THE
NMR
NEWSLETTER

No. 515

August 2001

PTS FREQUENCY SYNTHESIZERS

FREQUENCY GENERATORS, AGILE, QUIET, FAST

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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.

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FORTHCOMING NMR MEETINGS

ISMAR 2001, *Note change of meeting location:* Convention Center of Rodos Palace Hotel in Rhodes, Greece. **August 19-24, 2001**; See http://www.tau.ac.il/chemistry/ISMAR.html.

Sixth International Conference on Magnetic Resonance Microscopy, Nottingham, UK, **September 2-5, 2001**. http://www.magres.nottingham.ac.uk/conferences/2001/icmrm.

14th European Symposium on Polymer Spectroscopy, Dreikönigskirche – Haus der Kirche, Dresden, Germany, **September 2-5, 2001**. Contact: Institut für Polymerforschung Dresden c. 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: Dr. Andreas Kungl, Austrian Chemical Society (GÖCH), Biochemistry Subgroup, c/0 Institute of Pharmaceutical Chemistry, Univeristy of Graz, Universitätsplatz 1, A-8010 Graz, Austria. Tel: +43 316 380 5373; Faz: +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://www.ens-lyon.fr/STIM/alpineweb/html.

EMBO Practical Course: Structure Determination of Biological Macromolecules by Solution NMR, EMBL, Meyerhofstr. l, D-69117 Heidelberg, Germany, **September 12-19, 2001**; Email: nilges@EMBL-Heidelberg.de; sattler@EMBL-Heidelberg.de; http://www.embl-heidelberg.de/nmr/sattler/embo.

EMBO Workshop on NMR and Molecular Recognition, Ravello, Italy, **October 3-7, 2001**; Contact: Dr. T. Tancredi: ttancredi@icmib.na.cnr.it, or Dr. P. Amodeo: pamodeo@icmib.na.cnr.it. Information: http://www3.icmib.na.cnr.it/ravello2001.

43rd ENC (Experimental NMR Conference), Asilomar Conference Center, Pacific Grove, CA, **April 14-19, 2002.** Contact: ENC: 505-989-4573; 505-989-1073 fax; enc@enc-conference.org; http://www.enc-conference.org; See Newsletter 515, 26.

XXth International Conference on Magnetic Resonance in Biological Systems, Toronto, Ont., **August 25-30, 2002**. For further information check www.uso.ca/chem/icmrbs/, or contact: mgordon@julian.uso.ca.

Additional listings of meetings, etc., are invited.

THE NMR NEWSLETTER

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Notice.

After 43 years of consecutive monthly publication, the time has come to close down *The NMR Newsletter*. Accordingly, the September 2001 issue will be the last.

I must admit that I'd like to see a good full issue for the Newsletter's swan song, and I hope that several of our loyal readers will avail themselves of this final chance to tell colleagues of their latest research efforts via the Newsletter pages. The deadline date for receipt of material for issue No. 516 is August 27. As always, the nature of the contributions is wide open; whatever you wish, including graphics of any kind (including cartoons).

Barry Shapiro
Palo Alto, California
July 28, 2001.

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
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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.

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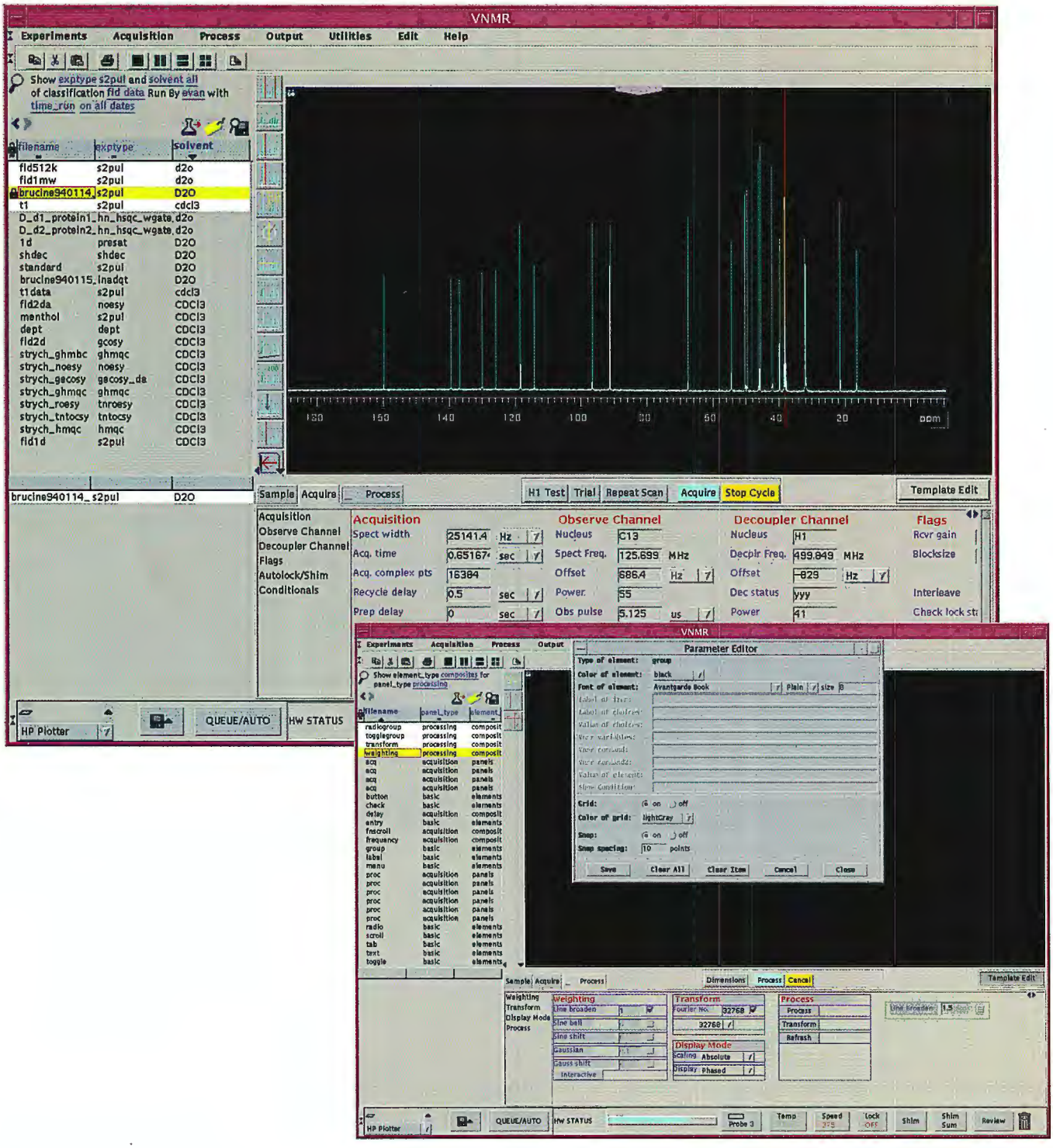

View and select objects, based on attributes you determine with Varian's innovative VnmrJ software and its ground-breaking LOCATOR. Unlike a filter, the LOCATOR reorders the list of objects so that you can see things that are "nearly the same." Use the LOCATOR to examine collected data, protocols, pulse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

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

Phase Inversion at Null Excitation

June 25, 2001
(received 6/26/2001)

Dear Dr. Shapiro,

In the excitation profile by the composite pulse $90^\circ_x 90^\circ_y 90^\circ_{-x} 90^\circ_{-y}$ (1), phase inversion occurs at each null (λ_1 to λ_4, in the range of $-2 \le \lambda \le 2$) as shown in Fig. 1. All the nulls correspond to overall rotations of $\Phi = 2j\pi$ ($j = 0, \pm 1, \pm 2, \ldots$) around certain axes. As a result, the initial magnetization returns to its original position along the z-axis and outputs a null. The phase inversion associated with the null can be revealed by the following analysis.

Assume a differential of the offset $d\Delta$ occurs at a null Δ_k, which introduces differentials of rotation angle $d\Phi$ and axis dN. The unitary rotation operator by the composite pulse can be expressed as

$$U = e^{-i\eta(N+dN)\cdot\sigma[(\Phi+d\Phi)/2]} = e^{-i\eta(N+dN)\cdot\sigma(d\Phi/2)} e^{-i\eta(N+dN)\cdot\sigma(\Phi/2)}$$
$$= e^{-i\eta(N+dN)\cdot\sigma(d\Phi/2)} \approx e^{-iN\cdot\sigma(d\Phi/2)} , \qquad [1]$$

where $\eta = 1/\sqrt{(N+dN)\cdot(N+dN)}$ is a normalization factor. In Eq. [1], the operator $e^{-i\eta(N+dN)\cdot\sigma(\Phi/2)}$, which corresponds to a $2j\pi$ rotation, is dropped and the high order term including $dNd\Phi$ is dropped in the last step. The excitation near null is then determined by an infinitesimal rotation,

$$\sigma = e^{-iN\cdot\sigma(d\Phi/2)} \sigma_z e^{iN\cdot\sigma(d\Phi/2)} \approx \sigma_z + i(d\Phi/2)[\sigma_z, N\cdot\sigma]$$
$$= \sigma_z + d\Phi(N_y\sigma_x - N_x\sigma_y) , \qquad [2]$$

In Eq. [2], the overall rotation axis $N \neq N_z$ is assumed, which is, however, not necessary in a more elaborate calculation if the rotation angle $\Phi \neq 2j\pi$.

