THE
NMR
NEWSLETTER

No. 497
February 2000

PTS FREQUENCY SYNTHESIZERS

FREQUENCY GENERATORS, AGILE, QUIET, FAST

Accurate, stable frequencies on command, μs switching. For NMR, Surveillance, ATE, Laser, Fluorescence. Low noise/jitter. Adapting to your needs with options.

PTS FREQUENCY SYNTHESIZERS

	Frequency Range	Resolution	Switching Time	Phase-Continuous Switching	Rack-Mount Cabinet Dim.[1]	Remote-Control Interface	Price Example[2]
PTS 040	.1-40 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 120	90-120 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$5,330.00 (1 Hz resol., OCXO freq. std.)
PTS 160	.1-160 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$6,495.00 (1 Hz resol., OCXO freq. std.)
PTS 250	1-250 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$7,440.00 (1 Hz resol., OCXO freq. std.)
Type 1 **PTS 310** Type 2	.1-310 MHz	1 Hz	1-20μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	1 Hz resol., OCXO: $6,425.00 1 Hz resol., OCXO: $5,850.00
PTS 500	1-500 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,720.00 (1 Hz resol., OCXO freq. std.)
PTS 620	1-620 MHz	optional .1 Hz to 100 KHz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$9,625.00 (1 Hz resol., OCXO freq. std.)
PTS 1000	0.1-1000 MHz	optional .1 Hz to 100 KHz	5-10μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$11,830.00 (1 Hz resol., OCXO freq. std.)
PTS 3200	1-3200 MHz	1 Hz	1-20μs	optional	5¼"H×19"W	BCD (std) or GPIB (opt)	$14,850.00 (1 Hz resol., OCXO freq. std.)
PTS x10	user specified 10 MHz decade	1 Hz	1-5μs	standard	3½"H×19"W	BCD (std) or GPIB (opt)	$3,000.00 (1 Hz resol., OCXO freq. std.)
PTS D310	two channels .1-310 MHz	.1 Hz	1-20μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$8,560.00 (.1 Hz resol., OCXO freq. std.)
PTS D620	two channels 1-620 MHz	.1 Hz/.2 Hz	1-20 μs	standard	5¼"H×19"W	BCD (std) or GPIB (opt)	$13,240.00 (.1 Hz/.2 Hz resol., OCXO freq. std.)

1 Bench cabinets are 17" wide.
2 Prices are U.S. only and include Manual and Remote (BCD) Control; PTS 3200 Digital Front Panel.

PTS CAN SUPPLY OEM-TYPE SYNTHESIZERS FOR ALL LEADING NMR-SPECTROMETER PRODUCTS.

PROGRAMMED TEST SOURCES, INC.

P.O. Box 517, 9 Beaver Brook Rd., Littleton, MA 01460 Tel: 978-486-3400 Fax: 978-486-4495
http: // www.programmedtest.com • e-mail: sales@programmedtest.com

SPONSORS OF *THE NMR NEWSLETTER*

Abbott Laboratories
Advanced Chemistry Development, Inc.
Agilent Technologies
Aldrich Chemical Company, Inc.
Amgen, Inc.
AMT
Anasazi Instruments, Inc.
AstraZeneca
Avanti Polar Lipids, Inc.
Bruker Instruments, Inc.
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

Biennial Meeting of the Australian and New Zealand Society for Magnetic Resonance (ANZMAG2000), Mt. Buller, Victoria, Australia; **February 13-17, 2000**; Contact: Dr. Jenny Wilson, Victorian College of Pharmacy, Monash University, Parkville, Victoria 3052, Australia; E-mail: anzmag@edda.vcp.monash.edu; vcp.monash.edu.au/chemistry/anzmag2k.

PITTCON 2000, New Orleans, LA, **March 12-17, 2000**; Contact: The Pittsburgh Conference, 300 Penn Center Blvd., Suite 332, Pittsburgh, PA 15235-5503; Phone: 412-825-3220; Fax: 412-825-3224; Email: expo@pittcon.org.

8th Scientific Meeting and Exhibition, International Society for Magnetic Resonance in Medicine, Denver, CO, **April 1-7, 2000**; Contact: ISMRM, 2118 Milvia Street, Suite 201, Berkeley, CA 94704. Tel. 510-841-1899; Fax. 510-841-2340; E-mail: info@ismrm.org; http://www.ismrm.org.

Symposium on Advances in NMR Applications, Naval Postgraduate School, Monterey, CA. Shuttle service to and from Asilomar will be provided. **April 9, 2000**; Contact: V. Davies, Nalorac Corporation, 837 Arnold Drive, Suite 600, Martinez, CA 94553; 925-229-3501; Fax: 925-229-1651; Email: victoria.davies@nalorac.com; http://www.nalorac.com. See Newsletter 495, 28.

41st ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, CA, **April 9-14, 2000**; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; E-mail: enc@enc-conference.org. Web: enc-conference.org

15th European Experimental NMR Conference, Leipzig, Germany, **June, 2000**. For information, see http://eenc.uni-leipzig.de.

SMASH-2000, Argonne, IL, **July 16-19, 2000**. Contact: G. E. Martin (gary.e.martin@amu.pnu.com). See Newsletter 493, 21.

XIX International Conference on Mag. Res. in Biological Systems, Florence, Italy, **August 20-25, 2000**. Contact: Profs. Ivano Bentini or Lucia Banci, Magnetic Resonance Center, Univ. of Florence, Via Luigi Sacconi 6, 50019 Sesto Fiorentino (Firenze), Italy; Phone: +39-055-2757600; Email: icmrbs@cerm.unifi.it; Fax: +39-0554-209253; http://www.cerm.unifi.it/icmrbs.html.

continued on inside back cover

Research Group for Antibiotics and NMR Laboratory
Department of Chemistry
University of Debrecen, Egyetem tér 1
H-4010 Debrecen P. O. Box 70, HUNGARY
e-mail: batta@tigris.klte.hu, fax: (36) 52 512 914
tel: (36) 52 512 900 ext. 2370 or 2234

January 10, 2000 (received 1/11/2000)

Professor B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto
CA 94303-3410
USA

Cool Carbohydrates

Dear Barry,

Well behaving proteins have resolved NH signals in water solution, which is invaluable for any stuctural studies. This is not the case with the OH groups in oligo-and polysaccharides since their exchange with solvent water is generally too fast.

Studying highly concentrated (1-2M) oligosaccharides in D_2O-H_2O mixtures we observed that the ^1H resolution improves (simultaneously with sharper deuterium lock signal) if the temperature is decreased to ca. 264K (just above freezing point) and OH signals exhibit resolved couplings. High concentration of carbohydrates has twofold benefit. Freezing temperature is decreased without supercooling, and NMR sensitivity is sufficient to measure homo and heteronuclear couplings of hydroxyl protons in simple 1D spectra or recent SE-HETLOC or HECADE spectra at natural isotopic abundance. Sensitivity is an issue, since the resolution is better at low H_2O content (3-10 %), and water suppression is not a must. Concerning the two polysaccharides we studied recently, in one we observed the OH groups, however, coupling constant analysis seems more difficult.

Yours truly,

Gyula Batta

1. L. Poppe, H. van Halbeek, *Nat. Struct. Biol.* 1(1994) 215-216, 2. Gy. Batta and K. E. Kövér, *Carbohydr. Res.* 320(1999) 267-272

Add a *NEW* NMR CASE™ Sample Changer and Make Your NMR Work All Night Long

Don't let your NMR spectrometer ***Stop*** *each night after just one sample!*

Bruker is proud to introduce the NMR CASE™ Sample Changer, a new, economical accessory that improves NMR laboratory productivity. It expands the maximum number of sequential samples your spectrometer can process unattended to 24.

Like its smaller sibling, the NMR SIXPACK™, the NMR CASE is simple to use. Setup takes only a few minutes (see installation sidebar). Then, just load the samples, enter your sample information into ICON-NMR*, and go. It's that simple!

Bruker's NMR CASE employs a unique, interchangeable 24 tube carousel. So while one carousel is feeding samples into your spectrometer, your colleagues can be loading another carousel for later acquisitions. The NMR CASE fits most magnet systems up to 600MHz, including Bruker's UltraShield Magnets.

With the NMR CASE, you'll never want to go back to loading samples into your spectrometer one-at-a-time! Both the Bruker NMR CASE and NMR SIXPACK are conveniently priced for laboratories that can't justify larger 60 or 120 tube changers. Contact your local Bruker representative today for more information.

** ICON-NMR™ is a component of Bruker NMR Suite™ software. ICON-NMR makes setting up experiments fast and easy, even for an NMR novice.*

Bruker's economical NMR CASE™ Sample Changer lets your spectrometer take spectra of up to 24 samples with no operator intervention.

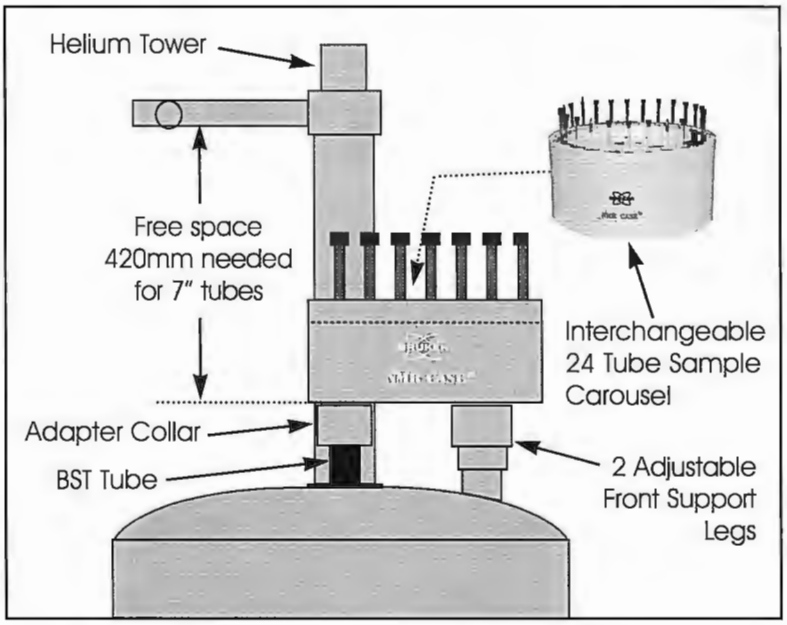
The Control Module of the NMR CASE can be mounted in the standard 19" console rack.

YOU CAN INSTALL AN NMR CASE YOURSELF

It takes just a few minutes! There are four simple steps.

- *Place the sample exchange module on top of the magnet sample transport tube and level with the adjustable front support legs.*
- *Attach a compressed air source to the pneumatic control module.*
- *Connect 5 air hoses between the sample exchange module and the pneumatic control module.*
- *Configure your XWINNMR software by entering two commands.*

It's that simple! No interface boards to install. No calibrations to run.



Sample Exchange Module on top of the magnet showing removable carousel being placed on three-legged base.

The NMR CASE can be used with most every existing magnet system, since the cylindrical carousel fits between the nitrogen fill ports and helium stacks. Some magnet systems are listed below. Don't see your magnet? Just measure the distance between your nitrogen fill ports and helium stack and contact Bruker to determine if the NMR CASE fits your magnet system.

Contact:

Bruker Instruments, Inc.
19 Fortune Drive
Billerica, MA 01821
Phone: 978-667-9580
FAX: 978-667-0985
e-mail: sales@nmr.bruker.com

or contact your nearest Bruker office.

NMR CASE™ Description

Samples:
Up to 24 NMR Tubes 5mm or 10mm OD

Requirements:

Site:
Compressed Air @ 4 - 5.5 bar (60 - 80 psi)

Spectrometers:
All AVANCE Systems
AMX & ARXs with BSMS and SGI computer.
(not compatible with AC, AM, or older models).

Requirements (cont'd.):

Software:
XWINNMR v2.0 or higher.

Magnets:
Bruker: Ultrashield Magnets up to 600 MHz and Long Hold & Ultra-Long Hold Magnets to 600 MHz (not compatible with 800MHz UltraStabilized).
Oxford and Magnex: Standard and Wide Bore Magnets, with some exceptions.

NMR CASE Part Numbers and Prices

Part No.	Description	Price
B2435	NMR CASE™ - Includes: 1 ea. 24 Tube Carousel 1 ea. Sample Exchange Module 1 ea. Pneumatic Control Module (rack mountable) 1 ea. Adapter Collar for BST* 1 ea. pneumatic hose assembly 1 ea. operation manual	$18,950.00
B2436	Additional 24 Tube Carousel (order separately)	$4,500.00

Part No.	Description	Price
B0625	Adapter collar for old SB BST* (order separately)	$400.00
B0636	Adapter collar for WB -> SB Adapter (order separately)	$500.00
Z42516	5mm Spinner (blue plastic†) Set of 24	$90.00 ea. $2,160.00
H00306	10mm Spinner (milky white plastic†) Set of 24	$135.00 ea. $3,240.00

** BST is the Bruker Sample Transport Tube through which samples are shuttled from the top of the magnet system to the probe.*
† Plastic spinners have limited operating temperature range (–50 to +80C). Inquire about ceramic spinners for extreme temperatures.

UNIVERSITY OF CALIFORNIA, SAN FRANCISCO

BERKELEY • DAVIS • IRVINE • LOS ANGELES • RIVERSIDE • SAN DIEGO • SAN FRANCISCO SANTA BARBARA • SANTA CRUZ

Department of Pharmaceutical Chemistry
SAN FRANCISCO, CALIFORNIA 94143-0446

January 26, 2000
(received 1/28/2000)

Dr. B.L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303-3410

Structure refinement of multiple, rapidly interconverting conformers

Dear Barry:

Recently, we proposed a new approach for refining structural conformers and determining their populations based on NMR data [1].

When distinct multiple conformers exchange rapidly on the NMR time scale, they give rise to a single set of NMR lines. In such a situation, plain inspection of spectra may give no clues about the presence of such conformers. Experimentally measured NMR parameters (such as NOE or scalar coupling data) correspond, nevertheless, to appropriate averages over all structures contributing to the signal. Conventional structural refinement using such data may lead to incorrect or even sterically impossible structures. If no single conformation can satisfy all experimental restraints, this can be an indication of the presence of multiple conformers. Furthermore, contradictions between experimental restraints and the theoretical force field used in structure refinement can serve as a basis for determination of high-resolution structures of individual conformers. But this task requires, of course, special approaches for refinement.

