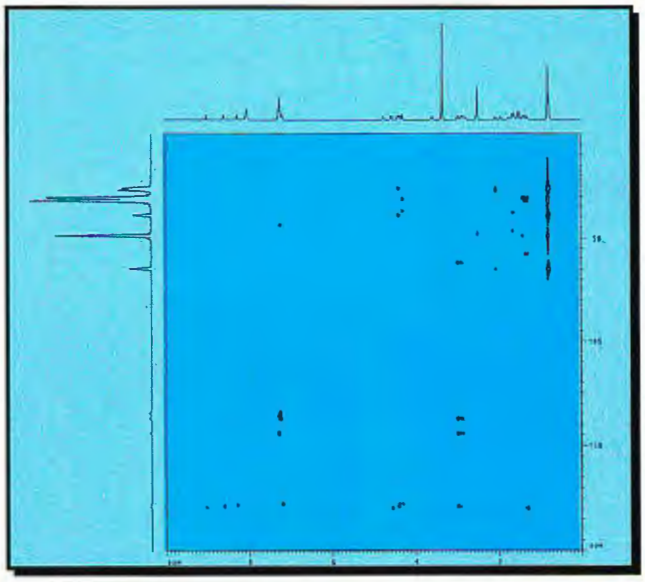


Figure 3: Gradient accelerated ^{1}H, ^{13}C shift correlation experiment HMBC via long-range couplings. 4 micromoles of Gramicidin S in DMSO. 4 transients were recorded for each of 256 t_1-increments. Experiment time 25 min, 2.5 mm TXI probe with Z-gradient.

For ultimate results with the 2.5 mm Microprobes, a shimsystem from our BOSS family is necessary.

Europe

Belgium N.V. BRUKER SPECTROSPIN S.A.
27, Avenue des Courses/Wedrennenlaan
B-1050 Bruxelles
Tel. (02) 648 53 99, Fax (02) 648 86 34

France BRUKER SPECTROSPIN S.A.
34, rue de l'Industrie, F-67160 Wissembourg
Tel. (88) 73 68 00, Fax (88) 73 68 79

Germany BRUKER
ANALYTISCHE MESSTECHNIK GMBH
Silberstreifen, D-7512 Rheinstetten 4
Tel. (07 21) 51 61-0, Fax (07 21) 51 71 01
Wikingerstraße 13, D-7500 Karlsruhe 21
Tel. (07 21) 59 87-0, Fax (07 21) 59 67-173

BRUKER-FRANZEN ANALYTIK GMBH
Fahrenheitstraße 4, D-2800 Bremen 33
Tel. (04 21) 22 05-0, Fax (04 21) 22 05-102

BRUKER SAXONIA Analytik GmbH
Permoserstraße 15, O-7050 Leipzig
Tel. (0037) 41 239 2453

Italy, BRUKER SPECTROSPIN SRL
Via Giovanni Pascoli 70/3, I-20133 Milano
Tel. (02) 70 63 63 70, Fax (02) 2 36 12 94

Netherlands BRUKER SPECTROSPIN NV
Bruynvisweg 18, NL-1530 AB Wormer
Tel. (75) 28 52 51, Fax (75) 28 97 71

Scandinavia BRUKER SPECTROSPIN AB
Enhagsslingan 6, S-183 39 Täby
Tel. (00 46) 87 58 03 35, Fax (00 46) 87 92 51 68

Spain BRUKER ESPAÑOLA S.A.
O'Donnell, 46-7º D, E-28009 Madrid
Tel. (1) 504 62 54, Fax (1) 504 47 86

Switzerland SPECTROSPIN AG
Industriestrasse 26, CH-8117 Fällanden
Tel. (01) 8 25 91 11, Fax (01) 8 25 96 95

United Kingdom, BRUKER SPECTROSPIN LTD.
Banner Lane, Coventry CV4 9GH
Tel. (0203) 855200, Fax (0203) 465317

Middle East

Israel, BRUKER SCIENTIFIC ISRAEL LTD.
Kiryat Weizmann-Science Park
P.O. Box 2445, Rehovot 76 123
Tel. (00972) 8-46 96 60, Fax (00972) 8-46 96 61

North America

Canada, BRUKER SPECTROSPIN LTD.
555 Steeles Avenue East, Milton
Ontario L9T 1Y6
Tel. (416) 876-46 41, Fax (416) 876-44 21

BRUKER SPECTROSPIN LTD.
9885 West Saanich Road, Sidney, B.C. V8L 3S1
Tel. (604) 656 1822, Fax (604) 656 7289

USA BRUKER INSTRUMENTS, INC.
Manning Park, 19, Fortune Drive
Billerica, Mass. 01821-3991
Tel. (508) 667-95 60, Fax (508) 667-39 54

BRUKER INSTRUMENTS, INC.
3411 Silverside Road
Webster Building, Suite 107
Concord Plaza, Wilmington, Delaware 19810
Tel. (302) 478-81 10, Fax (302) 478-14 31

BRUKER INSTRUMENTS, INC.
47697 Westinghouse Drive
Fremont, CA 94539
Tel. (510) 683 4300, Fax (510) 490 6588

BRUKER INSTRUMENTS, INC.
5111 Acadamy Drive, Lisle, Illinois 60532
Tel. (708) 971-43 00, Fax (708) 971-43 03

BRUKER INSTRUMENTS, INC.
9450 Grogan's Mill Road, Suite 115
The Woodlands / Houston, Texas 77 380
Tel. (713) 292-24 47, Fax (713) 292-24-74

Australia

Australia, BRUKER (AUSTRALIA) PTY. LTD.
P.O. Box 202
Unit 7, 163 Mc. Evoy Street
Alexandria NSW 2015
Tel. (02) 550-6422, Fax (02) 550-3687

Asia

P.R. China, BRUKER INSTRUMENTS, LTD.
Peking University, New Chemistry Building
100871 Beijing, P.R. China
Tel. 00861-2557531, Fax 00861-2557532

India, BRUKER INDIA SCIENTIFIC Pvt. Ltd.
48, B. Wing, Abhishek, Lokandwala Complex
Char Bungalows, Andheri (West)
Bombay 400058
Tel. (22) 626 2232, Fax (22) 826 8844

Japan BRUKER JAPAN CO. LTD.
21-5, Ninomiya 3-Chome
Tsukuba-shi, Ibaraki 305
Tel. (0298) 521 234, Fax (0298) 580 322

NMR-D729/09/93/b

CENTRE FOR MAGNETIC RESONANCE

PROFESSOR DAVID M. DODDRELL
DIRECTOR

THE UNIVERSITY OF QUEENSLAND
Brisbane Qld 4072 Australia
Telephone (07) 365 4100
International +61 7 365 4100
Facsimile (07) 365 3833
Telex UNIVQLD AA 40315

HIGH RESOLUTION HIGH FIELD RODENT CARDIAC IMAGING WITH FLOW ARTIFACT SUPPRESSION

(received 10/18/93)

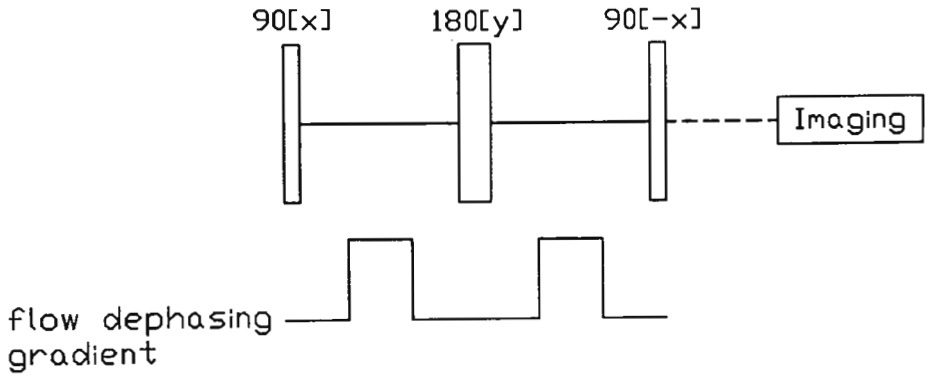
Dear Barry,

Recently we have been attempting some high resolution cardiac imaging studies using our Bruker AMX400 and 300 vertical wide bore (9.4T, WB 89 mm) and super wide bore (7T, SWB 150 mm) systems on both rats and mice.

Our initial investigations have been directed towards the use of respiratory/cardiac gated spin-echo imaging techniques coupled with blood flow suppression sequences. Blood flow artifacts can compromise anatomical detail especially in the rodent model where heart rates of up to 450 beats/min. are possible. In conjunction with this methodology, we have utilized custom designed hardware for vertical bore magnet systems for imaging and localized spectroscopy. This hardware consists of both WB and SWB shielded gradient sets capable of gradient strengths up to 19 G/cm (WB) and 25 G/cm (SWB), Birdcage resonator Rf probes, animal handling system and a fibre optical sensor for respiratory gating. All hardware was constructed at the C.M.R. With this equipment, mouse heart images with an in-plane resolution of 90 µm (1 mm slice thickness) were readily obtainable (see Fig. 2).

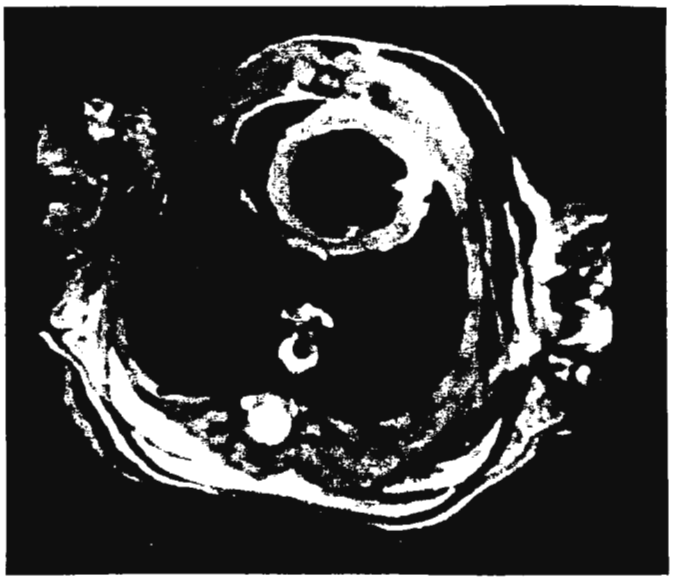
To overcome the problems associated with blood flow artifacts we have been experimenting with a number of ways of eliminating signal from flowing spins. One method that works effectively is to completely dephase the flowing spins prior to imaging. This can be achieved with the use of a combined spin-echo/gradient refocus preparation sequence first proposed by Nishimura[1] in 1986 for flow angiography (see Fig. 1).

Fig. (1).



In this case both flowing and stationary spins are rotated to the transverse plane by the first non-selective 90° pulse. The symmetrical gradients and 180° pulse refocus the stationary spins along the y axis where they can be returned to the z axis by the last 90° pulse (either +/- x phase). The flowing spins, however, are dephased by the gradient and are not refocused, and thus are subsequently spoilt by the first imaging slice gradient. The flow suppression gradients are applied in the slice direction to remove any image "stripe" artifacts[2]. The gradient duration

is kept to a minimum to reduce any additional T_2 weighting. The gradient amplitude required to suppress the signal depends on the complexity of the flow. This method is very effective in removing turbulent ventricular blood flow artifacts . It is interesting to note that the sequence can also be used for Angiographic imaging by replacing the last pulse with a y phased 90° pulse[3]. In this case the flowing spins are restored along the z axis while stationary spins are completely dephased by the imaging slice gradients.



References

1. D.G. Nishimura, A. Macouski and J.M. Pauly, *IEEE Trans. Med. Imaging*, MI-5, 140 (1986).

2. T.H. Foster, D.B. Plewes and J. Szumowski, "Society of Magnetic Resonance in Medicine, Book of Abstracts, Sixth Annual Meeting, p.30, 1987".

3. F.R. Korosec, T. Grist, J. Polzin and C. Mistretta, "Society of Magnetic Resonance in Medicine, Book of Abstracts, Twelfth Annual Meeting, p.571, 1993".

Yours sincerely,

STEPHEN ROSE **FERNANDO ZELAYA** **STUART CROZIER** **STEPHEN WILSON** **DAVID DODDRELL**

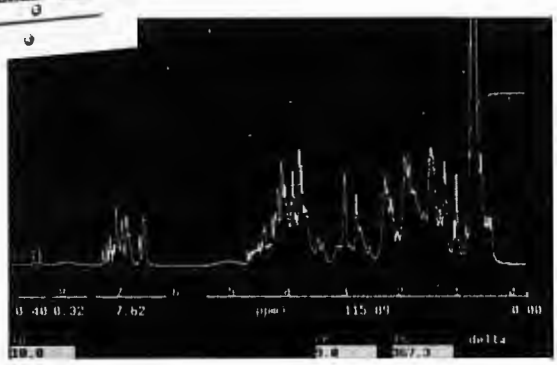
AMT Delivers More For Less!

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 ±1dB
- Noise blanking in less than 2 μs
- 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!

AMERICAN MICROWAVE TECHNOLOGY

3080 Enterprise Street ■ Brea, CA 92621 ■ (714) 993-0802 ■ Fax (714) 993-1619

AMERICAN MICROWAVE TECHNOLOGY

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 μs 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, ±10%, 50–60 Hz
AC power requirements	2000 watts
Package	Rack mount
Size (HWD, inches)	8.75×19×20.25
Net weight	100 lbs.

03/92

3080 Enterprise Street ■ Brea, CA 92621 ■ (714) 993-0802 ■ Fax (714) 993-1619

ENC 35th Experimental Nuclear Magnetic Resonance Conference

April 10-15, 1994, The Asilomar Conference Center, Pacific Grove, CA (USA)

Dr. Barry Shapiro
TAMU NMR Newsletter
966 Elsinore Court
Palo Alto, Ca 94363
USA

Dear Barry,

the program of the next Experimental NMR Conference is shaping up, and I want to inform you about the preliminary schedule of events. The meeting will take place at the familiar Asilomar conference grounds in California. Contrary to previous Asliomar conferences, this meeting will extend through Thursday afternoon. Lodging at Asilomar will be available for early arrival the weekend prior to the meeting (April 8 to 10).

