bruker avance 400 mhz nmr spectrometer manual

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Book Descriptions:

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The system we chose is a Bruker Avance II 400 MHz spectrometer system. The magnet is a 9.4T superconducting solenoid operating at a nominal proton frequency of 400 MHz. The magnet is actively shielded to produce a minimal magnetic footprint. The cryostat is an ultralong hold dewar with a nominal 180 day fill interval. It has exactly the same dimensions as our prior AC250 magnet and because of the active shielding, the magnetic footprint is also identical. The new spectrometer was installed in the spring of 2006, following decommissioning of our old AC250 system and renovation of S034A where it is now housed. The AVII400 is managed by Dr. Richard Fitch. The probe is broadbanded with automatic tuning and matching capability and pulsedfield gradient capacity. This facilitates remote operation and automation see below, as well as gradient and simplex shimming. The spectrometer is a fourchannel zgradient system, which is capable of the complete suite of 1D3D experiments not requiring horizontal gradients. The sample changer allows us to make maximum use of our instrument as it removes the need for the researcher or student to be present to place the sample in the magnet. This benefits researchers who may have large numbers of samples or need long acquisition times. It also benefits our laboratory course students, who can acquire highresolution spectra, including 13 C and 2D experiments, which are often not run due to time constraints. MaryoftheWoods College, and RoseHulman Institute of Technology. This allows students and researchers at these institutions convenient access to the instrument. These spectrometers are Bruker Avance AVII,AVIII, DMX and DRX series NMR spectrometers and all these are currently running BrukerTOPSPIN software.It has a Bruker US2 shielded magnet and Bruker Avance DRX console with three RF channels suitable for triple resonance experiments.http://decamiones.com/userfiles/car-service-manuals-for-sale.xml

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It has a triple axis gradinet amplifier and equipped with 5mm triple resonance 1H, 13C and 15N probe head with ATMA and Zgradient coil. This instrument is most suitable for structure determination of biomacrmolecules. It has a Bruker US2 shielded magnet and Bruker Avance DMX console with three RF channels suitable for triple resonance experiments. It has a triple axis gradinet amplifier and equipped with 5mm inverse broadband BB, 15N31P probe head with triple axis gradient coil.This system also has a 5mm BB observe ATMA probewith Zgradient,and a 10mm BB observe probe. This instrument is most suitable for structure determination of organic and inorganic small and macromolecules It has an Oxford unshielded magnet and Bruker Avance DRX console with three RF channels suitable for triple resonance experiments. It has a triple axis gradinet amplifier and equipped with 5mm QNP 1H, 13C, 19F and 31P probe head with zgradient coil. This instrument is most suitable for structure determination of organic and inorganic molecules and polymers. It has an Oxford unshielded magnet and Bruker AV II console with three RF channels suitable for double and triple resonance experiments. It has a zgradinet amplifier and equipped with 5mm BB observe ATM probe head with zgradient coil.It runs TOPSPIN 2.1 software on a linux workstation. This instrument is most suitable for structure determination of small organic molecules. It has an Oxford unshielded magnet and BrukerDMX console with Three RF channels suitable for double resonance experiments. This instrument is most suitable for structure determination of small organic molecules. It has a unshielded Oxford magnet and two chanel console.It runs TOPSPIN 2.1

on a linux workstation It has a Bruker shielded magnet with Bruker AVIII console and running TOPSPIN 2.1 sofware on pc workstation. It is a two channel spectrometer with gradient capability. This instrument is primarily used for 11B NMR.http://viaggi.abruzzo.it/img/car-service-manual-free-download.xml

It is an open access spectrometer to the users specially permitted to use this instrument. It has a Bruker unshielded magnet with Bruker Avance DMX console and running TOPSPIN on pc workstation. This is an open access spectrometer is used primarily for routine structure determination of small molecules. It has an Oxford unshielded magnet with Bruker Avance DMX console and running TOPSPIN 1.3 on a pc workstation. It is a two channel spectrometer without gradient capability. This is an open access spectrometer is used primarily for routine structure determination of small molecules. It is the ideal machine for ultra high sensitivity 13C and 1H experiments where sample quantity is limited. Connect to a qualified service provider using LabX Service. Use our directory to find and contact a service specialist. Technological innovations, prominent manufacturers and popular equipment all in one place. View All Applications Shop ReSellers Shop Featured ReSellers Shop All Stores Resources Resources, Guides and Articles Learn about equipment technologies and science in our resource center. Browse articles and infographics to get the latest industry insights. Topics Buying Guides Cannabis Laboratory Chromatography Infographics Mass Spectrometry Product Review Reasons to Upgrade Technical Insight View All Featured Infographics Featured Resources Auction Events Auction Events Check out upcoming equipment auctions on our event calendar. Score liquidation pricing on an incredible assortment of products. View All Applications Resources Resources, Guides and Articles Learn about equipment technologies and science in our resource center. The novel NanoBay design puts Bruker’s highperformance Avance III NMR spectrometer technology into an exceptionally compact enclosure.

