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<table>
<thead>
<tr>
<th>Cat. No.</th>
<th>Name</th>
<th>Formula</th>
<th>Min. Inst</th>
<th>MP (°C)</th>
<th>BP (°C)</th>
<th>$\chi \times 10^{6}$ @ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>D-11</td>
<td>DMSO-d$_6$</td>
<td>D$_2$O</td>
<td>0.999</td>
<td>56</td>
<td>62</td>
<td>0.551 (32)</td>
</tr>
<tr>
<td>D-12</td>
<td>Acetone-d$_6$</td>
<td>C$_3$H$_6$O$_6$</td>
<td>0.995</td>
<td>0.3</td>
<td>0.460 (20)</td>
<td></td>
</tr>
<tr>
<td>D-13</td>
<td>Acetone-d$_6$</td>
<td>C$_3$H$_6$O$_6$</td>
<td>0.995</td>
<td>0.3</td>
<td>0.460 (20)</td>
<td></td>
</tr>
<tr>
<td>D-121</td>
<td>Chloroform-d</td>
<td>C$_3$H$_5$O$_2$</td>
<td>0.898</td>
<td>1.50</td>
<td>0.746 (20)</td>
<td></td>
</tr>
<tr>
<td>D-122</td>
<td>Chloroform-d</td>
<td>C$_3$H$_5$O$_2$</td>
<td>0.898</td>
<td>1.50</td>
<td>0.746 (20)</td>
<td></td>
</tr>
<tr>
<td>D-21</td>
<td>Benzene-d$_6$</td>
<td>C$_6$H$_6$</td>
<td>0.999</td>
<td>544</td>
<td>544</td>
<td>0.611 (20)</td>
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<tr>
<td>D-129</td>
<td>Tetrachloroethane-d$_2$</td>
<td>C$_2$H$_4Cl$_4$</td>
<td>0.898</td>
<td>1.50</td>
<td>0.746 (20)</td>
<td></td>
</tr>
<tr>
<td>D-130</td>
<td>Octane-d$_8$</td>
<td>C$<em>8$H$</em>{16}$</td>
<td>0.999</td>
<td>1.50</td>
<td>0.746 (20)</td>
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<tr>
<td>D-28</td>
<td>Trifluoroacetic Acid-d</td>
<td>C$_3$F$_4$O$_2$</td>
<td>0.898</td>
<td>1.50</td>
<td>0.746 (20)</td>
<td></td>
</tr>
</tbody>
</table>

Cost-conscious quality NMR solvents offered by Wilmad, such as CDCl$_3$, are frequently priced lower than more traditional sources. Included in this offering are the most common solvents, like Acetone-d$_6$, Benzene-d$_6$, D$_2$O, and DMSO-d$_6$, as well as some of the most unusual solvents for specialty applications, like 1,1,2,2-Tetrachloroethane-d$_2$, Octane-d$_8$, and Trifluoroacetic Acid-d.

---

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<th>BOX/500 SHEETS</th>
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<tr>
<td>WCV-100</td>
<td>11&quot; x 16 3/4&quot;</td>
</tr>
<tr>
<td>WCV-60</td>
<td>11&quot; x 16 3/4&quot;</td>
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<tr>
<td>WCV-XL</td>
<td>11&quot; x 17&quot;</td>
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<tr>
<td>WCV-XL/T</td>
<td>11&quot; x 17&quot;</td>
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<tr>
<td>WCV-XL/20</td>
<td>11&quot; x 17&quot;</td>
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<tr>
<td>WCV-20 (CFT-20)</td>
<td>11&quot; x 16 3/4&quot;</td>
</tr>
<tr>
<td>CFT-20, FT-80, FT-80A</td>
<td>1&quot; x 17&quot;</td>
</tr>
</tbody>
</table>

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The Lilly Research Laboratories, Eli Lilly & Company
Merck Research Laboratories 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: Dr. Robin Hallock, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, California 94305-6535; Fax: (415) 723-2233; 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-334, 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; See TAMU NMR Newsletter 422, 47.

35th ENC (Experimental NMR Conference), Astirion Conference Center, Pacific Grove, California, April 10 - 15, 1994; Contact: ENC, 115 Don Guapir, 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, Stcacie Institute for Molecular Sciences, National Research Council of Canada, 100 Sussex Drive, Ottawa, ON K1A 0R6, Canada; (613) 993-1212; Fax: (613) 954-5242 See TAMU NMR Newsletter 422-9.

