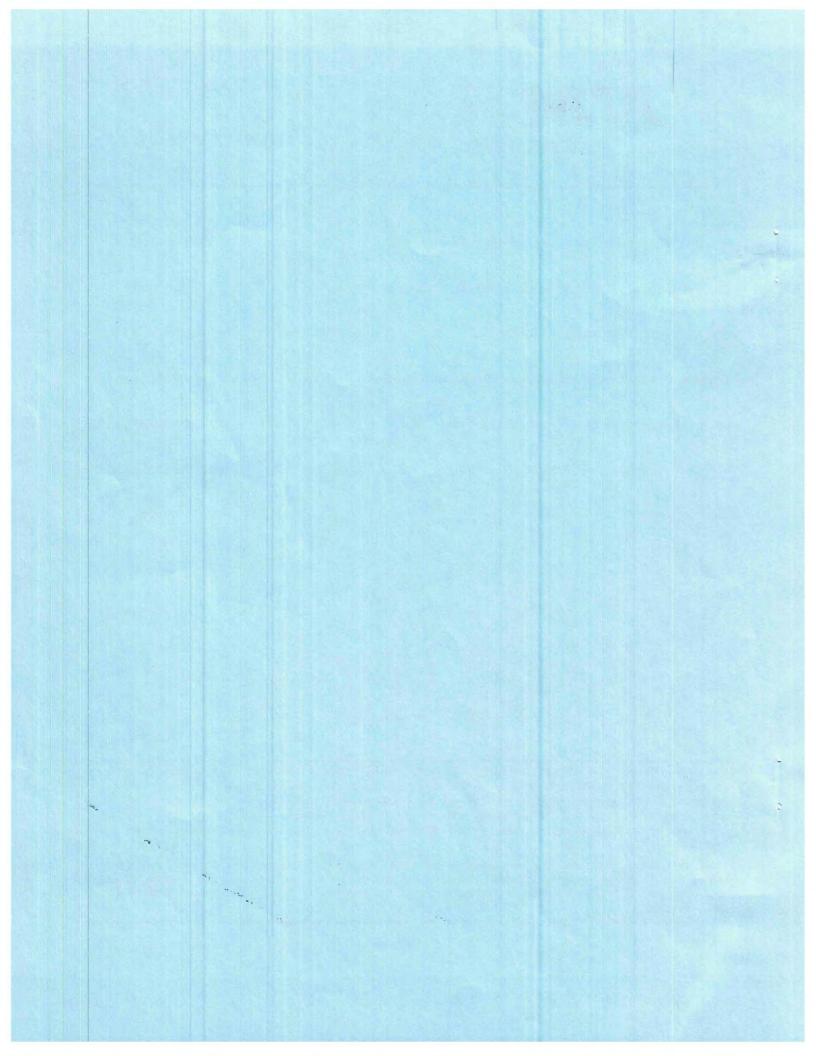


No. 508 January 2001

Shielded Magnets Revisited and ghmqd	Artifacts			. Nakas	hima, T.	2
Method to Obtain Higher Resolution M	RI Heart Mappi	ing .		. Кар	lan, J. I.	5
The Carlsberg Pulse Program Library				. Sørense	en, O. W.	9
RF Solenoidal Microcoil Performance	Armenean, M.,	Saint-Jalme	s, H., Fav	re, B., and Bri	guet, A.	13
Magic Angle Setting in Satellite Transit	ion MAS		Delevo	ye, L., and Bre	vard, C.	17
9th Annual "Advances in NMR Applica	tions" Symposi	um, March 11	, 2001	Nalorac/Casto	orina, P.	20
Position Available				Schu	bert, U.	21

A monthly collection of informal private letters from laboratories involved with NMR spectroscopy. Information contained herein is solely for the use of the reader. Quotation of material from the Newsletter is not permitted, except by direct arrangement with the author of the letter, in which case the material quoted must be referred to as a "Private Communication". Results, findings, and opinions appearing in the Newsletter are solely the responsibility of the author(s). Reference to The NMR Newsletter or its previous names in the open literature is strictly forbidden.

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



THE NMR NEWSLETTER	NO. 508, J	JANUARY 2001	AUTHOR INDEX		
Brevard, C 17 Delevo	ina, P 20 ye, L 17 B 13	Kaplan, J. I 5 Nakashima, T 2 Nalorac 20	Saint-Jalmes, H. 13 Schubert, U. 21 Sørensen, O. W. 9		
THE NMR NEWSLETTER	NO. 508, J	JANUARY 2001	ADVERTISER INDEX		
Bruker Instruments, Inc	15		outside back cover		

SPONSORS OF THE NMR NEWSLETTER

Abbott Laboratories

Advanced Chemistry Development, Inc.

Aldrich Chemical Company, Inc.

Amgen, Inc.

AMT

Anasazi Instruments, Inc.

AstraZeneca

Avanti Polar Lipids, Inc.

Bruker Instruments, Inc.

Bristol-Myers Squibb Company

Cambridge Isotope Laboratories

Cryomag Services, Inc.

The Dow Chemical Company

E. I. du Pont de Nemours & Company

Isotec, Inc.

JEOL (U.S.A.) Inc., Analytical Instruments Division

The Lilly Research Laboratories, Eli Lilly & Company

Merck Research Laboratories

Nalorac Corporation

Pharmacia & Upjohn, Inc.

Programmed Test Sources, Inc.

SINTEF Unimed MR Center, Trondheim, Norway

Tecmag

Unilever Research

Union Carbide Corporation

Varian, Inc.

FORTHCOMING NMR MEETINGS

Frontiers of NMR in Molecular Biology VII, Big Sky, Montana, **January 20-26, 2001**. Contact: Keystone Symposia, Drawer 1630, 221 Summit Place, Suite 272, Silverthorne, CO 80498. Tel: 800-253-0685 or 970-262-1230; Fax: 970-262-1525; E-mail: keystone@symposia.com. http://www.symposia.com.

PITTCON 2001, New Orleans, LA, March 4-9, 2001. Contact: The Pittsburgh Conference, Dept. CFP, 300 Penn Center Blvd., Suite 332, Pittsburgh, PA 15235-5503. Tel: 412-825-3220; Fax: 412-825-3224; E-mail: pittconinfo@pittcon.org.

42nd ENC (Experimental NMR Conference), Rosen Plaza Hotel, Orlando, Florida, March 11-16, 2001; Arthur G. Palmer, Chair: Agp6@columbia.edu; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; E-mail: enc@enc-conference.org; Web: www.enc-conference.org.