Several refinement tools have been proposed in the past to deal with this situation. We mention here the ensemble-average (or multiple-copy) molecular dynamics (MD) [2,3], because our own approach is similar to it in its main idea. Similar to conventional refinement schemes, multiple-copy MD refinement is driven by a pseudoenergy. The pseudoenergy is a sum of two terms: conformational energy defined by a theoretical force field, and a penalty term, which enforces the agreement between calculated and observed NMR parameters (such as NOE-derived inter-proton distances). The difference from the conventional restrained MD is that multiple-copy MD refines several copies of a molecule simultaneously. Individual molecules do not interact with each other during the MD simulation, but theoretical NMR parameters are calculated as appropriate averages over all copies. As a result, experimental restraints are not enforced for each individual molecule, but only for the ensemble as a whole. If a set of experimental restraints cannot be satisfied by any single conformation, multiple-copy MD will produce a set of distinct structures that satisfy experimental restraints as an ensemble.

One problem with this approach is the assumption that all members of the ensemble have equal populations, which somewhat limits its possible applications. In one variant of this method [4], individual structures are weighted with the Boltzmann factors derived from their conformational energies. However, relative free energies rather than conformational energies determine populations of conformers; attempts to calculate free energies during refinement would make this method impractical. We wanted to modify this method in such a way that populations of conformers are determined by experimental data rather than by theoretical force field. This can be done using the PDQPRO program, a part of the PARSE procedure [5]. For a given set of conformations, PDQPRO calculates their optimal populations by fitting theoretical ensembles' NMR parameters to the observed parameters. The fitting is done by a quadratic programming algorithm, which imposes a limitation on the type of NMR parameters that can be used: they must average in a linear fashion over the

ensemble. Suitable parameters are, e.g., scalar coupling constants and dipolar relaxation rates. It is relatively straightforward to calculate dipolar cross-relaxation rates for a theoretical ensemble of structures; experimental rates can be derived iteratively from NOE data, using, e.g., the program MARDIGRAS [6]. Derivation of experimental dipolar relaxation rates from NOE data is done independently of (prior to) refinement, so the required CPU time is not an issue here. Calculation of theoretical rates and PDQPRO calculations are relatively fast and they can be done at each refinement step.

For practical implementation of this idea, we used the DNAminiCarlo program [7] as the refinement engine, because we have the source code of this program in our hands. Instead of MD simulations, DNAminiCarlo performs energy minimization and Metropolis Monte Carlo calculations for nucleic acid systems. This choice limits, of course, possible applications of this method to nucleic acids. However, the calculations are very effective for these systems because of the use of highly specialized internal coordinates, generalized helical parameters. We modified DNAminiCarlo to handle multiple copies; dipolar relaxation rates are calculated for each ensemble-snapshot by invoking subroutines of the RELAX program [8]; "floating" populations are calculated for each snapshot by invoking subroutines of PDQPRO.

We extensively tested this approach using simulated data for DNA oligonucleotides [1]. We assumed that we have measured NOE data corresponding to typically observed cross-peaks for this type of molecule, and we did not use scalar coupling data. These data were sufficient to recover correct structures and correct populations for simulated equilibria involving two major conformers of sugar moieties: C2'*endo* and C3'*endo*. However, the same data were not sufficient to define reliably three-conformer equilibria with C2'*endo*, C3'*endo*, and an intermediate O4'*exo* conformers. This exercise showed that our approach can work successfully, but only if the system is defined sufficiently by experimental data. If all experimental data can be explained with a single structure, the method will produce a single "average" structure, even if multiple conformers contributed to observed data. Currently we are applying this method to an experimental RNA system. Also, we are interested in extending our approach to protein systems, but this will require using a different refinement engine.

---

1. A. Görler, N.B. Ulyanov & T.L. James, *J. Biomolec. NMR*, **in press** (2000).

2. A.M. Bonvin & A.T. Brönger, *J. Mol. Biol.*, **250**, 80-93 (1995).

3. J. Kemmink & R.M. Scheek, *J. Biomolec. NMR*, **6**, 33-40 (1995).

4. J. Fennen, A.E. Torda & W.F. van Gunsteren, *J. Biomolec. NMR*, **6**, 163-170 (1995).

5. N.B. Ulyanov, U. Schmitz, A. Kumar & T.L. James, *Biophys. J.*, **68**, 13-24 (1995).

6. B.A. Borgias & T.L. James, *J. Magn. Reson.*, **87**, 475-487 (1990).

7. V.B. Zhurkin, N.B. Ulyanov, A.A. Gorin & R.L. Jernigan, *Proc. Natl. Acad. Sci. U.S.A.*, **88**, 7046-50 (1991).

8. A. Görler & H.R. Kalbitzer, *J. Magn. Reson.*, **124**, 177-188 (1997).

Sincerely,

Nikolai Ulyanov

Nikolai B. Ulyanov

Adrian Görler

Tom

Thomas L. James
Professor & Chair

A d v a n c e d C h e m i s t r y D e v e l o p m e n t

133 Richmond St., W., Suite 605, Toronto, Ontario Canada, M5H 2L3 T: 416-368-3435 F: 416-368-5596
www.acdlabs.com info@acdlabs.com

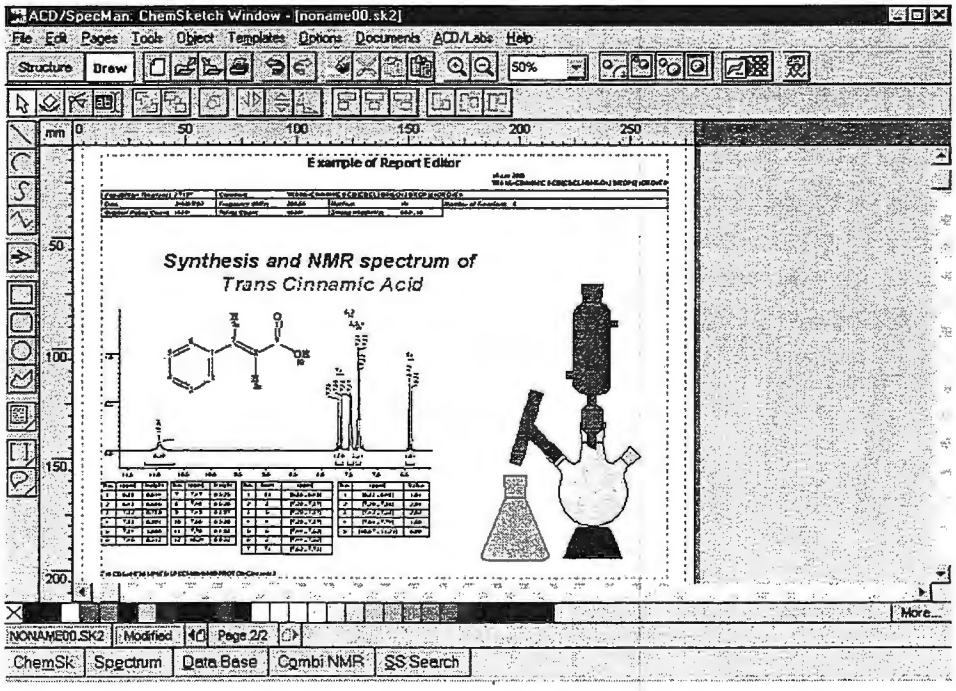
Sharing NMR Data and Making NMR Reports

January 11th, 2000 (received 1/12/2000)

Dear Barry,

During recent months I have been watching a large number of contributions which have been made to the NMR Newsletter and wonder whether or not there is an awareness regarding the ease of graphics inclusion in word processing documents. Recently there was a report regarding the movement of NMR data from a spectrometer through a desktop NMR processing package to a commercial graphics package From here an image file was generated for moving into a word processor. I would like to suggest that modern software packages offering standard OLE capabilities are more than sufficient for these tasks and that spectrometer vendors include such functionality in their standard software, especially when providing access to the data at the desktop whether it be via X-Windows emulation or Java tools.

As a provider of desktop processing tools we have chosen to use our chemical structure drawing package as the container for the graphical images generated by spectra, be they 1D or 2D. Since Chemsketch has both color capability and multiple tools in DRAW mode, the graphical displays of the spectra can be significantly enhanced prior to export to any of a number of graphics formats (MDL/ISIS skc, Windows BMP, Windows WMF, Paintbrush PCX, TIFF bitmap and GIF for web based graphics exchange). Such enhancements can include additional drawings such as laboratory apparatus, chemical structures and annotations as shown below inside the Report Editor of ACD/Chemsketch.

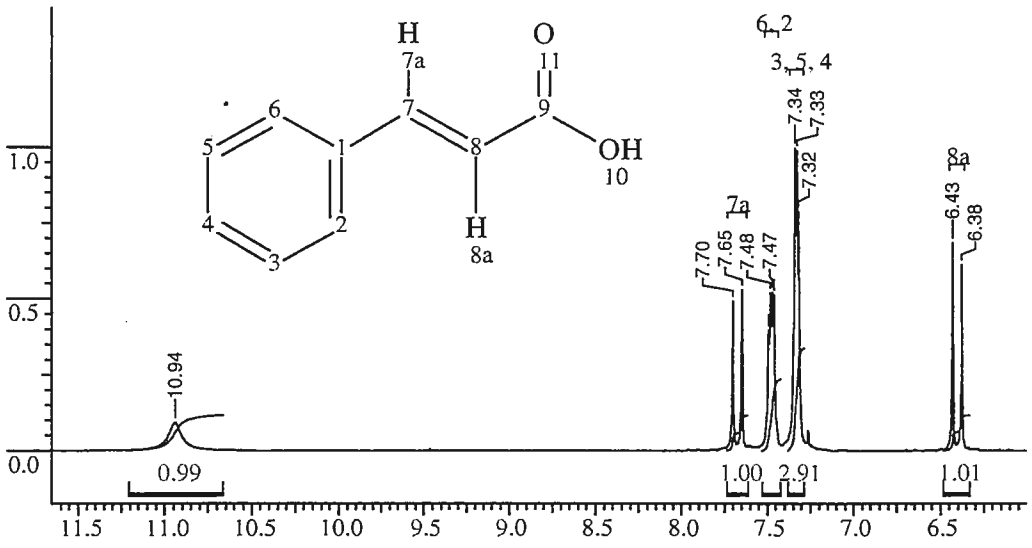


A d v a n c e d C h e m i s t r y D e v e l o p m e n t

133 Richmond St., W., Suite 605, Toronto, Ontario Canada, M5H 2L3 T: 416-368-3435 F: 416-368-5596
www.acdlabs.com info@acdlabs.com

Simply copy and pasting can move some or all of the images into Microsoft Word as shown below.

Synthesis and NMR spectrum of
Trans Cinnamic Acid



The ability to produce this type of graphics capabilities is of high value to chemists and spectroscopists alike for producing objects for inclusion in technical reports and publications. The drawing package ACD/Chemsketch can be freely downloaded from www.acdlabs.com/download. Also available is the free SpecViewer package (http://www.acdlabs.com/download/specview.html). This freeware can Import and display the major data formats (Galactic SPC, JCAMP-DX, ASCII) and ACD/ESP files. The freeware can analyse spectra without transforming the data (e.g., Reference, Integration, Peak Picking, Annotation), attach chemical structures to the spectrum and print spectra and create reports using all the power of ACD/ChemSketch (required). We have already had thousands of copies downloaded and used for the dissemination of pre-processed spectra across academic institutions. Professors are commonly processing the spectra using ACD/NMR Processor and putting the spectra up on web pages for download. We encourage everybody to take advantage of the freeware.

Best wishes,

Tony Williams

Your Source for RF Amplifiers

AMT

ISO 9001 CERTIFIED

2570 E. Cerritos Avenue
Anaheim, CA 92806

Phone: 714-456-0777
Fax: 714-456-0780
Email: amtinc.com

SCIENTIFIC PRODUCTS

AMT's scientific products are used extensively in Nuclear Magnetic Resonance (NMR) systems. These amplifiers cover the frequency ranges of 6 MHz to 500 MHz, with power levels as high as 2.0 kW peak power at 10% duty cycle.

MEDICAL PRODUCTS

AMT's medical products are employed in Magnetic Resonance Imaging (MRI) systems. These amplifiers cover the frequency range from 10 MHz to 200 MHz with power levels as high as 12 kW peak power at 10% duty cycle.

Your Source for RF Amplifiers

AMT IS:

⇒ LEADING SUPPLIER OF AMPLIFIERS

⇒ CUSTOMER SENSITIVE

⇒ ISO 9001 CERTIFIED

AMT AMPLIFIERS ARE:

⇒ PULSED OR CW MODE OPERATION

⇒ HIGHLY RELIABLE

⇒ SOLID STATE

⇒ COST EFFECTIVE

AMT

ISO 9001 CERTIFIED

2570 E. Cerritos Avenue
Anaheim, CA 92806

Phone: 714-456-0777
Fax: 714-456-0780
Email: amtinc.com

ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue, Argonne, Illinois 60439

January 11, 2000
(received 1/15/2000)

Bernard L. Shapiro
The NMR Newletter
966 Elsinore Court
Palo Alto, CA
USA 94303

Determination of Network Properties through Transverse (T_2) Relaxation Measurements

Dear Barry,

We have extended the scope of our investigation into solvent transport behavior to include transversal ^{1}H NMR relaxation studies. During the past decade, studies on polymeric networks have shown this method to be a useful tool for characterizing molecular dynamics. Since transverse ^{1}H relaxation times are dependent upon inter- and intra-molecular dipolar interactions of protons, useful properties such as crosslink density, molecular mobility and chain dynamics can be determined. Typically, rigid fractions such as crosslinks and physical entanglements are characterized by short T_2 relaxation times while mobile fractions such as dangling end chains are characterized by longer T_2.