The transverse magnetizations can be derived by,

$$M_x(\Delta_k + d\Delta) \propto Tr\{\sigma_x \sigma\} \propto N_y d\Phi = N_y \left.\frac{d\Phi}{d\Delta}\right|_{\Delta_k} d\Delta \quad , \qquad [3a]$$

$$M_y(\Delta_k + d\Delta) \propto Tr\{\sigma_x \sigma\} \propto -N_x d\Phi = -N_x \left.\frac{d\Phi}{d\Delta}\right|_{\Delta_k} d\Delta . \qquad [3b]$$

When $d\Delta$ varies from a small negative to a small positive both M_x and M_y change sign if $(d\Phi / d\Delta)|_{\Delta_k} \neq 0$. Consequently, a phase inversion occurs at null since the phase is defined as the angle between the vector of the magnetization, $M_x + iM_y$, and the x-axis in the rotating frame.

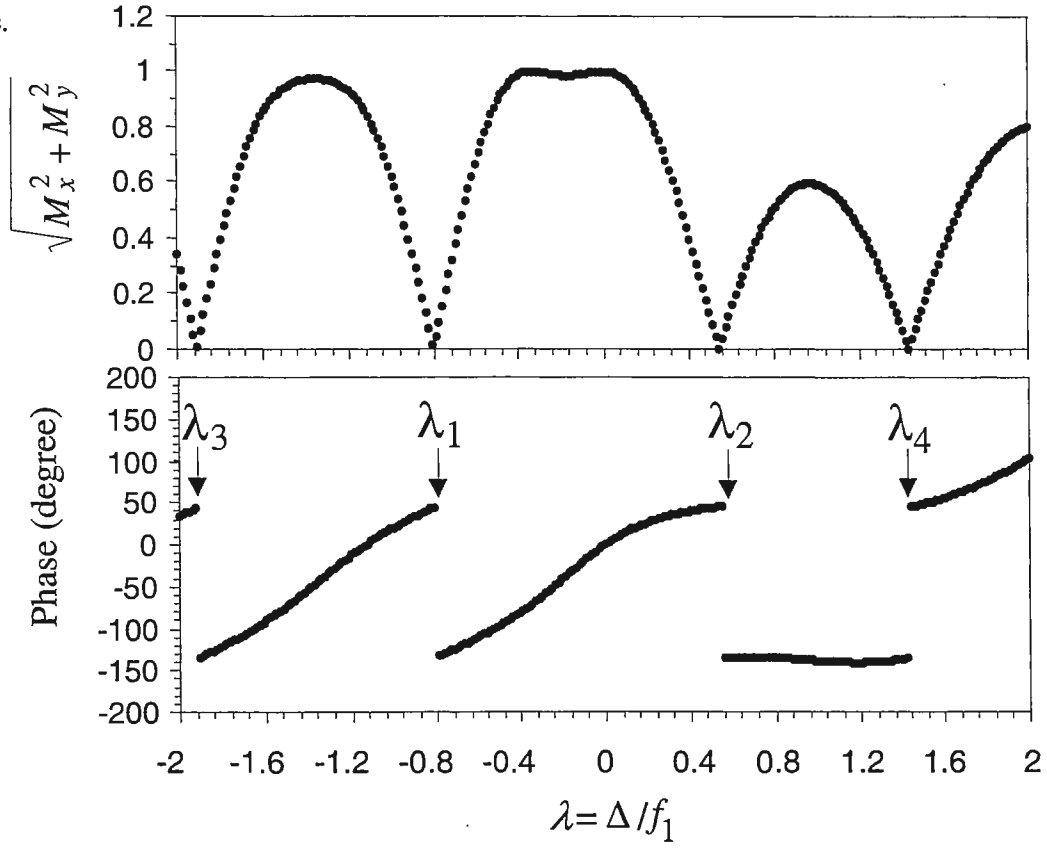


Fig. 1. The excitation profile by the composite pulse $90^\circ_x 90^\circ_y 90^\circ_{-x} 90^\circ_{-y}$(top) and the corresponding phase (bottom). A phase inversion, denoted by λ_1 to λ_4, occurs at each null.

Best regards,

Shanmin Zhang

David. G. Gorenstein

Reference:

1. A. Bax, *J. Magn. Reson.* **65**, 142 (1985).

800 US² : UltraShield™ and UltraStabilized™

… the new 800 MHz NMR siting solution

Overview

The 800 US²™ combines Bruker's innovative magnet technologies: UltraShield™ and UltraStabilized™. This new magnet system guarantees the ultimate performance demonstrated by its predecessors, including:

- Excellent field homogeneity and stability
- Minimal evaporation rates for sub-cooled magnets
- Small stray fields, and
- Efficient screening against external field perturbations

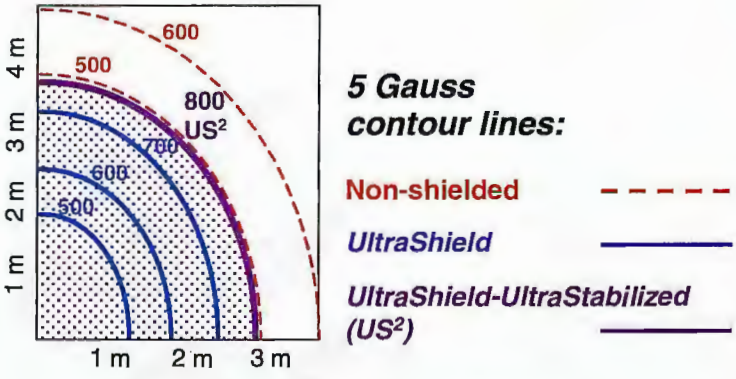
AVANCE 800 US² complete NMR system in operation at Bruker's application laboratory

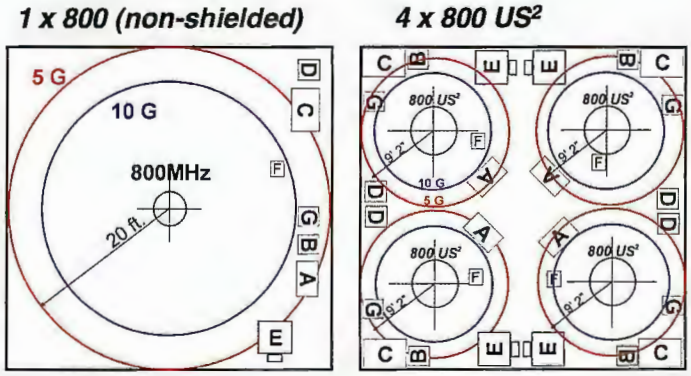
The Siting Solution

The 800 US² will reduce siting costs and provide more flexibility for future upgrades by reducing stray fields. This will enable customers to:

- **Utilize smaller labs** - the 800 US² will actually fit in a smaller footprint than a non-shielded 500.

- **Avoid the impact of stray fields** above and below the magnet.





- **Site multiple systems in the same lab** - up to four (4) Avance 800 US² can fit in the same space allocated to one 800 non-shielded system.