ACS National Meeting, "Symposium on High Resolution NMR Spectroscopy of Polymers," San Diego, CA, April 1-5, 2001; Contact: H. N. Cheng (hcheng@herc.com) or A. D. English (halan.d.English@usa.dupont.com); See Newsletter 505, 29.

<u>Magnetic Resonance in Chemistry and Biology, XIth International Conference</u>, Zvenigorod, Russia, **April 20-27,2001**. Contact: http://www.nmr.de/html/conf/zelino.shtml.

ISMRM 9th Scientific Meeting and Exhibition; ESMRMB 18th Annual Meeting and Exhibition, Joint Annual Meeting. Glasgow, Scotland, **April 21-27, 2001**.Contact: ISMRM Central Office, 2118 Melvia Street, Suite 201, Berkeley, CA 94704. Tel: 510-841-1899; Fax: 510-841-2340; E-mail: info@ismrm.org.

Computational Aspects of Biomolecular NMR, Gordon Conference, "Il Ciocco", Barga (Pisa) Italy, **May 6-11, 2001**. Contact: Michael Nilges nilges@embl-heidelberg.de, or Dave Cast case@scripps.edu.

ISMRM 9th Scientific Meeting and Exhibition, and ESMRMB 18th Annual Meeting and Exhibition, Joint Annual Meeting, April 21-27, 2001, 10th Annual Meeting of the Section for Magnetic Resonance Technologists, and 17th Annual Meeting of the British Association of MR Radiographers, April 20-22, 2001 Glasgow, Scotland, UK; Contact: ISMRM, P.O. Box 45690, San Francisco, CA 94145-0690; http://www.ismrm.org

Department of Chemistry Faculty of Science

Canada T6G 2G2

E3-43 Chemistry Building East, Telephone (403) 432-3254

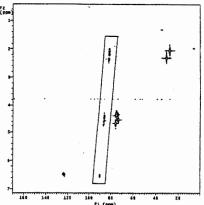
Dr. B. L. Shapiro The NMR Newsletter 966 Elsinore Court Palo Alto, California USA 94303 Nov. 29, 2000 (received 12/06/2000)

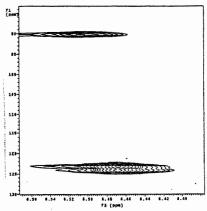
Re: Shielded Magnets Revisited and ghmqc Artifacts

Dear Barry:

Recently I reported on the observation that the magnetic field homogeneity of shielded magnets changes over the course of time and some of your readers may be interested in the drift rate of the homogeneity after five months. I repeated the magnetic field drift test again and the resolution of the line shape sample changed from 0.56, 6.3, 13 Hz non-spinning to 1.4, 8.9, 16.3 Hz over a time period of 16 hours. This is considerably better than previously reported but still unacceptable. I guess we will have to wait longer for the homogeneity to stop drifting (another specification for new systems delivered with shielded magnets).

On another note, I would like to ask the Newsletter readers if they have encountered artifacts that appear to be ¹H- ¹H autocorrelation responses in ghmqc experiments as shown in the left Figure. They appear as diagonals at a two dimensional frequency of $f_1/2=f_2$. Another curious thing about these artifacts is that they do not have the same f₂ proton frequency as the true H-C correlation as is shown in the expansion on the right. Product operator analysis tells us several things about these artifacts. They cannot be due to proton magnetization created by the first pulse because in order to detect these signals, H₁ must be formed by the 180° proton pulse. If H₊₁ were present after the carbon 90° pulse, then converted to H₁ by the ¹H 180, ¹H chemical shift information would be refocussed. If H₁ is present before the ¹H 180° pulse, then the artifact would be at a frequency f1=f2. The artifacts must arise from H₀ left by an imperfect 90° ¹H pulse which are converted to H₁ by the 180° proton pulse. If this magnetization were coupled to ¹³C then the next ¹³C 90° pulse should send this signal to two quantum land which is undetectable. Thus the artifact signal must pass through the ¹³C pulse and be detected during t₂ acquisition. (I have run ghmqc experiments on a neat D₂O sample and observed these artifacts from the residual proton signals). In the ghmqc experiment three gradients are applied to the sample for coherence selection and in this experiment the gradient levels were in a ratio of 2.2.-1. The only way for these artifact signals to come through is for these molecules to be in a position in the sample tube such that they experience the same amplitude from the second and third gradient. This can happen only if the amplitudes are zero and can occur (I suppose) for molecules outside the receiver coil which also explains the different f2 frequencies, suggesting that the artifact signals arise from molecules experiencing a slightly different Bo. One might wonder if these signals outside the receiver coil should show up in normal spectra but examination of the contour map shows them to be much smaller than the ¹³C satellite intensities. Anyway that was my best guess as to the origin of these signals and I would be interested to hear other explanations.





Sincerely, Tom Nakashima

e-mail: tom.nakashima@ ualberta.ca



A New Revolutionary Milestone: Shielded 800 magnet from Bruker

Continuing the tradition ... another World's First in Bruker Magnet History!



Shown in operation as part of a complete AVANCE 800 NMR System used for customer demonstrations at our application laboratory in Karlsruhe, Germany.

Bruker is proud to introduce the 800US^{2™}, the world's first shielded 800MHz high resolution NMR magnet. This superb system is a result of merging our latest and innovative magnet technologies: UltraShieldTM - Bruker's advanced self-shielding technology, and UltraStabilizedTM - Bruker's advanced sub-cooled technology for Ultra-High Field NMR. What was once thought almost impossible has now become a reality: siting an 800 MHz NMR magnet in a smaller footprint than a non-shielded 500!

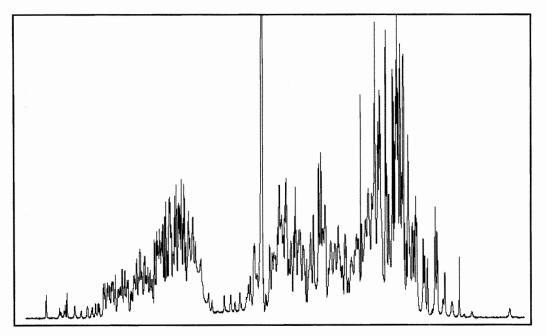
The 800US² comes with the same guarantee for outstanding performance demonstrated by the UltraStabilized magnet series over the past decade, and now with the added benefits of UltraShield technology. Its excellent field homogeneity and field stability along with the industry's best cryogenic performance for sub-cooled magnets reassures you of obtaining the highest quality NMR results with minimum maintenance costs.