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 435; Tel: (902) 484-3804; Fax: (902) 494-1130. See TAMU NMR Newsletter 424-11.

12th International Meeting on NMR Spectroscopy, Sponsored by the Royal Society of Chemistry, Manchester, England, July 2 - 7, 1995 [sic]; Contact: Dr. J. F. Cahn or Mr. G. B. Howlett - See TAMU NMR Newsletter 423-9; 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 422, 26.

Additional listings of meetings, etc., are invited.
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 an AM300 Bruker system equipped with a gradient unit. Figure 1 shows the temperature distribution across a 5 mm od, 46 mm height $[K_3(Co(CN)_6) / D_2O]$ 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.](image)

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

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 1H-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.

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decoupling coils
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  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 t1-noise and
  artifacts for GRASP experiments
• significantly boosted throughput due
to GRASP techniques

Figure 1: NOESY experi-
ment of 1 mg of Lysozyme
in 0.1 ml of 80% H2O/
10% D2O. Mixing time
150 msec, 64 scans were
recorded for each of
512 t1-increments. Expe-
riment time 12h 15 min,
2.5 mm TXI probe.
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.

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- triple resonance inverse (TXI, 'H observe, 'C and 'N dec.)

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

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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 \( \mu \)m (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](image)

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 $90^\circ$ pulse. In this case the flowing spins are restored along the $z$ axis while stationary spins are completely dephased by the imaging slice gradients.

References


Yours sincerely,

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<td>3130</td>
<td>200–500 MHz</td>
<td>50 to 300 W</td>
</tr>
<tr>
<td>3200</td>
<td>6–220 MHz</td>
<td>150 to 1000 W</td>
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<tr>
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<td>30–310 MHz</td>
<td>400 W</td>
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<tr>
<td>3420</td>
<td>10–90 MHz</td>
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- 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.

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

<table>
<thead>
<tr>
<th>Time</th>
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<tr>
<td>3 - 9 pm</td>
<td>Registration</td>
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<tr>
<td>6 - 8 pm</td>
<td>Western Cook-out</td>
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**Monday, April 11**

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<tr>
<th>Time</th>
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<tbody>
<tr>
<td>morning</td>
<td>Poster session I, H. Eckert, Chair</td>
</tr>
<tr>
<td></td>
<td>Opening remarks</td>
</tr>
<tr>
<td></td>
<td><em>In vivo</em> NMR, J. L. Ackerman, Chair</td>
</tr>
<tr>
<td>F. Wehrli, University of Pennsylvania, Philadelphia, PA, USA</td>
<td></td>
</tr>
<tr>
<td>D. Ailion, University of Utah, Salt Lake City, UT, USA</td>
<td></td>
</tr>
<tr>
<td>G. A. Johnson, Duke University, Durham, NC, USA</td>
<td></td>
</tr>
<tr>
<td>afternoon</td>
<td>Materials Imaging, D. G. Cory, Chair</td>
</tr>
<tr>
<td>L. D. Hall, University of Cambridge, Great Britain</td>
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**Tuesday, April 12**

<table>
<thead>
<tr>
<th>Time</th>
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<tbody>
<tr>
<td>morning</td>
<td>Selective Excitation in Liquid-State NMR, R. Freeman, Chair</td>
</tr>
<tr>
<td>D. Canet, C. Roumestand, Université de Nancy, France: DANTE-Z. A Robust and Easy-to-Implement Method for Selective Excitation</td>
<td></td>
</tr>
<tr>
<td>G. Bodenhausen, Université de Lausanne, Switzerland</td>
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</tr>
<tr>
<td>L. Lerner, University of Wisconsin, USA</td>
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</tr>
<tr>
<td>morning</td>
<td>Methods of High Resolution NMR for Macromolecules, G. Montelione, Chair</td>
</tr>
<tr>
<td>G. Wagner, Harvard Medical School, Cambridge, MA, USA</td>
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</tr>
<tr>
<td>A. Bax, National Institutes of Health, Bethesda, MD, USA</td>
<td></td>
</tr>
<tr>
<td>L. Kay, University of Toronto, Ontario, Canada</td>
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</tr>
</tbody>
</table>