Sincerely yours

Bernhard Blümich

Preliminary Program

Sunday, April 10

3 - 9 pm Registration
6 - 8 pm Western Cook-out

Monday, April 11

morning Poster session I, *H. Eckert, Chair*
Opening remarks
In vivo NMR, *J. L. Ackerman, Chair*
F. Wehrli, University of Pennsylvania, Philadelphia, PA, USA
D. Ailion, University of Utah, Salt Lake City, UT, USA
G. A. Johnson, Duke University, Durham, NC, USA

afternoon Materials Imaging, *D. G. Cory, Chair*
L. D. Hall, University of Cambridge, Great Britain

Tuesday, April 12

morning Selective Excitation in Liquid-State NMR, *R. Freemän, Chair*
D. Canet, C. Roumestand, Université de Nancy, France: *DANTE-Z. A Robust and Easy-to-Implement Method for Selective Excitation*
G. Bodenhausen, Université de Lausanne, Switzerland
L. Lerner, University of Wisconsin, USA

morning Methods of High Resolution NMR for Macromolecules, *G. Montelione, Chair*
G. Wagner, Harvard Medical School, Cambridge, MA, USA
A. Bax, National Institutes of Health, Bethesda, MD, USA
L. Kay, University of Toronto, Ontario, Canada

Executive Committee

Bernhard Blümich, Chair
Makromolekulare Chemie
RWTH Aachen
Worringer Weg 1
D-52056 Aachen, Germany
49 241 80 6421

Hellmüt Eckert, Chair Elect
Department of Chemistry
University of California
Santa Barbara, CA 93106
(805) 893-8163

Gaetano Montelione, Secretary
Rutgers University, CABM
679 Hoes Lane
Piscataway, NJ 08854
(908) 463-5321

James E. Roberts, Treasurer
Department of Chemistry
Lehigh University
6 East Packer Avenue
Bethlehem, PA 18015
(215) 758-4841

Jerome Ackerman
Laima M. Baltusis
Mary W. Baum
John Delayre
F. David Doty
Ray Freeman
Joel Garbow
Angela M. Gronenborn
Shaw Huang
Robert D. Johnson
Jacob Schaefer
Ruth E. Stark
Elizabeth A. Williams
Robert A. Wind

ENC 35th Experimental Nuclear Magnetic Resonance Conference

April 10-15, 1994, The Asilomar Conference Center, Pacific Grove, CA (USA)

W. Warren, Princeton University, New Haven, CO, USA

afternoon Software and Analysis: *A. Gronenborn, Chair*
D. Ziessow, Technische Universität Berlin, Germany
J. Markley, University of Wisconsin, Madison, WI, USA
D. Gorenstein, Purdue University, West Lafayette, IN, USA
A. Gronenborn, National Institutes of Health, Bethesda, MD, USA

afternoon Food Materials and Processes, *T. Eads, Chair*
K. J. Packer, University of Nottingham, Great Britain
M. J. McCarthy, University of California, Davis, CA
T. M. Eads, Purdue University, West Lafayette IN, USA

Wednesday, April 13

morning Poster session II, *H. Eckert, Chair*

afternoon Instrumentation, *R. Wind, Chair*
R. G. Griffin, Massachusetts Institute of Technology, Cambridge, MA, USA
J. Haw, Texas A&M University, College Station, TX, USA
G. Chingas, University of California, Berkeley, CA, USA

afternoon Miscellany, *A. N. Garroway, Chair*
A. G. Marshall, Ohio State University, Columbus, OH, USA
J. Wrachterup, Freie Universität Berlin, Germany

Thursday, April 14

morning Inorganic Solids, *R. D. Johnson, Chair*
R. Tycko, AT/T Bell Laboratories, Murray Hill, NJ, USA
H. Gondey, University of British Columbia, Vancouver, Canada
M. Mehring, Universität Stuttgart, Germany

morning Confined Spaces, *S. G. Huang, Chair*
P. T. Callaghan, Massey University, Palmerston North, New Zealand
F. Fujara, Universität Dortmund, Germany
J. Kärger, Universität Leipzig, Germany

morning Solid-State Methods, *E. A. Williams, Chair*
B. Meier, P. Robyr, M. Tomaselli, M. Baldus, S. Hediger, Eidgenössische Technische Hochschule, Zürich, Switzerland
W. Wu, S. Burns, K. W. Zilm, Yale University, New Haven, CT, USA
B. Chmelka, University of California at Santa Barbara, CA, USA

afternoon Biological Solids, *R. Stark, Chair*
C. Bronniman, Otsuka ELectronics, Fort Collins, CO, USA
A. McDermott, Columbia University, NY, USA
T. A. Cross, Florida State University, Tallahassee, FL, USA
Closing Remarks

evening Banquet
After Dinner Lecture: W. A. Anderson, Varian Associates, Palo Alto, CA, USA
Conference Adjourns

Friday, April 15

Departure

Executive Committee

Bernhard Blümich, Chair
Makromolekulare Chemie
RWTH Aachen
Worringer Weg 1
D-52056 Aachen, Germany
49 241 80 6421

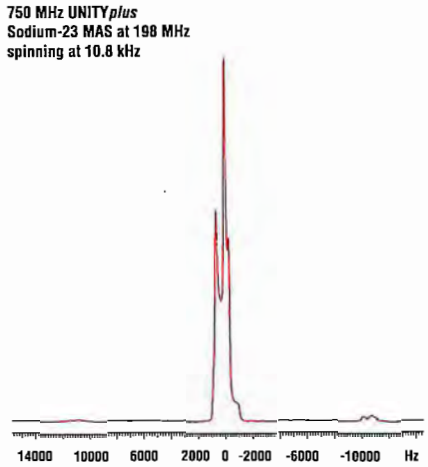
Hellmüt Eckert, Chair Elect
Department of Chemistry
University of California
Santa Barbara, CA 93106
(805) 893-8163

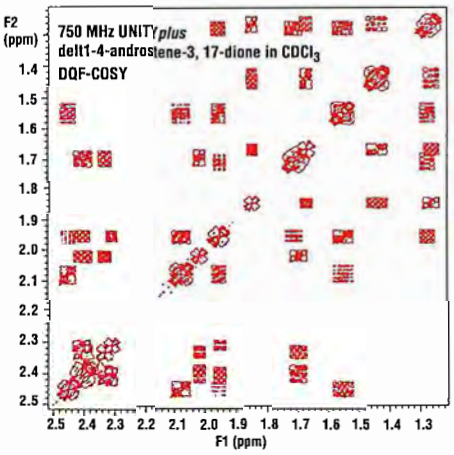
Gaetano Montelione, Secretary
Rutgers University, CABM
679 Hoes Lane
Piscataway, NJ 08854
(908) 463-5321

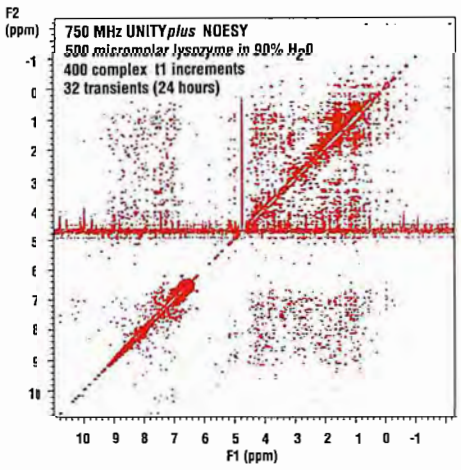
James E. Roberts, Treasurer
Department of Chemistry
Lehigh University
6 East Packer Avenue
Bethlehem, PA 18015
(215) 758-4841

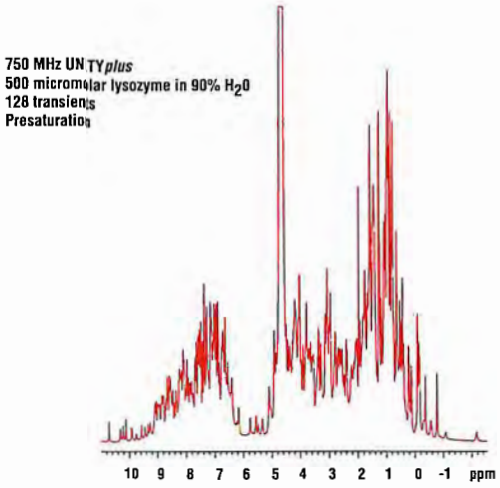
Jerome Ackerman
Laima M. Baltusis
Mary W. Baum
John Delayre
F. David Doty
Ray Freeman
Joel Garbow
Angela M. Gronenborn
Shaw Huang
Robert D. Johnson
Jacob Schaefer
Ruth E. Stark
Elizabeth A. Williams
Robert A. Wind

Varian Delivers 750 MHz Results.









*These spectra were obtained on a Varian 750 MHz UNITY*plus*™ NMR spectrometer.*

Leadership in NMR Technology

Varian Associates, Inc. and Oxford Instruments have jointly developed the first 750 MHz NMR system.

Ideal for biomolecular and chemical applications, Varian UNITY*plus* NMR spectrometers perform the most demanding experiments at all field strengths. Oxford 750 MHz magnets provide the best long-term stability and do not require cryo-pumping. Uniquely designed as a single, no-compromise platform for liquids, solids, and microimaging at the widest range of field strengths, the UNITY*plus* offers the most advanced technology to the NMR community. Varian's exceptional probes, pulsed field gradients, and flexible software provide the ideal system for leading-edge NMR research.

For additional information, contact the Varian office nearest 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

Varian's 750 MHz Spectrometers Provide Results

Advantages

- Highest chemical shift dispersion and sensitivity in a persistent, non-pumped magnet
- Outstanding performance and stability is delivered by the UNITY*plus* NMR system
- Greatest flexibility is provided by VNMR software

Applications

- Structure determination of biomolecules
- Detection of minute sample quantities
- MAS studies of quadrupolar nuclei
- Methods development at high field strengths

Call your sales representative. Australia (3) 543 8022. Austria (1) 69 55 450. Belgium (2) 721 4850. Brazil (11) 829 5444. Canada (416) 457 4130. Denmark (42) 84 6166. France (1) 69 86 38 38. Germany (6151) 70 30. Italy (2) 753 1651. Japan (3) 3204 2111. Korea (2) 561 1626. Mexico (5) 533 5985. Netherlands (3403) 50909. Norway (9) 86 74 70. Spain (01) 430 0414. Sweden (8) 82 00 30. Switzerland (42) 44 88 44. UK (932) 24 37 41. US 800-356-4437. Other International (415)424-5424.

MAG-6055/569

varian

2800 Plymouth Road Phone: 313-996-7000
Ann Arbor, MI
48105

PARKE-DAVIS PHARMACEUTICAL RESEARCH
People Who Care

September 28, 1993
(received 10/1/93)

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

Computer Program for Analyzing Protein Primary Sequence by Spin Systems

Dear Prof. Shapiro:

We have written a Macintosh based menu driven utility program to conceptually aid us in the cumbersome task of analyzing the protein primary sequence. It groups and totals the individual amino acids according to their respective spin systems. Identifies the amino acid residues whose occurence is unique in the sequence, as well as all of the di- and tri- amino acid segments that are also unique in occurence to delineate the scalar and NOE-based connectivities used in the sequential assignment procedure.

This program was written using the console package of Think C on the Macintosh to allow portability to an IBM PC and compatibles. Helpful features of this program include:

- Display of the sequence with spacing every ten.
- Ability to enter a sequence by keyboard or cutting and pasting from any Macintosh application, including Wordperfect, teachtext, MS Word, or even mail.
- Editing and updating of an already entered sequence.
- Display of unique amino acids, di- or tri- peptide seqments include the position number for easy determination of location in sequence.
- Printing of all data to the chooser chosen printer.

Here's an example of how the program works using BPTI [1-58] as the test protein.

```
                AMINO ACID SEQUENCE
      10        20        30        40        50        60
RPDFCLEPPY TGPCKARIIR YFANAKAGLC QTFVYGGCRA KRNNFKSAED CMRTCGGA


    Unique              AMX              Long Side Chains
  ----------          ----------          ----------
  G -  6              C -  6              P -  4
  A -  7              S -  1              R -  6
  T -  3              D -  2              K -  4
  V -  1              N -  3              E -  2
  L -  2              F -  4              Q -  1
  I -  2              H -  0              M -  1
                      W -  0
                      Y -  3
Total:  21          Total:  19          Total:  18
                    MENU OPTIONS
  ------------------------------------------------
    1) Total Amino's          6) Enter New Sequence
    2) Find Unique Di's       7) Change Amino Acid
    3) Find Unique Tri's
    4) Print All              COMMAND:
    5) Clear Sequence
```

Figure 1.

```
                AMINO ACID SEQUENCE
      10        20        30        40        50        60
RPDFCLEPPY TGPCKARIIR YFANAKAGLC QTFVYGGCRA KRNNFKSAED CMRTCGGA


 Unique POS    DI's  POS     DI's  POS     DI's  POS     DI's  POS
   Q 31        R - P  1      I - R  19     K - R  41
   V 34        X - L  5      A - X  23     K - S  45
   S 47        L - Z  6      A - G  27     S - A  47
   M 52        Z - P  7      G - L  28     A - Z  48
               P - P  8      L - X  29     Z - X  49
               X - T  10     Z - T  31     Z - A  52
               T - G  11     X - V  33     A - T  53
               G - P  12     V - X  34     O - A  57
               A - A  16     G - X  37
               A - I  17     X - A  38
               I - I  18     A - A  39
                    MENU OPTIONS
  ------------------------------------------------
    1) Total Amino's          6) Enter New Sequence
    2) Find Unique Di's       7) Change Amino Acid
    3) Find Unique Tri's
    4) Print All              COMMAND:
    5) Clear Sequence
```

Figure 2.

Figure 1. This sequence was imported from a PDB file using PALANTIR, an in-house program used to visualize, manipulate and analyze protein primary sequences, which converts the three-letter amino acid symbols to one-letter for use here. The amino acids are grouped into three spin systems: unique (G, A, V, L, I and T), AMX (S, C, D, N, F, Y, W and H) and long side chain (R, E, Q, M, K and P). Each column is sum totalled.

Figure 2. The uniquely occuring amino acids and the di- peptide segments with their positions are listed, where X represents any AMX residue and Z represents any long side chain residue (Q,E, or M) respectively. The uniquely occuring tri-peptide seqments can be displayed similarly.

All data can then be printed using menu item 4, figure 3.

```
                        Amino Acid Sequence
         10         20         30         40         50         60
RPDFCLEPPY TGPCKARIIR YFANAKAGLC QTFVYGGCRA KRNNFKSAED CMRTCGGA

Short Side Chains          AMX              Long Side Chains
-----------------      -----------------    ----------------
    G -   6                C -   6              P -   4
    A -   7                S -   1              R -   6
    T -   3                D -   2              K -   4
    V -   1                N -   3              E -   2
    L -   2                F -   4              Q -   1
    I -   2                H -   0              M -   1
                           W -   0
                           Y -   3
Total:   21            Total:   19          Total:   18

Uniqueness of Occurrence
------------------------
            Q 31
            V 34
            S 47
            M 52
Uniq Di Pos        Uniq Di Pos          Uniq Di Pos
-----------        -----------          -----------
  R - P  1           X - L  5             L - Z  6
  Z - P  7           P - P  8             X - T  10
  T - G  11          G - P  12            A - R  16
  R - I  17          I - I  18            I - R  19
  A - X  23          A - G  27            G - L  28
  L - X  29          Z - T  31            X - V  33
  V - X  34          G - X  37            X - R  38
  R - A  39          K - R  41            K - S  46
  S - A  47          A - Z  48            Z - X  49
  Z - R  52          R - T  53            G - A  57

Uni Tri  Pos       Uni Tri  Pos         Uni Tri  Pos
-----------        -----------          -----------
  R-P-X 1            P-X-X 2              X-X-L 4
  X-L-Z 5            L-Z-P 6              Z-P-P 7
  P-P-X 8            P-X-T 9              X-T-G 10
  T-G-P 11           G-P-X 12             P-X-K 13
  X-K-A 14           K-A-R 15             A-R-I 16
  R-I-I 17           I-I-R 18             I-R-X 19
  X-X-A 21           X-A-X 22             A-X-A 23
  X-A-K 24           A-K-A 25             K-A-G 26
  A-G-L 27           G-L-X 28             L-X-Z 29
  X-Z-T 30           Z-T-X 31             T-X-V 32
  X-V-X 33           V-X-G 34             G-G-X 36
  G-X-R 37           X-R-A 38             R-A-K 39
  A-K-R 40           K-R-X 41             X-X-K 44
  X-K-S 45           K-S-A 46             S-A-Z 47
  A-Z-X 48           Z-X-X 49             X-X-Z 50
  X-Z-R 51           Z-R-T 52             R-T-X 53
  T-X-G 54           G-G-A 56
```

Figure 3.

This program is available by sending a Macintosh formatted 3.5" disk to Parke-Davis/Warner-Lambert, attn. Chris Ingalls, 2800 Plymouth Rd., Ann Arbor, MI 48105. If requests are made an IBM PC version can be easily produced.

Please credit this contribution to D. Omecinsky.

Sincerely yours,

C. Ingalls D. Omecinsky M. D. Reily V. Thanabal

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

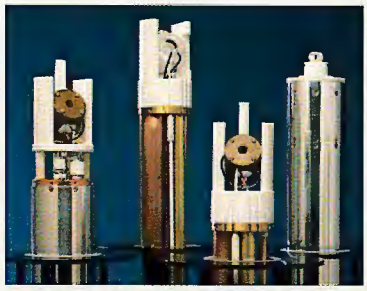
Advanced Console Design
- Full *channel modularity*
- Full channel expandability
- Easy customer access

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



Chemagnetics

Sun Microsystems Computer Corporation
Value Added Reseller

Dedicated to Design Excellence

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.

Chemagnetics™

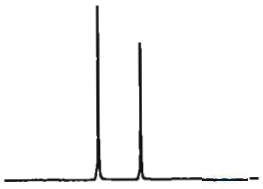
Variable Amplitude Cross Polarization – The Latest Technique from Chemagnetics

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.