The samples used in our initial investigation were cross-linked copolymers of poly(isobutylene)-co-poly(paramethyl-styrene) (PIB/PMS) (see *The NMR Newsletter* 478, 25, July 1998).

T_2 relaxation curves for a sample of PIB/PMS heated to 130 °C (squares) and the same sample swollen in d_{12}-cyclohexane for 60 minutes (circles) are shown in Figure 1. The relaxation measurements were acquired using the Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence with typical delay times between pulses in the range of 1 μs to 500 ms. Three distinct regions can be observed from the relaxation curves; an initial fast decaying component, an intermediate component and a slowly relaxing tail. The two inserts provide expanded views of regions between 0-100 μs and 200-1000 μs respectively. Although the samples have similar decay rates initially (up to 200 us), the sample heated to 130 °C appears to decay much quicker than the sample swollen in d_{12}-cyclohexane.

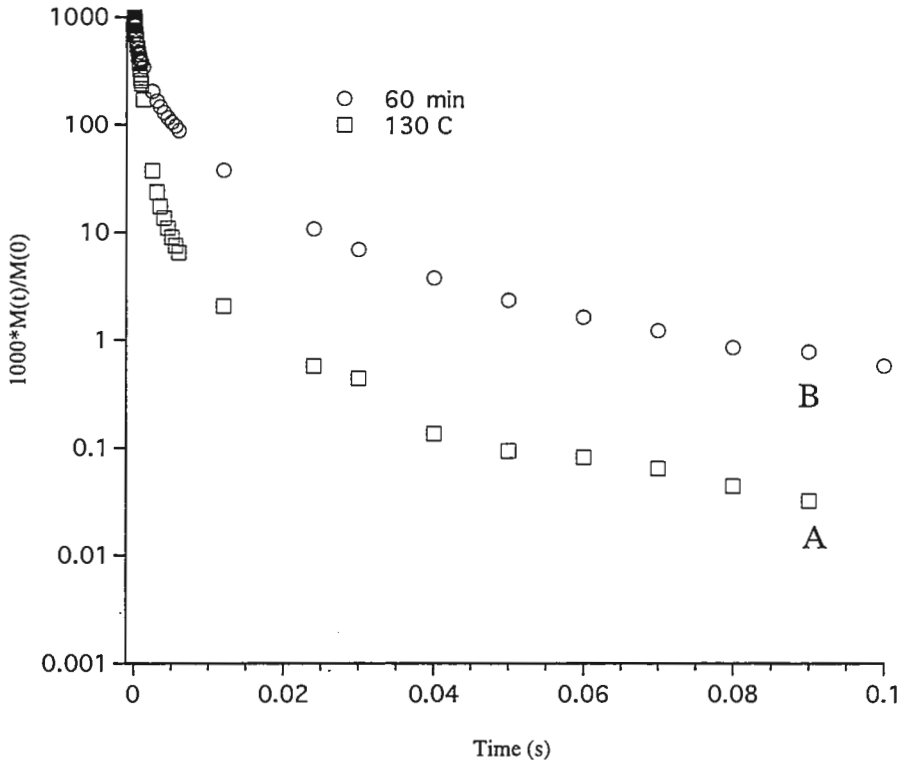
We have attempted to extract values for the fraction of inter-crosslink chains and dangling chain ends, in addition to determining the crosslink density as reported by Simon et al. *(Rubber Chemistry and Technology*, 65, 1, 1992). However, initial attempts at extracting these values using their empirical approach, based on second moment analysis, have been unsuccessful. One possible explanation for this maybe be due to the complex, multicomponent nature of our samples. We are currently investigating new theoretical approaches that will enable useful parameters such as crosslink density to be determined for complex polymeric materials. The ultimate goal is to determine the reliability and accuracy of relaxation measurements in relating physical structure to the energetic state of polymer systems such as rubbers and coals.

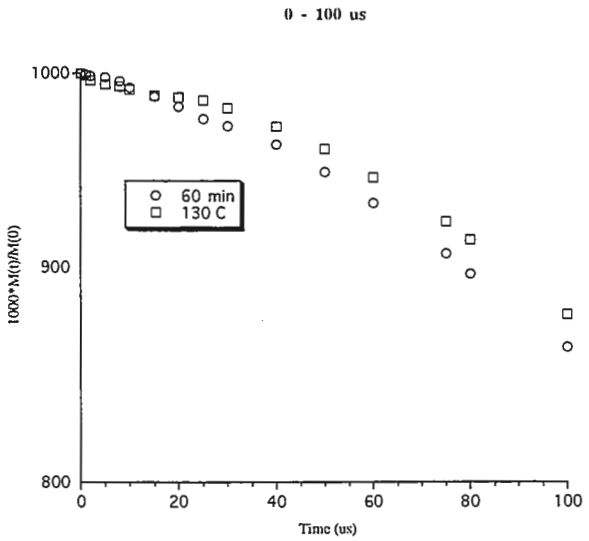
Sincerely,

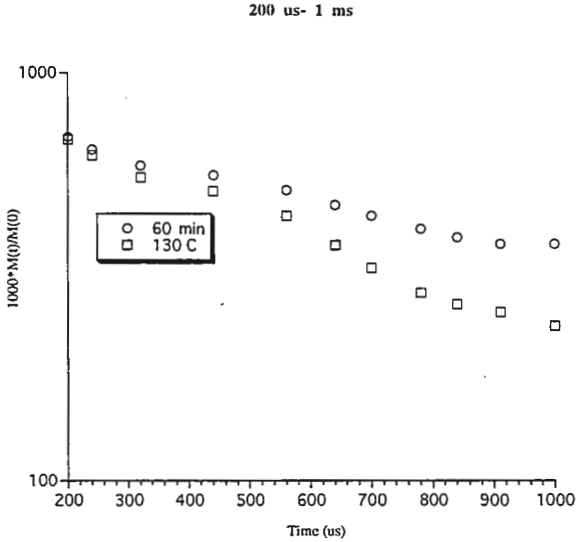
Donald F. Stec
Chemistry Division

Robert E. Botto
Chemistry Division

Figure 1. ^{1}H transverse relaxation curves for PIB/PMS heated to 130 °C and swollen with d_{12}-cyclohexane for 60 min.







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

January 6, 2000
(received 1/18/2000)

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

Streochemical *endo/exo* preference of 3-cyano-2-oxofuro[2,3-*b*]indoles

Dear Professor Shapiro:

3a-Alkyl-3-cyano-2-oxofuro[2,3-*b*]indoles **1a-d** are an important class of compounds, because they may be easily transformed into the corresponding fused furans, lactams, pyrroles, etc. These compounds are useful intermediates for the synthesis of biologically active natural products,[1] which have been isolated from diverse origins such as the *Physostigma* and *Flustra* alkaloids.[2] Preliminary investigations[3] about the configuration of **1a-d** demonstrated that compound **1a-c** exist as mixtures of *endo/exo* epimers in solution and undergo fast H-3 deuterium exchange with D_2O. In contrast, although H-3 in **1d** slowly exchanges with D_2O, a single diastereoisomer is present in solution.

The NOE experiments allowed unequivocal *endo/exo* assignment (Figure). For *endo* epimers NOEs of 17-27% were observed for the H-3 and H-8a protons, upon irradiation at the C(3a) methyl protons of the alkyl group of the major epimer in **1a**, and for the single epimer in **1d**. Whereas, a NOE was not detected for H-3 when the methyl protons of the minor *exo*-**1a** epimer were irradiated.

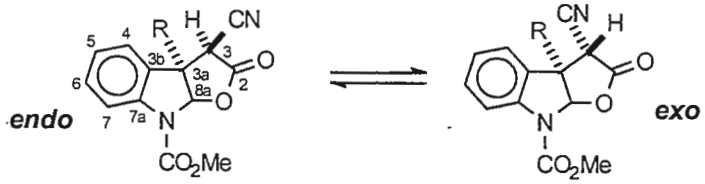
Concerning the furoindole **1a**, *ab initio* calculations performed at the HF/3-21G* level predict that the most stable structure corresponds to the *endo* epimer with a relative energy difference (E_{rel}) between the *endo* and *exo* epimers of 2.1 kJ mol^{-1}. Furthermore, if it is assumed that the entropic and solvation contributions are not important in the equilibrium between these epimers, the calculated difference in free energy (2.1 kJ mol^{-1}) means a value of 2.4 for the corresponding equilibrium constant, in agreement with the $\Delta G°$ value ($\Delta G° = -RT \ln K$, 25 °C) derived from the observed *endo/exo* populations in the ^1H NMR spectra measured in CD_2Cl_2 solvent.

REFERENCES

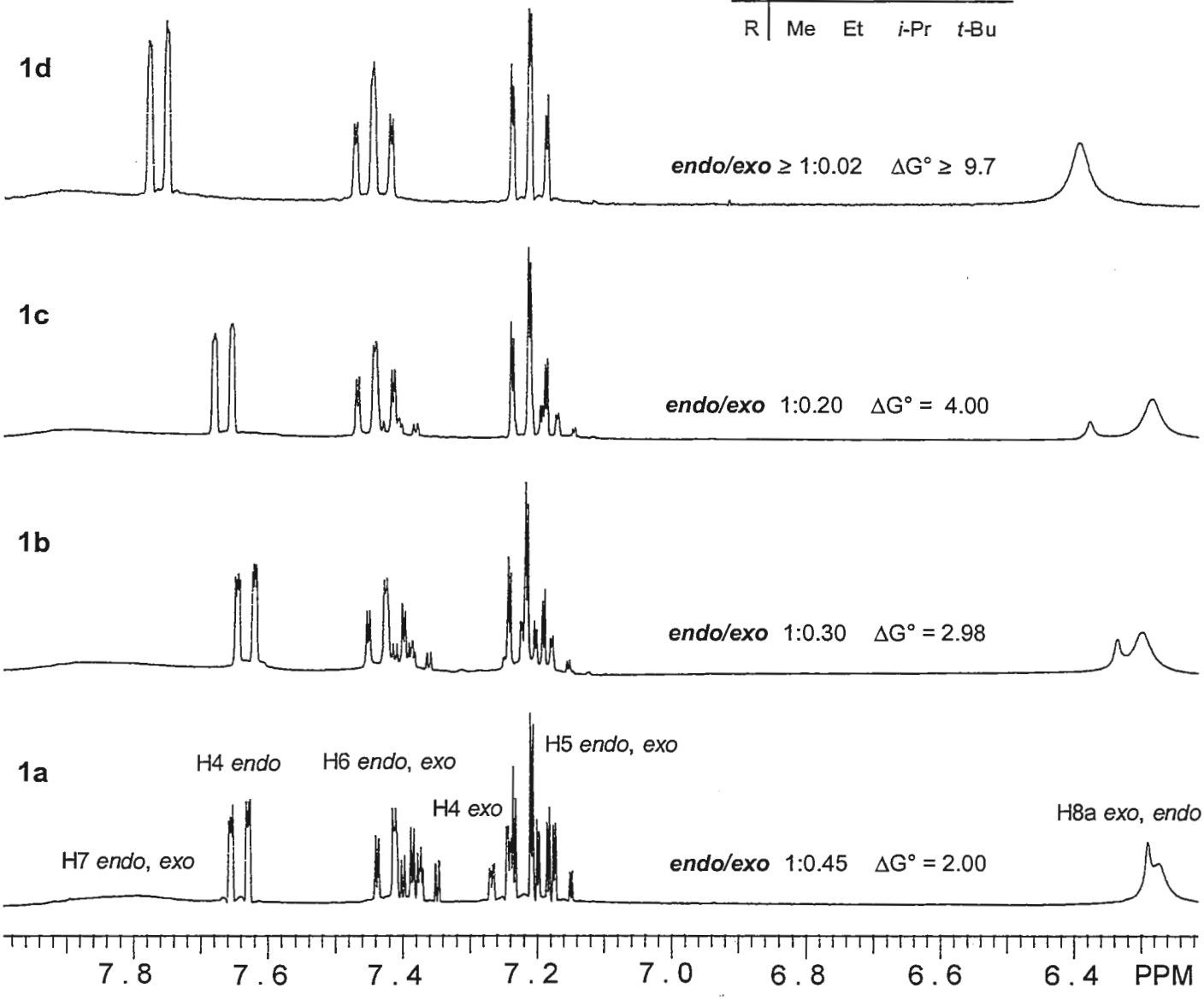
1. M. S. Morales-Ríos, O. R. Suárez-Castillo and P. Joseph-Nathan, *J. Org. Chem.*, 1999, **64**, 1086.
2. B. Witkop, *Heterocycl.*, 1998, **57**, 563; C. Christophersen, *Acta. Chem. Scand. B*, 1985, **39**, 517.
3. M. S. Morales-Ríos, O. R. Suárez-Castillo, C. García-Martínez and P. Joseph-Nathan, *Synthesis*, 1998, 1755.

Av. Instituto Politécnico Nacional 2508 Col. San Pedro Zacatenco México, D.F. C.P. 07360
Tels: 747-70-00 y 747-70-01 Fax: 747-70-03

in CD_2Cl_2
300 MHz



	1a	1b	1c	1d
R	Me	Et	*i*-Pr	*t*-Bu



ΔG° in kJ mol^{-1}

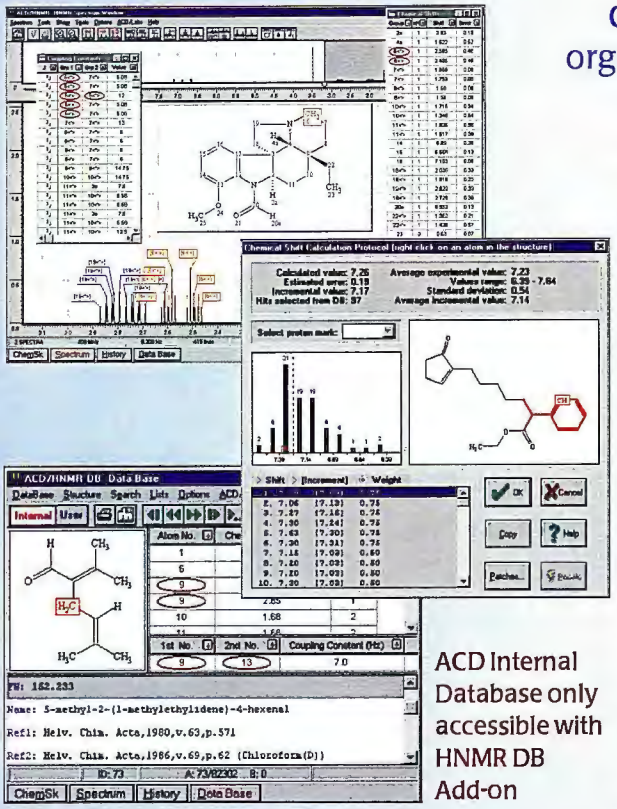
Sincerely yours,

Martha S. Morales-Ríos
Oscar R. Suárez-Castillo
Pedro Joseph-Nathan
pjoseph@nathan.chem.cinvestav.mx

Unparalleled NMR Prediction

Also Available F, P, & 2D NMR Predictors

ACD/HNMR



Calculate the proton NMR spectrum for any organic chemical structure to very high accuracy.