The very small stray fields of the 800US² helps you now take advantage of smaller lab spaces and provides you with more flexibility and options for future upgrades and additions as never before. These will cut future 800 siting costs by a significant margin due to a much smaller footprint required and no need for expensive building renovations.

In addition to its remarkably small stray fields, the 800US² incorporates UltraShield's optimized system design for compensating (screening) against external field perturbations. As already demonstrated by UltraShield magnets at numerous sites located near sources of large external field perturbations (e.g. subways, trains), the 800US² will respond similarly and perform better than a non-shielded magnet under the same site conditions.

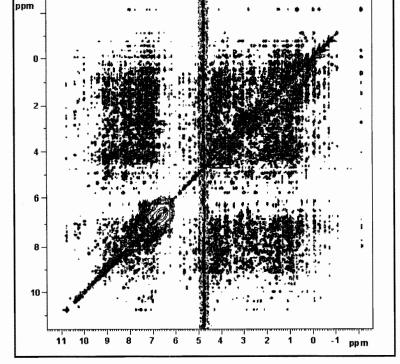
The 800US² magnet has been energized at full field in its final cryostat (customer system cryostat) on October 2, 2000. It is currently in operation as part of a complete AVANCE 800 spectrometer used for customer demonstrations at our application laboratory in Karlsruhe, Germany.

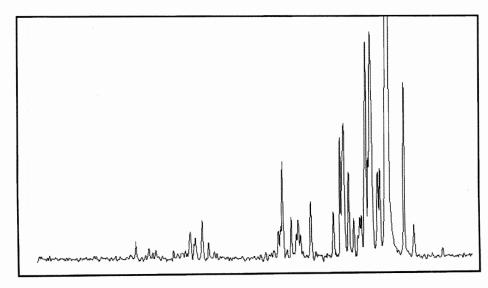




1D 1H spectrum w/presat 2mM Lysozyme

2D NOESY w/presat 2mM Lysozyme





2D NOESY w/presat 2mM Lysozyme

Column through methyl diagonal peak - showing almost no T1 noise!



INDIANA UNIVERSITY PURDUE UNIVERSITY INDIANAPOLIS

December 8, 2000

(received 12/22/2000)

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

Dear Barry,

SCHOOL OF SCIENCE

Method to Obtain Higher Resolution MRI Heart Mapping



Higher resolution MRI mapping of hearts might serve to reduce the need to do catherizations. It would also allow for a non-invasive follow-up after bypass surgery. For planer imaging one records the signal

$$M(t) = \int F(t, x', y') \rho(x', y') dx' dy'$$

and obtains the mapping density $\rho * (x, y)$ as

$$\rho * (x, y) = \sum E(t, x, y) M(t)$$

which can be expressed as

$$\rho * (x, y) = \int A(x, y, x', y') \rho(x', y') dx', dy'$$

where

A
$$(x, y, x', y') = \sum_{t} E(t, x, y) F(t, x', y')$$

Discretizing space one has

$$\rho*(x, y) = \sum_{x', y'} B(x, y, x', y') \rho(x', y')$$

which can be inverted to obtain the higher resolution density mapping as

$$\rho ** (x, y) = \sum_{x', y'} B^{-1}(x, y, x', y') \rho *(x', y')$$

Patent Pending

Plot showing increase in resolution is a computer simulation.

Sincerely,

Jerome I. Kaplan

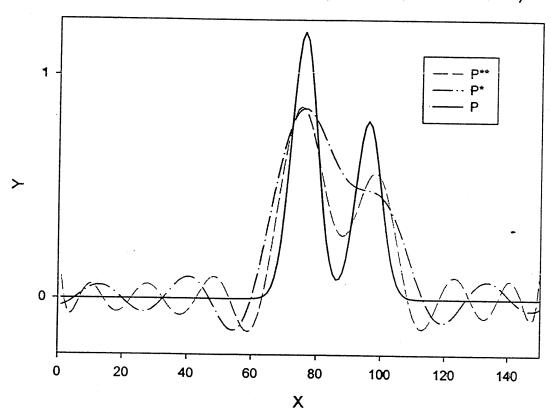
DEPARTMENT OF PHYSICS

LD 154 402 N. Blackford Street Indianapolis, Indiana 46202-3273

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

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

N=150, dx=0.0125, dp=0.25 (+lobe 28, bells 40 wide)



To **Venture**where no **Scientists**have gone before...



Introducing Varian's 900 MHz NMR systems

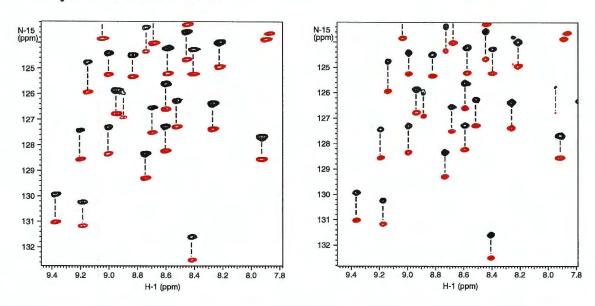
Does doubling the molecular weight limit of your NMR experiments sound like science fiction? The time is now. Varian and Oxford Instruments set the pace with the first 900 MHz system, producing unprecedented sensitivity and dispersion in biomolecular NMR. From 200 MHz to 900 MHz, Varian's technical leadership brings you the industry's fastest RF and digital systems. All this combined with easy to use software that simplifies the complexity of your most demanding experiments. Explore for yourself at www.varianinc.com/nmr/900



www.varianinc.com

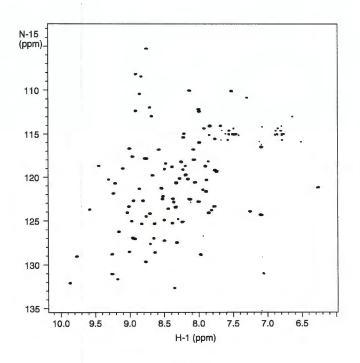
Varian NMR systems and Oxford Instruments are pleased to announce the acquisition of high resolution NMR data at 900 MHz! The Oxford 900 MHz magnet is now at field and providing stable operation with a low drift rate, and the homogeneity necessary for high quality NMR operation. The proof of this remarkable achievement is, of course, the NMR spectra included below and on our web-site at www.varianinc.com/900.