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**Conference Office:** 815 Don Gaspar, Santa Fe, NM 87501 (505) 989-4573
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
Software and Analysis; A. Gronenborn, Chair
D. Zieskow, 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
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
Afternoon: Software and Analysis
A. Gronenborn, Chair
D. Ziessow, Technische Universität Berlin, Germany
J. Markley, University of Wisconsin, Madison, WI, USA
A. Gronenborn, National Institutes of Health, Bethesda, MD, USA

Morning: Poster Session II, H. Eckert, Chair
Instrumentation, R. Wind, Chair
R. G. Griffin, Massachusetts Institute of Technology
J. Haw, Texas A&M University, College Station, TX, USA
G. Chingas, University of California, Berkeley, CA, 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

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

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

Solid-State Methods, E. A. Williams, Chair
B. Meier, P. Robyr, M. Tomasselli, 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

Biological Solids, R. Stark, Chair
C. Bronnimann, Otsuka EETronics, 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

Conference Office: 815 Don Gaspar, Santa Fe, NM 87501 (505) 989-4573
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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 occurrence is unique in the sequence, as well as all of the di- and tri-amino acid segments that are also unique in occurrence 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 segments 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.

![Figure 1](image1.png)

![Figure 2](image2.png)

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 occurring 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 occurring tri-peptide segments can be displayed similarly.
All data can then be printed using menu item 4, figure 3.

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<th>Amino Acid Sequence</th>
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<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
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<tr>
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<td>V 34</td>
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<tr>
<td>S 47</td>
</tr>
<tr>
<td>M 52</td>
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<td>E-P-P 7</td>
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</tr>
<tr>
<td>T-X-G 54</td>
<td>G-G-A 56</td>
<td></td>
</tr>
</tbody>
</table>

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

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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.

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Chemagnetics would like to thank Professor Steve Smith for suggesting this work and useful discussions during its implementation.
Dr. Bernard Shapiro,
968 Elsinore Court
Palo Alto, CA 94303

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,

Anaaya Majumdar

Erik Zuiderweg

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

Efficiency (%)
rfrf (\%) rf S (\%)

Inept

Efficiency (%)
rfrf (\%) rf S (\%)

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.

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Hitachi Instruments, Inc., 3102 N. First Street, San Jose, CA 95134-9903.
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 multidimensional 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$^2$O). Figs 3A and 3C correspond to $^{15}$N-filtered NOESY spectra with $^{13}$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.
Please credit this contribution to the account of David C. Fry.

Sincerely,

Steven Donald Emerson
Physical Chemistry Department

The new 750 MHz from Oxford, setting another new standard in NMR
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, $^3J_{HH}$, 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 $^1H$ 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 $^3J_{HH}$ but when the chemical shifts of the coupled $^1H$ pairs are very close, $^{13}C$ HMQC can be relied on to give good lineshape from which the $^3J_{HH}$ 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 $^3J_{CH}$ 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 $^1H$ 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 $^1H$ 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.
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 multiplier 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 1.5 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 t2 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 t2 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
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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, 19.9, and 24 kHz.

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• 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
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 (spin½), 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 (CF₃CHClBr) in CDCl₃. 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 CF₃ 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,

Anthony A. Ribeiro

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 \( \sigma_a = (2/3)\Delta \chi(c - a)/(a + 2c)(a^2 + c^2)\gamma \), 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 \( \sigma_a \) 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:

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

with \( f(A, C) = (2/3)(C - A)/(A + 2C)(A^2 + C^2)^{1/2} \) where \( \gamma \) 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, \( \chi_{iso}^{iso} \) 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 \( \sigma_a \) is not \( S_{LC} \cdot \Delta \chi \) but \( S_{LC} \cdot 3\chi_{iso}^{iso} \) because for most thermotropic liquid crystals \( 3\chi_{iso}^{iso} \) is 10 times larger than \( \Delta \chi \).

If the \( \sigma_a \)-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 \( ^{13}\text{C} \) with apparent anisotropies between 10 and 100ppm the errors are between 10 and 1%.


With kind regards

Prof. Dr. P. Diehl
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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.

