

No. 485

# February 1999

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#### FORTHCOMING NMR MEETINGS

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- <u>7th Annual "Advances in NMR Applications" Symposium</u>, Omni Rosen Hotel, Orlando, Florida, February 28, 1999; Contact: Annette Matamoroz at the Nalorac Corp.; 510-229-3501; annette.matamoroz @nalorac.com; See Newsletter <u>485</u>, 22.
- <u>40th ENC (Experimental NMR Conference)</u>, Clarion Plaza Hotel, Orlando, Florida, February 28 March 5, 1999, immediately preceding Pittcon in Orlando; Contact: ENC, 1201 Don Diego Avenue, Santa Fe, NM 87505; (505) 989-4573; Fax: (505) 989-1073; Email: enc@enc-conference.org.
- <u>Pittcon '99</u>, Orlando, FL, March 7-12, 1999 (50th year celebration of the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy.) Contact: The Pittsburgh Conference, Dept. CFP, 300 Penn Center Blvd., Suite 332, Pittsburgh, PA 15235-5503; 412-825-3220; Fax: 412-825-3224; e-mail: pittconinfo@pittcon.org:
- Spin Choreography a symposium in appreciation of Ray Freeman, Cambridge, England, April 8-11, 1999; web site: http://mchsg4.ch.man.ac.uk/mcmr/RF.html; fax: c/o M.H.Levitt +46-8-15 2187; email: mhl@physc.su.se.
- Seventh Scientific Meeting and Exhibition of the Intl. Soc. for Magnetic Resonance in Medicine (ISMRM), Philadelphia, PA, **May 22 28, 1999**; Contact: International Society for Magnetic Resonance in Medicine, 2118 Milvia St., Suite 201, Berkeley, CA 94704.
- International School of Structural Biology and Magnetic Resonance, 4th Course: Dynamics, Structure and Function of Biological Macromolecules; Erice, Sicily, Italy; May 25-June 5, 1999; Contact: Ms. Robin Holbrook, Stanford Magnetic Resonance Laboratory, Stanford University, Stanford, CA 94305-5055; (650) 723-6270; Fax: (650) 723-2253; Email: reh@stanford.edu. See Newsletter <u>483</u>, 8.
- Royal Society of Chemistry: 14th International Meeting on NMR Spectroscpy, Edinburgh, Scotland, **June 27 July 3**, **1999**; Contact: '99NMR14' c/o Mrs. Paula Whelan, The Royal Society of Chemistry, Burlingtom House, London W1V OBN, England; +44 0171 440 3316; Email: conferences@rsc.org\
- <u>SMASH No. 1</u> (Small Molecules Are Still Hot), Argonne, IL, **August 15-18, 1999**; Contact: Ms. Karen McCune, (mccune\_karen\_a@lilly.com, 317-276-9783) or S. R. Maple (maple\_steven\_r@lilly.com) or G.E.Martin (gary.e.martin@am.pnu.com) or A. G. Swanson (alistair\_swanson@sandwich.pfizer.com. See Newsletter <u>484</u>, 29

University of Nebraska Medical Center Nebraska's Health Science Center The Eppley Institute for Research In Cancer and Allied Diseases A National Cancer Institute Designated Laboratory Cancer Research Center 600 South 42nd Street Box 986805 Omaha, NE 68198-6805 (402) 559-4090 Fax: (402) 559-4651

> January 12, 1999 (received 1/16/99)

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

UCSF: A VAST Difference From Nebraska

#### Dear Barry:

I recently completed a half-year sabbatical at UCSF, amicably hosted by Tom James and his group. Although summer in San Francisco is as chilly as advertised, it was a refreshing change from the heat of Nebraska in the summer. While in the Bay area I made an appointment to visit the Varian applications lab, and they kindly let me in the door even though no new instrument purchase is on the horizon. As one of my focii is on the use of NMR Spectroscopy As A Tool To Investigate the Structural Basis of Anticancer Drugs (reviewed by yours truly in Current Medicinal Chemistry, 1998, 5, 115-135; reprints available) I asked Paul Keifer for a demonstration of the VAST accessory (versatile automated sample transfer). Using VAST and the flow probe, we rapidly screened a number of putative complexes between drugs and nucleic acids. I think this technology is really useful and recommend it to anyone with resources available to invest in drug discovery. As I don't know when I will have the opportunity of visiting UCSF or the Bay area again, I'd like to thank Tom James and his group for their help and insight, and Paul Keifer for showing me the VAST technology.