Partially Orientated Molecules



Measurement of residual dipolar N-H couplings at 900 MHz in partially oriented proteins with (right) and without the use of band-selective homonuclear decoupling.

TROSY at 900 MHz



The ¹⁵N-¹H TROSY correlation spectrum of 6F1 1F2 module pair from the gelatin-binding domain of fibronectin. Sample courtesy of Prof. .I.D. Campbell of Oxford University.



Carlsberg Laboratory

Danish Instrument Center for NMR Spectroscopy of Biological Macromolecules



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

(received 12/26/2000)

The Carlsberg Pulse Program Library

Dear Barry:

Pulse programs and related software developed at Carlsberg Research Center are now available via the WebPage http://www.crc.dk/nmr/. There are pulse programs both in Varian and Bruker format and we will keep adding new software as it is developed. Most programs are for protein applications but there is also a program package, MBOB, for 2D heteronuclear long-range correlation in small molecules.

Several authors have addressed the problem that it is impossible to achieve uniform excitation over a range of J coupling constants by a single delay in the HMBC pulse sequence. This comes about because the peak intensities are proportional to $\sin(\pi J \Delta)$ where J is the heteronuclear long-range J coupling constant and Δ is the excitation delay. A much used compromise is to set Δ slightly shorter than 1/J(max) where J(max) is the largest expected J coupling constant in the molecule under investigation, which typically corresponds to Δ = 65 ms. This value is often too small for the cross peaks associated with the smallest J values whilst choosing a larger value runs the risk that $\sin(\pi J \Delta)$ vanishes for some of the larger Js. It is also not possible to coadd spectra recorded with different Δ delays because the sine factor might change sign.

MBOB (Multiple-Bond and One-Bond correlation) combines HMBC spectra recorded with different Δ delays in absolute-value mode and includes a novel non-destructive low-pass J filter making it possible to extract a one-bond correlation spectrum from the same data set. In other words, MBOB yields a broadband HMBC spectrum and a one-bond correlation spectrum in the time it would typically take to record a single- Δ HMBC spectrum. Hence the time to record a separate HSQC or HMQC spectrum is saved.

On the next page is shown 500 MHz HMBC and edited MBOB spectra of ethyl *trans* cinnamate run with equal numbers of scans and the same instrument time. The long-range MBOB spectrum contains correlations that are absent in the Δ = 65 ms HMBC spectrum and the sensitivity is adequate for detection of one-bond correlations in the coupled mode.

Our MBOB software package runs in full automation performing the experiments, making the appropriate linear combinations and plotting the spectra.

Sincerely yours,

Ole W. Sørensen

MBOB reference: A. Meissner and O.W. Sørensen, Magn. Reson. Chem. 38, 981-984 (2000).

Carlsberg Laboratory
Department of Chemistry
Gamle Carlsberg Vej 10
DK-2500 Valby, Denmark

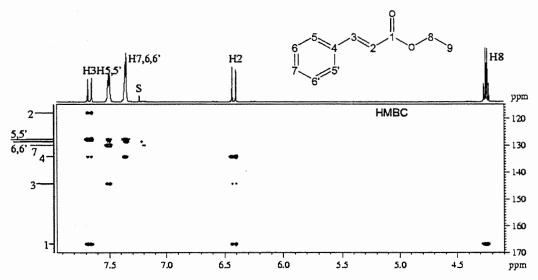
Email: kbo@crc.dk Email: jd@crc.dk Email: ows@crc.dk Tel.: Tel.:

Fax:

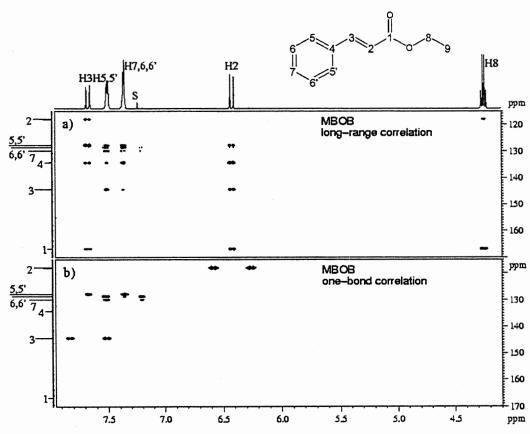
+45 33 27 52 20 +45 33 27 52 07

+45 33 27 47 08

Tel.: +45 33 27 52 07
Tel.: +45 33 27 52 09



Expansion of a conventional Δ = 65 ms 2nd order LPJF HMBC spectra of 15 μ l of ethyl *trans* cinnamate in 600 μ l CDCl3 at 25 °C.



Expansion of edited MBOB spectra of 15 μ l of ethyl *trans* cinnamate in 600 μ l CDCl3 at 25 °C. (a) MBOB multiple-bond correlation spectrum. (b) MBOB one-bond correlation spectrum. A 2nd order J filter and four Δ values (440.7, 343.4, 290.3, and 242.2 ms) was employed. The total number of scans leading to these spectra was the same as for the HMBC spectrum above.

ISOTECINE.

Cordially invites you to our hospitality suite.

Join us at the

Keystone Symposia 2001

Frontiers of NMR in Molecular Biology VII

Saturday, January 20

through

Wednesday, January 24

Big Sky, Montana

Solution NMR structure of the RNA-binding domain of NS1 from influenza virus. (Provided by A. Chien and G. Montelione, Rutgers University)

Stop by to discuss your stable isotope needs with representatives of the world's leading commercial producer of Labelled Biomolecular NMR Products.

We look forward to seeing you there!

SOTELING.

O A MATHESON TRI-GAS COMPANY

PROMOTING RESEARCH AND DISCOVERY

3858 Benner Rd. • Miamisburg, OH 45342 **Sales (800)448-9760** • Fax (937)859-4878 http://www.isotec.com • isosales@isotec.com

Biomolecular NMR Products

From the leader in labelled compound synthesis

TECHNICAL KNOWLEDGE

Isotec's knowledge and expertise allow the production of thousands of labelled products and the flexibility to meet any customers' needs. With 10 Ph.D. Chemists and 33 Masters- and Bachelors-level Chemists and Technicians, Isotec is fully committed to the development of **innovative ways to synthesize** both new and existing compounds.