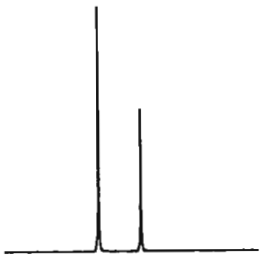
This power to implement new and future techniques lies at your fingertips when you invest in the CMX design philosophy. While others attempt to emulate its flexibility and modularity, the CMX continues to evolve into the most effective, versatile NMR spectrometer available.

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

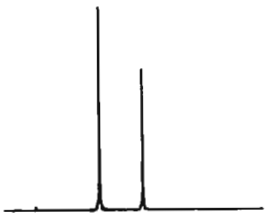


Variable amplitude cross polarization (VACP) *under* –2.6 KHz *mismatch condition*

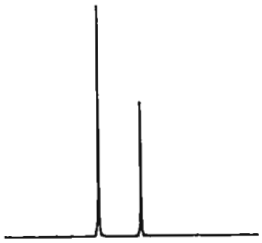
Deliberate "mismatch" of –1.9 KHz



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.

THE UNIVERSITY OF MICHIGAN
BIOPHYSICS RESEARCH DIVISION

930 NORTH UNIVERSITY AVENUE
ANN ARBOR, MICHIGAN
48109-1055

PHONE 313 764-5258
FAX 313 764-3323

October 19, 1993
(received 10/20/93)

Dr. Bernard Shapiro,
968 Elsinore Court
Palo Alto, CA 94303

Dear Dr. Shapiro:

In recent years we have been experimenting with different applications of multiple-pulse J-cross polarization (CP) in liquids. In early work by others and ourselves, we showed experimentally that these methods can yield better polarization transfer than pulsed-free precession methods (e.g. INEPT-style sequences). More recently, we found that the three-dimensional HCCH-TOCSY experiment also benefits from cross polarization as heteronuclear transfer steps. The question is now to determine what physical effect gives rise to these improvements. There should be no theoretical difference in efficiency between the two methods for ideal relaxation, r.f. offset and r.f. inhomogeneity. Here we show a computer simulation of the dependence of CP and INEPT efficiency on the radio frequency inhomogeneity.

Two independent r.f. coils were assumed. The r.f. inhomogeneity profile of each of these coils was assumed to have a Gaussian shape along the length of the sample. The r.f. field strengths at the crest of the Gaussians were assumed to be ideal for the experiments. Waltz-16 composite pulse sequences were used in the CP trains while no shift offset effects were included. The computations were done by using product-operator formalism for INEPT and by solving the Liouville-von Neumann equation for CP. For each given r.f. inhomogeneity distribution - say, field I drops off in a Gaussian way to 40% at the edge of the NMR sample while field S drops off to 60% at that point - calculations of the efficiency of transfer were made for many small volume elements in the sample. The signals in all volume elements were then added to a total signal. This quantity was plotted in the three-dimensional diagrams shown below. It is observed that CP and INEPT behave equally badly when one of the fields is perfectly homogeneous with the other field dropping off towards the edges of the sample. However, CP is seen to be much more effective in transfer than INEPT when both fields are approximately equally inhomogeneous; even when both fields fall off to 20% of their intensity at the end of the sample volume, CP retains approximately 50% of the transfer efficiency over the entire sample as compared to the situation that both fields would be completely homogeneous. The efficiency of the INEPT sequence is about 25% with this distribution.

Although this is only a model calculation, it clearly demonstrates that large differences in r.f. inhomogeneity sensitivity exist between INEPT and CP. This may explain part of the large sensitivity differences seen between the two methods.

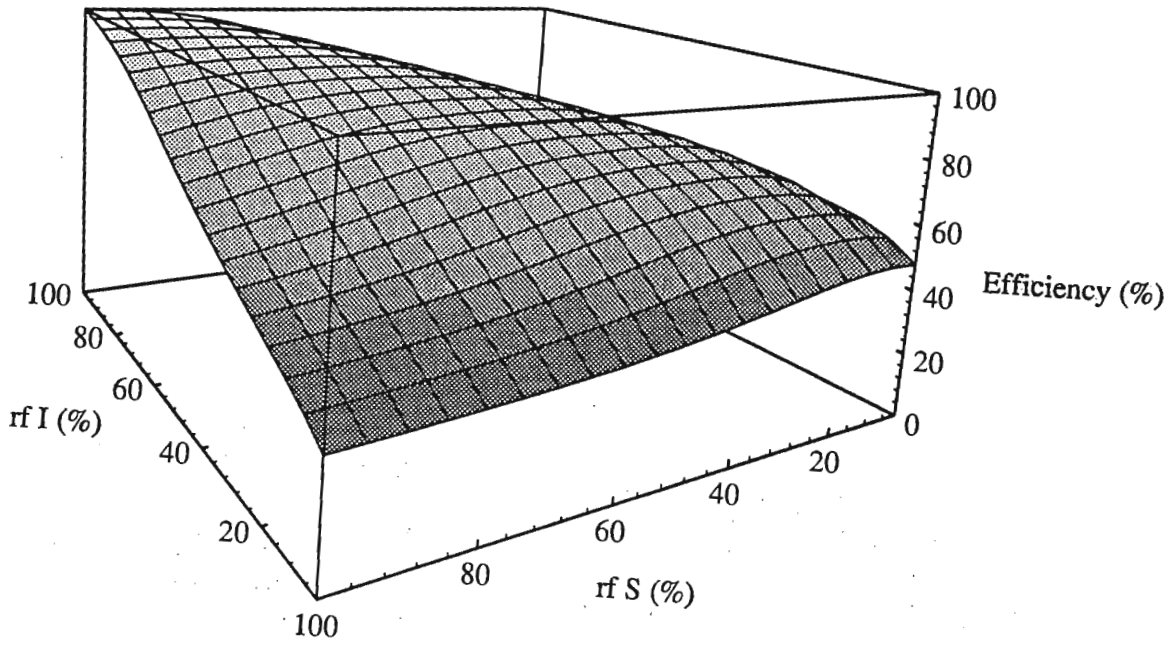
Details of these computations will be published.

Sincerely,

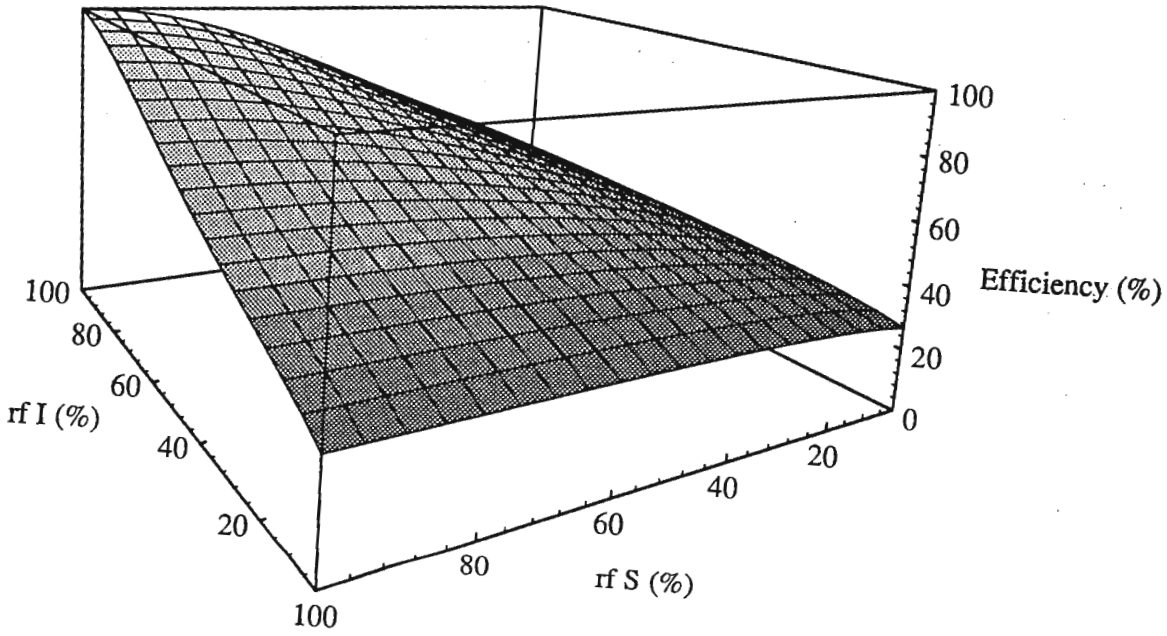
A Majumdar
Ananya Majumdar

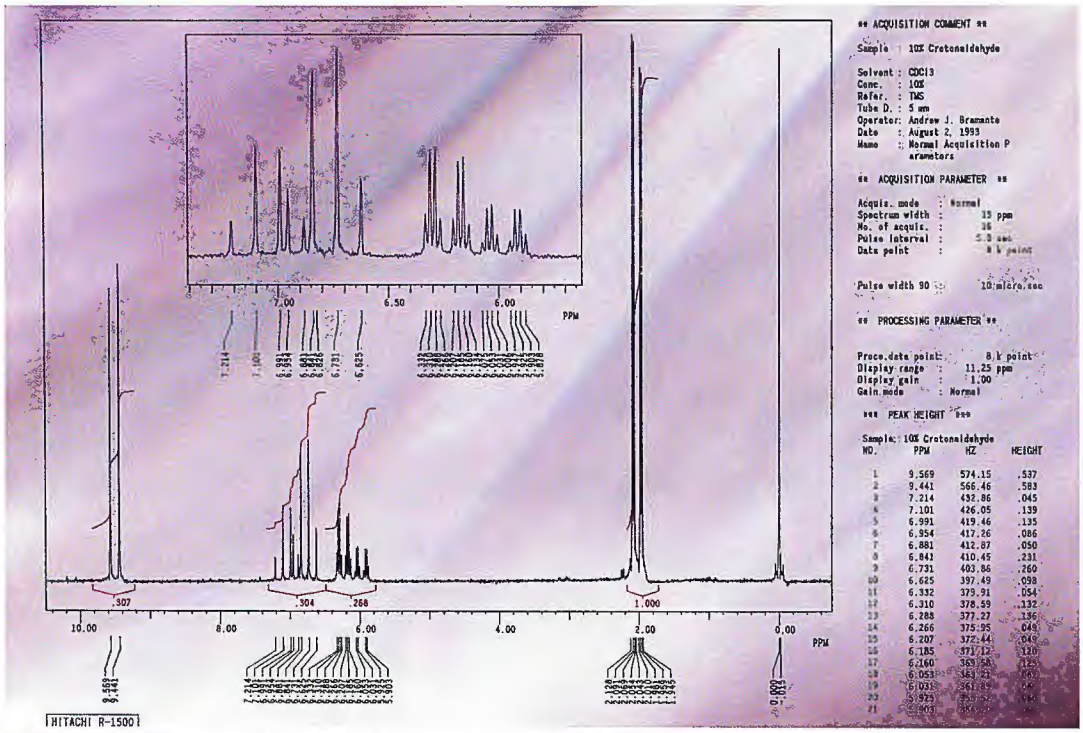
Erik Zuiderweg

Cross Polarization



Inept





This R-1500 FT-NMR spectrum of crotonaldehyde represents a 16 pulse acquisition; each pulse was 10 µsec with a pulse interval of 5 seconds.

HIGH RESOLUTION DIGITAL 60 MHz NMR. GET THE WHOLE STORY IN FIVE SECONDS.

Digital is the new wave in high-resolution 60 MHz NMR spectroscopy. That's because it's not only faster than old analog technology, but it extends the application beyond the acquisition of a simple spectrum. And only Hitachi has it.

Hitachi's R-1500 60 MHz FT-NMR acquires a full spectrum in five seconds. But speed is just part of the story.

The R-1500 will also perform FT experiments, such as solvent suppression, T_1 relaxation time measurement, and Gated Decoupling. And you can manipulate FID data by applying various apodization functions. The result: substantial increases in sensitivity and enhanced resolution.

For a simpler approach to digital 60 MHz NMR, Hitachi offers the R-1200 Rapid Scan NMR spectrometer. It can acquire a quality, high-resolution spectrum in only 10 seconds. The R-1200 is also remarkably sensitive, accumulating up to 256 scans for enhanced signal-to-noise.

Both systems come with a permanent magnet and our exclusive five-year warranty to ensure near-zero maintenance. Over time, the cost of ownership is among the lowest in the industry.

Faster speed, increased sensitivity and better resolution. Only in digital spectroscopy and only from Hitachi, the world leader in 60 MHz NMR, with the industry's best nationwide sales, service and applications support network. For the rest of the story, call **800-548-9001**.

HITACHI

Hitachi Instruments, Inc., 3100 N. First Street, San Jose, CA 95134-9953

© Hitachi Instruments, Inc., 1993 032

FAX FOR IMMEDIATE INFORMATION ON HITACHI'S DIGITAL 60 MHz NMR

I am interested in the:

❑ ***R-1200 Rapid Scan cw Spectrometer***

❑ ***R-1500 FT-NMR Spectrometer***

FAX to: 408-432-0704

Attn: S. Lee

NAME: _____

POSITION: _____

COMPANY: _____

ADDRESS: _____

CITY: _____ STATE: _____ ZIP: _____

BUSINESS PHONE: _____

DO YOU USE AN NMR SPECTROMETER IN YOUR WORK? YES ❑ NO ❑

IF YES, PLEASE LIST MANUFACTURER AND MODEL: _____

APPLICATION(S): _____

ARE YOU CONSIDERING AN NMR SPECTROMETER FOR PURCHASE? YES ❑ NO ❑

IF YES, AT WHAT FIELD STRENGTH? _____

IF YES, WHEN DO YOU NEED IT? _____

HAVE YOU DISCUSSED YOUR APPLICATION(S) WITH A HITACHI REPRESENTATIVE?
YES ❑ NO ❑

IF NO, WOULD YOU LIKE A HITACHI REPRESENTATIVE TO CONTACT YOU?
YES ❑ NO ❑

ALL NMR PRODUCTS ARE COVERED BY THE HITACHI 5 YEAR WARRANTY

HITACHI

Hitachi Instruments, Inc., 3100 N. First Street, San Jose, CA 95134-9953

Biomolecular NMR Laboratory
Bldg. 34, Room 211
Hoffmann-La Roche Inc.
340 Kingsland Street
Nutley, New Jersey 07110-1199

Direct Dial (201) 235-7663

ROCHE Hoffmann-La Roche

Dr. Bernard L. Shapiro
TAMU NMR News Letter
966 Elsinore Court
Palo Alto, CA 94303

October 11, 1993
(received 10/15/93)

Differentiation of Intra- from Intermolecular ^{15}N-filtered NOEs

Detailed characterizations of protein-ligand interactions are vital to rational drug design efforts. Conformational changes in both the protein and the ligand upon complexation often require NMR structure determinations to be performed on the complex. Early isotope-filtered NOE experiments have utilized samples where the small molecule is ^{13}C-^{15}N labeled while the protein is at natural abundance.[1,2] Typically, these experiments involve a series of four different datasets whose linear combinations allow differentiation between intramolecular and intermolecular interactions. Small molecule ligands are synthetic compounds which are generally difficult to produce as ^{13}C-^{15}N enriched materials. Conversely, recent advances in biotechnology and mutidimensional heteronuclear NMR have enhanced the accessibility and utility of ^{13}C-^{15}N doubly enriched proteins. For these reasons, more recent isotope-filtered NMR methods have emphasized application to complexes where the protein is enriched and the ligand is at natural abundance.[3,4] Here we report a simple method for differentiating intra- from intermolecular NOEs in ^{15}N-filtered NOESY by permuting the broad-band decoupling during t_1 between ^{15}N and ^{13}C. Fig. 1 illustrates the ^{15}N-filtered pulse sequence of Ikura and Bax[3] which has been modified to use GARP-1 decoupling during t_1 on either ^{15}N (Fig. 1A) or ^{13}C (Fig. 1B). Since the ^{15}N-filter is in F_2, NOEs will be generated from all ^{1}Hs but detected on only ^{14}N-attached ^{1}Hs. Comparison of the multiplicities of the NOESY cross-peaks between the two experiments allows clear differentiation of intramolecular and intermolecular interactions. The expected NOE cross-peak patterns are illustrated in Fig. 2. Fig. 3 illustrated comparisons of contour plots for two different ^{14}N-attached protons of a small molecule (which shall remain nameless) in complex with a ^{13}C-^{15}N enriched protein (173 residues, .7mM complex, 94% H_2O). Figs 3A and 3C correspond to ^{15}N-filtered NOESY spectra whith ^{13}C decoupling during t_1 while Figs 3B and 3D had ^{15}N decoupling in t_1. Comparison of Fig. 3A to 3B shows that the strong NOE at 2.7 ppm is an intramolecular NOE from a ^{12}C-attached ^{1}H to the ^{14}N-attached ^{1}H at 11.1 ppm. The small NOE at 10.7 ppm which appears only in Fig 3B is an intermolecular NOE from a ^{15}N-attached ^{1}H. Comparison of Fig. 3C to 3D clearly shows the intermolecular NOE at 7.4 ppm to originate from an aromatic ^{13}C-attached ^{1}H rather than a N-attached ^{1}H. The differentiation between intra- and intermolecular origins for these ^{15}N-filtered NOEs greatly enhances the interpretability of these interactions. This then allows determination of structural constraints for the bound conformation of the ligand as well as for the orientation and position of the ligand with respect to the protein.