Sincerely yours,

B,11

William H. Gmeiner, Ph.D. Associate Professor

P.S. - Thanks for letting me speak at BANG, it was a lot of fun.

Bill:

I had nothing to do with your invitation to talk at the BANG<sup>\*</sup> meeting - Paul Keifer and Gina Miner were the responsible parties. Your talk was very well received - come back again anytime!

\*Bay Area Nmr Group.

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January 22, 1999 (received 1/29/99)

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

Activation Energies for Molecular Motions in Asphalts

Dear Barry:

Many rheological properties of asphalt depend on the molecular motions of the many different carbon types in the asphalt.<sup>1</sup> From a knowledge of the activation energy for a particular type of molecular motion, it may be possible to use activation energies as an indicator of polar association and/or steric hindrance present in asphalts, and, which in turn, may be used to predict asphalt road performance.

The Arrhenius plots of the aromatic and aliphatic hydrogen relaxation rates,  $1/T_{1p}^{H}$  for three asphalts are shown in Figure 1. The activation energies for aromatic ring motions (6.5 to 8.2 kJ/mole) show a small increase from asphalts AAA-1 to AAM-1. Thus, the aromatic ring motions in asphalt AAA-1 have a lower barrier to motion than asphalt AAB-1, which in turn, have a lower barrier to motion than asphalt AAB-1. This is in agreement with the fact that AAA-1 has both a lower glass-transition temperature and lower number of condensed aromatic rings per average molecule than either AAB-1 and AAM-1, and, thus, has a greater degree of ring motions.



Figure 1. Arrhenius plots for the spin-lattice relaxation rate for hydrogens in the rotating frame for the aromatic and aliphatic hydrogens in asphalts AAA-1, AAB-1, and AAM-1

Dr. B. L. Shapiro Page 2 January 22, 1999

The low activation energies measured for the phenyl ring motions in asphalts (6.5 to 8.2 kJ/mole) over a temperature range from 20° to -45°C compared to phenyl ring motions in polymers suggest some type of rapid rotational motions of the small (3-5 rings) condensed aromatic structure or pendant phenyl exist in the solid state. Conceivably, the observed low activation energies are representative of fast in-plane molecular reorientation of the aromatic molecules in a frozen state because asphalts can be considered to be glass-like at low temperatures with low concentration of aromatic carbons (~30%). However, another explanation for the low activation energies for the motions of the aromatic structure in the solid state is based upon <sup>1</sup>H spin-diffusion between the aromatic and aliphatic molecular components in the asphalt. Because of the methyl and ethyl substituents on the phenyl rings <sup>1</sup>H spin-transfer of the methyl hydrogens to the hydrogens of the protonated aromatic carbons may be possible. The barrier to methyl rotation for methyl groups attached to an aromatic ring has been reported for o-xylene (5.8 kJ/mole), hemimellitine (6.06 kJ/mole), and isodurene (6.48 kJ/mole).<sup>2</sup> Thus, because of <sup>1</sup>H spin-transfer, the activation energies measured for the aromatic components in the asphalts may be directly influenced by the attached methyl group rotation.

The activation energies given in Figure 1 for the motions of aliphatic components in the asphalts over the temperature range from 20 to  $-45^{\circ}$ C are slightly higher than those observed for the aromatic ring motions. The activation energies (8.8 to 9.8 kJ/mole) for the motion of the aliphatic components in asphalts measure the combined average barrier to segmental motions of the methylene carbons and rotational motions of the various types of methyl groups (terminal, branched, and as an alkyl substituent on an aromatic ring). Based on the literature data for the activation energies associated with n-alkanes, the activation energies for the motions of the aliphatic components of the asphalts at temperatures above and below the glass-transition temperature are attributed mainly to methyl rotation. The activation energies for the segmental motion of the methylene carbons may contribute partly to the measured activation energy but can not be differentiated because of <sup>1</sup>H spin-diffusion of the methylene hydrogens is controlled by rapid methyl group rotation.

A more detailed discussion of the activation energies for molecular motions in asphalts is given in reference 3.