THE ISOTEC ADVANTAGE

Isotec continues to be the trusted source for reliable stable isotope labelled products for Biomolecular NMR Studies. Isotec offers a comprehensive line of ¹³C, ¹⁵N, and/or D labelled compounds for metabolic or rational drug design research. Isotec's **pioneering synthesis knowledge** enables us to synthesize compounds that help you reach your goals. Our list of products is extensive and includes the following:

Amino Acids

Amino Acids-¹³C,¹⁵N
Specifically labelled Amino Acids
N-t-Boc and F-MOC Protected Amino Acids

Ammonium Salts

Ammonium-¹⁵N Chloride Ammonium-¹⁵N₂ Sulfate

Growth Media

ISOGROTM - ¹³C Powder ISOGROTM - ¹³C, ¹⁵N Powder ISOGROTM - ¹⁵N Powder ISOGROTM - ¹³C, ¹⁵N, D Powder ISOGROTM - Powder (unlabelled)

*Deoxyribonucleotides

Ribonucleotides

*contact Isotec for availability

Glucose

D-Glucose-¹³C₆ D-Glucose-¹³C₆,C-d₇ D-Glucose-C-d₇

Buffers & Reagents

Acetic Acid- d_4 DL-1,4-Dithiothreitol- d_{10} Dodecylphosphocholine- d_{38} Ethylenediaminetetraacetic Acid- d_{12} (EDTA) Glycine- d_5 Sodium Dodecyl- d_{25} Sulfate Tris- d_{11}

NMR Solvents

Custom Synthesis

Call our Sales Office at 1-800-448-9760 or 1-937-859-1808 to request more information about how Isotec can meet your *labelled Biomolecular NMR Products* needs.

3858 Benner Road • Miamisburg, OH 45342 USA *E-mail:* isosales@isotec.com • *Internet:* www.isotec.com 800-448-9760 • 937-859-1808 • Fax 937-859-4878



LABORATOIRE DE RESONANCE MAGNETIQUE NUCLEAIRE METHODOLOGIE ET INSTRUMENTATION EN BIOPHYSIQUE

CNRS UMR 5012 - UNIVERSITE CLAUDE BERNARD LYON I - ESCPE

Bâtiment 308 - 43, Boulevard du 11 Novembre 1918 - 69622 VILLEURBANNE CEDEX Téléphone : (33) 04 72 44 82 67 Fax (33) 04 72 44 81 99 E-mail briguet@univ-lyon1.fr

Villeurbanne, december 14, 2000

RF solenoidal microcoils performances

(received 12/23/2000)

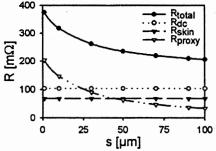
Dear Dr Shapiro,

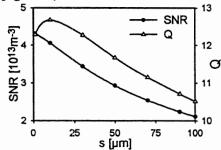
Solenoidal microcoils represent interesting tools for NMR spectroscopy and their optimization for an efficient use is unavoidable. The magnetic coupling between a RF coil and the studied sample must be improved since it corresponds directly to an optimal sensitivity for the signal reception. For sensitivity optimization, not only the quality factor (Q) but also the signal-to-noise ratio (SNR) and the electromagnetic parameters (R, L, B) of given coils have to be estimated. The SNR of an NMR experiment is defined as the peak signal divided by the root mean square (rms) noise [1]:

$$SNR = \frac{\frac{B_1}{i} V_S N \gamma \frac{h^2}{4\pi^2} I(I+1) \frac{\omega_0^2}{3\sqrt{2}kT}}{V_{noise}} \quad with \quad V_{noise} = \sqrt{4k TR_{noise} \Delta f}$$

where B_1/i is the magnetic field per unit current (*coil sensitivity*), V_S the sample volume, N the spin density, γ the gyromagnetic ratio, I the spin quantum number, ω_0 the nuclear precession frequency, T the sample temperature, K the Boltzmann's constant, K the Planck's constant and K is the spectral bandwidth. The microcoil itself is the dominant source of noise ($K_{noise} R_{coil}$) [2]. In the present simulations, based on the **ANSOFT Maxwell 2D/3D software**, we have varied the turn number ($K_{noise} R_{coil}$), the internal diameter of solenoidal microcoil ($K_{noise} R_{coil}$), the copper wire diameter ($K_{noise} R_{coil}$) and the interturn spacing ($K_{noise} R_{coil}$). The SNR per unit sample volume computation was done for proton in water at 2 teslas (85.13 MHz), $K_{noise} R_{coil}$).

Figures 1 (left) and 2 (right): Contributions of different effects (dc, skin, proximity) at the total resistance of microcoil for D_{int} =200 μ m, d=40 μ m, n=10 turns (figure 1). SNR per unit sample volume and Q dependencies in the same conditions (figure 2)





A decrease in the diameter of a solenoidal microcoil produces, as already mentioned [2], an increase in SNR per unit sample volume but simultaneously Q quickly decreases. For a solenoidal microcoil with n=10 turns and D_{int} =200 μ m if the interturn spacing s decreases from 100 μ m to 1 μ m the SNR per unit sample volume increases. It was also observed that the proximity effect on R_{noise} was compensated by a significant increase in sensitivity. For adjacent turns, Q reach then its maximum value if d=20 μ m or for few microns interturn spacing if d=40 to 100 μ m. Keeping the same D_{int} and s=1 μ m with d decreasing from 100 to 8 μ m, the SNR per unit sample volume increases to a maximum for d≈16 μ m, then it quickly decreases. If the wire radius of the coil is comparable to the skin depth, standard design rules [2] are no longer relevant, and the help of an electromagnetic simulation software is mandatory.

References: [1] Webb A. G. [1997], Progress in NMR Spectroscopy 31: 4
[2] Peck T. L., Magin R. L., Lauterbur P. C. [1995], J. Magn. Reson. Series B 108: 115.

Sincerely yours,

M. Armenean

H. Saint-Jalmes

Main! I has

B. Favre

A. Briguet

Mynul

APPING NEW FRONTIERS

FRONTIERS OF NMR IN MOLECULAR BIOLOGY VII

Participants of the Keystone Symposium on Molecular and Cellular Biology

Big Sky Resort in Big Sky, Montana January 20-26, 2001 In April of 1805, the Lewis & Clark Expedition mapped the frontiers of Montana. Their expedition created the foundation for the western development of America.