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TECHNICAL SPECIFICATIONS

Superconducting Magnet

NMR frequency (^{1}H)	800 MHz
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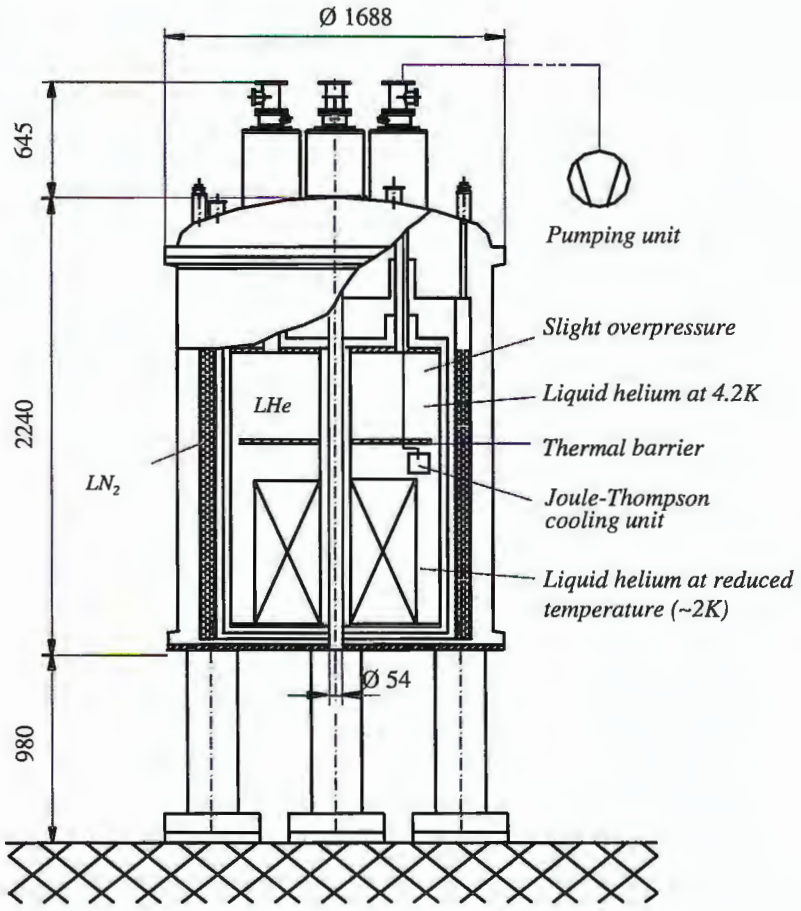
Field stability	< 8 Hz/hr
Supercondcuting shim coils	z, x, y, z^2, zx, zy, xy, x^2-y^2
5G Line from the magnetic center - radial distance - axial distance	 < 2.8 m < 3.9 m

Cryostat

Dewar type	UltraStabilized, sub-cooled
Room temperature bore	54 mm
Cryostat height (with magnet stand)	3865 mm
Cryostat diameter	1688 mm
He evaporation rate He refill volume Helium hold time	< 250 ml/hr ~ 350 liters > 60 days
N_2 evaporation rate N_2 refill volume N_2 hold time	< 700 ml/hr ~ 400 liters > 21 days
Magnet stand	included
Vibration dampers	included
Weight (including cryogens)	~ 7000 kg
Minimum ceiling height	< 5300 mm

800 MHz / 54 mm UltraShield-UltraStabilized (US$^{2™}$) NMR Magnet System



Dimensions in millimeters unless stated otherwise

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Date: July 10, 2001
(received 7/10/2001)

Prof. B.L. Shapiro
The NMR Newsletter,
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Artifacts caused by automatic DC correction

Dear Barry,

as we all know, NMR processing software provides a wide variety of parameters and options for the correction of experimental artifacts and for cosmetic operations on the NMR spectrum. Some of them has to applied explicitly by separate commands, however, there are some that are invoked automatically and might cause surprising effects.

A problem that sometimes happens to beginners is associated with the correction of the DC offset. The latter is imposed on the signal by the receiver electronics and shows up as a feature at the center of the frequency spectrum ("transmitter spike", "center glitch"). The normally applied CYCLOPS phase cycle is supposed to take care of the suppression of this feature by proper adding/subtracting signals with inverted pulse phases, however, in a number of occasion the artifact shows up nevertheless. As an example, we show a static solid-state spectrum of the synthetic polymer Polybutylmethacrylate. Fig. (a) displays the spectral expansion covering the overlapping powder patterns of the aliphatic lines between 0 and 100ppm and the carboxylic resonance between 110 and 270ppm. Since the latter is free from spectral overlap, the tensor elements of the chemical-shift tensor can easily be extracted from it.

Closer inspection reveals that there is a annoying spectral feature at 140ppm, see Fig (b). This frequency appears to be the center of the spectrum and thus, the artifact is identified as the "transmitter spike". The question came up why after completion of many CYCLOPS cycles, the latter did not took care of it. We identified the automatic correction of the DC offset by the NMR software as the source of the problem. The programmers might not trust the CYCLOPS (or had other good reasons) and thus the software performs a subtraction of the time-domain DC from the time-domain signal prior to each FT. It is done by assuming that the last points in the fid are just noise and the average over a couple of them is supposed to be the DC level of the signal which is subsequently subtracted from each fid data point. Now, if the acquisition time was by mistake chosen a bit too short such that the fid was cut off, this procedure leads to

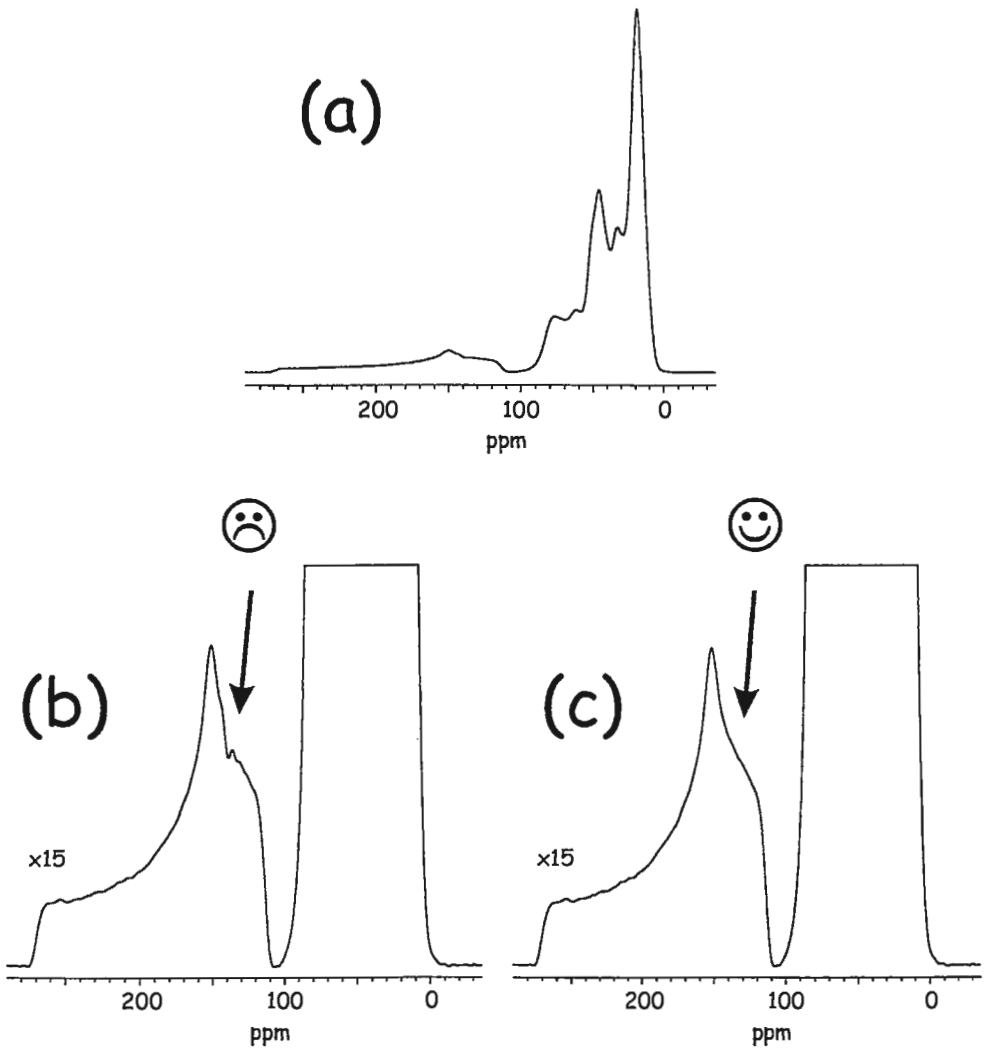
an erroneous DC level that is imposed in the fid and the "transmitter spike" reappears in the spectrum. An easy solution is to switch off the DC correction prior to FT which in our case could be done by providing an additional argument for the FT command, see Fig. (c).

This example might serve to demonstrate that the automatic and hidden execution of data manipulations by software not only hampers the insight into the meaning and working of this operations but also might cause faulty results.

Please credit this contribution to Horst Schneider.

Best Regards,

Detlef Reichert

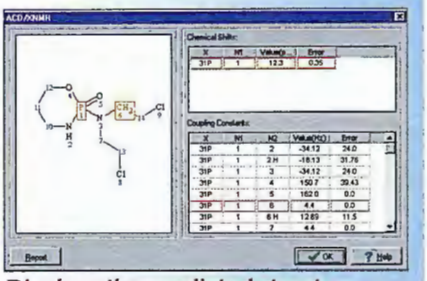


NMR IR UV-VIS MS Raman

ACD/PNMR Predictor & DB

^{31}P NMR Spectra

Look up structures and predict spectra from the same interface!