#### References

- Netzel, D. A.; Miknis, F. P.; Wallace, J. C.; Butcher, C. H.; and Thomas, K. P. Asphalt Science and Technology, Usmani, A., ed.; Marcel Dekker: New York, 1997, Chapter 2. Molecular Motions and Rheological Properties of Asphalts: An NMR Study.
- 2. Mann, B. E. Dynamic <sup>13</sup>C NMR Spectroscopy, Progress in NMR Spectroscopy, <u>11(2)</u>: 95 (1977).
- Netzel, D. A.; Miknis, F. P.; Soule, J. L.; Taylor, A. E.; and Serres, M. L. Handbook of Asphalt Binder Technology, Youtcheff, J., ed.; Marcel Dekker: New York (in press), Chapter ??. Solid-State <sup>13</sup>C NMR Studies of the Molecular Structure and Dynamics of Asphalt Binders.

Sincerely,

Dan Netzel

Fran Miknis



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> January 11, 1999 (received 1/14/99)

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

### **SLF vs PISEMA**

Dear Dr. Shapiro,

Heteronuclear dipolar coupling of <sup>13</sup>C-<sup>1</sup>H and <sup>15</sup>N-<sup>1</sup>H spin pairs directly reflects the orientation of the bond vector. For the measurement of the heteronuclear dipolar coupling, the PISEMA technique introduced by Hu, Ramamoorthy and Opella [J. Magn. Reson. A **109**, 270 (1994)] shows great improvement on spectral resolution over the separated-local-field (SLF) experiment (see Figure 1). In this letter, we report that the truncation of weak heteronuclear dipolar coupling to remote protons is the primary cause of this improvement.



Figure 1. a) 2D PISEMA and b) 2D SLF spectra of methyl- $\alpha$ -D-glucopyranoside single crystal at an arbitrary sample orientation.

In essence, PISEMA is a cross polarization experiment with proton homonuclear decoupling and the Hamiltonian is a sum of the flip-flop terms of the heteronuclear dipolar interaction  $H_{Si} = sD_{Si}(S_{+}I_{-}^{i} + S_{-}I_{+}^{i}), (s = \sin 54.7^{\circ})$  with all surrounding protons. The flip-flop Hamiltonians do not commute with each other,  $[H_{Si}, H_{Sj}] \neq 0$  for  $i \neq j$ . Therefore, the weak dipolar coupling from remote protons is truncated because of the presence of the strong coupling term from the directly bonded proton. In the SLF experiment, however, the dipolar Hamiltonian terms  $H_{Si} = sD_{Si}2S_{2}I_{z}^{i}$  $(s = \cos 54.7^{\circ}$  for FSLG homonuclear decoupling) commutes with each other  $[H_{Si}, H_{Sj}] = 0$ . Each weak dipolar coupling splits the dipolar spectrum and it contributes the linebroadening. Figure 2 shows the numerical simulation of PISEMA and SLF spectra which demonstrates this truncation effect. The



Figure 2. PISEMA (left) and SLF (right) dipolar spectra calculated for a  ${}^{13}C$  spin coupled to one, two and three protons. The  ${}^{13}C{}^{-1}H$  dipolar coupling are 20, 1.5 and 1.0 kHz, respectively.

addition of weakly coupled protons (1.5 and 1.0kHz) has nearly no effect to the PISEMA spectrum of the strongly coupled (20kHz)  ${}^{13}C{}^{-1}H$  pair.

The analytical solution of cross polarization with flip-flop Hamiltonian has been solved for simple spin systems: CH,  $CH_2$ , and fast rotating  $CH_3$  group. The PISEMA spectra of more complicated systems have been simulated numerically. These results will be presented in a poster at this year's ENC in Orlando.

Please credit this contribution to Dr. Nagarajan Murali's account. Professor David Grant is gratefully acknowledged for providing the methyl- $\alpha$ -D-glucopyranoside single crystal sample.

Best regards,

Zhehong Gan Center of Interdisciplinary Magnetic Resonance (CIMAR) National High Magnetic Field Laboratory (NHMFL) email: gan@magnet.fsu.edu, phone: 850-644-4662, fax: 850-644-1366