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- **Research in Pharmaceutical Industry**
- **Materials Science Research**

### The BIOSPEC CSI™ Magnets

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<th>BIOSPEC CSI™</th>
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<th>24/40</th>
<th>47/40</th>
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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.
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 $T_1$'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 $[\text{PCr}]/[\text{ATP}] = 4.0$. The steady state saturation transfer experiment was performed by "infinite" irradiation of the $\gamma$-ATP signal. Spillover saturation was appropriately accounted for. The product $k_1T_1([\text{PCr}])$ was 0.22, and from the measurement of the SF's at the repetition time of 0.84 s we calculated $T_1(\gamma\text{-ATP}) = 1.7$ s and $T_1(\text{PCr}) = 2.9$ 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,

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

References:

Please credit this contribution to Gunther L. Eichhorn’s subscription.
Dear Barry,

We recently upgraded our GE (now Bruker) GN 300 Spectrometer with a Tecmag Scorpio 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 $^{13}\text{C}$ 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 broadband decoupling routines we were able to successfully acquire $^1\text{H}$ and $^{13}\text{C}$ HMQC data. See Figure 1.

Sincerely,

Kirsten Berghmans

Chuck Gasparovic

Lisa Theisen

Figure 1: $^1\text{H}$-$^{13}\text{C}$ HMQC of D-(1-$^{13}$C) Mannose in D$_2$O (cross peaks are for alpha and beta anomers).

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SHIGEMI SYMMETRICAL 5mm NMR MICROTUBE SYSTEM

<table>
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<th>Insert OD (mm)</th>
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</tr>
</tbody>
</table>

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

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The spectra of 20mm sucrose in D<sub>2</sub>O 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 confirm a sensitivity enhancement of about 10%.

---

### PST-001 and PST-002

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<td>50/15</td>
<td>9.98 ± 0.00 - 0.01</td>
<td>9.52 ± 0.01</td>
</tr>
</tbody>
</table>

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SHIGEMI, INC.
Suite 21, 4790 Route 8 • Allison Park, PA 15101 • USA
Tel: (412)444-3011 • Fax (412)444-3020
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

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Eppendorf tube

Glass vial with cap lined with white rubber
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<table>
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<th>Isotopologue</th>
<th>Formula</th>
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<td>99 atom%</td>
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</table>

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

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<table>
<thead>
<tr>
<th>Code</th>
<th>Compound</th>
<th>Isotopic Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>83-52009-2</td>
<td>Acrylamide-$^{13}C_3$</td>
<td>99 atom%</td>
</tr>
<tr>
<td>82-20219-3</td>
<td>Choline-$d_4$ Chloride (Trimethyl-$d_6$)</td>
<td>98 atom%</td>
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<tr>
<td>85-12272-9</td>
<td>L-Cysteine-$^{15}N$</td>
<td>99 atom%</td>
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<tr>
<td>82-70041-0</td>
<td>Deuterium Oxide ULTRA-D</td>
<td>99.999 atom%</td>
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<tr>
<td>82-00815-2</td>
<td>DL-1,4-Dithiothreitol-$d_{10}$ (Cleland’s Reagent)</td>
<td>98 atom%</td>
</tr>
<tr>
<td>81-61002-4</td>
<td>ISOGRO™-$^{13}C,^{15}N$ Powder-Growth Medium</td>
<td>99 atom% $^{13}C,^{15}N$</td>
</tr>
<tr>
<td></td>
<td>2-Mercaptoethanol-1,1,2,2-$d_4$</td>
<td>98 atom%</td>
</tr>
</tbody>
</table>

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Hey Barry,

Greetings from Galveston! We have just finished the installation of our new 400 MHz UNITYplus 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 D₂O 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 spatial 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 spatial resolution was limited by sensitivity. We hope to increase our sensitivity and spatial 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

230 UNIVERSITY BOULEVARD SUITE 610 GALVESTON, TEXAS 77555-0843 (409) 772-2101 FAX (409) 762-9362
Dear Dr. Shapiro:

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 $^2$H NMR spectrum recorded for $[^2$H$_6]$ 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,

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

Something to "Peak" your interest

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 Tocys 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
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](image1)

![After. Peaks A&B have been merged, while peak C displays good c-of-mass adjustment.](image2)

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 Jarzetzky's 65th Birthday
March 24-26, 1994
Stanford University, Stanford, California
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(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
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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
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Dr. K. Darrell Berlin  
Department of Chemistry  
Oklahoma State University  
Stillwater, OK 74078  

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Contact: D. M. Wilson (510) 242-2415  
Fax (510) 242-4647
Dear Dr. Shapiro,

We currently operate three Sun-based Varian spectrometers - a VXR-300s and 2 Unity 500s - 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 CDC13, 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 effectiveness 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
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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
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No. 426 (March) 18 February 1994
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