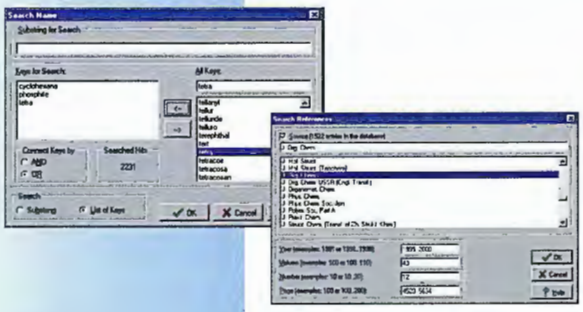
Displays the predicted structure, interactively related to assigned shifts & coupling constants

Save time and resources: predict results with confidence limits or use an excellent database source to search for compounds containing the same structural backbone or functional groups.

ACD/PNMR is the most complete package available today for scientists working in the areas of agrochemicals, surfactants or other areas of chemistry requiring the application of ^{31}P NMR.

Each entry in the ACD/PNMR database includes original literature references, molecular formula, molecular weight, assigned peaks and IUPAC name. You can search on any of these fields. Search capabilities also include structure and substructure, and searching by exact value or range of values for chemical shifts and coupling constants.

- **NEW**- The substructure search has been improved and significantly speeded up.
- **NEW**- Search for several structure fragments simultaneously.
- **NEW**- Output your predicted results, or print a database record in PDF format.
- **NEW**- The internal ACD/PNMR database contains more entries (over 19,000 records) and represents more classes of structure types than previously. This means over 24,000 chemical shifts and 9,000 coupling constants. Over 100 entries are from references published in 2000.
- **NEW**- Tile View of database entries can be done after a search, thereby assisting comparison and summary. The displayed data fields, the font, and the zoom level can be chosen after right-clicking to access the pop-up menu.

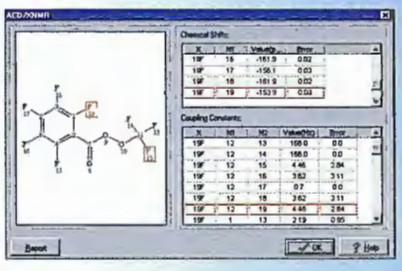


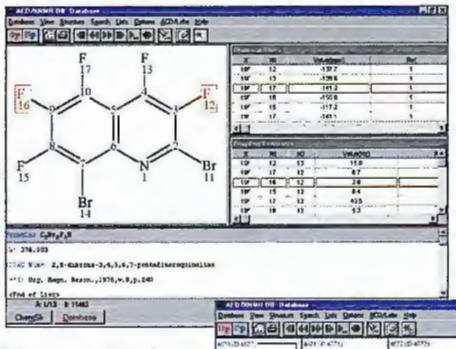
Phosphorus database window summarizes, and can be searched by, chemical shifts, coupling constants, references, formula and IUPAC name

ACD/FNMR Predictor & DB

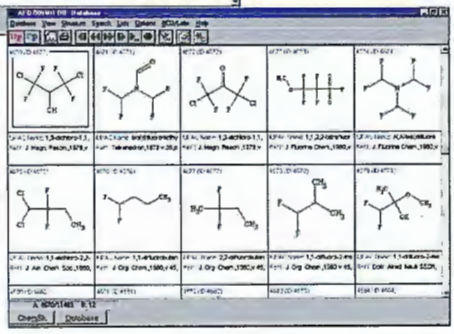
^{19}F NMR Spectra

If it's not in the database, our prediction is the next best thing.





Fluorine database window showing chemical shifts, coupling constants & references

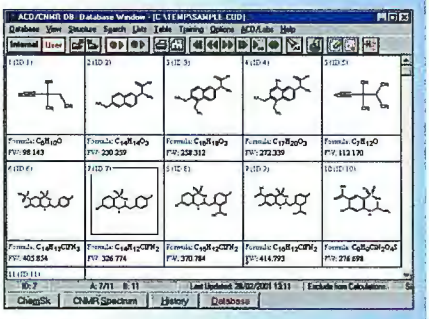


ALSO AVAILABLE! ACD/FNMR contains the same interface, with full search and prediction capabilities and the latest improvements for speed and ease of use. Its database comprises ^{19}F NMR spectra for over 11,400 structures with over 25,200 chemical shifts and 15,300 coupling constants.

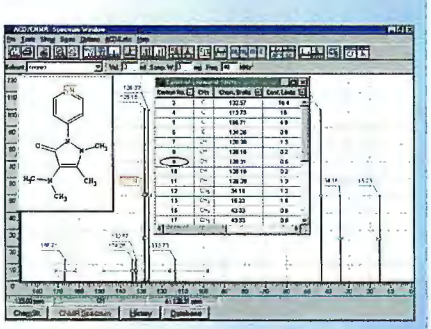
ACD/CNMR Predictor

^{13}C NMR Spectra

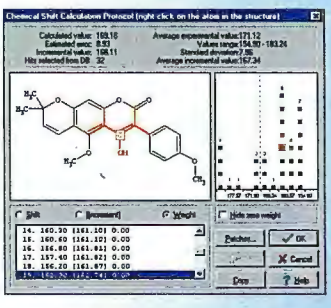
Train the system with your own data and don't be limited by a restricted set!



Tile view shows multiple hits from database search



Verify shifts with on-screen structural relation



Protocol window explains the calculation procedure

Draw a molecule and predict the ^{13}C spectrum at the touch of a button.

ACD/CNMR enables you to:

- Calculate spin-spin interactions, simulate off-resonance, DEPT, and J-modulation.
- Show predicted chemical shifts as a table or on the chemical structure.
- **NEW**- The accuracy of prediction is even greater. The DAT file has been expanded by over 20% and now contains over 1,500,000 experimental chemical shifts.
- Algorithm includes cases such as diastereotopic carbon atoms.
- Modify or delete shifts in the internal DAT file.
- **NEW**- File Associations for CNMR-specific file extensions can be set through a menu command.

Database Capability

- Create your own database of structures and observed chemical shifts and coupling constants.
- Enter ^{13}C-X coupling constants, where X = ^{19}F, ^{31}P, etc.
- Enter unassigned shifts or automatically transfer the peak table into the CNMR database from ACD/NMR Manager.
- Self-training system will access user-defined databases for improved prediction accuracy of compounds important to your lab.
- Export database entries as printed reports, PDF file, or JCAMP.
- Data Forms Manager streamlines and standardizes record entries as you build your own databases.
- Include multiple-user databases in system training.
- Search database by chemical shift, coupling constant, structure and substructure, formula, MW, or any one of the data fields you specify when designing your own database.
- **NEW**- Several entries in the database can be seen in a single window, with the new Tile View feature.

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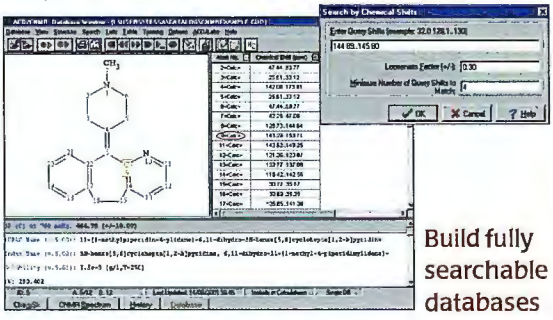

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Leonard D. Spicer, Director
Anthony A. Ribeiro, Manager

919 684 4327
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Dr. B.L.Shapiro
The NMR Newsletter
966 Elsinore Court
Palo Alto, CA 94303

July 23, 2001
(received 7/26/2001)

Re: Calibration of ^{195}Pt Chemical Shift Scale on a Unity 500 Spectrometer

DearBarry,

For a collaborative project on platinum complexes with Mark Grinstaff (Duke, Chemistry), we recently calibrated the ^{195}Pt chemical shift scale on our Unity 500 spectrometer. From the sensitivity viewpoint, recording of ^{195}Pt NMR spectra (^{195}Pt nucleus: spin 1/2, 33.7% natural abundance, relative sensitivity 9.94 $x10^{-3}$) is similar to recording of ^{13}C NMR spectra (^{13}C nucleus: spin 1/2, 1.1% natural abundance, relative sensitivity 1.59 $x10^{-2}$) (1). However, while ^{13}C NMR shifts span a 220 ppm window, ^{195}Pt NMR shifts span a 5500 ppm range (1), which is about 0.59 MHz on a 500 MHz spectrometer. Since the Unity console has a maximum spectral window of 100 kHz, the platinum NMR spectra were recorded in small spectral windows while systematically tracking spectral frequencies and offsets.