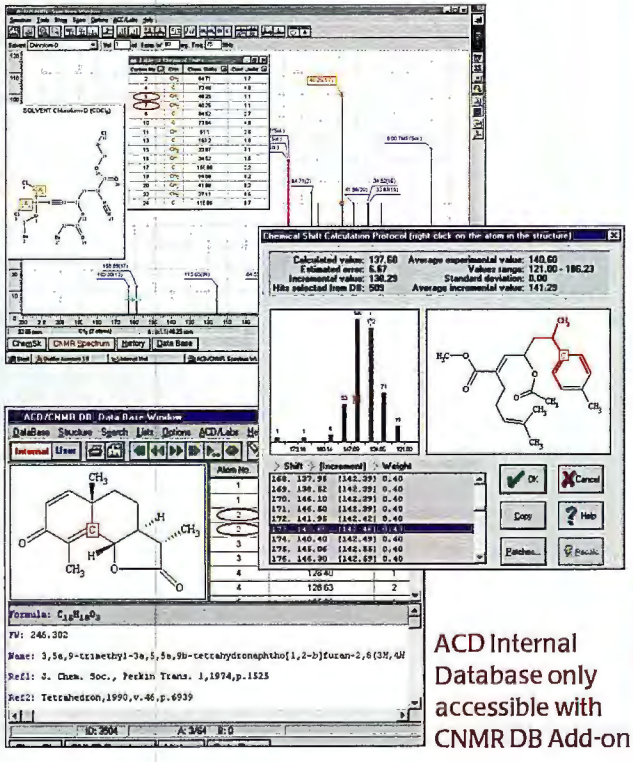
- Takes into account second order interactions (strong coupling effects) and long-range coupling constants.
- Uses 3D molecular structure minimization and Karplus relationships to predict proton-proton coupling constants.
- Allows you to calculate exact spectra for any strongly coupled system with up to 8 magnetically non-equivalent nuclei (spin 1/2) or more if some nuclei are magnetically equivalent.
- Prediction based on an internal data file with over 600 000 experimental chemical shifts and 110 000 coupling constants.
- Self-training system lets you create your own database of chemical shifts, for improved prediction accuracy .
- **NEW!** Data Forms Manager streamlines and standardizes record entry as you build your own databases.
- **NEW!** Include multiple user databases in system training
- **NEW!** Calculate missing shifts in Database Window.
- **NEW!** Enter unassigned shifts or automatically transfer peak table into HNMR database from SpecManager.
- **NEW!** Modify or delete shifts in the internal DAT file.

ACD Internal Database only accessible with HNMR DB Add-on

ACD/CNMR

Calculate the carbon-13 NMR spectrum for any organic molecule to very high accuracy.

- Calculates spin-spin interactions, simulates off-resonance, DEPT, J-modulation and much more.
- Prediction based on an internal data file with over 900 000 experimental chemical shifts.
- Self-training system lets you create your own database of chemical shifts, for improved prediction accuracy.
- **NEW!** Data Forms Manager streamlines and standardizes record entry as you build your own databases.
- **NEW!** Include multiple user databases in system training
- **NEW!** Calculate missing shifts in Database Window.
- **NEW!** Modify or delete shifts in the internal DAT file.
- **NEW!** Enter ^{13}C-X coupling constants, where X = ^{19}F, ^{31}P, etc.
- **NEW!** Enter unassigned shifts or automatically transfer peak table into CNMR database from SpecManager.



ACD Internal Database only accessible with CNMR DB Add-on

NMR
IR
UV-VIS
MS
Raman

Visionary Software for Scientists

Advanced Chemistry Development

NMR

IR

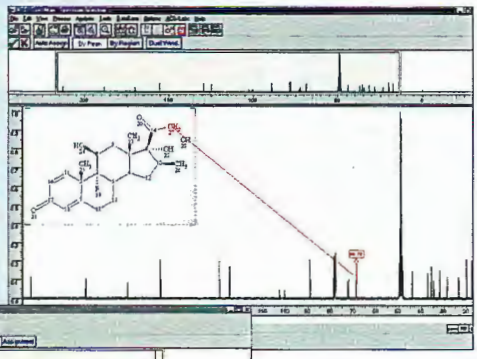
UV-Vis

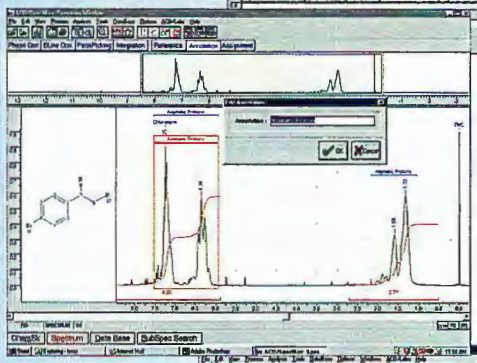
MS

Raman

ACD/NMR Processor

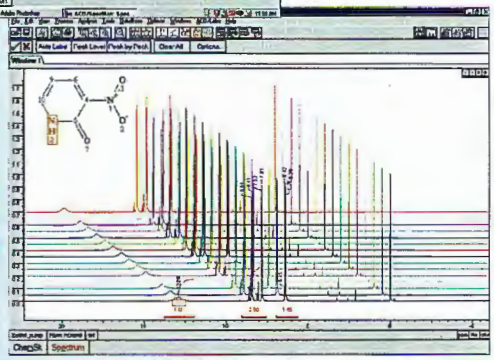
Assign a spectrum by selecting either peak or region–simply select the nucleus and drag towards the peak





On-screen annotations: ideal for identifying solvents, impurities, and polymer details including sequencing and tacticity

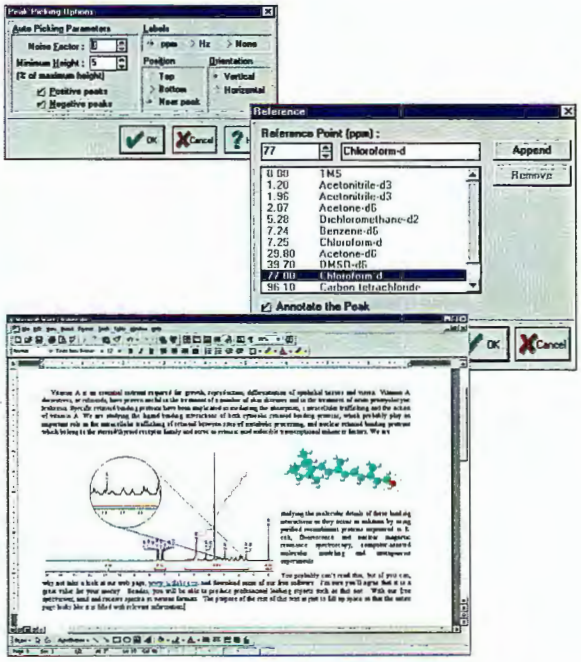
Overlay multiple spectra



Process raw NMR data using a wide array of tools. Creation of professional reports is just a button click away!

Both NMR Processor and NMR Manager provide a complete solution for processing experimental NMR data, including:

- Import filters for Varian, Bruker, JEOL, Nicolet Chemagnetics, WinNMR, NUTS, Lybrics, JCAMP, etc.
- Fourier Transform, phase correction, baseline correction, peak referencing, peak integration, and peak picking
- Manual and auto-phasing
- Manual and auto-integration
- Rectangular zoom
- Peak-by-peak assignment between structure and spectrum
- Annotation of each spectrum using either peak or region selection is possible, offering unique capabilities to assign and annotate polymer spectra and other complex spectral curves
- Full integration with the ChemSketch structure drawing package permits automatic layout and cut-and-paste of spectra, structures, annotations and tables.



Create professional slides and reports

New features in NMR Manager and NMR Processor

- New Weighting Functions,
- Interactive Weighting Functions,
- Multiple Window Display,
- Spectral Subtraction and Addition

Visit Us At PITTCON! Booth #4029

Advanced Chemistry Development
133 Richmond St. West, Suite 605
Toronto, Ontario, M5H 2L3
T: 416 368·3435
F: 416 368·5596
Toll Free: 1 800 304·3988
info@acdlabs.com
www.acdlabs.com

We are pleased to invite you to attend the....

8th Annual Nalorac

ADVANCES IN NMR APPLICATIONS SYMPOSIUM

Sunday April 9, 2000
1:00 to 6:00 p.m.
Naval Post Graduate School
Monterey, California
Transportation provided to and from Asilomar

Featuring the latest developments in experimental techniques presented by leading NMR experimentalists.

including....

- **Sample Automation**
- **Small Sample Characterization**
- **New Experimental Methods**
- **Molecular Ordering**
- **Large and Small Molecular Systems**
- **Cryogenic Probes**

NALORAC'S 25th ANNIVERSARY SYMPOSIUM 2000

NALORAC

Register and request a detailed program:

Nalorac Corporation, 837 Arnold Drive Suite 600, Martinez, CA 94553
Phone: (925) 229-3501 Fax: (925) 229-1651
Email: symposium@nalorac.com
Website: http://www.nalorac.com

HERCULES

Hercules Incorporated
Research Center
500 Hercules Road
Wilmington, DE 19808-1599
Phone: (302) 995-3505
FAX: (302) 995-4135
email: hcheng@herc.com

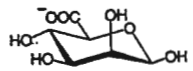
Dr. B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303-3410

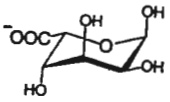
January 8, 2000
(received 1/15/2000)

Dear Dr. Shapiro:

NMR Analysis of Alginates

Alginates are natural polysaccharides extracted from brown algae and used commercially as thickeners, binders, encapsulants, stabilizers, film-formers, and suspending agents[1]. Structurally, they are unbranched copolymers of (1-4)-linked β-D-mannuronic acid (M) and α-L-guluronic acid (G) residues.

M = 

G = 

These comonomers are known to form both blocky and alternating sequences in alginates, and the polymeric microstructure is essential for their properties.

Alginates have been extensively studied by NMR[2-6], especially by Grasdalen, Larsen, Smidsrod, Skjak-Braek, and their coworkers[3-6]. They have provided spectral assignments and also recommended a computational procedure to obtain M-and G-centered triads. Analysis has often been done with the help of conventional statistical models, viz., Bernoullian, first- and second-order Markovian models[1,6]. Because alginates are compositionally heterogeneous, Bernoullian and Markovian models may not be appropriate. I have recently[7] used alternative models to analyze the NMR data of alginates.

As an example, the NMR triad data (I_{obsd}) of an alginate sample, taken from a paper in the literature[3], are shown in column 2 of Table 1. It is clear (from columns 3 and 4 in Table 1) that single-component Bernoullian (**B**) and first-order Markovian (**M1**) models[8] do not fit the data very well (mean deviation > 1.0). Two analytical methodologies were then employed[7] to analyze this data set: 1) perturbed Markovian (continuous) model, and 2) multicomponent (discrete) model.

Table 1. NMR data of alginate and analysis by different statistical models

		one-component models		perturbed Markovian models		multicomponent models	
triad	I_{obsd}	I_{calc}(**B**)	I_{calc}(**M1**)	I_{calc}(EMG/**B**)	I_{calc}(EMG/**M1**)	I_{calc}(**B**/**B**)	I_{calc}(**M1**/**M1**)
MMM	38	38.0	38.0	38.0	35.7	38.0	38.0
MMG	16	28.9	18.3	18.1	16.0	16.4	16.0
GMG	6	5.5	2.2	4.3	6.0	5.1	6.0
MGM	10	14.5	3.1	9.1	9.8	8.2	10.0
GGM	8	11.0	16.5	8.5	8.3	10.3	8.0
GGG	22	2.1	22.0	22.0	24.2	22.0	22.0
mean dev.		6.8	3.6	0.9	0.8	0.9	0.0
		P_M=0.724	P_{GM}=0.272	P_M=0.643	P_{GM}=0.695	component 1	component 1
			P_{MG}=0.194	σ =0.294	P_{MG}=0.437	w_1=0.344	w_1= 0.344
				τ = -0.039	σ = 0.367	P_M=0.143	P_{GM}=0.144
					τ = -0.042		P_{MG}=0.996
						component 2	component 2
						w_2=0.656	w_2= 0.656
						P_M=0.833	P_{GM}=0.970
							P_{MG}=0.174

Perturbed Markovian Model

Previously the perturbed Markovian models have been developed to treat compositionally heterogeneous polymers, using symmetric function[9], non-symmetric functions[10], or a function-free approach[10]. For convenience, the exponentially modified Gaussian (EMG) function has been used for alginates[7]. Thus, the Bernoullian probability is represented not by one value (P_M), but by a distribution:

$$f(z) = \frac{N}{\tau\sigma\sqrt{(2\pi)}} \int_0^\infty \exp\left[-\frac{(z-P_M-z')^2}{2\sigma^2} - \frac{z'}{\tau}\right] dz' \qquad (1)$$

where z is the Bernoullian probability, N the area under the Gaussian, σ the standard deviation, τ the skew factor, z' the dummy variable of integration, and P_M the average value of Bernoullian probability without the exponential modification. Equation 1 gives the Bernoullian EMG function (EMG/**B**). The EMG function for the first-order Markovian probabilities (EMG/**M1**) can be expressed similarly[10].