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Magnet Type (Standard or shielded)	Stand	dard	Standard	5tano	lard	Standard	Standard	Actively Shielded	Actively Shielded	Actively Shielded	Actively Shielded
Field Stability (Hz/hour 'H)	<	2 2		<	3	<3	<15	<8	<10	<10	<10
Axial 5 Gauss Stray Field Contour (Metres)	1.8	31	2.65	2.1	9	2.75	4.2	1.5	1.8	1.8	2.5
Radial 5 Gauss Stray Field Contour (Metres)	1.4	12	2.0	1.3	7	2.2	3.3	1.0	1.3	1.3	1.75
Cryostat Type	Compact	Т3	T3	Compact	T3	T3	TS	T3	T4FB	T4FB	T5F8
Minimum Helium Refill Interval (Days)	80	235	203	80	235	203	120	183	150	150	140
Helium Refill Volume (Litres)	26	79	68	26	79	68	101	62	83	83	120
Year Hold Cryostat Option Available	x	1	1	x	1	1	x	х	x	x	x
Nitrogen Refill Interval (Days)	14	14	14	14	14	14	22	14	15	15	14
Minimum Nitrogen Refill Volume (Litres)	32	61	61	32	61	61	135	61	81	81	136
* Minimum Operational Ceiling Height (Metres)	2.69	2.92	2.92	2.69	2.92	2.92	4.16	2.9	3.1	3.1	3.16
System Weight (kg) Including Cryogen's	120	315	391	133	325	399	1050	400	610	625	1200

NMR Operating Frequency (MHz1H)	600		750		00	900	
Field Strength (Tesla)	14	.0	17.6	18	.8	21.1	
Nominal Room Temperature Bore Access (mm)	51	89	51	6	3		
Magnet	Activalu		1000		(7) 7/1	(2.2K)	Pumped
Type (Standard or shielded)	Shielded	Standard	Standard	Standard	Pumped	Standard	With Iron Shield
Field Stability (Hz/hour 'H)	<10	<12	<15	<15	<15	<15	<15
Axial 5 Gauss Stray Field Contour (Metres)	2.5	5.0	7.6	8.69	6.3	12.2	8.73
Radial 5 Gauss Stray Field Contour (Metres)	1.75	3.9	6.1	6.89	5.0	9.7	3.81
Cryostat Type	T5F8	T4FBL	Т6	T6L	77		Т8
Minimum Helium Refill Interval (Days)	120	90	60	60	60		60
Cryostat Helium Refill Volume (Litres)	101	60	187	216	328	1	200
Minimum Nitrogen Refill Interval (Days)	15	15	14	14	14		15
Nitrogen Refill Volume (Litres)	136	100	137	162	167	1	800
* Minimum Operational Ceiling Height (Metres)	3.16	3.4	3.78	3.97	3.97	8.75	
System Weight (kg) Including Cryogen's	1180	1200	3000	4000	4000	18	3000

### **Room Temperature Shim Specifications**

	1	Dime	ensions
Shim Type (Model)	Number of Channels	External Diameter (Cryostat Bore Size)	Internal Diameter (NMR Probe Diameter)
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18/89/73	18	89mm	73mm
26/89/73	26	89mm	73mm
28/51/40	28	51mm	40mm
40/51/40	40	51mm	40mm
29/51/45	29	51mm	45mm
36/63/51	36	63mm	51mm

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To: Dr. Barry Shapiro The NMR Newsletter 966 Elsinore Court Palo Alto, CA 94303 SEARLE 4901 SEARLE PARKWAY SKOKIE, ILLINOIS 60077 PHONE (847) 982-7000 FAX (847) 982-4701

(received 1/11/99)

Subject: Practical Results with NMRSAMS, a CASE tool

Dear Barry,

After eighteen years at the Frederick Cancer Research & Development Center in Frederick, Maryland I have relocated to G. D. Searle in Skokie, Illinois. This is a change from a government contracting position operating an NMR laboratory as a Center resource to a private pharmaceutical company, where I am working with Roy Bible, Bob Dykstra, Dave Lankin, Pat Finnegan, Elisabeth Hajdu and other heroes of the private sector. This is not just a change in geography and organization; I am also changing from many years with Varian equipment to working primarily with Bruker spectrometers. As a result I am in a unusual position to make comparisons between the two vendors at least as small molecule structure determination goes. I will defer any definite statements however until a later letter since I don't believe I have given the Bruker approach enough time to "sink in". I am interested in hearing from other people who have for one reason or another experienced what amounts to a vendor 180 pulse. In the meantime I want to make some comments on my experiences in Frederick with NMRSAMS, a CASE (computer assisted structure elucidation) program. I will start with some general comments.