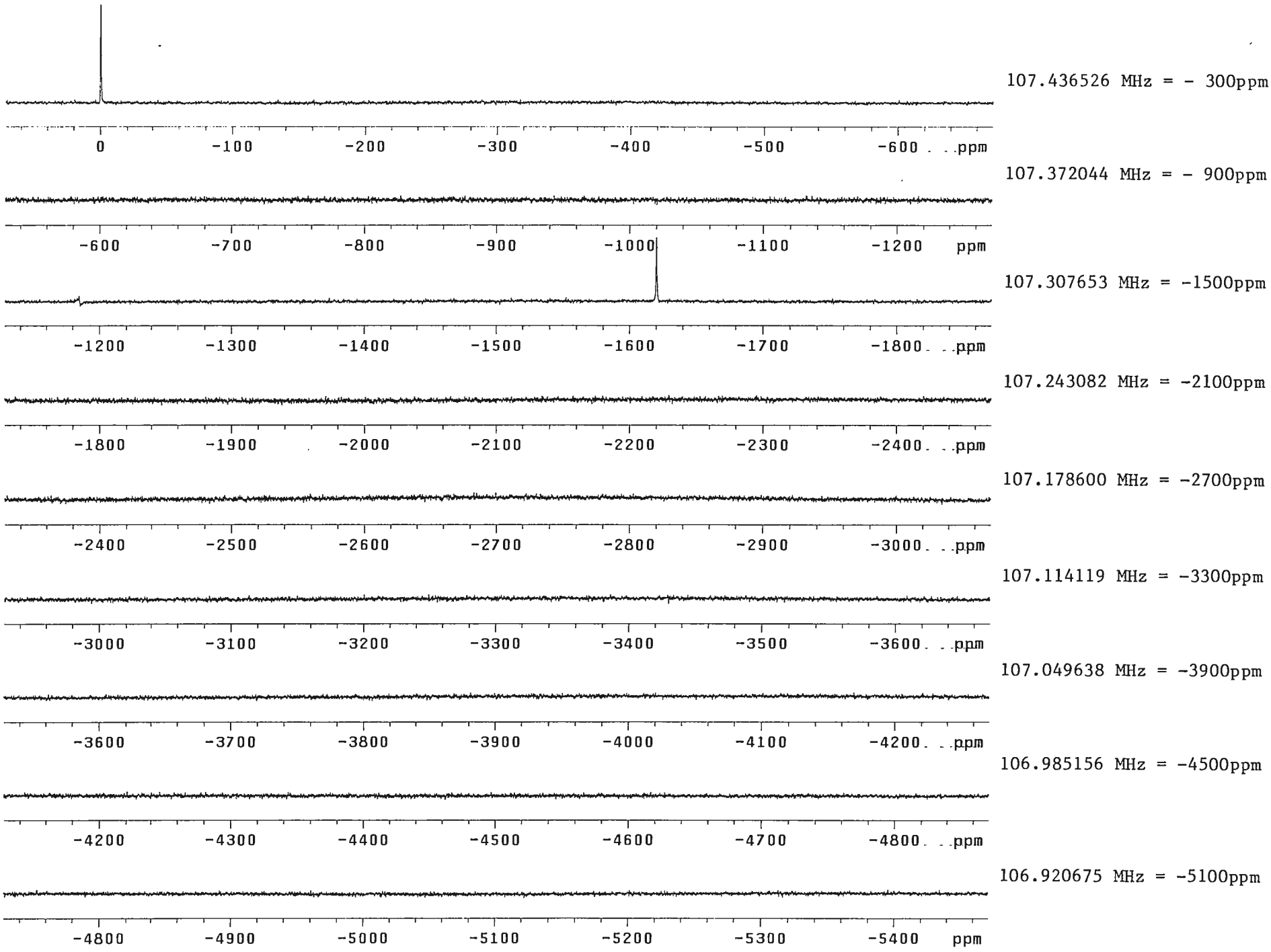
The figure shows our resultant calibrated ^{195}Pt shift scale. The nine NMR spectra were recorded with an 80 kHz spectral width (SW), a 45° pulse flip angle (8 usec), a 1.5 sec repeat time, and digitized into 120320 complex points to give a digital resolution of 1.33 Hz/pt. A tuned K and E bandpass filter was connected in series to the 5mm Varian probe, and a 60-130 MHz 1/4-λ cable was used in the low band preamp. The ^{195}Pt chemical shift scale (1) was calibrated by setting the low field ^{195}Pt signal of a 2M sample of Na_2PtCl_6 in d_2o to 0 ppm (exact resonance at 107.468767 MHz on the 500 MHz system). The transmitter was adjusted upfield to -300 ppm (107.436526 MHz) and a spectrum (spectrum 1) recorded between 70 ppm and -670 ppm (80 kHz SW = 740 ppm). The transmitter frequency was systematically decreased in 600 ppm steps to cover the full 0.59 MHz ^{195}Pt range with the most upfield spectrum corresponding to the transmitter at -5100 ppm (106.920675 MHz) and spectral range covering -4730 to -5470 ppm. Note that these are explicit frequencies and we have bypassed Varian's "tn" transmitter nucleus macro which had a more limited range. An experimental spectrum of 1M Na_2PtCl_4 in d_2o (spectrum 3) obtained over the 80 kHz SW with transmitter at the calibrated -1500 ppm position showed ^{195}Pt resonance at -1620 ppm [1] and added confidence to the achievement of a systematic calibration of the ^{195}Pt chemical shift scale.

Regards,

Tony

Anthony A. Ribeiro (A^2R)

{1} P. S. Pregosin, (1982) *Coordination Chemistry Reviews* **44**, 247-291.


107.436526 MHz = - 300ppm
0 -100 -200 -300 -400 -500 -600 . . .ppm
107.372044 MHz = - 900ppm
-600 -700 -800 -900 -1000 -1100 -1200 ppm
107.307653 MHz = -1500ppm
-1200 -1300 -1400 -1500 -1600 -1700 -1800. . .ppm
107.243082 MHz = -2100ppm
-1800 -1900 -2000 -2100 -2200 -2300 -2400. . .ppm
107.178600 MHz = -2700ppm
-2400 -2500 -2600 -2700 -2800 -2900 -3000. . .ppm
107.114119 MHz = -3300ppm
-3000 -3100 -3200 -3300 -3400 -3500 -3600. . .ppm
107.049638 MHz = -3900ppm
-3600 -3700 -3800 -3900 -4000 -4100 -4200. . .ppm
106.985156 MHz = -4500ppm
-4200 -4300 -4400 -4500 -4600 -4700 -4800. . .ppm
106.920675 MHz = -5100ppm
-4800 -4900 -5000 -5100 -5200 -5300 -5400 ppm

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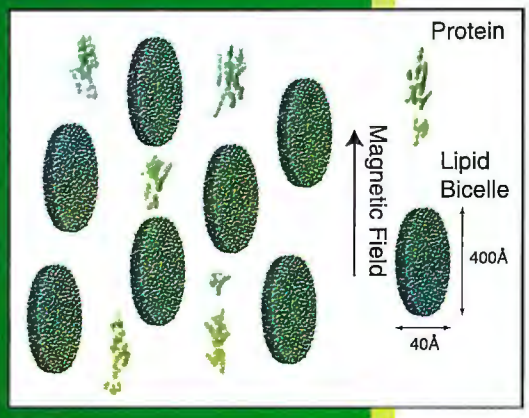

Tjandra & Bax[1] recently developed a new nuclear magnetic resonance (NMR) technique that gently aligns protein molecules in a bath of liquid crystals, allowing researchers to determine how each bond between neighboring atoms is oriented with respect to the rest of the molecule. By compiling all such orientations between atoms,a precise map of the protein can be derived. In aqueous solution, just above room temperature, the lipids switch from a gel to a Liquid Crystal (LC) phase, where they form disc-shaped particles, often referred to as bicelles[2], with diameters of several hundred angstroms and thicknesses of ~40Å. The lipids are diamagnetic, and, as a result, the bicelles orient with their normal orthogonal to the magnetic field. However, the lifetimes and temperature ranges of orientation for these samples are critically dependent on sample composition and experimental conditions. Losonczi & Prestegard[3] demonstrated that doping dilute bicelle solutions with small amounts of charged amphiphiles substantially improves the stability and degree of alignment, as well as extends the temperature range of orientation for these systems.