figure 1

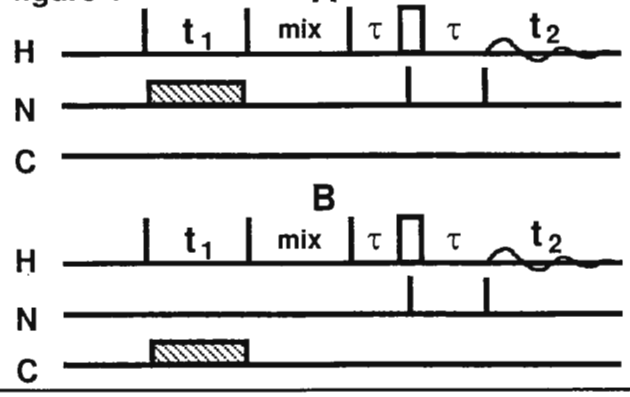


figure 2

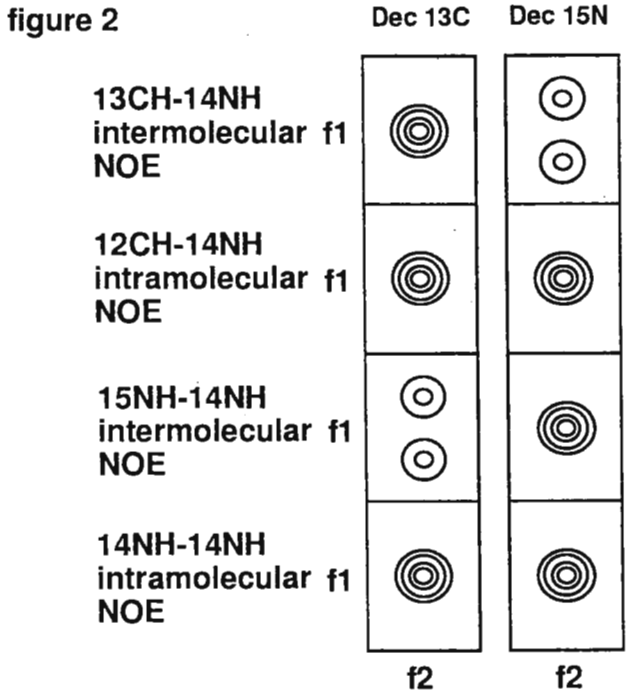
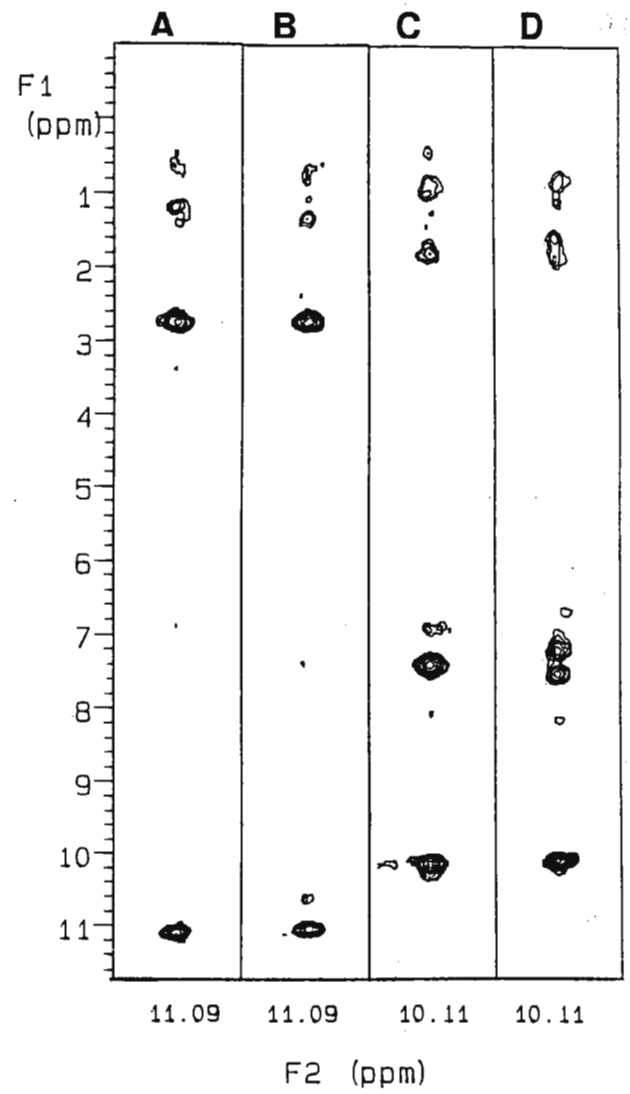


figure 3



1. G. Wider, C. Weber, R. Traber, W. Braun, H. Widmer, K. Wuthrich, *J Am Chem Soc*, **1990**, *112*, 9015-9016.
2. S.W. Fesik, R.T.Gampe, H.L. Eaton, G. Gemmecker, E.T. Oljniczak, P. Neri, T.F. Holzman, D.A. Egan, R. Edalji, R. Simmer, R. Helfrich, J. Hochlowski, M. Jackson; *Biochemistry*, **1991**, *30*, 6574-6583.
3. M. Ikura and A. Bax; *J Am Chem Soc*, **1992**, *114*, 2433-2440.
4. A.M. Petros, M. Kawai, J.R. Luly, and S.W. Fesik; *FEBS*, **1992**, *308#3*, 309-314.

Please credit this contribution to the account of David C. Fry.

Sincerely,

Steven Donald Emerson

Steven Donald Emerson
Physical Chemistry Department

NMR 750

The new 750 MHz from Oxford, setting another new standard in NMR

OXFORD

Oxford Instruments, NMR Instruments
Osney Mead, Oxford OX2 0DX, England
Telephone +44 (0) 865 269500 Fax +44 (0) 865 269501

Identification of Uncommon Sugars in Polysaccharides by NMR Spectroscopy

G. P. Reddy and C. Allen Bush

Department of Chemistry and Biochemistry
University of Maryland Baltimore County
Baltimore, Maryland 21228 (received 10/18/83)

In structure determinations of the cell surface polysaccharides of pathogenic vibrios (*V. cholerae, V. vulnificus* etc.) we and others have encountered a number of uncommon sugars, mostly amino sugar derivatives. In our laboratory we begin with standard carbohydrate analysis – acid hydrolysis followed by chromatographic identification of the resulting monosaccharides. High performance anion exchange chromatography gives excellent separations and electrochemical detection by pulsed amperometry is very sensitive. In analysis of vibrio capsular polysaccharides we observe peaks which are believed to represent sugars on the basis of the electrochemical detection. But alas, if the retention time fails to agree with that of our standard sugars from the Sigma catalog, the identities of the peaks remain unknown.

Let the powerful forces of high resolution NMR come to the rescue, bringing light where darkness would otherwise prevail. The monosaccharides, generally in the pyranoside ring form, adopt fixed chair conformations characteristic of 6-membered rings in which bulky substituents are predominantly equatorial. Thus assignment of the chemical shift and homonuclear coupling constant, $^{3}J_{HH}$, for each proton of the ring indicates whether a proton is axial or equatorial identifying the relative stereochemistry of each carbon center.

This information can be extracted from DQF-COSY except in the case of ^{1}H strong coupling, which is in fact rather common in sugars. If strong coupling is not too serious, TOCSY can provide the assignment and the values of $^{3}J_{HH}$ but when the chemical shifts of the coupled ^{1}H pairs are very close, ^{13}C HMQC can be relied on to give good lineshape from which the $^{3}J_{HH}$ values may be extracted. Finally ^{13}C HMBC gives cross peaks indicating long-range C-H coupling whose size can also be correlated with pyranoside stereochemistry. A trans relationship between a C-H pair gives a large $^{3}J_{CH}$ while gauche pairs give little or no cross peak. Knowledge of the stereochemistry at each center identifies the sugar configuration as gluco, manno, galacto etc...

Amino sugars are recognized by the characteristic upfield chemical shift of the ^{13}C bearing the nitrogen atom. Thus sugar ring carbon signals near 50 ppm in the HMQC are tentatively assigned as amino sugars and the identification of N-acylated amino sugars is confirmed by the resonance of the amide proton in water-suppressed ^{1}H NMR spectra. 6-deoxy sugars such as rhamnose or fucose derivatives are recognized by the methyl doublet of H6 around 1.3 ppm and deoxy positions in the ring (eg. C2, C3 or C4) are identified as methylene resonances by DEPT in the ^{13}C spectra or by their characteristic upfield chemical shift in ^{13}C or ^{1}H spectra.

For all the rare amino sugars we have encountered so far (QuiNAc, RhaNAc, FucNAc, GalNAcA, GlcNAcA) we have been able to obtain authentic samples once the interpretation of the NMR spectra told us what to ask for. Thus we could confirm the identity of the sugar by HPLC to calm the skepticism of narrow-minded reviewers who failed to share our confidence in the power of NMR for identification of these uncommon sugars.

UNIVERSITY OF VIRGINIA
DEPARTMENT OF CHEMISTRY
McCORMICK ROAD
CHARLOTTESVILLE. VIRGINIA 22901

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

Oct. 20, 1993
(received 10/22/93)

Fixing 2D baselines with Felix

Dear Dr. Shapiro:

Two Varian Unity Plus 500's were installed here several months ago and are happily cranking out data. Following acquisition of two or higher dimensional datasets, the data are transferred to a remote computer and processed with Felix. We have made use of a procedure for baseline adjustment of two dimensional data originally described by Otting et al. (JMR 66 187) and later in the Varian System Operation Manual (1/93 version, p.166-7). This procedure involves controlling the baseline level by changing the amplitude of the first point of fids. The best value of the first point multplier for t2 fids should be determined empirically. The following briefly describes applying this procedure in the t2 dimension of 2D spectra when using Felix.

After starting Felix, open a database and set absint to 1. Read the first t2 fid, apodize, FT, and phase, then pick baseline points. Correct the baseline with the spline or polynomial function and record the baseline level by positioning the mouse-controlled arrow so that it points at the baseline level.

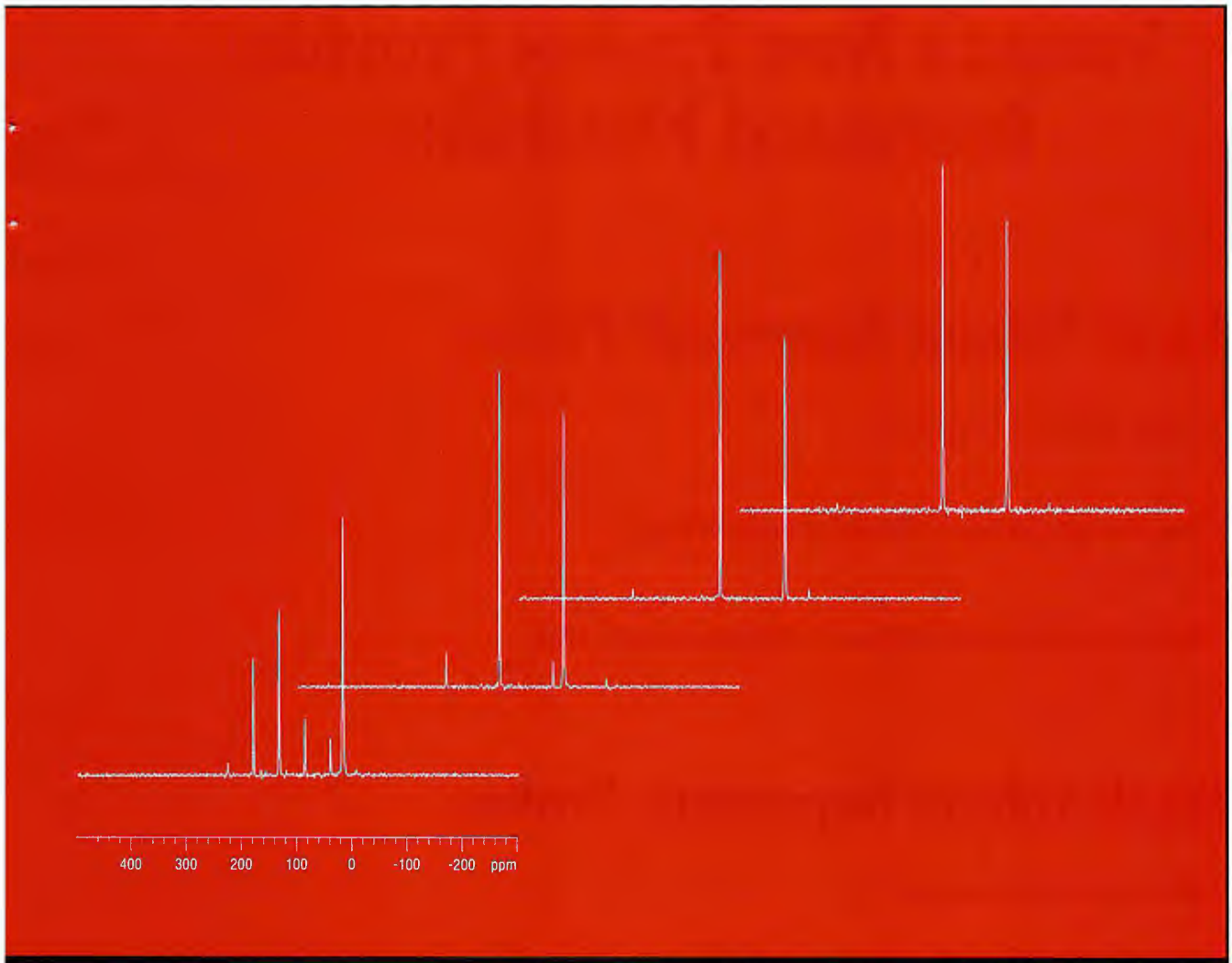
Define a function that will change the amplitude of the first t2 point by using the set and srv commands (ie. set 1, srv 1 1 1.5 1.5, could be used to multiply the first complex point by 1.5). Store the function in a buffer. Obtain the first t2 fid again and multiply it by the function that adjusts the first point amplitude (mwb). After processing, compare the level of the baseline with the level of the mouse arrow position. If the baseline is at a different level than the arrow, change the first point multiplier (ie. the last two values of the srv command) and repeat the above procedure until the level of the f2 baseline and the arrow coincide. All the t2 fids (all values of t1) can then be adjusted by using the set, srv, stb, and mwb commands in a macro.

We often find the above procedure to be more efficient than using a spline or polynomial correction subsequent to FT in t2. The above procedure will raise or lower the entire baseline; baseline roll in f2 should be eliminated prior to spectrum acquisition by adjusting the delay between the end of the pulse sequence and the start of fid collection.

Jeff Ellena

Gordon Rule

Supersonic Spinning for Solid Samples



These spectra of Hexamethyl-benzene were acquired with a Varian 500 MHz NMR spectrometer and 3.5 mm supersonic MAS probe with spinning speeds of 5.8, 12.0, 19.8, and 24 kHz.