The idealized goal of a CASE tool is to provide raw data to a program which then cranks out structures consistent with the data, such that the program could be used in combination with an autosampling machine. With sufficient concentrations (not unreasonable in a pharmaceutical company) one could fairly rapidly get 1D proton and carbon and 2D COSY, HMQC and HMBC data, (preferably gradient enhanced but that is not the point here). Though the principle is apparently straightforward I think this goal is not a realistic one. A far more practical approach is to conceive of a "toolbox" which allows rapid correlation of existing data by an interactive procedure. The advantages of the CASE program would not be automation, but rather vastly improved data handling and a much more thorough checking of the structure space when appropriate. The spectroscopist is allowed (maybe required is a better word) to examine the data in detail, but has a much better, faster grasp of the connection between his opinion of a particular data item and the consequences of his decisions in the target structure(s).

My experience with NMRSAMS bears out the value of the latter approach. Over the past year and a half or so working with Dr. Gwen Chmurny at Frederick we adopted the approach of running ALL novel structure problems through NMRSAMS. In not a single instance was the program able to generate a new structure on the first go-round. The central problem was that the sheer amount of data being generated even for small molecules meant that invariably there were cross peaks that were misinterpreted, typographical errors, etc. which prevented an immediate solution. Nevertheless I considered the program invaluable. I took the following approach: in general we had some idea of the structure, in some cases even a complete target. I would then start the program and begin building a structure atom by atom, in essence creating user defined constraints as I merrily built a likely structure. Since the program immediately reports conflicts in the data set and the particular data leading to the inconsistency I could generally proceed quite rapidly, while examining data in detail as necessary and massaging/fixing the raw data as necessary. Eventually I would arrive at a complete structure, by which time I had by force produced a carefully cleaned up set of NMR data. Then - and only then - I removed my user constructed constraints (the bonds) I had put in to create the structure, and started the structure generation search using only data justified constraints. It is difficult to describe the increase in the confidence in the result I experienced when, as frequently happened, the program produced a unique, or nearly unique result. Or the surprize when it produced an unexpected result completely consistent with the data. I regret not being able to show some results as in general the structures are proprietary.

The program is not perfect by any means. It struggles with aromatic ring systems, for example. Fortunately the experienced human can often see these possibilities and provide a helping hand in the form of user constraints. The point I want to make is not with regard to the value of NMRSAMS vs other CASE tools, but rather that providing experienced spectroscopists with tools that help them control the data that modern 2D NMR produces is very useful, while providing them with black boxes that tend to hide what is going on is pretty much worthless. I am very curious as to other peoples opinions.

Bruce D. Hilton

México, D.F. 07000

G. D. Searle

Opinions expressed are my own and not representative of G. D. Searle, the National Cancer Institute, or any other organization.

Please credit this submission to: Robert Dykstra



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#### HELP REQUEST-PAPER FOR ZETA 8 PLOTTER

Dear Professor Shapiro:

Please be so kind as to include in The NMR Newsletter our HELP REQUEST:

If any reader of the Newsletter knows where to purchase paper for Zeta 8 plotters, please inform to me.

Many thanks in advance

Pedro Joseph-Nathan pjoseph@nathan.chem.cinvestav.mx



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### Shift Assignment Errors in the Published Literature - the Impact on Computational Shift Prediction

January 6th, 1999 (received 1/22/99)

Dear Barry,

As you are well aware, our NMR shift prediction software has been available for over four years and has been well accepted by the marketplace. To date, we have prediction software and chemical shift assignment databases available for H1, C13, F19 and P31 nuclei. One of the common concerns for anyone considering the implementation of our software is both the quality of our prediction algorithms as well as the quality of the database which was used as the basis of our correlation algorithms and is updated on an ongoing basis with fresh data from the literature. As all NMR spectroscopists will be aware, there are "considerable" misassignment errors in the literature resulting from simple typographical errors, poor interpretation of experimental data and errors which have been carried through the literature when based on an initial poor assignment.

Our quality checking procedures include careful examination of the shift assignments and comparison with those entries already existing within our database. Following entry of the data into our database and two stages of typographical error checking we will then look for inconsistencies across the compiled database. In this way we have identified many thousands of poor assignments. Where necessary we will work with our collaborators to obtain fresh experimental data in order to clear up confusions.