Today, the participants of the Keystone Symposia, map the frontiers in genomics, proteomics and drug discovery. CIL is proud of our association with this esteemed group of pioneers. We appreciate the opportunity to provide researchers with the isotope labeled compounds required to explore these new areas of research.





Cambridge Isotope Laboratories, Inc.

50 Frontage Road, Andover, MA 01810-5413 USA PH: 800.322.1174 (USA) PH: 978.749.8000 FAX: 978.749.2768 URL: http://www.isotope.com



PROTECTED AMINO ACIDS (U-13C,U-15N)

Solid phase peptide synthesis, while not a novel process, continues to emerge as an essential tool for many areas of protein research. From structure-function studies to antibody research to drug candidate screening, the synthesis of target peptides is accel² erating the rate of pharmaceutical development. To assist researchers in performing peptide structure analysis by NMR, Cambridge Isotope Laboratories, Inc. (CIL) has assembled an extensive list of protected uniformly Carbon-13, Nitrogen-15 labeled amino acids. With more than 15 years of synthetic experience, CIL continues to produce the highest quality protected amino acids availables. Remember, quality in means quality out. Let CIL provide a quote for the compounds listed below. If your experiment requires a protected uniformly labeled residue that is not listed below, CIL is always interested in expanding this group of products. Please ask for a custom quotation.

N-FMOC PROTECTED

CNLM-4226	L-Arginine-N-FMOC, PMC	(U-13C6,98%+;U-15N4,96-99%)
CNLM-4345	L-Leucine-N-FMOC	(U- ¹³ C ₆ ,98%+; ¹⁵ N,96-99%)
CNLM-4346	L-Isoleucine-N-FMOC	(U-¹³C₀,98%+;¹⁵N,96-99%)
CNLM-4347	L-Proline-N-FMOC	(U-13C ₅ ,98%+;15N,96-99%)
CNLM-4348	L-Valine-N-FMOC	(U-13C ₅ ,98%+;15N,96-99%)
CNLM-4349	L-Tyrosine-N-FMOC, O-t-Butyl Ether	(U-13C,,98%+;15N,96-99%)
CNLM-4354	L-Asparagine-N-FMOC	(U-13C ₄ ,98%;U-15N ₂ ,96-99%)
CNLM-4355	L-Alanine-N-FMOC	(U-13C3,98%;15N,96-99%)
CNLM-4356	L-Glutamine-N-FMOC	(U-13C5,98%;U-15N2,96-99%)
CNLM-4357	Glycine-N-FMOC	(U-13C2,98%;15N,96-99%)
CNLM-4358	L-Methionine-N-FMOC	(U-13C ₅ ,98%;15N,96-99%)
CNLM-4362	L-Phenylalanine-N-FMOC	(U-13C,,98%;15N,96-99%)
CNLM-4722	L-Cysteine-N-FMOC, S-TRITYL	(U-13C3,98%+;15N,96-99%)
CNLM-4752	L-Aspartic Acid-N-FMOC, β-O-t-Butyl Ether	(U-13C4,98%+;15N,96-99%)
CNLM-4753	L-Glutamic Acid-N-FMOC, y-t-Butyl Ether	(U-13C ₅ ,98%+;15N,96-99%)
CNLM-4754	L-Lysine-α-N-FMOC, ε-N-t-BOC	(U-13C6,98%+;15N2,96-99%)
CNLM-4755	L-Serine-N-FMOC, O-t-Butyl Ether	(U-13C3,98%+;15N,96-99%)
CNLM-4788	L-Aspartic Acid-N-FMOC	(U-13C4,98%+;15N,96-99%)
CNLM-4789	L-Aspartic Acid-N-FMOC, α-O-t-Butyl Ether	(U-13C4,98%+;15N,96-99%)

N-T-BOC PROTECTED

CNLM-2392	L-Aspartic Acid-N-t-BOC, β-Benzyl Ester	(U-¹³C₄,98%+;¹⁵N,96-99%)
CNLM-2393	L-Phenylalanine-N-t-BOC	(U-¹³C ₉ ,98%+;¹⁵N,96-99%)
CNLM-2394	L-Alanine-N-t-BOC	(U-¹³C₃,98%+;¹⁵N,96-99%)
CNLM-2395	L-Valine-N-t-BOC	(U- ¹³ C ₅ ,98%+; ¹⁵ N,96-99%)
CNLM-2396	L-Leucine-N-t-BOC:H2O	(U-¹³C₀,98%+;¹⁵N,96-99%)
CNLM-2412	Glycine-N-t-BOC	(U-13C ₂ ,98%+;15N,96-99%)

50 Frontage Road, Andover, MA 01810-5413 USA

PH: 800.322.1174 (USA) PH: 800.643.7239 (CANADA) PH: 978.749.8000 FAX: 978.749.2768 URL: http://www.isotope.com



BRUKER S.A. - 34, rue de l'Industrie - 67166 WISSEMBOURG cedex - FRANCE

Tél. +(33) 03 88 73 68 00 - Télécopieur +(33) 03 88 73 68 79 - Télex 870 639 BRUSPIN

http://www.bruker.com - e-mail : sadis@bruker.fr

AGENCE RÉGION PARISIENNE : 3, avenue du Général de Gaulle - 91090 LISSES
Tél. +(33) 01 69 89 85 20 - Télécopieur +(33) 01 60 86 34 90 - e-mail : savlisse@bruker.fr

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

(received 12/06/2000)

Magic-Angle Setting in Satellite Transition MAS

Dear Barry,

Since the introduction of the MQMAS sequence by Frydman $et\ al.^1$ in 1995, the acquisition of isotropic spectra of half-integer quadrupolar nuclei is straightforward and allows for a good structural characterisation of elements e.g. 27 Al, 23 Na, 11 B, and 17 O in a variety of materials, such as catalysts, glasses or minerals. It is well known that the efficiency of the multiple-quantum excitation and conversion is rather poor and this problem has been widely discussed in the scientic community. Developments by Madhu $et\ al.^2$ and Kentgens and Verhagen³ grandly improved the overall efficiency of the sequence by the use of amplitude-modulated pulses. More recently, Gan^4 presented a new approach based on the same principles than MQMAS, but correlating satellite and central transitions for the complete averaging of anisotropic second-order quadrupolar terms. The gain in sensitivity compared to MQMAS seems to be very important but the sequence requires that (1) the spinning axis be precisely aligned with the magic-angle, and that (2) the dwell time in the t_1 evolution be set to the rotor period.