The experimentally observed sequence intensities can be fitted to the theoretical sequence intensities to obtain P_M (or P_{MG} and P_{GM}), σ, and τ. The results are shown in columns 5 and 6 of Table 1. Indeed both perturbed models (EMG/**B** and EMG/**M1**) produce acceptable goodness-of-fit (mean deviation $\leq$ 1.0%, comparable to experimental precision). Although the EMG/**M1** function introduces one additional probability parameter, the improvement in the fit over EMG/**B** function is only marginal (mean deviation 0.8 versus 0.9). Using the values of the EMG/**B** function, we can plot the chemical composition distribution (CCD) curve for this polymer (Figure 1).

Multicomponent Model

In the multicomponent models[11,12], the polymer is considered to be the mixture of two or more discrete components. No assumption is made of the nature of the components: they may be separate chains or joined together as block copolymers. In this case, each experimentally observed sequence (composition, diad, triad, or higher n-ad) is the weighted average of the corresponding sequences of all the components. For a two-component polymer, the observed intensity for each triad ($I_{i,obsd}$) is:

$$I_{i,obsd} = w_{1i} f_{1i} + w_{2i} f_{2i} \qquad (2)$$

where $w_{\alpha i}$ is the weight factor, and $f_{\alpha i}$ is the intensity for triad i and component α.

This discrete two-component model has been used to analyze the alginate data (Table 1, columns 7 and 8). The two-component **B/B** model provides a good fit to the data, and the two-component (**M1/M1**) model gives even a better fit. Thus, this alginate sample contains at least two components. The two **M1/M1** components can be depicted in the chemical composition distribution plot (Figure 1).

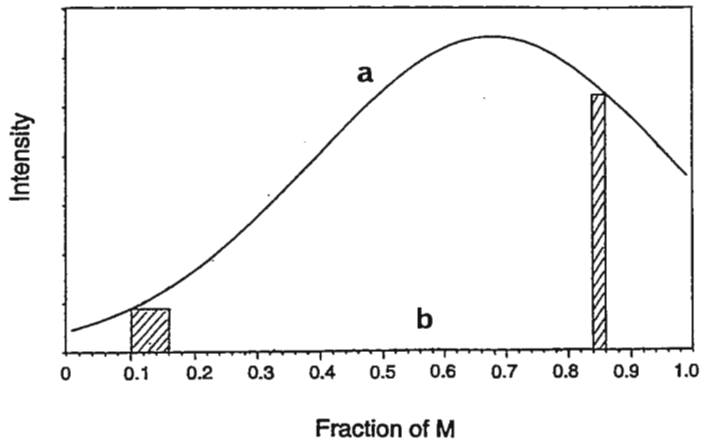


Figure 1. Calculated chemical composition distribution (CCD) for alginate isolated from *Laminaria digitata*: (a) continuous function approach, (b) two-component approximation (discrete model); each component is shown as separate polymer chains for convenience. The area under each bar corresponds to the weight factor.

It is of interest that the same sample can be represented in two different ways (continuous versus discrete functions). For a polymer with a complex microstructure, often the NMR data on the polymer alone do not enable us to tell if one representation is preferred over another. Additional data from other analytical techniques (e.g., fractionation and molecular weight) are needed. In the case of alginates, it is useful to fractionate the polymer and to analyze the NMR data of polymer fractions. This has indeed been done[7]; interested readers may consult the recent paper[7] for more detailed studies.

In summary, alginates are compositionally heterogeneous, and the NMR data reflect this heterogeneity. The NMR data should therefore be analyzed not by conventional single-component models, but by alternative methods, e.g., multicomponent and perturbed Markovian models.

Very truly yours,

H. N. Cheng

References

1. S. T. Moe, K. I. Draget, G. Skjak-Braek, O. Smidsrod, "Alginates", in A. M. Stephen (ed), *Food Polysaccharides and their Applications*, Dekker, New York, Basel, Hong Kong, 1995, pp. 245-286
2. Recent NMR papers include (a) C. A. Steginsky, et al, *Carbohydr. Res.*, **225**,11 (1992), (b) A. Heyraud, et al., *Carbohydr. Res.*, **289**,11 (1996), (c) R. Pannikar, D.J. Brasch, *Carbohydr. Res.*, **293**, 119 (1996), and **300**, 229 (1997), (d) F. Llanes, F. Sauriol, F. G. Morin, A. S. Perlin, *Can J. Chem.*, **75**,585 (1997).
3. H. Grasdalen, B. Larsen, O. Smidsrod, *Carbohydr. Res.*, **89**, 179 (1981).
4. H. Grasdalen, *Carbohydr. Res.*, **118**, 255 (1983).
5. G. Skjak-Braek, H. Grasdalen, B. Larsen B, *Carbohydr. Res.*, **154**, 239 (1986).
6. B. T. Stokke, O. Smidsrod, P. Bruheim, G. Skjak-Braek, *Macromolecules*, **24**, 4637 (1991)
7. H. N. Cheng, *Polymer Bull.*, **43**, 247 (1999).
8. H. N. Cheng, "Polymerization and Statistical Models", in D. M.Grant, R. K. Harris (eds), *Encyclopedia of NMR*, Wiley, New York, 1995, pp. 3713-3721
9. H. N. Cheng, *Macromolecules*, **25**, 2351 (1992).
10. H. N. Cheng, *Macromolecules*, **30**, 4117 (1997).
11. H. N. Cheng, *J. Appl. Polym. Sci.*, **35**, 1639 (1988).
12. For example, H. N. Cheng, *Makromol. Chem. Theor. Simul.*, **1**,415 (1992), and **2**, 901 (1993).

Simplicity

The LOCATOR has replaced your spectrometer logbook

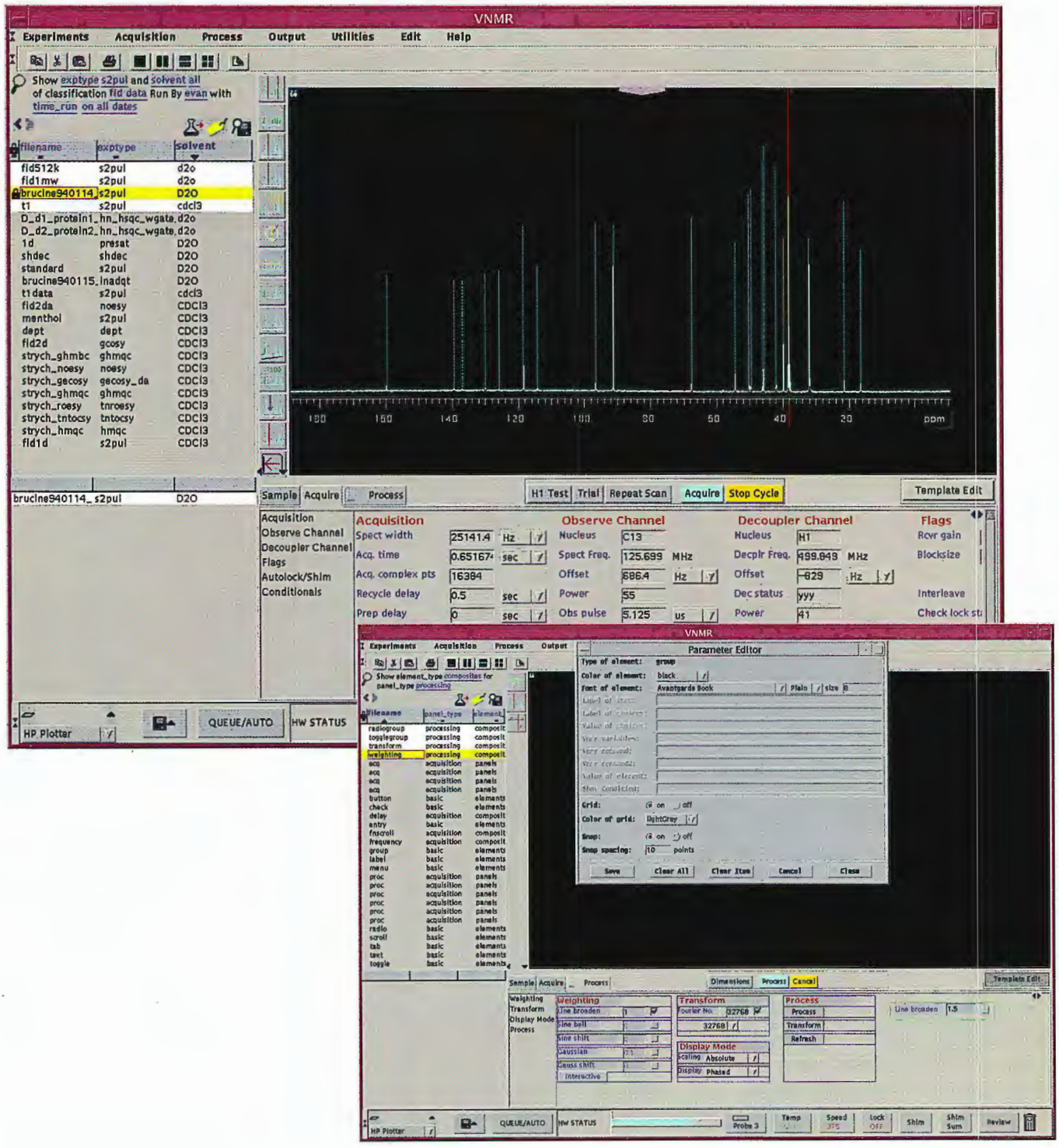
VnmrJ's new and intuitive information retrieval system, the LOCATOR, has driven the spectrometer logbook to join the antique typewriter and old fountain pen as a recording collectible.

Customized Solution
Now you have the luxury of finding your data in a way that makes sense to you. VnmrJ's LOCATOR helps you organize your data the way you want it, ready for instant access. Forget confusing file systems and complicated directory structures.

Query Capabilities
Where is that COSY you ran last Wednesday? You didn't process it, and it was run on the product of the reaction that you described on page 10/14703 of your lab notebook. You can't remember the filename you gave it, but you need it now. The LOCATOR will find it immediately.

So, find out for yourself how easy it is to simplify your workday and improve your productivity with VnmrJ's LOCATOR. For more information, please call 800-356-4437, or check out our Web site at www.varianinc.com.

VARIAN



View and select objects, based on attributes you determine with Varian's innovative VnmrJ software and its groundbreaking LOCATOR. Unlike a filter, the LOCATOR reorders the list of objects so that you can see things that are "nearly the same." Use the LOCATOR to examine collected data, protocols, pulse sequences, shim sets or any other object on disk without having to remember operating system specific incantations.

VnmrJ's LOCATOR uses a powerful SQL database manager running in the background. It invisibly combs your disk system, cataloging your files, ready to respond immediately to your changing requirements. You interact with the LOCATOR with an intuitive interface developed for your needs and written in Java based technology.

VARIAN

MAG-8828/369 11/00

WESTERN RESEARCH INSTITUTE
"Providing solutions to energy and environmental problems"
365 No. 9th St., Laramie, WY 82072-3380 • Phone: (307) 721-2011 • Fax: (307) 721-2345

January 14, 2000
(received 1/18/2000)

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

Other Uses of MAS

Dear Barry,

We were expecting your missive about our not having sent in a contribution to the NMR newsletter. We've been in somewhat of a holding pattern while we got our FHWA asphalt contract in place, so have not been doing a lot of spectroscopy. Consequently, our contribution is only marginally related to NMR, but we hope it might be of interest to the readers just the same.

The seeds of this idea were planted about 20 years ago when we were working with Gary Maciel at Colorado State University on applications of CP/MAS ^{13}C NMR to characterize fossil fuels. At that time, Vic Bartuska had built a large-volume spinner, ~14 mm in diameter, for this purpose. After Vic had used it to acquire spectra of oil shales and coals, we decided to look at tar sands. I remember one day Gary telling me how Vic had run a tar sand sample and when he took the cap off the spinner there was only sand left in the center, the bitumen having centrifuged out and coated the walls. Well 20 years later we are looking into whether we can use high-speed spinning as a way to study the adhesive properties of asphalt with aggregates.

We purchased a large-volume (14 mm dia) bench top spinning module from Varian/Chemagnetics and have been using it to test combinations of different asphalts on different aggregates. The results of one of our exploratory experiments are shown in the Figure. As you can see, a significant fraction of the asphalt can be removed by spinning, leaving the more tightly bound material coating the aggregate. In this case the glass beads. We have done similar experiments with pieces of aggregate (~1/4 inch).

This gives us an opportunity to now extract the remaining material from the asphalt to determine what classes of compound types and functional groups adhere most strongly to different aggregates. We found that the fraction of material remaining on the aggregate is different for different asphalts, but this may be just a viscosity effect as the asphalts have different viscosities at room temperature. Hopefully, we'll have more to report about these experiments in future contributions.

In the meantime we hope Y2K is a good one for the Newsletter,

Best regards,

Fran Miknis Dan Netzel

a) b) c)

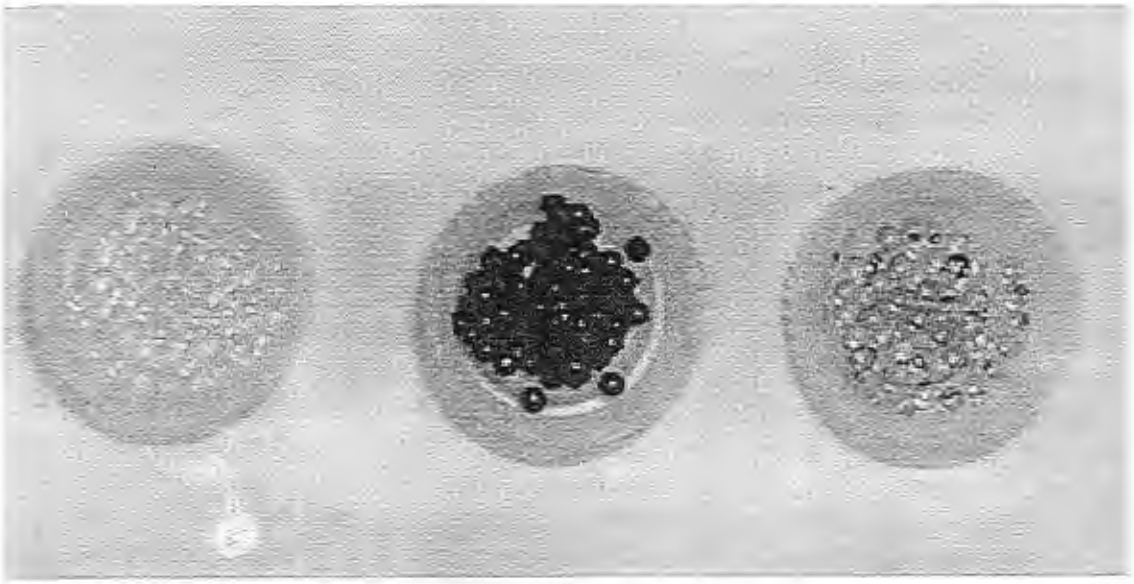


Figure 1. Asphalt coated onto 3 mm glass beads. a) glass beads, b) glass beads coated with asphalt, c) glass beads after spinning at 3 kHz for 30 min, then at 4 KHz for 60 min.