One example of a substructure specific error which we have unearthed as a part of our database quality checking is shown below. We have noticed there have been a number of contrary assignments made for the quaternary aromatic carbons for alkoxy substituted tosylates of the form below:



For example, for the ethyl substituted system, the predicted C-13 spectrum is given in the table on the following page



Notice the large error in chemical shift (95% confidence interval) for C-1 and C-4 (6ppm and 5ppm respectively) compared to the small confidence limits for the other predicted chemical shifts. In general C-13 chemical shifts are predicted to better than +/- 3ppm with our C13 NMR prediction program. Within the program it is possible to directly view assigned fragments from within our database of 67,000 assigned structures. These fragments are shown within a Calculation Protocol window. The displays for both C-1 and C-4 are shown on the next page. As can be seen in each of the windows there are a number of representative fragments for both nuclear environments (each of the black squares represents a structure and by passing the mouse over a square displays the related structure and highlights the associated fragment in red). Consideration of the calculation protocol windows shows that for each of the two nuclei, C-1 and C-4, there are TWO distinct columns of related structures displayed, one around 132ppm and one around 145ppm. The conclusion from this data is that ongoing assignments for C-1 and C-4 have been confused in a number cases. These confusions have not arisen from one particular publication that we can identify. For example the shifts at ca. 132.30 for C-4 are were obtained from a number of sources including the following two references:

Tetrahedron 1992, vol 48, page 9753 Chem. Ber. 1979, vol 112, page 2815



Carbon-1 Calculation Shift Protocol

Carbon-4 Calculation Shift Protocol

(continued on P. 21)



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- Use first-order or higher-order interactions for prediction of the H,H J-resolved experiment
- Use heteronuclear couplings for all the experiments



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To use Advanced/Structure Elucidator, you will require a <sup>13</sup>C NMR spectrum. It is helpful (but not necessary) to have multiplicity information available from DEPT or APT experiments, and <sup>1</sup>H NMR and IR spectral data. Molecular weight and elemental composition data provide further refinement.

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In order to clarify and correct the appropriate shift assignment for such structures we obtained NMR spectra for Ethyl p-Toluenesulfonate using a combination of H-1, C-13, HETCOR and HMBC experiments we were able to produce the following corrected assignments (see the picture on the next page). The spectra were measured on a Bruker AM-300 at room temperature in CDCI3 solutions at 300 MHz for H-1 and 75.47 MHz for C-13, respectively. These corrections have been included in our prediction algorithms thereby impacting the predictions for such related structures in the future.

With this information we have been able to make assignments for the structures in the following references:

Tetrahedron 1990, vol 46, page 3061 Tetrahedron 1984, vol 40, page 905 Khim. Prir. Soedin., 1991, page 707 Helv. Chim. Acta., 1990, vol 73, page 2090

and have been able to change assignments in the literature including in Tetrahedron 1994, vol 50, page 11039.



This is not the only example of such a problem that we have identified with literature data and have had to resolve. However, it is an indication of some of the approaches we are taking to building a high quality and appropriate database to be used as reference data for prediction and searching by structure and substructure.

We welcome clarification from any of your readers of any literature assignments that they have noted to be incorrectly carried throughout the literature. Please forward any comments to me directly at tony@acdlabs.com.

Yours sincerely.

Antony Williams

P.S. Your readers may wish to take advantage of the FREE NMR viewer now posted at our website (www.acdlabs.com/download). This includes an integrated Structure Drawing Package, Chemsketch and can read and display Varian, Bruker and JCAMP spectra. JEOL format is presently being implemented.

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34,716-7	99.9%	Deuterium oxide	100g	screw-cap bottle	81.30
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(received 1/4/99) December 14, 1998

### NMR Cooler Interrupt System

Dear Dr. Shapiro:

We have a Bruker 500MHz DMX spectrometer equipped with a BCU 05 refrigeration unit that refrigerates the air supplied to the NMR probe to approximately -40C. In the probe, this refrigerated air is typically reheated to the desired working temperature via a heater in the probe. On our system, an aqueous NMR sample left in the probe can accidentally freeze when the air supplied to the NMR console and cooling unit is unexpectedly shut off (due to a compressor failure, e.g.) and then resumed. Such freezing results because a sensor in the console VT unit shuts off power to the probe heater once the air flow stops. Since this heater remains off even after the air supply resumes (unless manually turned back on), the probe air is quite cold since the BCU 05 stays on despite the fact that the probe heater is now off.

To avoid NMR sample freezing and tube fracture due to an air supply interruption, we designed an electronic device that monitors the supply air pressure and automatically shuts off power to the BCU 05 unit as well as air flow to the probe once the air pressure drops below a preset value. Below is a functional diagram of the device we have designed. We call it the "Cooler Interrupt System". It has been demonstrated to work successfully and is currently installed on our DMX-500 spectrometer. This device gives both an audible and visual alarm when triggered by a sudden loss of air flow. It requires a manual reset to turn the cooling unit and air flow to the probe back on. In general, cooling units similar to the Bruker BCU 05 are typically used to pre-cool the probe supply air in order to regulate the NMR probe and sample temperature. It is therefore likely that air supply failures of this sort can also potentially lead to NMR sample freezing and related problems with other, similarly configured NMR systems.