In the process of testing this latter sequence in our application laboratory, we opted for the two-pulse sequence, selecting the $p \to -1 \to -1$ coherence transfer pathway using the adequate phase cycle (see ref.⁴ for more experimental details). We set the magic-angle maximizing ⁷⁹Br rotational echo in KBr powder. But, as expected, the accuracy of this setting was far below that was required to run Gan's experiment successfully. Nevertheless, we found that this setting was sufficient to optimize experimentally the pulse lengths in the sequence (figure 1). It can be noted that the conditions of excitation and conversion of the satellite transitions are broad compared to those obtained in MQMAS.

In a second phase, we decided to set the angle of rotation precisely on the magic-angle by running a series of 2D spectra, just *slightly* changing the orientation of the spinning axis between each experiment. Thanks to the high efficiency of the satellite transition excitation, each 2D ²³Na spectrum shown in figure 2 were acquired in less than 15 minutes. It turns out that in the case of a highly-crystallized sample, the splitting of the resonances in figures 2–a, b and c, is a very good indicator of the orientation of the spinning axis.

Of course, acquiring 2D satellite transition MAS NMR of unknown samples is rather difficult if the previous setting is not reliable over several automatic rotor insertions and ejections. Therefore, we also tested the stability of the rotor orientation by ejecting and inserting the same rotor 5 times between each experiment, as well as by manipulating the probes (2.5mm and 4mm) outside the magnet. Results revealed no splitting of the resonances that would have been a sign of a slight change of stator angle. The second critical parameter is the spinning speed, which must be very stable as the t_1 evolution time must be set to equal the rotor period. Indeed, a variation of a few hertz can lead to a drastic loss of the satellite signal. In our experiments, it in fact happened once that the echo signal disappeared in the 2D dataset, due to a variation of the spinning speed of more than 5 Hz. However, after repacking the rotor, the variation of the spinning speed remained within 1 Hz.

We believe that this simple 2D acquisition is an elegant and fast method to align the rotor axis exactly on the magic angle. However, there are still many problems to be solved in the acquisition of high-resolution MAS spectra of quadrupolar nuclei, using satellite transitions. Gan mentioned the presence of central transition signals that could be minimized using a presaturation pulse. In our case, we did not observe additional signals from the central transition and thus, we did not use a presaturation pulse. But, our 2D spectra were recorded by a selection of the echo pathway only, and the processing was made in absolute value mode. The main challenge in the near future will be to acquire pure absorption 2D spectra in a hypercomplex mode without introducing too much of the unwanted central transition signal.

Sincerely,

L. Delevoye

C. Brevard

1. FRYDMAN L. AND HARWOOD J. S. J. Am. Chem. Soc. 117 (1995), pp. 5367–5368.

- 2. MADHU P., GOLDBOURT A., FRYDMAN L. AND VEGA S. Chem. Phys. Lett. 307 (1999), pp. 41–47.
- 3. KENTGENS A. AND VERHAGEN R. Chem. Phys. Lett. 300 (1999), pp. 435-443.
- 4. GAN Z. J. Am. Chem. Soc. 122 (2000), pp. 3242-3243.

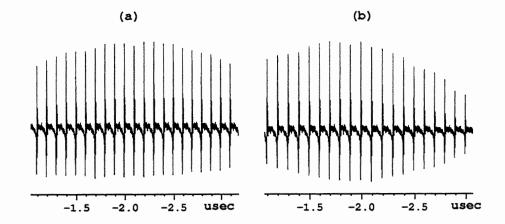


Figure 1 – Experimental optimisation of pulse lengths p_1 (a) and p_2 (b) of a ²³Na 2D satellite transition MAS sequence. The echo signal was acquired after an evolution period equal to several rotor periods in order to separate the satellite transition signal from any other undesired signal (possibly central transition). No phasing was applied during processing.

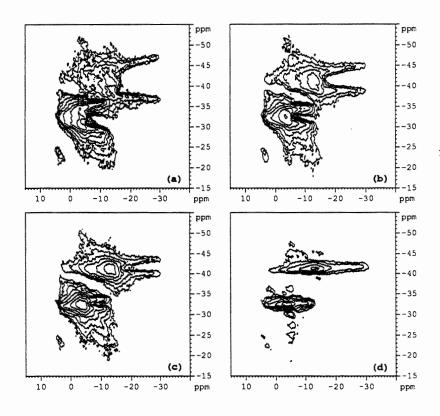


Figure 2 – 23 Na 2D satellite transition MAS spectra of Na₂HPO₄. Spectrum 1-a correponds to the first spectrum obtained after setting the spinning angle with the KBr sample. Each following spectrum was obtained after slightly changing the spinning angle until the splitting of the resonances disappeared.

Winter Holiday

You are invited to attend the

Welcome the Year 2001!

9th ANNUAL ADVANCES IN NMR APPLICATIONS SYMPOSIUM

Featuring the Latest Developments in Experimental Techniques

To be held prior to ENC at the The Rosen Centre Hotel Orlando, Florida

Sunday, March 11, 2001 1:00 to 6:00 p.m.

The agenda includes a presentation of recent results by leading NMR experimentalists concerning applications of pulsed field gradient and classical NMR techniques with both large and small molecular systems.

The results obtained will be of interest to all liquid state NMR spectroscopists.

Request a detailed program or RSVP by contacting Peggy Castorina, Nalorac's ENC Coordinator.

Transportation will be provided between The Rosen Plaza Hotel and The Rosen Centre Hotel.



837 Arnold Drive, Martinez, CA 94553
Phone: (925) 229-3501 Fax: (925) 229-1651
Email: peggy.castorina@nalorac.com
Website: http://www.nalorac.com

Position Available

A postdoctoral position starting immediately is available at the Institute of Inorganic Chemistry at the University of Technology/ Vienna.

The scientific work will be related to all kinds of NMR-spectroscopic problems in organometallic and inorganic materials chemistry within a very interdisciplinary and highly interactive environment. The candidate is therefore required to work in a team and should have very good communication skills. The position provides an excellent opportunity for publication and professional growth. The institute is equipped with four Bruker NMR spectrometers operating between 250 and 400 MHz and with different probe heads. A strong background in NMR (theory, as well as hands on experience in liquid and, preferentially, solid state NMR) is required. Experience with the implementation of novel pulse sequences, using UNIX and NT systems, as well as the coordination of technical maintenance is preferred. The work also includes user training and computer system management.