It delivers high productivity and highestquality NMR information for pharmaceutical and industrial chemists, for academic research and teaching, as well as for food analysis, diagnostics research and other small molecule applications. Bruker NMR systems has a product line that includes Fourier, NanoBay and AVANCE spectrometers. These are quality products manufactured by a trusted name in NMR. Browse ads and contact the seller directly or request a quote for more details. Buy and Sell Bruker Equipment and Accessories today on LabX. Up for sale is a complete Bruker DPX300 NMR Spectrometer with Avance 300 Power Supply and control system. The magnet was deenergized about five years ago. All of the kits for moving the magnet are included. This is a 2channel system with a solids accessory rack with high power 1H and X transmitters. It operates under XWINNMR from a Linux workstation. It is used, good for parts, and is being sold asis with no warranty. We encourage you to contact us to speak with one of our expert and professional team. The sample injection valve is powering on. We do not have the ability to test this unit further. Please see photos for details. What you see pictured is exactly what you will receive. Shipping Attention Buyers!Please note that this item is quite large and will need to be shipped by freight. Please contact the seller at for a shipping quote as the shipping costs are not included in the purchase price. Shipping Attention Buyers. Please note that this item is quite large and will need to be shipped by freight. Attention International Buyers. We will not state a price on customs or shipping forms other than the actual final bid price. We ask our customers not to make such requests as these requests cannot be honored. Please bid accordingly. The sale of this item may be subject to regulation by the U.S. Food and Drug Administration and state and local regulatory agencies. If so, do not bid on this item unless you are an authorized purchaser.

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not meet your expectation in certain areas of the service, please kindly send me an email on how you feel and how i can improve that area of service. You comment and advice is very much appreciated. I strive to earn a 5 star feedback from you. Designated trademarks and brands are the property of their respective owners. Use of this Web site constitutes acceptance of the LabX User Agreement. The SmartProbe does both direct and indirect detection well and allows pulsing on any nucleus in the group 15 N 31 P, 19 F on one channel and 1 H on the other channel. This instrument is also equipped with Bruker SampleXPress casette autosampler with 60 positions, and operated by IconNMR.

To utilize the instrument, obtain training and an account from Dr. Teymoori. Many pulse sequences are available for one and twodimensional spectra. Experiments are acquired near room temperature only no VT operation. This instrument is fully capable of performing any 1D or 2D NMR experiment possible on a 2channel NMR spectrometer The instrument is equipped with a single 5mm BBFO probe, with Variable Temperature capability. This means that you can setup any sample or samples to acquire multiple broadband nuclei, with no user intervention required in terms of probe tuning or any changing of cables. Samples can be submitted on a firstcome, firstserved basis.This is our most sensitive instrument for 19F and 31P. The user can use offline processing software, such as MestReNova, to analyze the data wherever they are. Note we have a campus sitelicense for MestReNova. There is also a broadband probe tunable to nuclei in the NMR frequency range between 187 Os and 31 P, which we use for multinuclear measurements. This probe has no gradients and no autoswitch from one nucleus to another. Therefore only manual operation is possible after changing from the QNP probe. There is a 24position sample changer. By continuing without changing your cookie settings, you agree to this collection. For more information, please see our University Websites Privacy Notice. The system is controlled by a PC Windows computer operated by Bruker Topspin 1.3 software allowing for 1D, 2D and 3D NMR experiments This instrument also has a double resonance 5 mm multinuclear broadband probe for liquid samples. The system is controlled by a PC Windows computer operated by Bruker Topspin 1.3 software allowing for 1D and 2D NMR experiments The system is controlled by a PC Linux computer operated by Bruker Topspin 3.0 software allowing for 1D, 2D and 3D NMR experiments. We recommend you update your browser Chrome Firefox Internet Explorer Safari.