Further information and details regarding this device can be requested by contacting us at the address above or at the phone numbers and email addresses below. Please credit this to the account of Prof. Mark Rance.

Pearl Tsang 513 556-2301, pearl.tsang@uc.edu

Bob Voorhees 513 556-9297, robert.voorhees@uc.edu

Sincerely.

Elwood Brooks 513 556-9211, elwood.brooks@email.uc.edu

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### The NMR Newsletter - Book Reviews

Book Review Editor: István Pelczer, Dept. of Chemistry, Princeton University, Princeton, NJ 08544

### "Methods for Structure Elucidation by High-Resolution NMR"

#### Edited by

### Gy. Batta, K. E. Kövér and Cs. Szántay, Jr.

Elsevier, Amsterdam 1997, 357p, ISBN 0-444-82157-0, US \$244.-; (www.elsevier.com)

Reading the editorial preface of this volume I could not help but get suspicious. The editors aimed at "presenting 16 loosely connected papers,... different from basic educational texts, hard-core scientific papers and regular reviews" and with "slight overlap in the topics". What are we really getting after spending \$244, which is not a trivial amount of money? We do get formally 347 pages with an additional subject index; taking into account the generous layout with an empty cover page and back page for each article the largest chapter has 37 printed pages and the smallest just 5 pages. Checking further into the content of these 16 articles, I find that about the half of the chapters may essentially be considered as reprints from the original literature of the authors. A lot of the articles deal with selective pulses and there is not only "slight" but considerable overlap. Although in the editorial it is claimed that much emphasis has been given to pulsed field gradients, I find only two contributions, which address them. Thus, from a first glance through this book I have to admit I was very disappointed.

There are, however, some very nice short reviews or personal accounts in this book, which are indeed worth reading. Some of these really make the impression of having been newly written for this particular volume, e.g., the report by G. Otting on high power spin-lock purge pulses. Also very well written is the account on chemical exchange measurements by A. Bain and G. J. Duns, who address specifically the regions outside the traditional line shape measurements. Finally, I might mention the review on ROESY type measurements by Jurani, Zolnai and Macura, which is a timely report on the pertinent questions.

Thus, this volume leaves for me a very mixed impression. Duplication of known features interleaves with some more original reports. Why put this together in a rather expensive book? There is no visible editorial concept with respect to NMR methods or the chemistry involved. In times of severe tightening of library budgets publishers must adopt a different attitude towards scientific publishing.

#### **Stefan Berger**

Institut für Analytische Chemie Universität Leipzig Linnéstr. 3 04103 Leipzig Germany.



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BROOKLYN CAMPUS UNIVERSITY PLAZA, BROOKLYN, NEW YORK 11201



(received 1/21/99) January 19, 1999

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

#### A Reliable and Quiet Air Compressor That Can Handle the NMR and Other Air Requirements

Dear Barry,

In 1997, Long Island University took possession of a Varian INOVA 400 MHz NMR, obtained through NSF grant HRD 9628573. As originally configured, this machine was supplied air through a Jun-Air 12-20 (1HP) quiet (49 dB) oil based compressor, with the feed air being dried by a combination of Balston oil/particulate filters and a Balston membrane filter. After much use, this system proved to be very high maintenance, especially with regard to oil mist contamination. Therefore, as part of a large, multi-instrument Department of Defense grant (F49620-98-1-0464), a new, oil free scroll type compressor was purchased. This system was designed to not only supply air to the NMR, but also to eliminate the need for compressed air for all instruments in the LIU instrumentation facility.