UMEÅ UNIVERSITET
Kemiska Institutionen/Organisk Kemi
Dan Johnels

UMEÅ UNIVERSITY
Dept. of Chemistry/Organic Chemistry

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

January 25, 2000
(received 1/26/2000)

^{13}C solid state NMR of polymerised C70.

Many different fullerene derivatives have been prepared since the discovery of the fullerenes in 1985[1]. These are prepared by chemical modifications, but also by subjecting the fullerenes to high temperatures and pressures[2]. The structure and properties of the materials prepared by physical methods, starting from C60, were not fully understood until the materials were investigated by spectroscopic methods, where the application of solid state NMR spectroscopy clearly revealed that the fullerene units are linked together by covalent bonds to polymeric chains or sheets[3].
We are currently investigating the structure of high pressure/temperature treated C70, where several structural possibilities exist due to the reduced symmetry. The material we have investigated has been treated at 2.5 GPa and 580 K. The solid state ^{13}C MAS NMR spectrum of this material, presented in Figure 1A, clearly show that the polymer again is formed by covalent bonds between the C70 units, as indicated from the signals at 67 and 71 ppm, stemming from sp^3 carbons. The signal at 110 ppm arises from the end caps of the rotor. The sp^2 region corresponds well to what is expected from C70 derivatives[4]. In a simulation, based on ^{13}C solution NMR data of the recently prepared C70 dimer[5], is presented in Figure 1B. There are some obvious variations of the intensities, where as the regions of the signals are similar. This allows us to assume a presence of other structural units than dimers in the sample. The working hypothesis for the structure of the polymeric materials, based on NMR and other spectroscopic data (Raman, IR), is that it is a 1D (chain) polymer.

UMEÅ UNIVERSITET, SE-901 87 UMEÅ, SWEDEN Telephfone: int+46+90-786 68 37 Telefax: int+46+90-13 88 85 E-mail: Dan.Johnels@chem.umu.se

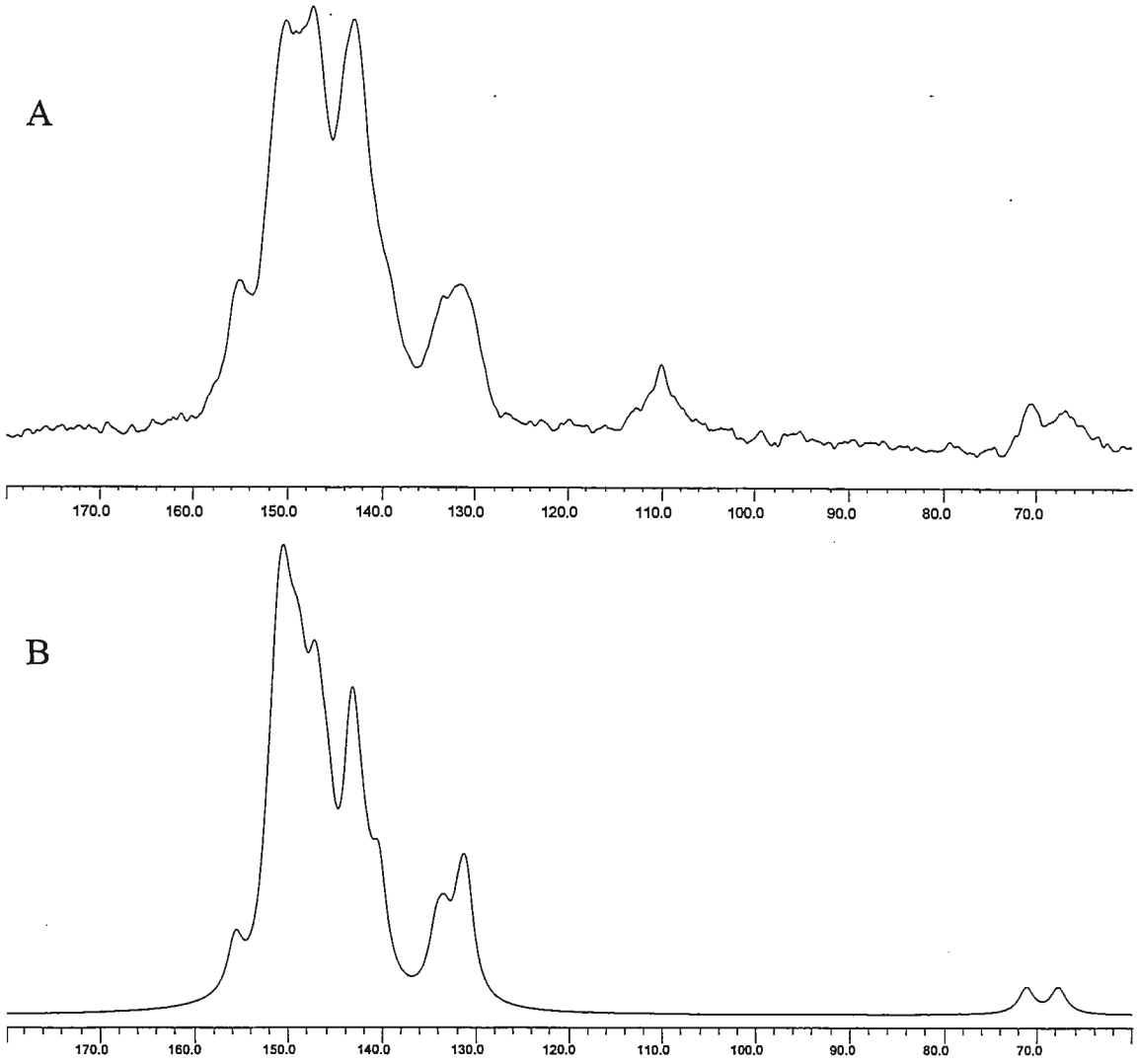


Figure 1. A. Experimental spectrum of P/T treated C70. B. Simulation based on solution NMR data of dimeric C70, a line width of 200 Hz was used.

Alexander Soldatov
Dept. of Experimental Physics
Umeå University
SE-901 87 Umeå
Sweden

Dan Johnels
Dept. of Chemistry/Organic Chemistry
Umeå University
SE-901 87 Umeå
Sweden

P.S. Please credit this contribution to the account of Prof. Ulf Edlund.

References:

1. H.W. Kroto, J.R. Heath, S.C. O'Brien, R.F. Curl and R.E. Smally, Nature, **318**, 162 (1985).
2. B. Sundqvist, Adv. Phys., **48**, 1 (1999).
3. P.-A. Persson, U. Edlund, P. Jacobsson, D. Johnels, A. Soldatov and B. Sundqvist. Chem. Phys. Lett., 258 (1996) 540-546.
4. A. Herrmann, M. R+ttimann, C. Thilgen and F. Diedrich, Helv. Chim. Acta, **78**, 1673 (1995).
5. S. Lebedkin, W.E. Hull, A. Soldatov, B. Renker and M.M. Kappeś, to be published.

Aldrich Stable Isotopes

Visit our booth at Pittcon 2000 or return the fax form below to enter our prize drawings at Pittcon.

NEW **10 x 0.6mL Ampules**

52,201-5	**Chloroform**-*d*, 99.8 atom % D
52,196-5	**Deuterium oxide**, 99.9 atom % D
52,203-1	**Dichloromethane**-d_2, 99.8 atom % D
52,202-3	**Methyl**-d_3 **alcohol**-*d*, 99.8 atom % D
52,205-8	**(Methyl sulfoxide)**-d_6, 99.9 atom % D

NEW **10g Screw-Cap Bottles**

52,209-0	**Acetone**-d_6, 99.9 atom % D, contains 0.03% v/v TMS
52,214-7	**Acetonitrile**-d_3, 99.6 atom % D
52,210-4	**Benzene**-d_6, 99.6 atom % D
52,213-9	**Methyl**-d_3 **alcohol**-*d*, 99.8 atom % D
52,212-0	**(Methyl sulfoxide)**-d_6, 99.9 atom % D

Visit our Web site at www.sigma-aldrich.com

Fax us this page to enter our prize drawings!

- Deuterated Solvents
- Isotopically Labeled Fatty Acids
- Standards for Solid State NMR
- Deuterated Buffers
- Deuterated Aliphatic Hydrocarbons
- Deuterated Products for Environmental Studies
- Books
- NMR Tubes and NMR Tube Cleaners

Fax form to Aldrich Stable Isotopes at 1-414-298-6327

Name _____

Institution _____

Department _____

Address _____

City _____

State _____ Zip Code _____

Phone ( ) _____ E-mail _____

Please indicate your areas of interest

- ☐ Deuterated Solvents
- ☐ ^{13}C, ^{15}N, or other isotopically labeled compounds
- ☐ NMR tubes and NMR tube cleaners

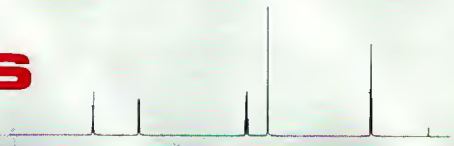
Stop by our booth at Pittcon and enter to win our contest, or return this form by fax to be included in the drawings!

Prizes Include:
Aldrich Letterman's Jacket
Sigma-Aldrich Periodic Table Umbrella
And Much More

ALDRICH® *chemists helping chemists in research and industry*

P.O. Box 355, Milwaukee, WI 53201 USA **Telephone:** 414-273-3850 • 800-558-9160 **Fax:** 414-273-4979 • 800-962-9591 **Web Site:** www.sigma-aldrich.com

Aldrich NMR Tubes



Aldrich Statement of Conformance

Every Aldrich premium-quality NMR tube has been individually inspected and gauged to meet published specifications. This statement is stamped on each box of tubes.

Premium Quality

Made of ASTM Type I thin-wall borosilicate glass* for compatibility with ground joints, valves, and vacuum racks. Precision ground and highly polished for spinning stability and highest resolution.

Economical Price

Multi-pack discount.

*Except for disposable grade NMR tubes.

NMR Tube Selection Guide

Choose the NMR tube grade that most closely matches the frequency (in MHz) of the spectrometer.

Aldrich Grade	MHz	Applications	Cross Reference No. (5mm tubes) Wilmad	Kontes
Disposable	30	Student labs, basic studies	WG-Economy	–
Series 30	30	Student labs, basic studies	505-PS	897200
Series 200	60 to 270	Lower-field studies	506-PP	897205
Series 300	60 to 360	Routine applications	507-PP	897220
Series 400	200 to 500	High-field, multiple-scan FT-NMR	528-PP	897235
Series 500	500+	Ultra-high resolution	535-PP	897240
Series 800	600 to 800	Highest resolution and performance	541-PP	897245

Grade	L (in.)	Wall Uniformity TIR (in.)†	Camber (in.)	Cat. No.	Pkg/5	20 Pkg's
3mm microsample NMR tubes						
Series 300	7	0.0020	0.0010	**Z41,290-2**	**$44.60**	**$712.10**
	8	0.0020	0.0011	**Z41,291-0**	**50.70**	**810.30**
Series 400	7	0.0010	0.0005	**Z41,292-9**	**69.50**	**1,111.40**
	8	0.0010	0.0006	**Z41,293-7**	**78.80**	**1,259.90**
Series 500	7	0.0005	0.00025	**Z41,294-5**	**113.10**	**1,808.70**
	8	0.0005	0.00035	**Z41,295-3**	**128.50**	**2,055.40**
5mm NMR tubes						
Disposable	7	–	–	**Z41,277-5**	**$12.40**	**–**
Series 30	7	0.0030	0.002	**Z41,278-3**	**28.70**	**$457.00**
Series 200	7	0.0025	0.002	**Z41,279-1**	**31.40**	**501.90**
Series 300	7	0.0020	0.0010	**Z41,280-5**	**40.50**	**646.20**
	8	0.0020	0.0011	**Z41,281-3**	**45.40**	**725.70**
	9	0.0020	0.0012	**Z41,282-1**	**50.70**	**810.30**
Series 400	7	0.0010	0.0005	**Z41,284-8**	**61.20**	**977.60**
	8	0.0010	0.0006	**Z41,285-6**	**67.20**	**1,074.80**
Series 500	7	0.0005	0.00025	**Z41,286-4**	**86.90**	**1,389.50**
	8	0.0005	0.00035	**Z41,287-2**	**97.20**	**1,553.60**
Series 800	7	0.0002	0.00015	**Z41,288-0**	**194.45**	**3,112.00**
	8	0.0002	0.00015	**Z41,289-9**	**218.00**	**3,486.70**
10mm NMR tubes						
Series 200	7	0.0030	0.0015	**Z41,296-1**	**$19.70/ea**	**$393.50/25**
Series 400	7	0.0015	0.0005	**Z41,298-8**	**27.20/ea**	**542.50/25**

†TIR=Total Indicator Reading

ALDRICH® *chemists helping chemists in research and industry*

P.O. Box 355, Milwaukee, WI 53201 USA **Telephone:** 414-273-3850 • 800-558-9160 **Fax:** 414-273-4979 • 800-962-9591 **Web Site:** www.sigma-aldrich.com

Voltronics non-magnetic trimmer capacitors:

More than 34 years of fine tuning

The first choice for NMR and MRI probes

Every NMR and MRI Test Depends on One Moveable Part!