Remarkable Flexibility in Spectrometers and Probes

Varian's UNITY*plus*™ NMR spectrometers provide the ultimate in experimental flexibility and performance. Now, you can select from hundreds of new solid probe designs including supersonic MAS, supersonic CP/MAS, DAS, DOR, and REDOR probes.

Small volume supersonic probes offer the fastest spin rates for MAS experiments. And large volume supersonic MAS probes provide the highest sensitivity while simultaneously maintaining a spin rate which keeps spinning sidebands outside of the region of interest.

For more information, contact the Varian office nearest 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

Varian's New Probes Provide Increased Flexibility

Large Volume Supersonic Probes

- Provide the highest sensitivity
- Keep the region of interest clear of spinning sidebands
- Maintain excellent pulse width and performance specifications

Small Volume Supersonic Probes

- Provide the fastest spin rates
- Attain the shortest pulse widths
- Permit high decoupling field strengths

Call your sales representative. Australia (3) 543 8022. Austria (1) 69 55 450. Belgium (2) 721 4850. Brazil (11) 829 5444. Canada (416) 457 4130. Denmark (42) 84 6166. France (1) 69 86 38 38. Germany (6151) 70 30. Italy (2) 753 1651. Japan (3) 3204 2111. Korea (2) 561 1626. Mexico (5) 533 5985. Netherlands (3403) 50909. Norway (9) 86 74 70. Spain (01) 430 0414. Sweden (8) 82 00 30. Switzerland (42) 44 88 44. UK (932) 24 37 41. US 800-356-4437. Other International (415)424-5424.

MAG-7016/857

varian

Duke University

NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY CENTER

LEONARD D. SPICER, DIRECTOR
ANTHONY A. RIBEIRO, MANAGER

(919) 684-4327
(919) 684-6287

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

September 28, 1993
(received 10/1/93)

Determination of Coupling Constant Signs in Halothane

Dear Barry,

Heteronuclear 2D AX correlation schemes yield sharp, single cross peaks connecting individual X (e.g. 13C) to A (e.g. 1H) spins in most cases. However, if a third passive heteronucleus M is present (spin ½), the peak positions are further determined by the heteronuclear coupling with the M spin. This modulation of AX cross peaks by the M spin allows the determination of the relative signs and magnitudes of the relevant coupling constants in a simple manner (1).

Fig. 1 shows the 1H-13C direct bond correlation (HETCOR) map of the anesthetic halothane ($CF_3CHClBr$) in $CDCl_3$. The simultaneous coupling of the three 19F methyls (M spin) to the 1H (A spin) and the 13C (X spin) of the CH group leads to a set of four cross peaks separated and skewed by $^3J_{HF}$ and $^2J_{CF}$ in the 2D map. The highest frequency 1H cross peak near 5.81 ppm occurs at the highest 13C frequency near 50.4 ppm. $^2J_{CF}$ is thus determined to have the same relative sign as $^3J_{HF}$ in halothane. Wong and co-workers (2) and Cholli (3) described similar effects in other compounds.

Fig. 2 shows the analogous 1H-13C multi-bond correlation (LRHETCOR) map of halothane correlating the methine proton to the CF_3 carbon. A set of four cross peaks is observed separated and skewed by $^3J_{HF}$ and $^1J_{CF}$. In contrast to the direct bond results, the highest frequency 1H cross peak near 5.81 ppm occurs at the lowest 13C frequency near 118 ppm. $^1J_{CF}$ is thus determined to have the opposite sign to $^3J_{HF}$ in halothane. We are not aware of a previous application of LRHETCOR to derive coupling constant signs in this manner.

Sincerely, Tony

Anthony A. Ribeiro

1. A. Bax and R. Freeman, J. Magn. Reson. 45, 177 (1981).
2. T. C. Wong, V. Rutar, and J. S. Wang, J. Amer. Chem. Soc. 106, 7046 (1984).
3. A. L. Cholli, Appl. Spectrosc. 45, 839 (1991).

Fig. 1

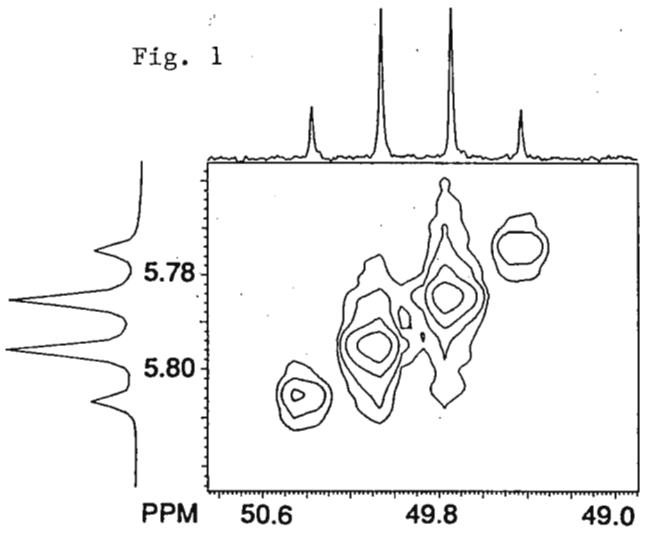
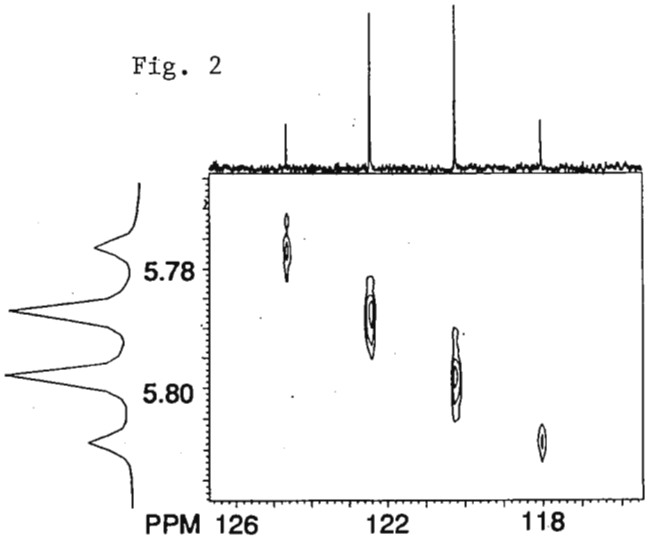


Fig. 2



BOX 3711, DUKE UNIVERSITY MEDICAL CENTER
DURHAM, NC 27710

Universität Basel

Institut für Physik
Experimentelle Kernphysik

Institut für Physik
Klingelbergstrasse 82
CH-4056 Basel
Switchboard +41 61 267 31 11
Telefax 061/ 267 37 84

Prof. Dr. P. Diehl
Direct Dial Number 004161 293703

Electronic Mail Address:
diehl@urz.unibas.ch

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

September 30, 1993
(received 10/4/93)

Anisotropic solvent shifts in anisotropic media

Dear Barry,

Already more than 30 years ago [1] it was pointed out that in isotropic liquids solute protons experience average local magnetic fields on the surface of non spherical solvent molecules with anisotropic magnetic properties. For disk-shaped solvents $\sigma_a = -2n(\Delta\chi/r^3)$ where $\Delta\chi$ is the anisotropy of the solvent diamagnetic susceptibility and n is the number of solvent molecules in the relevant range r. For rod-shaped solvents $\sigma_a = n(\Delta\chi/r^3)$. Subsequently the neglect of the sides or faces of the solvent cylinder was corrected [2] and the relation $\sigma_a = (2/3)\Delta\chi(c-a)/(a+2c)(a^2+c^2)^{2/3}$, where a is the solvent cylinder radius and $2c$ is the cylinder height, was derived. For anisotropic media the anisotropic solvent shifts turned out to be important for the measurement of shift anisotropies [3], however a general relation for σ_a corresponding to the ones mentioned above was not derived. Furthermore it was often wrongly assumed that for cases with $\Delta\chi = 0, \sigma_a$ could be neglected. We have derived this "missing" relation. It is:

$$\sigma_a = n(\Delta\chi + P_2(cos\gamma)S_{LC}(3\chi^{iso} + \Delta\chi)f(A,C)$$

with $f(A,C) = (2/3)(C-A)/(A+2C)(A^2+C^2)^{2/3}$
where γ is the angle between the director of the liquid crystal solvent and the applied magnetic field, S_{LC} is the degree of order of the liquid crystal, χ^{iso} is the diamagnetic susceptibility of the solvent in its isotropic state, $A = a + R$ and $C = c + R$ with a and c as defined above and R is the radius of a spherical solute.

The relation shows that if in anisotropic solvents the S_{LC} value is varied as is necessary for the determination of shift anisotropies, the dominant term in the change in σ_a is not $S_{LC} \cdot \Delta\chi$ but $S_{LC} \cdot 3\chi^{iso}$ because for most thermotropic liquid crystals $3\chi^{iso}$ is 10 times larger than $\Delta\chi$.

If the σ_a-effects are not corrected for, the determination of shift anisotropies by the liquid crystal methods can be seriously in error depending upon the size of the anisotropy. For protons e.g. where the apparent anisotropy is smaller than 10ppm the error is between 10 and 100%. For ^{13}C with apparent anisotropies between 10 and 100ppm the errors are between 10 and 1%.

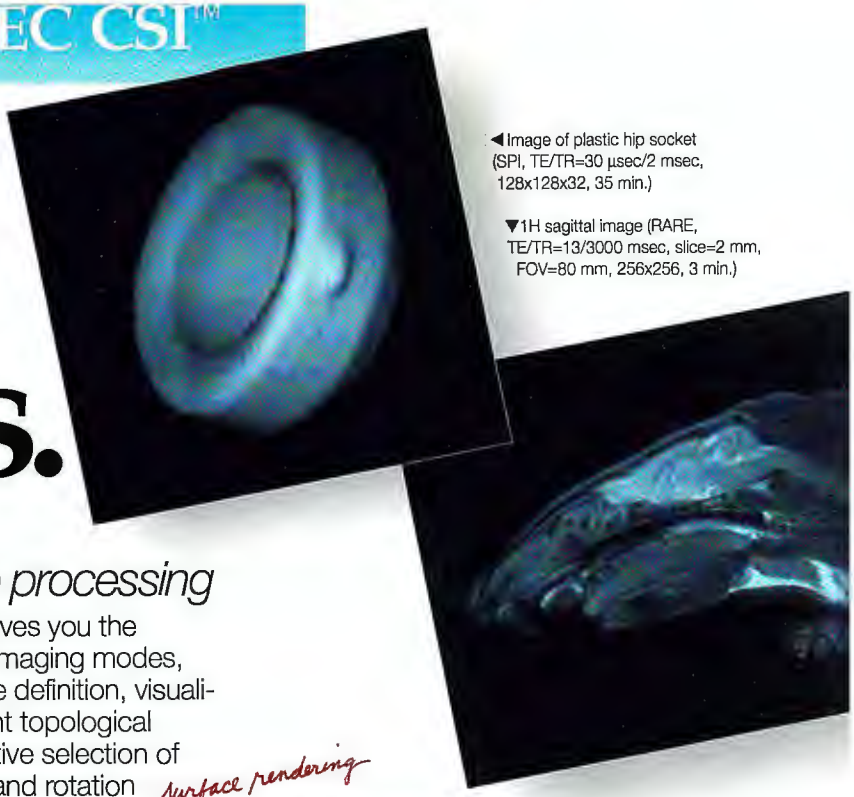
[1] A.D. Buckingham, T. Schaefer and W.G. Schneider, J.Chem.Phys.32,1227(1960)
[2] R.J. Abraham, Molec.Phys.4,369(1961)
[3] A.D. Buckingham and F.E. Burnell, J.Am.Chem.Soc.89,3341(1967)

With kind regards

Prof. Dr. P. Diehl

BIOSPEC CSI™

Real Advances.



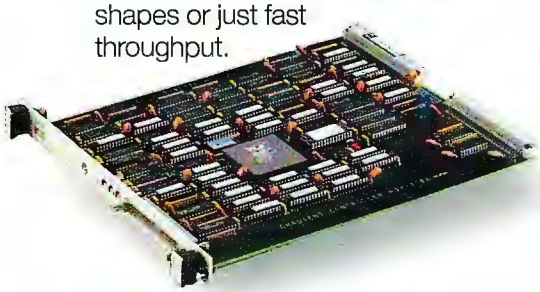
◀ Image of plastic hip socket (SPI, TE/TR=30 µsec/2 msec, 128x128x32, 35 min.)

▼ 1H sagittal image (RARE, TE/TR=13/3000 msec, slice=2 mm, FOV=80 mm, 256x256, 3 min.)

With well over 200 systems in operation and over 30 years of technological leadership in NMR spectroscopy it's more than just a commitment to support CSI and Bruker systems. Our innovations drive the advances in research imaging. Here are four recent examples:

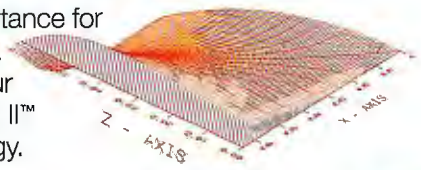
Real-time "on the fly" gradient calculations

for virtually unlimited gradient sequences can now be clocked out in less than 30 µs/point. Our new "intelligent" Gradient Controller with an Intel i960 processor gives you the flexibility to control waveforms which can be configured for complex gradient shapes or just fast throughput.



Optimized gradient design, as illustrated here,

which incorporates minimized gradient coil inductance for shortest switching times thanks to our leading ACUSTAR II™ gradient technology.



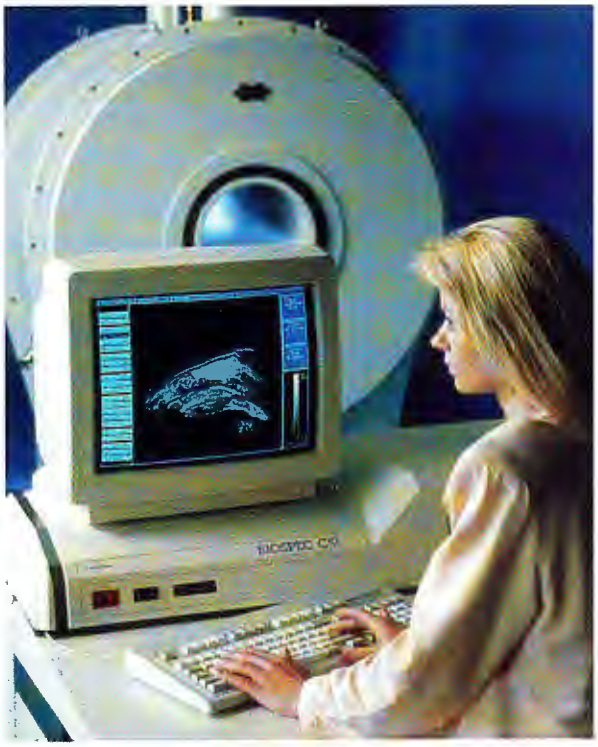
3-D image processing software gives you the whole range of imaging modes, including volume definition, visualization of different topological classes, interactive selection of viewing angles and rotation axes, angiographic reprojection, real-time section selection and volume measurements in 3D space. A powerful imaging tool kit for applications as diverse as plant physiology, medicine, materials and polymer sciences or petrochemical products.

N-Dimensional pipeline acquisition software for high throughput enables you to adjust parameters efficiently and have complete control of the experiment during measurement. User-definable functions include: dataflow and storage, Fourier transformation, window multiplication, phase correction, magnitude calculation, etc., and implementation is easy and user-oriented.

BIOSPEC CSI™ delivers.

So, if you are looking for real answers in imaging today or tomorrow, take advantage of our real advances. We don't mind sharing.