1. Tjandra, N. and Bax, A. (1997). Direct measurement of distances and angles in biomolecules by NMR in a dilute liquid crystalline medium. *Science* 278:1111-3.
2. Sanders, C.R. II and Schwonek, J.P. (1992). Characterization of magnetically orientable bilayers in mixtures of dihexanoylphosphatidylcholine and dimyristoylphosphotidylcholine by solid-state NMR. *Biochemistry* 31:37,8898-905.
3. Losonczi, J.A. and Prestegard, J.H. (1998). Improved dilute bicelle solutions for high-resolution NMR of biological macromolecules. *J Biomol NMR* 12:447-51.

Product	M.W.	25mg	200mg	500mg	1 gram	Catalog Number
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Bicelle Preparation

Buffer:
An effective and convenient method for preparing bicelles makes use of a buffer solution containing 10mM phosphate buffer, pH 6.6, 0.15 mM sodium azide, 93% H_2O (HPLC-grade), 7% D_2O (99.9%). Below, this solution will simply be referred to as buffer.

Bicelle Formation:
DMPC/DHPC stock solutions containing a total of 15% w/v (150mg lipid/ml) are prepared as follows: *Add buffer to the lyophilized lipid mixture* - 50mg lipid mixture/280µg buffer or 200mg lipid mixture/1130µg buffer. *Let the mixtures hydrate at room temperature (18-22°C) for several hours.*
Lipid mixtures with a "q" of 2.8 - 3.0, the hydration is complete in 2 - 3 hours. Lipid mixtures with a "q" of 3.25 - 3.5 require 24 hours for complete hydration. Accelerated hydration (one hour) may be effected by heating any mixture to 40°C for 10 minutes and cycling to 18°C twice, then briefly vortexing.
Protein-Bicelle Mix: Two volumes of protein solution are added to one volume of bicelle solution.

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Magnetic Alignment of Biological Membranes

Blue, green, and red stylized molecules represent DMPC, DMPG, and DMPE-DTPA, respectively, and yellow represents the Yb^{3+} ion DHPC, which is believed to be sequestered in curvature defect regions, is not shown in this figure.

We thank Biophysical Journal for permission to use this graphic.

A phospholipid chelate complexed with ytterbium (DMPE-DTPA:Yb^{3+}) is shown to be readily incorporated into a model membrane system, which may then be aligned in a magnetic field such that the average bilayer normal lies along the field. This so-called positively ordered smectic phase, whose lipids consist of less than 1% DMPE-DTPA:Yb^{3+}, is ideally suited to structural studies of membrane proteins by solid-state NMR, low-angle diffraction, and spectroscopic techniques that require oriented samples. The chelate, 1,2-dimyristoyl-*sn*-glycero-3-phospho-ethanolamine diethylenetriaminepentaacetic acid, which strongly binds the lanthanide ions and serves to orient the membrane in a magnetic field, prevents direct lanthanide-protein interactions and significantly reduces paramagnetic shifts and line broadening. Similar low-spin lanthanide chelates may have applications in field-ordered solution NMR studies of water-soluble proteins and in the design of new magnetically aligned liquid crystalline phases[1].

The addition of lanthanides (Tm^{3+}, Yb^{3+}, Er^{3+}, or Eu^{3+}) to a solution of long-chain phospholipids such as DMPC and short-chain phospholipids such as DHPC is known to result in a bilayer phase in which the average bilayer normal aligns parallel to an applied magnetic field. Lanthanide-doped bilayers have enormous potential for the study of membrane proteins by solid-state NMR, low-angle diffraction, and a variety of optical spectroscopic techniques[2].

The equimolar complex, consisting of the lipid-like, amphiphilic chelating agent DTPA-18 and Tm^{3+}, is shown by deuterium NMR to be useful in aligning bicelle-like model membranes, consisting of DMPC and DHPC. As shown previously, in the absence of chelate, the lanthanide ions bind loosely with the lipid phosphate groups and confer the membrane with a sufficient positive magnetic anisotropy to result in parallel alignment. Two conclusions could be drawn from this study: 1. The addition of Tm^{3+} to the bicelle system is consistent with a conformational change in the surface associated peptide, and this effect is shown to be reversed by addition of the chelate, and 2. The paramagnetic shifts are shown to be significantly reduced by addition of chelate[3].

1. Prosser, R.S., et al. (1998). Novel chelate-induced magnetic alignment of biological membranes. *Biophys J* 75:2163-9.
2. Prosser, R.S., et al. (1998). Solid-state NMR studies of magnetically aligned phospholipid membranes: taming lanthanides for membrane protein studies. *Biochem Cell Biol* 76:443-51.
3. Prosser, R.S., et al. (1999). Lanthanide chelates os bilayer alignment tools in NMR studies of membrane-associated peptides. *J Magn Reson* 141:256-60.

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National Institutes of Health
Laboratory of Chemical Physics, NIDDK
Building 5, Room 112
Bethesda, Maryland 20892-0520
July 23, 2001

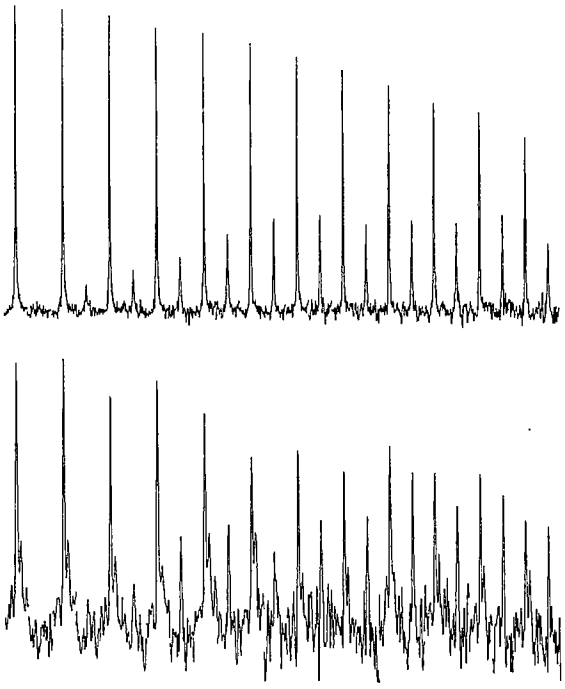
Big Sensitivity Gains in Dipolar Recoupling Measurements Through Pulsed Spin Locking

Dear Barry:

Structural measurements on specifically isotopically labeled compounds in solid state NMR typically require 0.1 to 1.0 micromoles of labeled molecules and many hours or days of signal averaging at room temperature and fields in the 7-14 T range. Often, especially in the case of biological macromolecules, sufficient sample quantities are not available. We have found that the sensitivity of many structural measurements, including dipolar recoupling measurements to determine internuclear distances and tensor correlation measurements to determine torsion angles, can be enhanced quite substantially by detecting NMR signals under pulsed spin locking conditions. In the case of ^{13}C magic angle spinning experiments, pulsed spin locking can effectively narrow the NMR lines of interest from 100-600 Hz to 10-20 Hz, producing signal-to-noise enhancements of 2 to 8. One example is shown below. More examples and experimental details will be given in a forthcoming paper.

Sincerely yours,

Dr. Robert Tycko
e-mail: tycko@helix.nih.gov



^{13}C-detected $^{13}C/^{15}N$ rotational echo double resonance (REDOR) data obtained with pulsed spin lock detection (top) and conventional FID detection (bottom) for the peptide Ac-KLVFFAE-NH_2 in the form of amyloid fibrils (see *Biochemistry*, vol. 39, p. 13748, 2000). All other data acquisition conditions are identical. Undephased (S_0) and REDOR difference (ΔS) peaks are plotted alternately, with the dephasing period increasing from 6.8 ms to 77.2 ms in 6.4 ms increments. Peaks are plotted in spectral widths of 400 Hz (top) and 4000 Hz (bottom).

DEPARTMENT OF PHYSICS
AND
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INDIAN INSTITUTE OF SCIENCE
BANGALORE - 560 012. INDIA

Prof. ANIL KUMAR
Convener, SIF

13 July 2001

(received 7/25/2001)

Selective Saturation of Antisymmetric States (SSAS)

Dear Berry,

The proton NMR spectrum of CH_3CN oriented in a liquid crystal matrix is shown in Fig. 1(a). The 3:6:3 intensity pattern arises from a 3:4:3 pattern from transitions of symmetric eigenstates and two transitions of unit intensity from antisymmetric eigenstates overlapped with the central transition(Fig.1(b)). To treat the 4 symmetric eigenstates of this spin system as a 2 qubit system, we needed to saturate away the antisymmetric domains. This proved easy, since the matrix element of the I_X operator connecting states 2 and 3 is twice as large as that connecting states 5 and 6, and 7 and 8. That is,

$$< 2|I_X|3 >= 2 < 5|I_X|6 >= 2 < 7|I_X|8 > . \qquad (1)$$

Therefore the nutation angle for a given r.f., selectively applied at the central transition frequency, is twice for symmetric domain compared to the antisymmetric domains. If the flip angle is adjusted for π for symmetric domain, the r.f. acts as a $\pi/2$ pulse for both transitions of antisymmetric domains. Following such an r.f. pulse, a gradient pulse destroys the magnetization of antisymmetric domains. A subsequent π pulse at central transition frequency restores the equilibrium magnetization of the symmetric domain Fig.1(c). The resulting spectrum (measured by small flip angle pulse) has intensity pattern 3:4:3 (Fig.1(d)). We have used oriented CH_3CN and $^{13}CH_3CN$ as 2 and 3 qubit systems after SSAS and implemented several logic gates. This work has just been submitted for publication.