Features

- *They're truly non-magnetic,* with magnetic field distortion less than 1 part per 600 million.
- *Lifetime is far greater* and RF power handling capability higher thanks to our non-rotating piston design.
- *Tuning is linear* - no reversals.
- *Positive stops* at minimum and maximum capacitance.
- *Extended shafts* can be specified because the tuning screw does not move in or out.

Specifications

Frequency range	to 1.5 GHz
Working Voltage	to 20 kV
Capacitance ranges	0.45 pF min. to 120 pF max.
Sizes	From 0.12 in. to 1 in. dia.
Mounting styles	All common types
Magnetic field distortion	<1 part per 600 million

Custom is Standard at Voltronics

Every NMR and MRI system has unique requirements, and we address them all. In fact we built our entire line of non-magnetic trimmers based on specific requests from our customers. We'll gladly modify an existing trimmer design or create a new one to meet the exact needs of your system.

So if you're building NMR or MRI systems, you should be talking to Voltronics. For 25 years, we've delivered the best-performing, most reliable non-magnetic trimmer capacitors available.

Call (973) 586-8585 and discuss your needs with one of our applications engineers.

Voltronics CORPORATION

The Trimmer Capacitor Company

100-10 Ford Road • Denville, NJ 07834
973.586.8585 • FAX : 973.586.3404
e-mail: info@voltronicscorp.com
Our complete catalog can be found at:
http://www.voltronicscorp.com

Universität Bern

Departement für
Chemie und Biochemie
Labor für NMR-Spektroskopie

Freiestrasse 3
CH - 3012 Bern

Tel: +41 (31) 631 39 48
Fax: +41 (31) 631 34 24
E-mail: bigler@ioc.unibe.ch

Prof. Dr. Peter Bigler

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

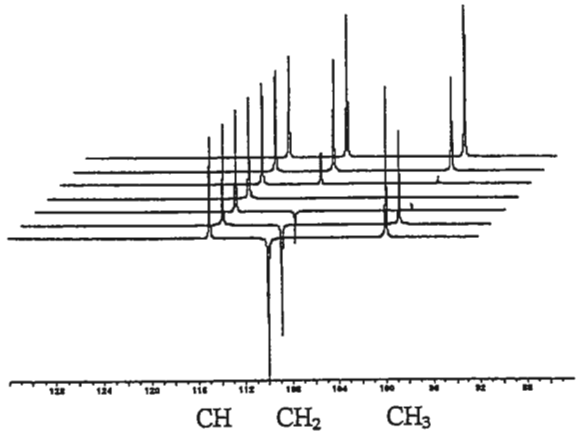
december, 14th 1999
(received 12/20/99)

DEPT: What most textbooks do not mention

Testing the ^{19}F-capabilities of our BRUKER DRX-spectrometers we experimentally compared several pulse sequences dedicated for the detection of multiplicity selective ^{13}C spectra, such as DEPT, INEPT (PENDANT) and JMOD (APT). We had to realize that the $^2J_{FF}$ coupling constants of CF_2 and CF_3 groups are close or even larger than the corresponding $^1J_{CF}$ coupling constants. So the common assumption made for the ^1H-^{13}C case ($^1J_{CH} >> ^2J_{HH}$) that homonuclear coupling can be neglected is no longer fulfilled for the ^{19}F -^{13}C case. As a consequence and with the delays adjusted solely to the CF coupling the intensities of CF_2 and CF_3 signals may heavily be distorted, may even disappear or may be wrong in sign (CF_2).
The simplified expressions as mentioned in most textbooks are no longer valid and must be replaced by the full expressions. For the DEPT experiment with ^{19}F broadband decoupling and with P0 and D2 set equal to 135° and $(2*^1J_{CF})^{-1}$ respectively they are:

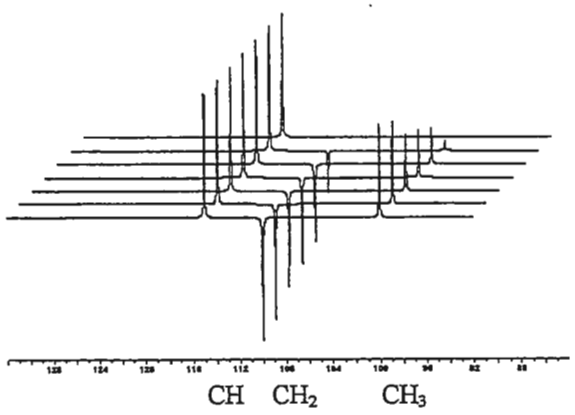
$$I_{CF3} \sim -1.06 * \sin(\Pi\, {}^1J_{CF}\, D2)^6 [\cos(\Pi\, {}^2J_{FF}\, D2)^4 - 2* \cos(\Pi\, {}^2J_{FF}\, D2)^2 * \sin(\Pi\, {}^2J_{FF}\, D2)^2 + \sin(\Pi\, {}^2J_{FF}\, D2)^4]$$
$$I_{CF2} \sim -1.00 * \sin(\Pi\, {}^1J_{CF}\, D2)^4 * \cos(2\Pi\, {}^2J_{FF}\, D2)$$
$$I_{CF} \sim -0.71 * \sin(\Pi\, {}^1J_{CF}\, D2)^2$$

According to experimental data and simulations (below) corresponding problems are most severe for DEPT and less severe for INEPT (or PENDANT). Characterizing a series of perfluorated compounds it turned out that JMOD (APT), which is obviously not affected by this "$^1J_{CF}$-$^2J_{FF}$-problem", is the best alternative for this kind of investigations although its well-known higher $^1J_{CF}$ sensitivity must be taken into account.



DEPT-135

D2=$(2*^1J_{CF})^{-1}$



INEPT

D4=$(4*^1J_{CF})^{-1}$; D5=$(3*^1J_{CF})^{-1}$

$^1J_{CF}$: 280Hz
$^2J_{FF}$: 0, 60, 120, 140, 160, 220, 280 Hz (bottom ⇨ top)

Yours sincerely
Christian Schorn and Peter Bigler

Discotic Phospholipid Particles (Bicelles) Revolutionize Structural Analysis of Macromolecules by NMR

Avanti - your partner in research

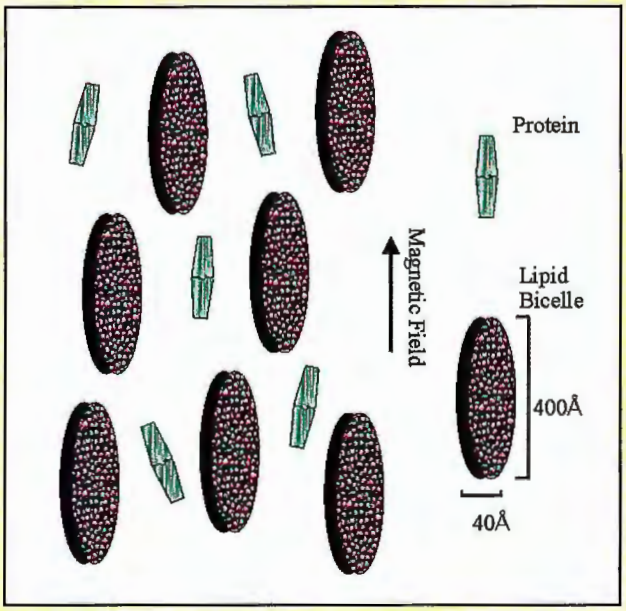
Exciting new research products for:

- Determination of water soluble protein structure
- Determination of lipid soluble protein structure
- Determination of carbohydrate structure

Available NOW

- Ester bonded lipids for bicelle preparation
- Ether bonded lipids for bicelle preparation (pH stable)
- Anionic/Cationic lipids for bicelle preparation
- Lanthanide chelating lipid
- Pre-mixed lipids for bicelle preparation

(see over for prices)



References:

- Tjandra, N; & Bax, A, (1997) Direct Measurement of Distances and Angles in Biomoleculesby NMR in a Dilute Liquid Crystalline Medium., Science, 278, 1111-3.
- Sanders CR 2d; Schwonek JP, (1992) Characterization of magnetically orientable bilayers in mixtures of dihexanoylphosphatidylcholine and dimyristoylphosphatidylcholine by solid-state NMR., Biochemistry, 31:37, 8898-905
- Losonczi JA; Prestegard JH, (1998) Improved dilute bicelle solutions for high-resolution NMR of biological macromolecules., J Biomol NMR,12:3, 447-51
- Crowell KJ; Macdonald PM, (1999) Surface charge response of the phosphatidylcholine head group in bilayered micelles from phosphorus and deuterium nuclear magnetic resonance., Biochim Biophys Acta, 1416:1-2, 21-30 dimyristoylphosphatidylcholine by solid-state NMR., Biochemistry, 31:37, 8898-905
- Ottiger M; Bax A, (1999) Bicelle-based liquid crystals for NMR-measurement of dipolar couplings at acidic and basic pH values., J Biomol NMR, 13:2, 187-91
- Cavagnero S; Dyson HJ; Wright PE, (1999) Improved low pH bicelle system for orienting macromolecules over a wide temperature range., J Biomol NMR, 13:4, 387-91
- Struppe J; Komives EA; Taylor SS; Vold RR, (1998) 2H NMR studies of a myristoylated peptide in neutral and acidic phospholipid bicelles., Biochemistry, 37:44, 15523-7
- Prosser RS; Volkov VB; and Shiyanovskaya IV, (1998) Novel Chelate-Induced Magnetic Alignment of Biological Membranes. Biophys J. 75: 2163-2169

Avanti®
Polar Lipids, Inc.

E-mail avanti@quicklink.net or visit our Web site www.avantilipids.com

For information about Avanti please contact our Product Hotline: 800-227-0651 (USA & Canada) or 205-663-2494 International

PRICE LIST

Lipids for Bicelle Formation

Product	M.W.	Quantity/Price				Catalog No.
		25mg	200mg	500mg	1 gram	
Acyl Zwitterionic Lipids						
DHPC*	453.51	$21	$40	$75	$120	850305
DMPC	677.94	$10	$20	$40	$60	850345
Acyl Anionic Lipids						
DMPG•Na	688.85	$20	$70	$135	$200	840445
DMPS•Na	701.85	$40	$265	$425	$675	830033
Acyl Cationic Lipid						
DMTAP•Cl	590.37	$40	$110	$215	$362	890860
Ether Zwitterionic Lipids						
6-0-PC	425.54	$83	$280	$560	$900	999998
12-0-PC	593.86	$83	$280	$560	$900	999994
13-0-PC	621.92	$120	$360	$720	$1,200	790579
14-0-PC	649.97	$83	$280	$560	$900	999993
Ether Anionic Lipid						
14-0-PG•Na	660.89	$160	$560	$1,120	$1,800	999800
Ether Cationic Lipid						
14-0-Ethyl-DMPC•Cl	742.46	$65	$235	$450	$750	890701
DDAB•Br	630.96	$28.50	$71.50	$115.50	$192.50	890810
Lanthanide Chelating Lipid		1mg	5mg	10mg	25mg	
DMPE-DTPA•$(NH_4)_5$	1096.35	$10	$45	$80	$175	790535

*DHPC IS EXTREMELY HYGROSCOPIC; PREPARE SOLUTIONS IN DRY BOX OR DILUTE WITH BUFFER IMMEDIATELY AFTER OPENING.

DEUTERATED LIPIDS ARE ALSO AVAILABLE. PLEASE INQUIRE.

Premixed Lipids for Bicelle Formation

DMPC:DHPC Ratio (q)	50mg units			200mg units			Catalog No.
	1	5x	10x	1	5x	10x	
2.8	$ 50	$ 195	$ 325	$ 150	$ 625	$ 1,000	790590
3.0							790573
3.25							790574
3.5							790575

Many more products available from stock. Please call our product Hot-line 800-227-0651 (Dept. 109) for more information or to place an order.

Avanti® POLAR LIPIDS, INC.

A d v a n c e d C h e m i s t r y D e v e l o p m e n t

133 Richmond St., W., Suite 605, Toronto, Ontario Canada, M5H 2L3 T: 416-368-3435 F: 416-368-5596
www.acdlabs.com info@acdlabs.com

Offline Processing of Bruker Digitally Filtered NMR Data

January 16th 2000
(received 1/24/2000)

Dear Barry,

The benefits of the oversampling procedure applied to NMR data is well known and certainly one of the most valuable aspects is the increase of the effective dynamic range of a spectrum. However, this requires collecting a FID with a great number of points to provide sufficient digital resolution. The *large* FID is digitally filtered to remove the signal outside the spectral region of interest and decimated to the desired spectral width, the whole procedure taking place on-the-fly by means of high-speed digital signal processors [1]. Unfortunately the procedure may have its own drawbacks. Commercial digital filters are proprietary, so one does not really know what has been done to the data, and some resultant processed spectra can assume a strange form after digital filtering. This seems to be the case with digital filtering employed on Bruker Avance spectrometers (Figure 1).