It is used for VT, reaction monitoring and heteronuclear NMR experiments.It is used for extended variable temperature and heteronuclear NMR experiments in additon to regular proton and carbon ones.It is fully automated with a SampleXpress autosampler. It is located at B530 of Silverman Hall.In addtion, it has ultrahigh 19F sensitivity, the best around Chicago area.It is dedicated for highthroughput application. All the heteronuclear NMR experiments can benefit from the high sensitivity and convinience of the SampleXpress auto sampler.It also has wide variable temperature capability. We have two solidstate probes on this system. The Bruker 4 mm HX probe is for most of route work. The Phoenix 1.6 mm HFX probe can be used for 19F and 1H with spinning rate up to 40K Hz. Only tubes rated for 400 MHz or higher may be used with this instrument. We recommend Wilmad 507PP7 or equivalent tubes. Lower quality tubes can damage the instrument. For this reason, those found using lower quality tubes such as disposable tubes will have their accounts suspended. During the hours of 9 AM 6 PM, only short experiments i.e., within one working day; individuals repeatedly leaving samples behind will have their accounts suspended, as this is a safety hazard.Only tubes rated for 600 MHz or higher may be used with this instrument. We recommend Wilmad 535PP7 or equivalent tubes. The same restrictions about lowquality NMR tubes as stated above apply to this instrument. When requesting a reservation of the 600 MHz instrument, please specify the probe you would like to use, the experiments you intend to conduct, and your sample type. Note that some justification as to why the 600 MHz instrument may be requested, as opposed to the 400 MHz instrument for apparent routine sample requests, may be required. NMR reservations must be canceled at least 24 hours in advance to avoid being charged for the

reservation Please see the training rates. NIHMS754144supplementSupplemental.

docx 195K GUID 406CD3BFF1544E848A4CAF0056948AD5 Abstract We have investigated and compared a number of sample conditions on different NMR platforms in the search of maximum SNR and optimal experiment time efficiency for structure elucidation and quantitation of natural products. Using restricted volume 3 mm Shigemi microcell assembly in conjunction with a 900 MHz NMR spectrometer equipped with a 5 mm carbonsensitive inverse cryoprobe, it was possible to achieve a substantial increase in SNR 46fold as compared with a conventional room temperature 400 MHz instrument. Switching from standard 5 mm NMR tube to 3 mm Shigemi microcell assembly typically improved SNR by threefold on either 600 or 900 MHz cryoplatform. A quantitation method that relies on a calibrated residual protonated NMR solvent signal as internal standard was developed using the same hardware setup and restricted sample volume tubes. Linearity of the method spans over 3 orders of magnitude, from low microgram to milligram quantities. Over the past several decades many technical improvements have been introduced to increase the intrinsic NMR sensitivity optimizing the sample volume for specific availability or solubility of a given sample, increasing the external magnetic field strength currently standing at 23.5 T, and optimizing the probe quality factor Q factor and RF coil geometry. Restrictive volume NMR tubes are readily available from Shigemi Inc.Microcell assemblies, consisting of a glass tube and a plunger, reduce the solvent volume by approximately half for the same tube diameter. Glass material matched to the magnetic susceptibility properties of the solvent is used to fill the bottom compartment of the tube as well as the plunger. In addition, lowabundant samples microgram or nanogram are difficult to analyze by classical analytical methods.

Unless thoroughly dried or handled in an anhydrous environment, residual solvent or moisture may contribute to inaccuracies in the determination of sample weight. Herein, we present a study of the sensitivity enhancement achieved using reduced diameter and restricted volume tubes when applied to standard 5 mm diameter cryoprobes, as well as a quantification method for samples that cannot be accurately measured using a microanalytical balance. The quantitation method in restricted volume tubes is based on comparison of integrated intensity of the residual proton signal DMSO d 5 in DMSO d 6, in this particular case to the protons of the dissolved analyte in order to determine the concentration in solution. The demonstrated method has a linear response over 10 3 fold concentration range and limit of detection in nanomolar range. Experimental section General experimental procedures 1 H NMR spectra were recorded on a Bruker Avance AV 400 equipped with BBO ATM 5 mm Zgradient probe, Avance DRX 600 equipped with CPTXI 5 mm Zgradient probe, and Avance AV 900 spectrometer equipped with CPTCI ATM 5 mm Zgradient probe. The same batch of NMR solvent DMSO d 6 99.96% D, SigmaAldrichFluka, Corp. was used for all measurements. Restricted volume 3 and 5 mm NMR microcell assemblies were purchased from Shigemi, Inc. Standard 5mm tubes and 3mm tubes were from WilmadLabglass 535PP7 and 335PP7, respectively, purchased through SigmaAldrichFluka Corp. All spectra were collected in triplicate. Standard solutions of paclitaxel in DMSO d 6 for qNMR measurements were made by serial dilutions of stock solutions prepared by dissolving accurately weighed samples of paclitaxel 1.40, 2.50, and 4.80 mg in the calculated volumes of DMSO d 6. Selection gradients GPZ1 and GPZ2 were set to 28 and 20% of maximum gradient power. Free induction decays were processed in XwinNMR 3.5 or Topspin 1.3 as follows Fourier transformation with exponential apodization of 0.