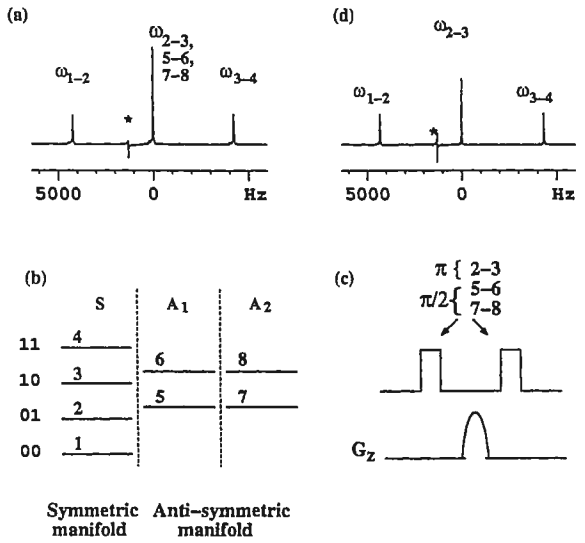

Fig. 1

Anil Kumar K.V.Ramanathan T.S.Mahesh Neeraj Sinha

p.s.: Please credit this contribution to the account of Prof. C.L.Khetrapal.

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T82-00144	40% p-Dioxane, 5mg/ml Cr(acac)$_3$ in Benzene-d_6(99.6%)	^{13}C PW90
T82-00141	40% p-Dioxane in Benzene-d_6(99.6%)	^{13}C Sensitivity/Resolution
T81-00029	0.2% Cr(acac)$_3$, 2% Benzamide-^{15}N(99%) in DMSO-d_6"100%"(99.96%)	^{15}N Sensitivity
T81-00028	1% $^{13}CH_3I$(99%)/1% Trimethylphosphite, 0.2% Cr(acac)$_3$ in $CDCl_3$(99.8%)	^{31}P Sensitivity
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Department of Chemistry

PO Box 117200
Gainesville, FL 32611-7200

July 20, 2001
(received 7/27/2001)

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

Fluorinated Ethers

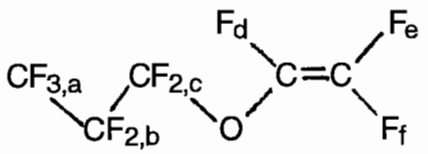
Dear Barry:

We have been continuing our exploration of the NMR characteristics of interesting fluorocarbon derivatives, and recently we have been compiling results on a number of fluoroethers, of which samples have been provided to us by several different laboratories.

The simplest molecules are the derivatives of dimethyl ether in which various numbers of fluorines and chlorines are substituted for hydrogens. In hexafluoro-, pentafluoro- and tetrafluoro- derivatives, the one-bond C-F coupling values are usually 260-265 Hz, while in one -OCF_2Cl group, J is as large as 294 Hz. Across the oxygen, the three-bond C-F couplings are about 5 Hz, and the four-bond F-F J values are 4 to 8 Hz. ^2J (H-F) values in –$OCHF_2$ groups are 69-75 Hz, but in groups with only one F, such as –OCHFCl and –OCH_2F, the value is substantially less, 53-59 Hz.

In saturated, longer-chain fluoro-ethers, with CF_2 groups on each side of the oxygen, the value of ^4J(F-F) across the oxygen is larger, 10-15 Hz. This coupling constant is reduced by substitution of a hydrogen for one of the four fluorines and increased by substitution of an iodine atom.

Trifluorovinyl ethers offer some interesting spin-spin coupling results. Within the vinyl group itself, the trans F-F coupling is always about 110-120 Hz, so that it is quite characteristic. The geminal and cis vicinal couplings vary over a wide range, depending on what is attached to the oxygen. Some interesting aspects of F-F indirect coupling are illustrated by the following molecule:



The coupling from (a) to (c) is 7.8 Hz, rather small. Fluorines (c) are coupled by 6 Hz to both (d) and (f), but not coupled to (e), the atom trans to the fluoroalkoxy

group, a pattern which seems to be followed by all the molecules in which a trifluorovinyl group is on one side of the oxygen and a fluorine-containing entity is on the other side. This effect might be explained by a combination of "through-space" coupling with the geometry established for trifluoromethyl vinyl ethers by other spectroscopic techniques in which the methyl group is directed almost perpendicular to the plane of the vinyloxy group. Also interesting in this respect is a coupling of 0.7 Hz from (a) to (d).

If we examine the molecule perfluorovinyllethyl ether, in which there is an ethyl instead of an n-propyl group, the $-OCF_2-$ fluorines are again coupled by 6 Hz to two of the three fluorines in the vinyl group, but the CF_3 fluorines show no resolvable coupling. However, in $CF_3CH_2OCF{=}CF_2$, the CF_3 fluorines are coupled by about 2 Hz to the CF fluorine. These and other effects, which we are in process of compiling for a manuscript to be submitted for publication, lead to interesting questions about the relative contributions to J of bonding and geometrical variations, which the theoreticians will perhaps soon be able to explain.

Yours sincerely,

Wallace

Wallace S. Brey

RSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRSRF

25 July 2001

Bruker MAS Controller Available

Dear Barry,

We have an old Bruker pneumatic MAS controller (model MAS-DB, MSL vintage) and a transformer (120 V to 220 V) for running Bruker equipment. Present condition is unknown, though these were used within the past 5 years. They are available free to good home, preference given to educational institutions.

Sincerely,

Al

A. N. Garroway, Head
Polymer Diagnostics Section
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Chemistry Division

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NMR in Microbiology. Theory and Applications

(Edited by Jean-Noël Barbotin and Jean-Charles Portais)
Horizon Scientific Press (www.horizonpress.com), 2000.
ISBN 1-898486-21-2, pp. 496, $169.99

NMR has been used to study metabolic processes and profile metabolism for decades, but – in this era of *genomics*, *post-genomics*, *proteomics*, and so on – *metabonomics* is another new term we can make friends with now. As it was presented, for example, recently in Princeton by Jeremy Nicholson and his group (Imperial College, London, UK) (www.genomics.princeton.edu/html/events.htm), this field is undergoing a rapid and truly impressive metamorphosis, and is becoming a revolutionary chapter in NMR applications. It is due, in large part, to amazing technical developments over the last few years, which have increased the sensitivity of NMR by magnitudes, and have introduced hyphenated and HR-MAS NMR techniques as very competitive tools. And, I believe, this is only the overture.

Publications are mushrooming on this subject, and academic research groups and pharmaceutical companies alike rush to benefit from this expanding field. Hence it is particularly timely and pleasant to have these two books available.

Interdisciplinary developments often show the character of intercultural engagement, if I may say so, when the participants have to learn each other's language. In both books there is multitude of introductions of one or the other technique, such as basics of NMR spectroscopy, mathematical analysis of metabolic flux, etc., sometimes in partial repeat. This, however, does not take away from the great value of the subject and presentations overall.

In the fifteenth volume of the great series "Biological Magnetic Resonance," *in vivo* ^{13}C NMR studies are discussed. Such experiments can be done either on tissues, or on perfused organs, or using imaging methods. The book presents seven chapters on these subjects, extended with the list of titles of the previous volumes, and three pages of index.

The first chapter is a well-structured extensive introduction to tracer theory, various structural and kinetic models, which also gives an overview of how ^{13}C NMR spectroscopy is applied in this field. Various experimental protocols for following metabolic pathways and kinetics are discussed, including condensation reactions by analysis of multiplets due to ^{13}C-^{13}C couplings. Technical aspects of ^{13}C NMR spectroscopy, isotope labelling, signal detection, and enrichment schemes and sensitivity considerations are also presented. Theory and mathematical analysis of metabolic flux are further discussed in chapters 3 and 4.

continued

There are two chapters which deal with particular metabolic cycles. In Chapter 2 metabolic pathways which intersect in the citric acid cycle, are probed by NMR, while Chapter 7 presents results on the tricarboxylic acid cycle. Chapter 5 tells about pathological studies on perfused heart using both dynamic ^{13}P and ^{13}C NMR. Metabolic processes and functional behaviour of the brain can also be studied by imaging methods following ^{13}C-labelled compounds, either using surface coils or resonant cavity coils are presented in Chapter 6.