Bruker Instruments, Inc. Manning Park, 15 Fortune Drive, Billerica, MA 01821 (508) 667-9580 Fax: (508) 663-9177
In Europe: Bruker Medizintechnik GmbH, Silberstreifen, D-7512 Rheinstetten 4, Germany (0721) 5161-0



Comprehensive Support for Innovative Systems

BRUKER

The Bruker BIOSPEC CSI™ Series

The BIOSPEC CSI™ product range offers a series of multi-purpose NMR systems designed primarily for biomedical research. All instruments use horizontal axis superconducting magnets with bore diameters of 21 to 60 cm with field strengths from 2.35T to 7.0T (see below). Various RF probes can be used for a wide range of applications in the fields of imaging and spectroscopy.

Double-tuned and frequency-selective preamplifiers under software control make in-vivo spectroscopy easier by allowing shimming on protons and acquisition of the X-nucleus spectrum with the same coil. As part of our development program Bruker works closely with customers when problems arise that are associated with particular applications or the details of RF coil design. This cooperation is an integral part of the support we provide for our BIOSPEC CSI™ customers.

The use of an RF generation/receiving system based on an ultra-high intermediate frequency of 451 MHz eliminates the need for all narrow IF band-pass and image rejection filters, and ensures that all spurious signals are minimized.

Bruker's ASPECT X32 workstation, a high-performance 32-bit computer, is based upon the UNIX™ operating system and allows the operator to define investigational protocols, monitor the system performance and still retain complete control over the instrument. Archiving, post-processing of data and system administration can be carried out in parallel with data acquisition.

The design concept of the ASPECT X32 aims to achieve a great degree of standardization, both in the hardware and in the software. This strategy is demonstrated through the incorporation of the VME System Bus, standard interfaces for peripheral devices, and control units for communication with other computer systems in local or public networks.

To accommodate the broad range of NMR/MRI/MRS applications within one single self-contained module of ASPECT X32 software, BRUKER has developed a comprehensive program package: UXNMR. This program covers all aspects of automation, acquisition, processing and data visualization.

Despite its wide range of applications, the system is relatively simple to operate due to automation features. For research applications the MR software can be used in its full flexibility; for routine applications a menu version of the program can be implemented which permits the execution of standardized experiments with only a few simple commands.

Typical Applications

- Basic Physiology
- Biomedical Research
- Agriculture
- Metabolism
- Petrogeology
- Research in Pharmaceutical Industry
- Materials Science Research

The Biospec CSI™ Magnets

BIOSPEC CSI™	70/20	24/40	47/40	30/60
Bore Size (cm)	21	40	40	60
Field (Tesla)	7.05	2.35	4.70	2.94
^{1}H frequency (MHz)	300	100	200	125
Max. object diameter for imaging (cm)	9	18	18	27
Magnet center to outer flange (cm)	55	57	69	83
Flange diameter (cm)	113	113	120	166
Total height (cm)	215	215	225	230
Total length (cm)	110	121	138	170

All Biospec CSI™ magnets are available with ACUSTAR II actively shielded gradient systems. Bruker is willing to discuss the provision of any non-standard magnet requirements.

For complete details or to arrange a demonstration please contact your Bruker representative.

In the U.S.A. and Canada:

Bruker Instruments, Inc.
Manning Park
Billerica, MA 01821
(508) 667-9580
Fax (508) 667-3954

Bruker Instruments, Inc.
47697 Westinghouse Drive
Fremont, CA 94539
(510) 683-4300
Fax (510) 490-6586

In Europe:

Bruker Medizintechnik GmbH
Silberstreifen
D-76287 Rheinstetten, Germany
(0721) 5161-0
Fax (0721) 5161-305

DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

National Institutes of Health
National Institute on Aging
Gerontology Research Center
4940 Eastern Avenue
Baltimore, Maryland 21224

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

October 14, 1993
(received 10/18/93)

Subject: **Accurate Measurement of T_1 Relaxation Time in the Presence of Chemical Exchange**

Dear Dr. Shapiro:

In order to obtain the T_1 relaxation times of species undergoing chemical exchange one has the option of performing a steady state saturation transfer experiment together with a transient experiment, such as inversion-recovery, or else performing a transient magnetization transfer experiment. In an attempt to avoid these time consuming and complicated procedures, we applied the theory of saturation factors (SF) for exchanging systems (1) to measure the T_1 relaxation times in the presence of chemical exchange without transient experiments.

The advantage of using saturation factors for this measurement is that the T_1's of both components can be obtained from a single measurement of saturation factors with a short repetition time, as in the case of systems with no exchange, combined with a single steady-state saturation transfer experiment which provides the product kT_1. To obtain the T_1 values the equations for SF's as a function of T_i 's given in (1) have to be inverted numerically.

We have applied the method to the creatine kinase reaction, using a Bruker AMX 360 MHz instrument. The sample was prepared according to Kantor et al. (2) with equilibrium ratio of PCr/ATP = 4.0. The steady state saturation transfer experiment was performed by "infinite" irradiation of the γ-ATP signal. Spillover saturation was appropriately accounted for. The product k_+T_1(PCr) was 0.22, and from the measurement of the SF's at the repetition time of 0.84 s we calculated T_1 (γ-ATP) = 1.7 s and T_1 (PCr) = 2.9 s.

We believe that this method can be of practical use if the experiment is performed with all obvious precautions, e.g. precise determination of the flip angle and spoiling of transverse magnetization prior to each pulse.

Yours sincerely,

A. Horská

Alena Horská Jiří Horský Richard G. S. Spencer
NIH/National Institute on Aging, Baltimore, MD 21224

References:

1. R.G.S. Spencer, J.A. Ferretti and G.H. Weiss, J.Magn.Reson. **84**,223 (1989)
2. H.L. Kantor, J.A. Ferretti and R.S. Balaban, Biochim.Biophys.Acta **789**,128 (1984)

Please credit this contribution to Gunther L. Eichhorn's subscription.

422-34

The University of New Mexico

Center for Non-Invasive Diagnosis
1201 Yale Blvd. NE
Albuquerque, NM 87131-5021
Telephone (505) 277-8512
FAX (505) 277-4056

September 27, 1993
(received 9/29/93)

INDIRECT DETECTION WITH TECMAG SYSTEM ON A GN300

Dear Barry,

We recently upgraded our GE (now Bruker) GN 300 Spectrometer with a Tecmag Scorpio[1] pulse programmer and data acquisition station. We wanted the ability to do indirect detection experiments such as HMQC, with our new system. Unfortunately, the spectrometer could only acquire with the observe transmitter and the decoupler transmitter (what would otherwise be the indirect detection transmitter) only operated at the proton frequency.

We were able to work around this by letting the spectrometer think it was pulsing and broad band decoupling at the proton frequency on the decoupler channel and externally rerouting and modifying the signal to the ^{13}C frequency (75 MHz). The decoupler transmitter (at 300 MHz) and an external variable PTS 250 (set to 225 MHz) were hooked up to a double balanced mixer (Anzac MD-143 5-500 MHz). The resulting output, being the sum and difference of the two frequencies (525 and 75 MHz), was put through a 75 MHz band pass filter and hooked to an external power amplifier (EIN model 325LA RF power amplifier). The PTS was synchronized with the spectrometer's 10 MHz clock. From the power amplifier the signal was passed through crossed diodes (Pamona Electronics model 3232), to eliminate pulse break through and RF leakage from the amp, and connected directly to the broad band observe port on the probe. We used the observe transmitter for proton pulsing and acquisition. Using predetermined carbon 90's and broad band decoupling routines we were able to successfully acquire 1H and 13C HMQC data. See Figure 1.

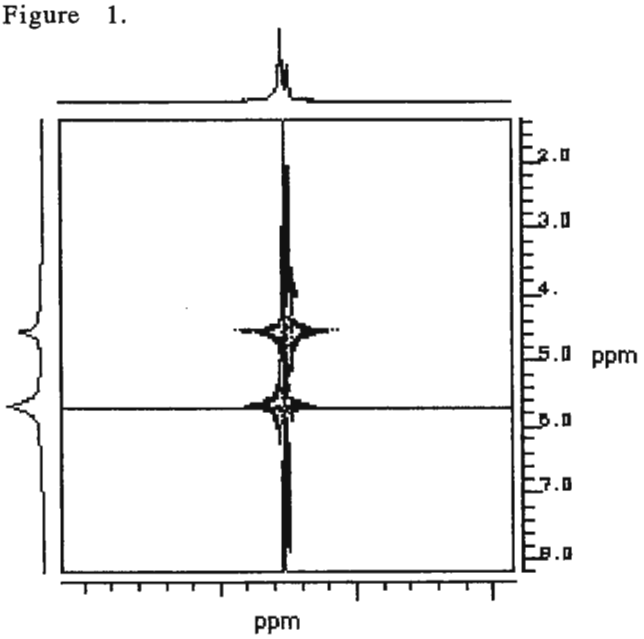


Figure 1: 1H-13C HMQC of D-(1-13C) Mannose in D20 (cross peaks are for alpha and beta anomers).

Sincerely,

Kirsten Berghmans, Kirsten

Chuck Gasparovic (on sabbatical)

Lisa Theisen

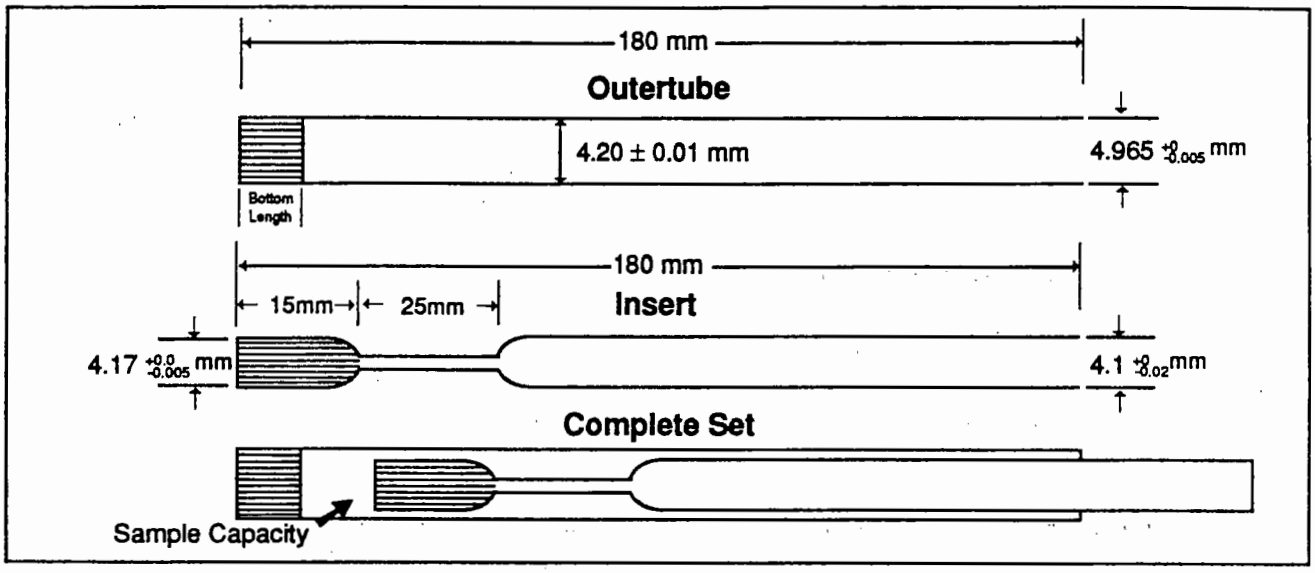
1. Tecmag 6006 Bellaire Blvd., Houston, TX 77081

Specially designed

SYMMETRICAL NMR MICROTUBES

for Aqueous samples

This unique NMR microtube is made of a special type of hard glass which has an excellent chemical durability and **a magnetic susceptibility which matches that of D_2O.** Therefore, the best resolution of a sample can be obtained in a D_2O or H_2O solution.



SHIGEMI SYMMETRICAL 5mm NMR MICROTUBE SYSTEM

Complete Set	Insert length (mm)	Insert ID (mm)	Insert OD (mm)	Outertube length (mm)	Outertube ID (mm)	Outertube OD (mm)	Bottom* length (mm)
BMS-005B	180	2.6	4.1	180	4.2	4.965	8
BMS-005V	180	2.6	4.1	180	4.2	4.965	15

***For best results, choose the one that matched your probe coil height most closely.**

SHIGEMI, INC.

4790 Route 8 • Allison Park, PA 15101 • USA

Tel: (412)444-3011 • Fax: (412)444-3020

Specially designed

Thin Wall NMR Sample Tube

Shigemi's high precision thin wall NMR sample tube has a unique construction. The wall thickness of this particular tube is reduced only around the position of the detection coil. The result of this new invention allows an increase in the sample volume and higher sensitivity without sacrificing its mechanical strength. Therefore, there is no need for special handling during routine usage of our Shigemi NMR tubes.

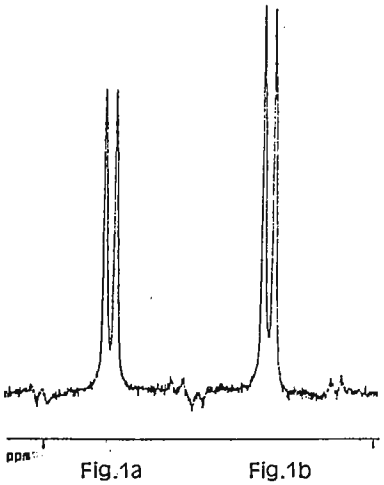
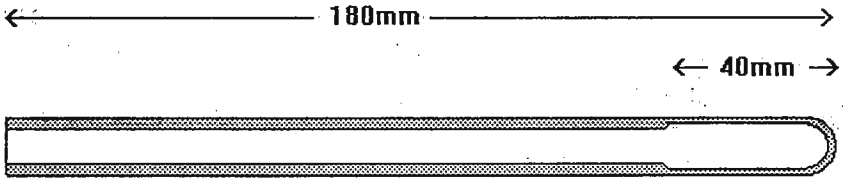
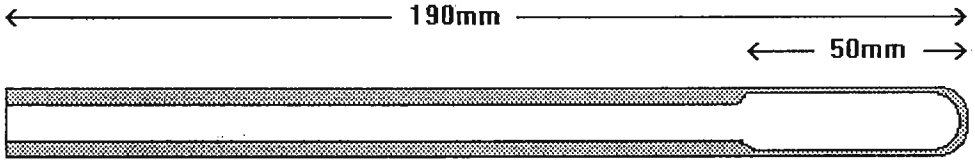


Fig.1a Fig.1b

The spectra of 20mm sucrose in D_2O were obtained with a single scan without apodization prior to Fourier transformation on a Bruker AMX-600 spectrometer at 298 K. By using Shigemi high quality 5mm standard tube (Fig.1a) and the Shigemi highly sensitive thin wall 5mm tube (Fig.1b), the spectra confirms a sensitivity enhancement of about 10%.



PST-001 and PST-002



ST8-001,ST8-002, ST10-001, and ST10-002

O.D. (mm)	Product Number	Wall (mm)	Concen - tricity/ Camber (μ)	OD (mm/in)	ID (mm/in)
5.0	PST-001	0.21	20/ 8	4.96 + 0.00 – 0.01	4.54 ± 0.01
	PST-002	0.21	40/15		
8.0	ST8-001	0.25	40/ 8	8.00 + 0.00 – 0.01	7.52 ± 0.01
	ST8-002	0.25	50/15		
10.0	ST10-001	0.25	40/ 8	9.98 + 0.00 – 0.01	9.52 ± 0.01
	ST10-002	0.25	50/15		

SHIGEMI, INC.

Suite 21, 4790 Route 8 • Allison Park, PA 15101• USA

Tel:(412)444-3011• Fax(412)444-3020

Acorn NMR

46560 Fremont Blvd., #418
Fremont CA 94538-6491
Telephone: (510) 683-8595
FAX: (510) 683-6784

October 20, 1993
(received 10/22/93)

Re: Vile Vials and Crappy Caps

Dear Barry,

On the principle that you should learn from the mistakes of others because you won't live long enough to make them all yourself, we thought your readers might benefit from our recent experience.

Many of our spectroscopy service customers find it convenient to send samples in small vials, frequently using those plastic vials which are so near and dear to the hearts of biochemists. Because these samples often consist of only 1-2 mg, to minimize sample loss and the possibility of spilling, we often add solvent to the vial and then pipette the solution into an NMR tube. The appearance of impurity peaks in the spectrum of a customer's sample led us to reconsider this procedure.