Inquiries and applications to:

Prof. Dr. U. Schubert

Institute of Inorganic Chemistry Vienna University of Technology Getreidemarkt 9/153

A-1060 Vienna

Austria

tel.: 0043-1-58801-15320 fax.: 0043-1-58801-15399

email: uschuber@mail.zserv.tuwien.ac.at

Forthcoming NMR Meetings, continued from page 1:

- Gordon Research Conference on Magnetic Resonance, June 17-22, 2001, Roger Williams University, Bristol, Rhode Island (note the new, improved location !!!). Contacts: Rob Tycko, Chair, 301-402-8272, tycko@helix.nih.gov, and Kurt Zilm, Vice-Chair, kurt.zilm@yale.edu. Site description and application information available at http://www.grc.uri.edu.
- IXth International Symposium on Magnetic Resonance in Colloid and Interface Science, St. Petersburg, Russia, June 26-30, 2001. Contact: Mrs. L. Ya. Startseva, Secretariat of ISMRCIS, Boreskov Institute of Catalysis, 5, Prosp. Akad. Lavrentieva, Novosibirsk, 630090, Russia. Tel: +7 (3832) 34-12-97; Fax: +7 (3832) 34-30-56; E-mail: star@catalysis.nsk.su.
- Royal Society of Chemistry: 15th International Meeting on NMR Spectroscopy, Durham, England, **July 8-12, 2001**; Contact: Mrs. Paula Whelan, The Royal Society of Chemistry, Burlington House, London W1J 0BA, England; tel: +44 (0) 207-437-8656; fax: +44 (0) 207-734-1227; Email: conferences@rsc.org\; Use the subject header '01NMR15'
- ESR and Solid State NMR in High Magnetic Fields, Stuttgart, Germany, **July 22-26, 2001**. Contact: Prof. Hans Paus, 2 Physikalisches Institut, Universität Stuttgart, Pfaffenwaldring 57, D-70550 Stuttgart, Germany. Tel: ++49-711-685-5223 or -5217; Fax: ++40-711-685-5285; E-mail: ampere2001@physik.uni-stuttgart.de.
- ISMAR 2001, Jerusalem, Israel, August 19-24, 2001; See http://www.tau.ac.il/chemistry/ISMAR.html.
- 14th European Symposium on Polymer Spectroscopy, Dresden, Germany, **September 2-5, 2001**. Contact: Institut für Polymerforschung Dresden e. V., ESOPS 14, Postfach 12 04 11, 01005 Dresden, Germany. Tel: +49 351 4658-282; Fax: +49 351-4658-214; E-mail: espos@ipfdd.de.
- Fourth International Conference on Molecular Structural Biology, Vienna, Austria, **September 5-9, 2001**. Contact: Andreas Kungl, Austrian Chemical Society (GÖCH), Biochemistry Subgroup, c/o Institute of Pharmaceutical Chemistry, University of Graz, Universitätsplatz 1, A-8010 Graz, Austria. Tel: +43 316 380 5373; Fax: +43 316 382541; E-mail: andreas.kungl@kfunigraz.ac.at.
- 2nd Alpine Conference on Solid-State NMR, Chamonix-Mont Blanc, France, **September 9-13, 2001**; Contact: Alpine Conference Secretariat, Laboratoire STIM, Ecole Normale Supérieure de Lyon, 46 allée d'Italie, 69364 Lyon Cedex 7, France; alpine.SSNMR@ens-lyon.fr; Tel. +33-(0)4 72-72-84-86/83 84; Fax. +33 (0)4 72 72 84 83; http://ens-lyon.fr/STIM/alpineweb,html
- XXth International Conference on Magnetic Resonance in Biological Systems, Toronto, Ont., August 25-30, 2002. For further information check www.uwo.ca/chem/icmrbs/, or contact: mgordon@julian.uwo.ca

Additional listings of meetings, etc., are invited.

Address all Newsletter correspondence to:

Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303.
650-493-5971* - Please call
only between 8:00 am and
10:00 pm, Pacific Coast time.

Deadline Dates

No. 509 (Feb.)	26 Jan. 2001
No. 510 (Mar.)	23 Feb. 2001
No. 511 (Apr.)	23 Mar. 2001
No. 512 (May)	27 Apr. 2001
No. 513 (June)	25 May 2001

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 is adorned with a large <u>red dot</u>: this decoration means that you will not be mailed any more issues until a technical contribution has been received.

^{*} Fax: 650-493-1348, at any hour. Do not use fax for technical contributions to the Newsletter, for the received fax quality is very inadequate.

^{*} E-mail: shapiro@nmrnewsletter.com

JEOL Can Give You the Data You Need From Your Desktop PC or MAC

Detta : Automation Mode Sample						
Connect:ecp300.jeol.com			0		0[Hz]	
		XH	22.9[dC]	1	TULE	
Connect View	Remove	Iter	0	Seans	0	
Sample ID 123-Isolation				Solvents		
Comment Basic Prep. ACETIC_ACID-D3 ACETONE-D6						
Silot 0 Sample State LOADED ACETONITRILE-D3 BENZENE-D6 CHLOROFORM-D						
Cur. Temp. 22.9[dC] Lock States IDLE CHLOROFORM - D CYCLOHEXANE-D12 Temp. Set 25[dC] Temp. State TEMP OFF						
Proton Presaluration						
Carbon			Proton_and_Carhon			
Carbon and Dept 135 Carbon and APT			100			
Edited DEPT Proton and COSY						
ecp300.jeol.com : INFO : Job 00_012 is now completed on 123-Isolation						
ecp300.jeol.com : INFO : Queue is Held on sample 123-Isolation ecp300.jeol.com : INFO : Queue no longer Held Class						
ecpsuu jeol.com : INFO : Que	He He Hong	eres (c	7.0		Clase	

The **Eclipse+** NMR Spectrometer can be operated anywhere there is a computer on the local network. The **Single Window Automation** pictured above can be used with a single mouse click to select the sample from the auto-sample changer, gradient shim on any probe, run the selected experiment, and plot the data on any network postscript printer. Need more data, click another button and the **Eclipse+** is off to do your work - and you have not left your office. Contact us at nmr@jeol.com or visit or web site at www.jeol.com.

JEOL USA, Inc., 11 Dearborn Road, Peabody, MA 01960
Tel: 978-535-5900 Fax: 978-536-2205
email: nmr@jeol.com www.jeol.com