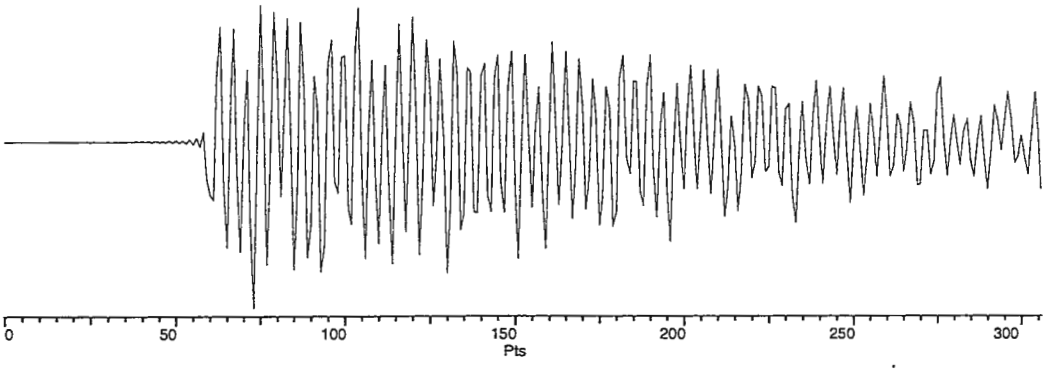
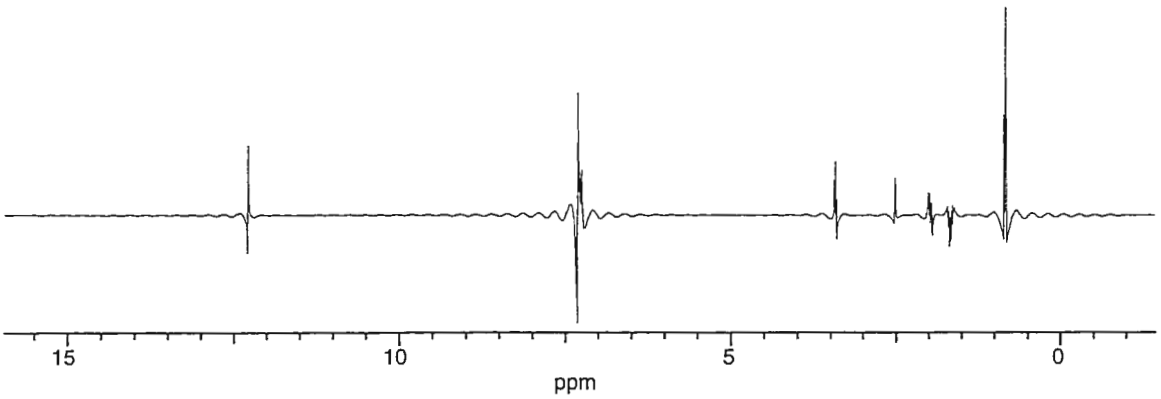


Figure 1. Beginning of typical digitally processed FID obtained on a Bruker DMX spectrometer.

Shown on the Figure 1 is the beginning of a typical digitally processed FID obtained on a Bruker DMX spectrometer. A rising signal starting from zero at the first data point and achieving a maximum at approximately the 60-th point is undoubtedly an artifact of digital filtering. The FID is correctly processed on XWINNMR, a component of the Bruker software suite but standard Fourier transformation leads to a spectrum with wiggles as shown below (Figure 2).



Advanced Chemistry Development

133 Richmond St., W., Suite 605, Toronto, Ontario Canada, M5H 2L3 T: 416-368-3435 F: 416-368-5596
www.acdlabs.com info@acdlabs.com

Figure 2. ^{1}H NMR spectrum of phenylbutyric acid in DMSO (DMX-400) obtained by straightforward Fourier transformation of digitally processed FID.

The observation that the spectrum shown in Figure 2. can be made to resemble a *normal* spectrum by applying several thousand degrees of linear phase correction gave a key to correct processing of such data. As was shown by W. M. Westler and F. Abildgaard [2] an appropriate number of initial points of the FID should be circularly left shifted before Fourier transformation. The resulting spectrum is almost identical to that processed by XWINNMR (Figure 3A). It is important to perform the circular left shifting after zero-filling and/or apodization to keep the flat baseline. Shown in Figure 3B is the spectrum obtained when exponential multiplication (with LB = 1 Hz) was applied after the circular left shift. Retaining the non-zero points at the end of FID is however a very cumbersome business. Besides this can interact in an unexpected way with some offline processing steps. For example the spectrum shown in Figure 3B would result from the spectrum shown in Figure 3A by inverse Fourier transformation followed by exponential multiplication (with LB = 1 Hz).

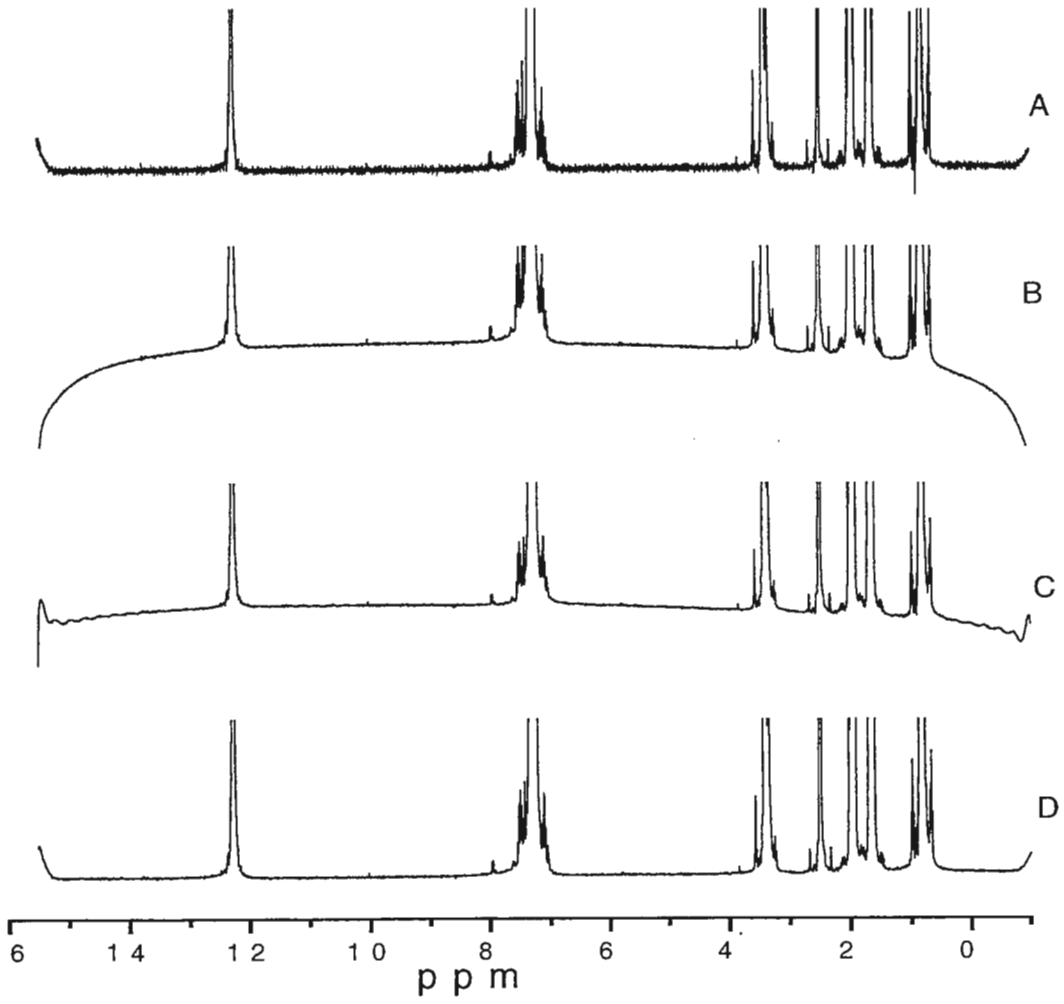


Figure 3. ^{1}H NMR spectrum of phenylbutyric acid in DMSO (DMX-400). A - Fourier transformed by XWINMR, B - exponential multiplication (with LB = 1 Hz) was done after circular left shift of initial points, C- exponential multiplication was done after 'digital to analog' conversion by XWINNMR, D - exponential multiplication was done after digital FID conversion by our technique

Our challenge was to think of a conversion of some kind that would allow us to treat afterwards digitally filtered data like 'normal' data. Bruker's own conversion routine "convdta" is applicable only to acquisition data [3] and sometimes significantly distorts baseline (Figure 3C) and appears to be unsuitable.

A d v a n c e d C h e m i s t r y D e v e l o p m e n t

133 Richmond St., W., Suite 605, Toronto, Ontario Canada, M5H 2L3 T: 416-368-3435 F: 416-368-5596
www.acdlabs.com info@acdlabs.com

We would like to describe our empirical treatment that was found to eliminate all resulting nuisances generated by digital filtering. The basic steps for the digitally filtered FID are the following:

1) zero-filling,
2) circular left shift of appropriate number of points,
3) Fourier transformation,
4) phasing
5) extracting real part and its inverse Fourier transformation,
6) truncating the obtained FID

The resultant FID can be processed further without any complications. The first step of the procedure is necessary to keep the number of spectral points while step 2 is that proposed by W. M. Westler and F. Abildgaard [2].

Shown in Figure 3D is the Fourier transformed result of exponential multiplication (with LB = 1 Hz) of such "converted" data. Please note that a flat baseline is retained as displayed in Figure 3A. The quality of the baseline after this conversion is dependent on phasing quality. The best baseline is achieved when the purely adsorptive part is taken as real (Figure 3D) and the worst one - when the purely dispersive part is taken as real (result is very similar to Figure 3B). The protocol for conversion of Bruker-processed 1R files is very similar and has the advantage that the result does not depend on the effectiveness of the phasing routine.

More and more institutions and laboratories are utilizing third party tools for access and processing of NMR data. We believe that a thorough examination of the quality of data generated away from the spectrometer using these tools is necessary to take into account different approaches taken by the vendors.

Yours sincerely,

C. Golotvin

Sergey Golotvin
Advanced Chemistry Development

References:

1. Hoch J.C. and Stern A.S.: NMR data processing, p. 196, New-York, 1996, Wiley-Liss.
2. W. M. Westler and F. Abildgaard, DMX Digital Filters and Non-Bruker Offline Processing II, private communication, 1995.
3. XWINNMR Software Manual, Bruker Analytische Messetechnik GmbH, p131, 1995

Research Group for Antibiotics and NMR Laboratory
Department of Chemistry
University of Debrecen, Egyetem tér 1
H-4010 Debrecen P. O. Box 70, HUNGARY
e-mail: batta@tigris.klte.hu, fax: (36) 52 512 914
tel: (36) 52 512 900 ext. 2370 or 2234

January 10, 2000 (received 1/11/2000)

Professor B. L. Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto
CA 94303-3410
USA

Renewable Resources

Dear Barry,

Since the first deployement of a supercon (200) NMR in Hungary in 1980 we collect the evaporating He gas at low pressure in a 1 m^3 bell floating on an oil bath and then we regularly compress the gas in cylinders. Now, we have a 360 and a 500 magnet connected to the same recovery system. It turned out, that the 500 is sensitive to both the change in atmospheric pressure (the wheather in Central Europe is often changeable...) and the recovery process (lock level is unstable). So, when sensitive measurements were carried out we did not use the recovery system. Now, we installed an electronic absolute pressure stabilizer for the magnet He cryostat. The excess gas is fed to the same bell. The weight of the bell is a cause of an extra 20 mbar pressure in addition to the atmospheric pressure. However, the total excess pressure over the atmospheric pressure is generally less than 40 mbar, which is less than the third of the total excess pressure during a regular helium refill procedure, which shouldn't harm the magnet. The investment costs USD 2000,- and we enjoy a more stable instrument and better milage.

Yours truly,

Gyula Batta

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. 498 (Mar.) 25 Feb. 2000

No. 499 (Apr.) 24 Mar. 2000

No. 500 (May) 28 Apr. 2000

No. 501 (June) 24 May 2000

No. 502 (July) 21 June 2000

* 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

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 **red dot**: this decoration means that you will not be mailed any more issues until a technical contribution has been received.

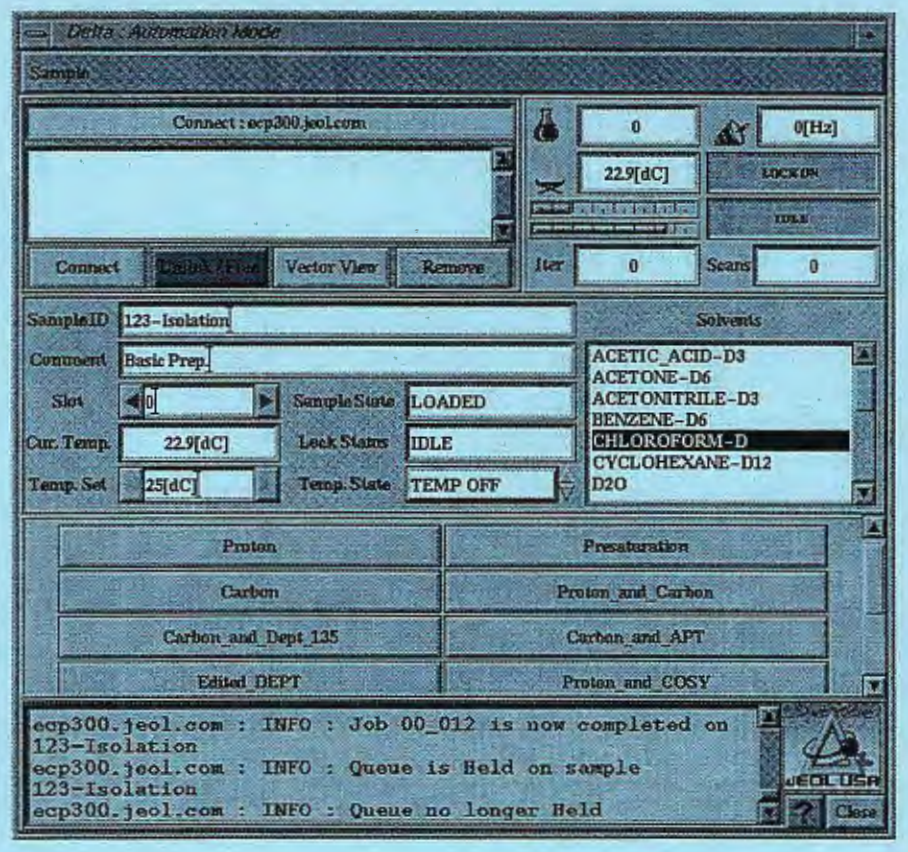
Forthcoming NMR Meetings, continued from page 1:

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.

Royal Society of Chemistry: 15th International Meeting on NMR Spectroscopy, Durham, England, **week of July 8-13, 2001**; Contact: Mrs. Paula Whelan, The Royal Society of Chemistry, Burlington House, London W1V 0BN, England; +44 0171 440 3316; Email: conferences@rsc.org\

Additional listings of meetings, etc., are invited.

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



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

JEOL