It turns out that $CDCl_3$ leaches "stuff" out of the plastic vials. We tested 3 different small plastic sample vials supplied by customers. $CDCl_3$ was allowed to stand in each vial for a couple of hours and then a spectrum was acquired. The results are shown in the first 3 spectra below. The same experiment using MeOH and DMSO showed no impurity peaks.

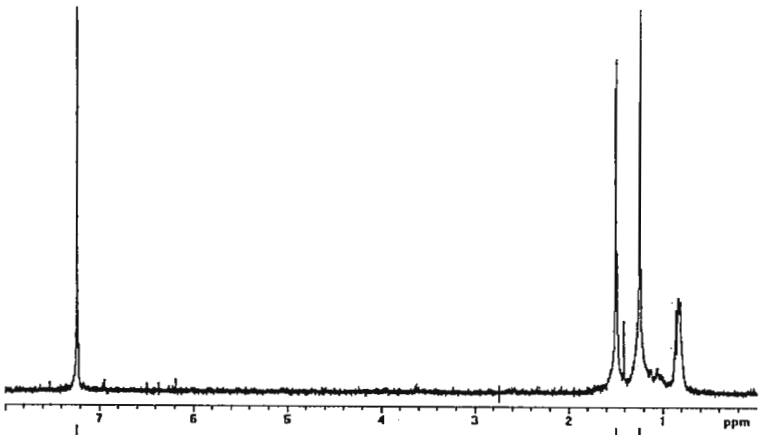
These results led us to wonder about the screw caps on our glass vials, which can be obtained with liners made of various materials. The bottom spectrum is the result of $CDCl_3$ which was allowed to stand in a glass vial whose cap was lined with white rubber. (Note that no TMS was added to that sample.) Similar results were obtained with acetone. We consulted VWR about this problem, who recommended teflon lined caps, but even those are not recommended for $CDCl_3$. We have teflon lined caps on order, so have not yet had the opportunity to test them. If any of your readers has a better solution, we would appreciate hearing from him / her.

All of this reminded us of an experience familiar to most chemists: the isolation and identification of dioctyl phthalate from a reaction mixture (the plasticizer in Tygon tubing).

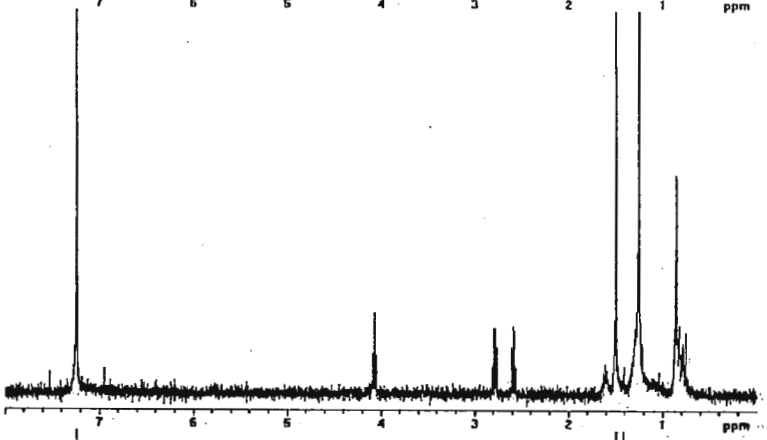
Regards,

Gina Miner

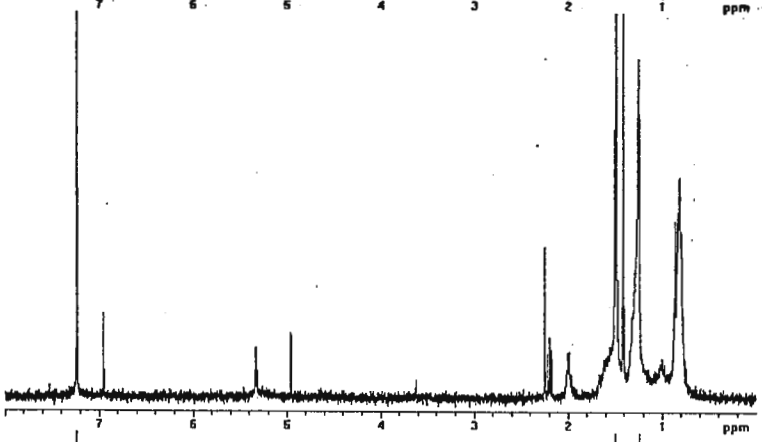
Woody Conover



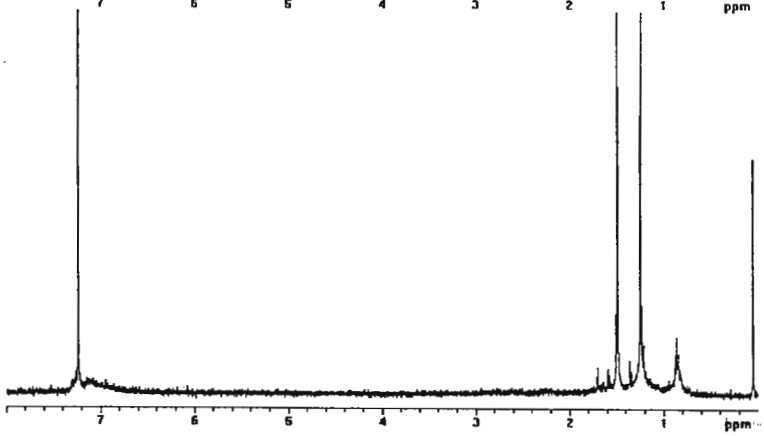
Corning cryo-tube



"Mini-centrifuge" tube



Eppendorf tube



Glass vial with cap lined with white rubber

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-3,3,3-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

NEW from Isotec!

☞0.6ml sizes for bulk orders of the following NMR solvents:

Acetone-d_6	99.9 atom%
Benzene-d_6	99.6 atom%
Dimethyl-d_6 Sulfoxide	99.9 atom%
Methyl Alcohol-d_4	99.8 atom%

+ other deuterated NMR solvents!

☞Under Production!

Water-^{17}O, enriched to 50%
Dimethyl-d_6 Sulfoxide ULTRA, enriched to 99.999%

☞ISOTEC *has the largest in-house inventory of stable isotope enriched metals!*

ISOTEC continues to serve the growing demands of the research community for new, interesting compounds. Below is a list of a few such compounds under production or now available:

83-82009-2	Acrylamide-$^{13}C_3$	99 atom%
82-20219-3	Choline-d_9 Chloride (Trimethyl-d_9)	98 atom%
85-12272-9	L-Cysteine-^{15}N	99 atom%
82-70041-0	Deuterium Oxide ULTRA-D	99.999 atom%
82-00815-2	DL-1,4-Dithiothreitol-d_{10} (Cleland's Reagent)	98 atom%
81-61002-4	ISOGRO™-^{13}C,^{15}N Powder-Growth Medium	99 atom% ^{13}C, ^{15}N
	2-Mercaptoethanol-1,1,2,2,-d_4	98 atom%

To recieve 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

The University of Texas Medical Branch at Galveston

School of Medicine
Graduate School of Biomedical Sciences
School of Allied Health Sciences
School of Nursing

Marine Biomedical Institute
Institute for the Medical Humanities
UTMB Hospitals

Marine Biomedical Institute

MR MICROSCOPY OF FIXED CEPHALOPOD BRAINS

10/11/93
(received 10/19/93)

Hey Barry,

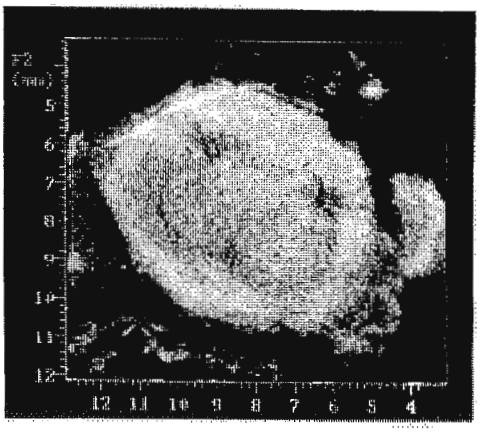
Greetings from Galveston! We have just finished the installation of our new 400 MHz UNITY*plus* system, complete with liquids, solids and imaging capabilities. This is the first of three instruments, which include a 600 and a 750, that the Molecular Science Institute at UTMB is getting from Varian, mostly to do protein structure determination. Although we are generally happy with the performance of the 400, we were surprised and a little dismayed that our SISCO pulse sequences and macros don't just "plug in" to the Unity. Even though both systems run VNMR software, there seem to be as many differences as there are similarities in their execution of imaging. We are grateful to Dr. Simon Chu of Varian who has initiated the integration of SISCO routines into Varian software on the Unity system.

In my laboratory we are mainly interested in imaging, so we have tested the 400's μ-imaging capabilities on fixed cuttlefish brain specimens, placed in D_2O for a susceptibility-matched low intensity background. The cuttlefish is a cephalopod with a fairly advanced brain for such a primitive creature. We are working with Dr. B.U. Budelmann and his student Heike Neumeister to elucidate brain pathways. In certain cases, MR microscopy offers an alternative to histological analyses, if one is willing to compromise on spacial resolution. In our initial runs we have obtained 30 μm in plane resolution (zero-filled once in each dimension) with 0.45 mm slices. In an unlabeled, formalin fixed specimen (see image below), areas rich in cell bodies are higher in signal intensity, while areas of lower intensity indicate axonal fibers connecting the various regions of the brain. These contrast patterns are analogous to those observed in T2-weighted images of mammalian brains where grey matter is of higher signal intensity than white matter. The high intensity perimeter of this slice is separated from the middle by fiber tracts or commissures (reduced intensity).

One goal of the project is to resolve individual cell bodies within the brain matrix which have diameters up to around 60 μm. Already our images hint at resolving cell bodies which appear as bright dots (not visible in this low quality reproduction). These dots are in the correct location and have approximately the right diameters to be cell bodies. In most cases these dots are probably projections of more than one cell. We'll have to do better on slice thickness to resolve single cells.

In these initial experiments we have used the Varian 25 mm saddle-style coil. Our spacial resolution was limited by sensitivity. We hope to increase our sensitivity and spacial resolution further by using a transversely oriented solenoid rf coil.

Please credit this correspondence to the account of Dr. L. L. Smith.



Mike Quast Ed Ezell

200 UNIVERSITY BOULEVARD SUITE 610 GALVESTON, TEXAS 77555-0843 (409) 772-2101 FAX (409) 762-9382

INDIANA UNIVERSITY
PURDUE UNIVERSITY
INDIANAPOLIS

October 4, 1993
(received 10/9/93)

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

IBM Clone Data Acquisition System

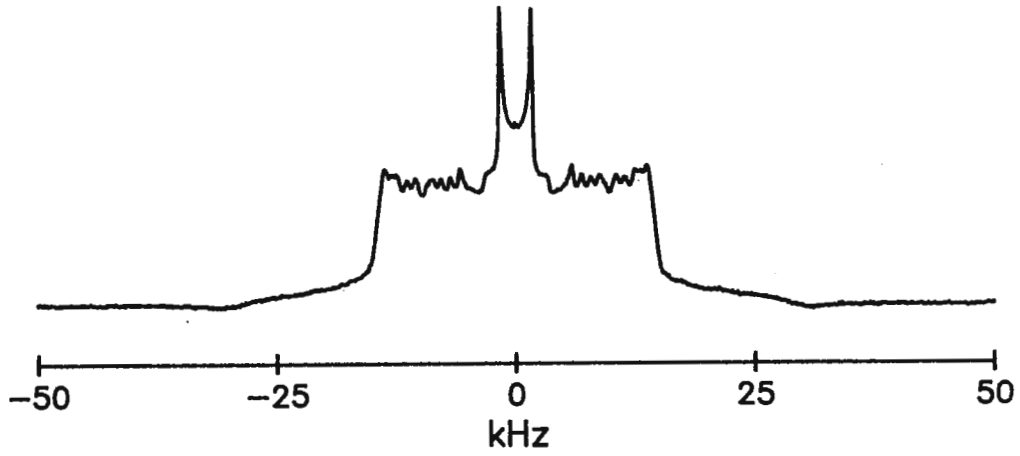
Dear Dr. Shapiro:

SCHOOL OF SCIENCE

IUPUI

As part of the continuing development of our homebuilt broadline NMR spectrometer (TAMU 362-23), we have finally replaced an antiquated Nicolet 1080 computer based pulse programming and data acquisition system with a Renaissance 486 computer based system. Pulse programming is performed with a UBC programmable pulse generator, while data is acquired *via* a Rapid Systems R1200M dual channel digital oscilloscope. The graphical interface software was written in Borland C++ for Windows, utilizing Microsoft Windows version 3.1 on a DOS 5.0 platform. Apart from its relatively modest cost, virtues of the system include: downloadable pulse programming with tremendous flexibility; fast digitization up to a 1 MHz sampling rate; multiple display modes and online FFT.

An example of a ^2H NMR spectrum recorded for [$^2H_{62}$] 16:0-16:0 PC bilayers



with our system, which is currently operational but far from bug free, is shown. Once we have finished debugging, we intend to publish a description and shall be glad to provide copies of the program.

Please credit this contribution to the account of B. D. Nageswara Rao.

Yours sincerely,

DEPARTMENT OF PHYSICS

LD 3154
402 N. Blackford Street
Indianapolis, Indiana
46202-3273

317-274-6900
Fax: 317-274-2393

George L. Griffith M. Alan McCabe Bruce D. Ray Stephen R. Wassall

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 Fluorescense
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 QC
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 anayzer
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 flourometer
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.

SERVING THE SCIENTIFIC COMMUNITY FOR 13 YEARS 1980-1993

International Equipment Trading, Ltd.
960 Woodlands Parkway • Vernon Hills, Illinois 60061 **(708) 913-0777** **Fax (708) 913-0785**

TRIPOS
Associates Inc.
A subsidiary of EVANS & SUTHERLAND

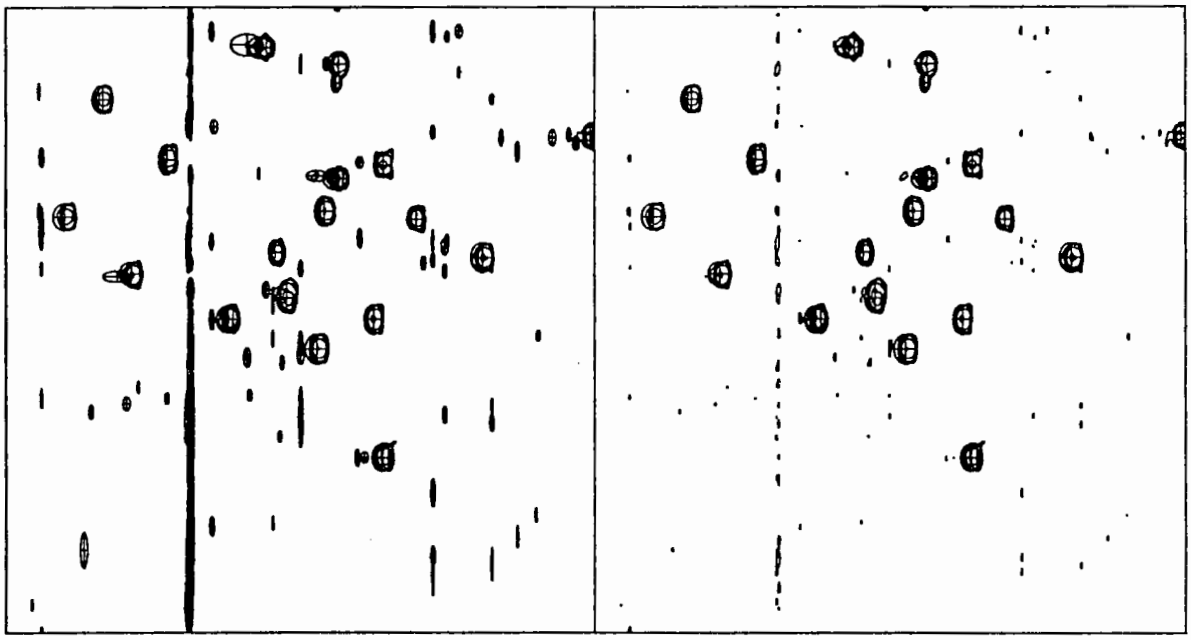
(received 10/2/93)

Something to "Peak" your interest

Dear Barry,

It's been a while since I contributed something, and what a long strange trip it's been. In keeping with my interests in the automation of NMR analyses, and in general attempts to make more useful software to deal with data, here are some interesting (I think) applications of the problem of getting high quality peak tables for automated NMR assignments or structure determination. What I've done is implement some macros in the Sybyl/NMR Triad software package that turn a rather ugly automatically picked peak table into something of beauty. Others may recognize that it usually is not very useful to use automatic peak picking, since one must then manually edit the peak table to remove noise, artifacts, and deal with other problems common in automatic peak pickers. Here I will show it is possible to use automatic peak picking and mostly-automated peak table refinement to get good peak data.