The more recent book, "NMR in Microbiology" gives a great overview of various NMR techniques applied in this field, mostly by authors from Europe. It is extended by a couple of pages listing web-sites of some of these authors and others, such as educational sites, data banks, etc., and also with four pages of index.

The book is organized into five sections. The first one, in six chapters, is about NMR methods and microbiology. Basic NMR theory, practical aspects of NMR spectroscopy and microbiology, ^{13}C labelling and mathematical methods for analysis are discussed. The closing chapter of this section is about two-dimensional NMR by Jack Skalicki and Thomas Szyperski (who also contributed to another chapter about future perspectives).

The section "NMR and Macromolecules" opens with an elegant presentation from Uhrín and Brisson about solving the structure of microbial polysaccharides using (selective) 1D, and multidimensional NMR methods. It is followed by a chapter, which discusses NMR studies of the bacterial sugar transport system, determining 3D structure, and complexation behaviour of relevant proteins. An interesting chapter on protein hydration and *in situ* water activity in enzyme reactions is next, followed by a contribution which talks about *in situ* monitoring of enzyme activity by ^{1}H, ^{31}P, and ^{13}C NMR. The last chapter in this section is an overview of solid state NMR studies of biological membranes, membrane peptides and proteins.

A relatively short section (three chapters) presents investigations of microbial metabolic pathways in both bacteria and yeast, which is then followed by an extensive section of case studies. This is a very impressive collection of applications, such as studies of polymer biosynthesis, sugar transport and metabolism, NMR on bacterial biofilms and model systems, pattern analysis in spectra of stable isotope-labelled metabolites, biodegradation of xenobiotics using ^{19}F, ^{1}H, ^{13}C, and ^{2}H (and also applying LC-NMR), and solid state NMR of soils. The book closes with a section of two chapters discussing general perspectives of integration of genomic and physiological data by metabolic engineering, and future perspectives.

This book is trying to cover a very big area of methods and applications, and present it to a diverse audience – and does so with great success. Perhaps the typography is something to complain about; the font is small for the text, while the titles are too large, and the appearance of the figures is uneven.

I recommend both books to a wide audience, including many, who have never used NMR before, as we are at the doorstep of a significant expansion of utilizing this spectroscopic method in life sciences. The recent, and anticipated improvements in hardware (cryo-probes, and also very high fields), technology (hyphenated applications), and data analysis open a new chapter both in academic and industrial research, as well as in direct medical applications. I am sure we shall see many similarly good books on this subject in the near future.

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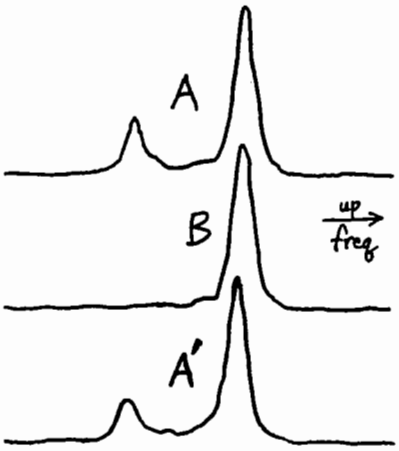
27 June 2001

Dr. Shapiro,

Mea culpa.

We noticed a report that nanocrystalline palladium displays two well-resolved proton NMR lines after exposure to hydrogen gas. The nanocrystalline material has a grain size of about 10 nm, so about half the Pd atoms are within 1-2 nm of the surface. Thus the authors attributed the two lines to H atoms in the interior of the nanocrystallites and in the grain boundary regions.[1] This assignment is problematic – H atoms jumping 10^7-10^8 hops per second (as indicated by narrow lines and T_1) would be expected to exchange between the interior and grain boundary regions on the 10^{-3} second NMR timescale, leading to a single merged line. Possibly the authors were seduced: spectroscopic separation of these two kinds of sites is a big goal in nanocrystalline research.



For this reason, we re-examined the proton NMR. We hydrided some nano-Pd by H_2 gas exposure at 1 atm to yield the uppermost spectrum (A) with two NMR lines about 30 ppm apart. Upon evacuation, both lines disappear. Subsequent exposure to H_2 yields only the single resonance shown as B; the single resonance is nearly at the frequency (30 ppm up-frequency Knight shift, relative to water) of H in coarse-grain Pd metal. Further cycles of evacuation and exposure to H_2 gas only yield spectrum B.

We found a way to retrieve the two-line spectrum, similar to A. The material yielding spectrum B was evacuated and then exposed to O_2 at 1 atm for 18 hours. Then, after a 1 hour evacuation, exposure to H_2 at 1 atm returned spectrum A'. Exposure to air (instead of O_2) works, too. Here's what is happening: the O_2 bonds to the high surface area of nano-Pd. Despite Pd's reputation as a non-oxidizing metal, the O_2 evidently bonds sufficiently that it is not removed by subsequent evacuation. When H_2 is next introduced, the catalytic Pd surface splits the H_2 into H atoms. Some H dissolve into the Pd and some extract the oxygen from the Pd surface to form H_2O. The H_2O is a surface species (i.e., it does not freeze out even at 200 K). Evacuation removes the dissolved H *and* the H_2O, so that subsequent hydriding cycles find no oxygen, so no second proton NMR line occurs. We note that the H atoms bond oxygen more tightly than does the Pd surface, but the Pd surface binds the H_2O molecules more weakly than the oxygen atoms.

If there is a lesson here, it involves the careless handling of the nano-Pd ("Pd is not an oxidizing metal" – wrong). And the lesson would be easier to take had we not been the authors of the incorrect assignment of the two peaks.[1] Mea maxima culpa. Maybe, on the bright side, there is a message here about testing out the simple explanations before invoking exotic phenomena.

Mark S Conradi

D. Blane Baker Ivan Salazar Mark S. Conradi

[1] J.W. Hanneken, D.B. Baker, M.S. Conradi, and J.A. Eastman, J. Alloys and Compounds (in press).

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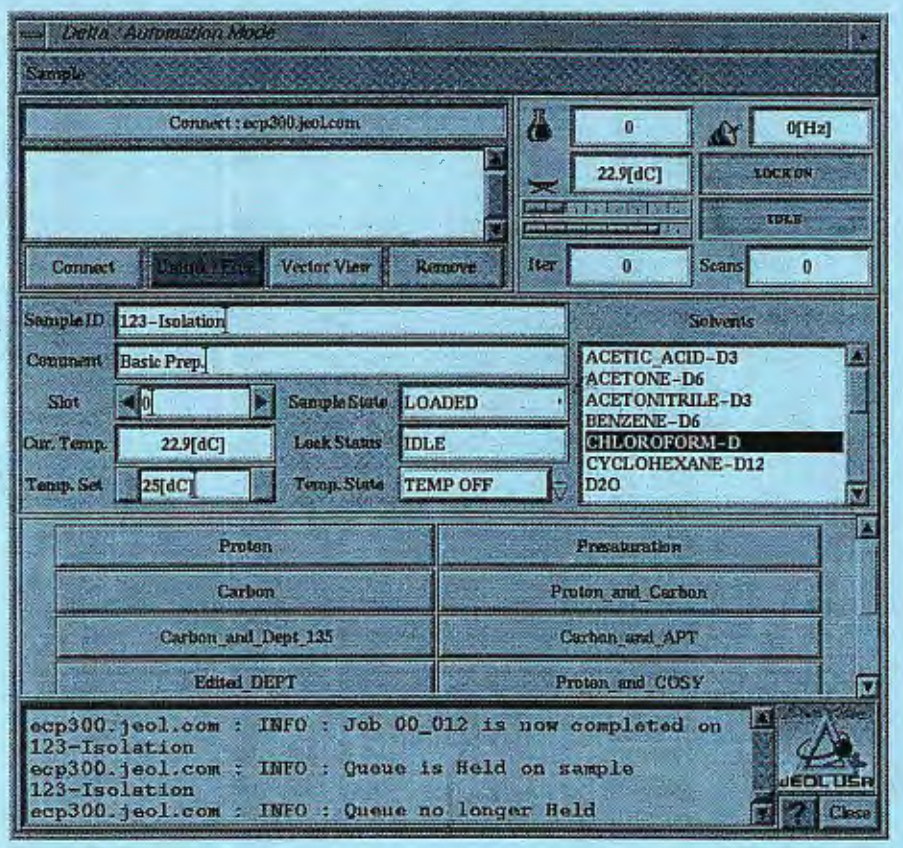

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