The compressor system is graphically represented above and contains the following components. The heart of the system is a 5 HP Powerex model SES050821 scroll compressor (A). This compressor delivers 14 cfm at 100 p.s.i. while generating less than 50 dB of noise. This rate of supply is more than four times the air flow required to perform solid state NMR, leaving ample capacity for the remainder of the machinery. The scroll unit itself is fully enclosed, with the enclosure containing the compressor, a ten gallon expansion tank, and a magnetic starter valve which is preset to turn on at 90 p.s.i and off at 110 p.s.i. The air is dried to a dew point of  $-100^{\circ}$ F through a combination active/passive system. After leaving the expansion tank, the air passes through a Deltech model HX25 refrigerated air dryer (B). This component, which has a capacity of drying up to 25 cfm, reduces the air to a dew point of  $-40^{\circ}$ F. The air then passes through a Deltech model D0020 CF coalescing filter (C) which removes any condensation, as well as particulates. After

this, the -100°F dewpoint is achieved by passing the air through a Deltech model HB51-6 heatless desiccant system (D). This dessicant unit consists of two twenty four inch long, eight inch wide, canisters containing a mixture of activated alumina and molecular sieves. These tandem desiccant beds cycle automatically at three minute intervals. At all times, one cartridge is absorbing moisture from the inlet air while the other is regenerated by a portion of the dry air from the active bed. No heating is required in this process. The final purification is achieved by then passing the now dry air through a Deltech D0020PF (E) which is designed to remove any particulate dust from the desiccant bed at a flow rate of up to 20 cfm. The air then passes into an eighty gallon holding tank (F), which can be isolated at both ends and contains a 200 p.s.i. pressure relief valve as well as a pressure gauge. The unit contains one-way check valves between each component to prevent back-flow damage. In addition, each leg of the tee'd outlet is also protected by a check valve. Finally, a 2.5 HP Thomas oil free compressor, in place as an emergency back-up, is mounted atop the main storage tank and is connected by means of a tee to the drying system before the refrigerator.

This compressor system has been operational for six months without a breakdown. Besides supplying air to the NMR, it also supplies air to three gas chromatographs, a TGA/DTA/DSC thermal analysis cluster, a GC/MS, an HPLC, an AA, and an FTIR whose optical bench requires a constant purge of dry air. The use of this system has eliminated the need for no less than seven compressed air tanks, a fact which not only saves money but also greatly improves the aesthetics of the instrument room. While performing all of these tasks, the compressor maintains a leisurely 11% duty cycle. In addition, the system is much more compact than the schematic indicates. The compressor itself has a footprint of approximately 3' x 2'. The refrigerator is approximately 18" on a side and all of the filters and desiccants are mounted on a dexion frame above the refrigerator, saving additional space. Routine maintenance consists of draining the ten gallon expansion tank daily, dumping the condensation bucket weekly, and lubricating the compressor every 500 hours of operation. In addition, tests have indicated that the back-up compressor, which is actually stripped from a portable pneumatic station, can handle the same load, albeit at a 50% duty cycle and nearly four times the noise.

All plumbing consists of 3/8" copper tubing and there is a pressure regulator at each station. The original Balston membrane filter remains in the loop, as it was already wall-mounted in the NMR room. The entire compressor system as assembled cost approximately \$7,000 and took about 2 days to set up and plumb. Also, it should be noted that the placement of the main receiving tank after the desiccant eliminates the possibility of rust, thereby extending the life of the tank and greatly simplifying the task of removing particulate.

Below are the addresses of the vendors mentioned in this letter:

Powerex, Inc: 150 Production Drive, Harrison, OH, 45030; (800) 544-0350

Deltech, A United Dominion Co., 344 Churchmans Road, P.O. Box 667, New Castle, DE, 19720-0667; (302) 328-1345

Balston, Inc: 260 Neck rd., Box 8223, Haverhill, MA 01835; (800)343-4048

Sincerely.

Fernando Commodari, Ph.D. Fern@hornet.liunet.edu

Edward J. Donahue, Ph.D. Edonahue@hornet.liunet.edu

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Deadling	e Dates
No. 486 (Mar.)	19 Feb. 1999
No. 487 (Apr.)	26 Mar. 1999
No. 488 (May)	23 Apr. 1999
No. 489 (June)	21 May 1999
No. 490 (July)	25 June 1999

- \* 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.
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#### Forthcoming NMR Meetings, continued from page 1:

"Applications of NMR to Complex Systems", Symposium at the American Chemical Society Meeting, New Orleans, LA, August 22-26, 1999; Contact: R. E. Botto, Symposium Chair, Chemistry Division, Argonne National Laboratory, 9700 South Cass Ave., Argonne, IL 60439; 630-252-3524; Fax: 630-252-9288; Email: robert\_botto@qmgate.anl.gov

<u>41st ENC (Experimental NMR Conference)</u>, 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.

Additional listings of meetings, etc., are invited.

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