The data are a 2D Tocsy acquired on ubiquitin in water. The first steps performed (and displayed below) are (1) automatic picking of all peaks with maxima above a given value; (2) elimination of all peaks with widths <3.5 points and half-widths less than 2.5 points where the data intensity was less than twice the threshold. This second step is very effective in removing noise peaks, and is easily performed using the spreadsheet operations supported. The minimum peak widths and half-widths were determined by examining a 3D scatter graph of either the widths or half-widths versus the peak intensity.



After autopicking: 118 peaks in this region **After noise removal: 23 peaks remain**

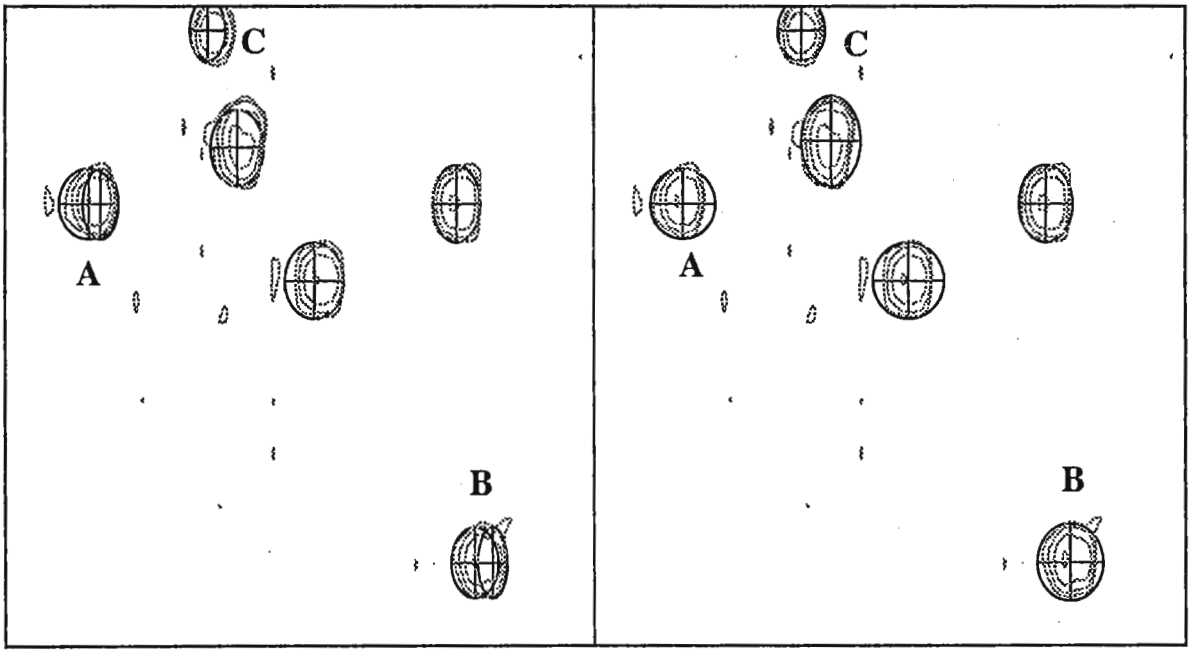
Next, peaks were removed from the diagonal, and peaks without a symmetry-related partner, that are also less than 7.5 points wide are removed. Luciano Mueller's group at Bristol-Meyers Squibb

HEADQUARTERS: 1699 South Hanley Road, Suite 303 St. Louis, Missouri, 63144, U.S.A.
(314)647-1099 FAX-(314)647-9241 800-323-2960

OVERSEAS OFFICES: England, France, Germany, Japan

made the suggestion not to remove peaks that have reasonable peak widths, but are missing a symmetry-related partner, since not all experiments, samples and spectrometers are perfect.

The last peak table refinements were performed to deal with some of the nuances of TOCSY data when subjected to automatic peak picking. The peaks shown are from the "fingerprint" region, and it is not uncommon that when the HN coupling is quite strong, the peak exhibits a doublet character in F2. An automatic peak picker will usually find two peaks (not one), but these cases are readily distinguished and corrected. I wrote a macro that will look for peaks that have nearly identical F1 shifts, and with F2 shifts within 15Hz (or so). These peaks are collapsed into a single peak. The final step is to adjust all peak positions and "footprints" using a center of mass correction, which will alleviate some of the problems of poorly-phased data, etc. This last step also is performed using another Sybyl/Triad macro. Note that the peak merging steps and the center of mass adjustments are also done in the EASY program developed in Prof. Dr. Kurt Wüthrich's lab.



Before peak merging and center-of-mass adjustment

After. Peaks A&B have been merged, while peak C displays good c-of-mass adjustment.

In summary, it is possible to save a lot of time using automatic peak picking to generate a peak table, and to clean it up with mostly automated peak refinements. The only manual intervention required was to determine the parameters to define "noise" peaks, but this need only be done once for data of a particular matrix size and sample linewidth. [I will provide all macros used to any Triad users (email: weber@tripos.com)].

Sincerely,

Dr. Paul Weber
NMR Product Manager

International Symposium on Biological NMR

On the Occasion of Professor Oleg Jardetzky's 65th Birthday

March 24-26, 1994

Stanford University, Stanford, California

Organizers: J. L. Markley (Wisconsin) and S. J. Opella (Pennsylvania)

Preliminary List of Speakers: K. Akasaka (Kobe), R. B. Altman (Stanford), C. H. Arrowsmith (Toronto), S. Chan (CalTech), J. S. Cohen (Georgetown U.), M. Cohn (Pennsylvania), P. Cozzone (Marseille), C. M. Dobson (Oxford), O. Jardetzky (Stanford), T. S. Jardetzky (Harvard), J.-F. Lefèvre (Strasbourg), M. Levitt (Stanford), W. N. Lipscomb (Harvard), L. Litt (UC San Francisco), J. L. Markley (Wisconsin), H. M. McConnell (Stanford), S. J. Opella (Pennsylvania), W. D. Phillips (Washington U.), G. C. K. Roberts (Leicester), J. K. M. Roberts (UC Riverside), R. G. Shulman (Yale), D. Wemmer (UC Berkeley)

For information and registration fax or write to: Ms. Robin Holbrook, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, CA 94305-5055, USA; Fax: +415/723-2253

18 October, 1993

Symposium on *In Vivo* Magnetic Resonance Spectroscopy VII.

The *In Vivo* Magnetic Resonance Spectroscopy Symposium is an advanced workshop emphasizing experimental methods and techniques directed towards *in vivo* magnetic resonance spectroscopy, with ample time intended for discussion. The Symposium will be held at the Hyatt Regency Hotel, Monterey, California, on April 9 - 10th, 1994, just prior to the 35th ENC meeting. For further information contact Radiology Postgraduate Education, Room LS-105, University of California, San Francisco, CA 94143-0742, tel (415) 476-5731, fax (415) 476-9213. Those wishing to make presentations at the Symposium should send a 1/2 page abstract to Dr. Gerald Matson, Department of Veterans Affairs Medical Center, 4150 Clement St. 11M, San Francisco, CA 94121, before March 4, 1994.

WANTED: One good used magnet-Dewar for a Varian XL-300 (50 mm bore) NMR spectrometer. O-Ring seals preferred but will consider the older version with indium seals if in good working condition. The Dewar should have proper connections to interface with the probes. If probes are also available, we would considerr them as well. Please contact:

Dr. K. Darrell Berlin
Department of Chemistry
Oklahoma State University
Stillwater, OK 74078

PHONE: (405) 744-5950
FAX: (405) 744-6007

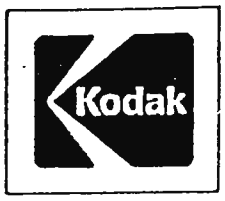
Chevron

Chevron Research and Technology Company
P.O. Box 1627
Richmond, CA 94802-0627
Phone (510) 242-3000
Fax (510) 242-4647

Equipment Available

The following equipment is available to any nonprofit research, charitable, or educational institution willing to pay shipment costs: a WH-90 stand-alone magnet system (no console or computer). High-resolution 2-cm gap iron magnet and power supply in base; Haskris cooler; probe arm, probes, and preamplifier; ^{2}H lock; variable temperature unit; homogeneity system. All working in 1990. Good for prototype process analyzer applications. Ready for high resolution experiments two hours after activating. Entire system only, no parts.

Contact: D. M. Wilson (510) 242-2415
Fax (510) 242-4647



Eastman Kodak Company

September 19, 1993
(received 10/1/93)

Hypershim: A New Autoshim Method

Dear Dr. Shapiro,

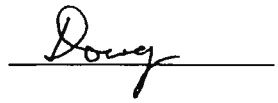
We currently operate three Sun-based Varian spectrometers - a VXR-300s and 2 Unity 500's - and have had some problems with Varian's autoshim routine on these instruments. When performing even "simple" optimizations such as a z1, z2 correction on a sample in CDCl3, the Varian routine will sometimes stop at a point that can be significantly improved by hand (mouse?) and occasionally jump to worse values and never recover. Also, autoshimming of the higher order spin and nonspin shims is usually ineffective. At least some of these problems can be traced to the optimization routine Varian uses.

However, Varian supplies a "sethw" command that can be used to set hardware shim values directly and a "readlk" command to read the lock level. Along with the Magical II macro language, this is all that is needed to create a custom shimming routine. We have recently implemented an autoshim macro that features the Hypersphere optimization method (Brown, et al, JMR, 85:15, 1989). This method uses a multidimensional spherical search algorithm that we have found over the past few years to be significantly better than Simplex and various steepest-descent nonlinear optimization methods in a variety of applications. The algorithm takes a lot of "bad" steps and so generally converges more slowly than other methods, but the randomness adds a function similar to that of simulated annealing by allowing these "bad" iterations to continue and therefore skip over local minima. In several large (hundreds of variables) optimizations we have performed over the years, the performance of the Hypersphere method can best be described as relentless and effective.

The macro system we have constructed for autoshimming we call *Hypershim*. Hypershimming is accomplished by entering the macro name followed by the shims to be optimized - for example *hypershim('z1c','z2c','z3')*. The early results look very promising. We have tested the effectivness of the method by finding starting conditions that the Varian algorithm bogs down with and then starting Hypershim from the same conditions. Hypershim usually ends with a lock level that cannot be improved manually. So far the routine has been kept pretty simple, with crude methods to account for solvent relaxation times and varying step sizes but it does seem to function well. A problem noted is that with difficult samples such as lineshape standards, the lock level will initially increase then decrease as the method cannot keep up with the hysteresis involved. Also, Hypershim on our Sun 4/110 computer (VXR-300s spectrometer) will occasionally abort in the middle of an optimization with an error stating the acquisition system is too slow. Another nice feature of Hypershim is that it has been set up as a shell program to do the bookkeeping and call the optimization routine, so another optimization method can be easily substituted for the Hypersphere routine.

Hypershim has been submitted to Varian's Userlib.

Sincerely,



Douglas E. Brown
Eastman Kodak Company 82/c204
Rochester, NY 14650-2132
(716)477-5175
debrown@kodak.com

Table of Contents, cont'd.

MR Microscopy of Fixed Cephalopod Brains . . . . . **Quast, M.,** and **Ezell, E.** 41

IBM Clone Data Acquisition System **Griffith, G. L., McCabe, M. A., Ray, B. D.,** and **Wassall, S. R.** 42

Something to "Peak" Your Interest . . . . . **Weber, P.** 45

International Symposium on Biological NMR, On the Occasion of Professor Oleg Jardetzky's 65th Birthday; Stanford, California, March 24-26, 1994 . . . . . **Holbrook, R.** 47

Symposium on *In Vivo* Magnetic Resonance Spectroscopy, VII; Monterey, CA, April 9-10, 1994 **Matson, G.** 47

Equipment Wanted . . . . . **Berlin, K. D.** 48

Equipment Available . . . . . **Wilson, D. M.** 48

Hypershim: A New Autoshim Method . . . . . **Brown, D. E.** 49

♣ ♦ ♥ ♠ ♣ ♦ ♥ ♠

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. 424 (Jan. 1994)	17 December 1993
No. 425 (February)	21 January 1994
No. 426 (March)	18 February 1994
No. 427 (April)	25 March 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.

The Leader

in stable isotope labeled compounds & research products

13C

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

15N

CIL

Please call for product information and to receive a catalog

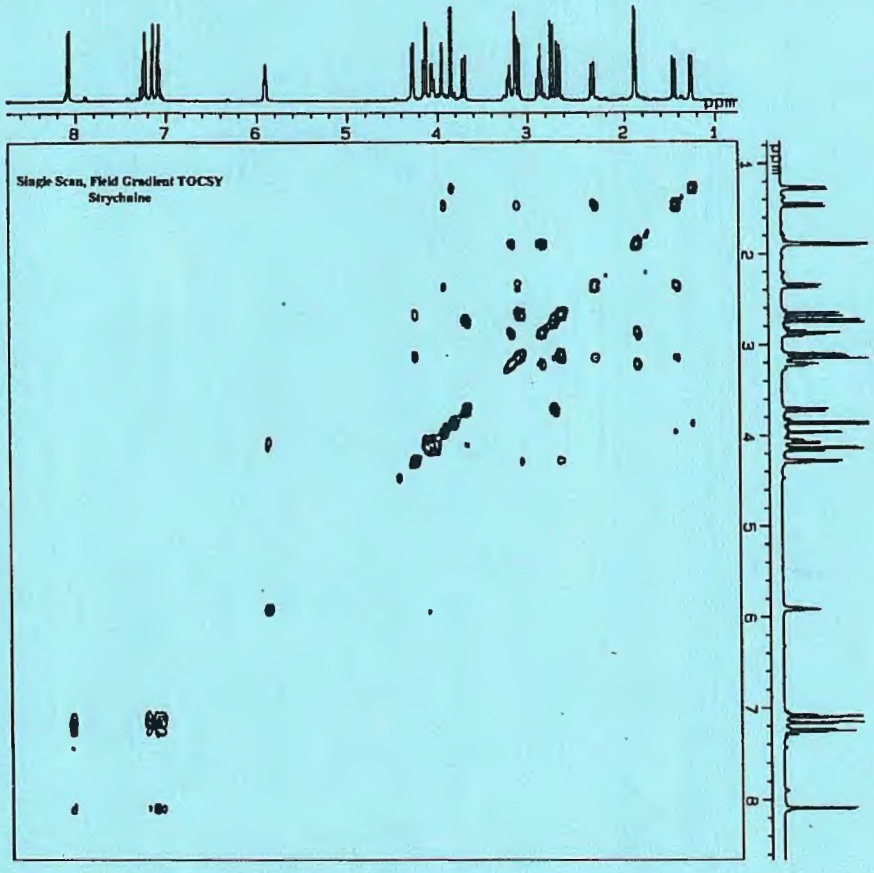
CAMBRIDGE ISOTOPE LABORATORIES
20 Commerce Way, Woburn, Massachusetts 01801

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

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
908/254-7026
FAX 908/254-8850

JEOL, CA
3500 West Bayshore Rd
Palo Alto, CA 94303
415/493-2600
FAX 415/493-0581

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

JEOL NMR

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.
525/515-2616
FAX 525/515-3261