30 Hz, followed by manual phase correction and autobaseline correction fifthorder polynomial fit. Gradient selection in DEPTQ was set to detect all carbon signals. In particular, we were interested in obtaining further sensitivity gains by switching to smaller diameter NMR tubes, especially on the 900 MHz system equipped with 5 mm carbon sensitive cryoprobe CPTCI, and how these compared with the 600 MHz cryoplatform and 400 MHz room temperature hardware setups. This corresponds to a reduction in data collection time by more than 2000fold, for a sample of the same mass. This

gain was derived purely from the difference in magnetic field strength and possible small differences in probe construction the 600 system cryoprobe was rebuilt in 2007, while the 900 probe was among the first ever built. When using 5 mm tubes, the 600 MHz platform appeared to be as sensitive as the 900 MHz system. With the room temperature probe, the filling factor dominated resulting in actual decrease in SNR, as observed for 3 mm tubes in 400 MHz room temperature system. We examined its possible application with 5 mm cryoprobes. A typical sequence of NMR experiments used in structure elucidation consists of a COSY, TOCSY, HSQC, and HMBC experiment, or their variants. Direct 13 C spectra may be collected, if concentration permits, to augment data gathered from the inverse experiments. Recorded on a Bruker Avance AV 900 spectrometer equipped with a 5 mm TCI triple resonance inverse cryoprobe using DMSOmatched 3 mm Shigemi microcell assembly. Data were linear predicted in the second frequency domain to 128 points. Data were linear predicted in the second frequency domain to 512 points. Open in a separate window Figure 4 1D F2 slices of the 1 H 13 C HMBC spectrum in Fig. 3, with SNR values for selected signals at 5.39 ppm top and 1.80 ppm bottom. Noise region was set to 2 ppm. Open in a separate window Figure 8 1D F2 slices of the 1 H 15 N HMBC spectrum in Fig.

7, with SNR values for selected signals at 6.20, 3.92, 3.27, and 2.42 ppm. The majority of the isolated samples we encountered in our research were soluble in DMSO and hence could be analyzed using DMSO d 6. DMSO is a versatile solvent not only for the purposes of recording NMR spectra but also for various wellplate bioassays kits. The use of 0.25 ml ampules, in our case, minimized the overall amount of water residue introduced in the samples. Residual solvent peak was reduced twofold to threefold compared with 99.9% D solvent, reducing potential dynamic range problem. The solvent peak still dominated the spectrum and with proper shimming 13 Csatellites were well distinguished from the baseline. However, we decided to exclude them in the process of peak integration. This translated into a need to determine the exact concentration of DMSO d 5 in each batch of solvent used for qNMR determinations. The concentration of DMSO d 5 in DMSO d 6 was determined by construction of a calibration curve using a known standard. Commercially available pyrimidine was used as internal standard for this purpose. As a liquid miscible with DMSO, it was easily transferred via an automatic pipette into an NMR tube. The signal of choice H2, singlet, appeared in a less crowded region of the spectrum, therefore permitting more accurate integration. Using the integral values of the residual DMSO d 5 resulted in a similar linear response curve Fig. 9 . Subsequent quantitations were carried after the DMSO batches had been calibrated with pyrimidine solution. This reduced the dynamic range problems introduced by large pyrimidine signals in dilute sample solutions. The method can be used for concentration measurements spanning 3 orders of magnitude. Open in a separate window Figure 9 NMR detector response recorded for a range of concentrations of paclitaxel standards solutions in DMSO d 6 with pyrimidine as internal standard, prepared in 3 mm NMR tubes.

At the same time, this sample was used to investigate the lower limit of detection on the 900 MHz cryoplatform. Both limit of detection and limit of quantitation are functions of the magnetic field, as the shape and the sharpness of the peaks greatly affect the quality of the integral measurement; i.e. the higher the resolution of the spectrometer, the lower the limits of detection and quantitation Table 3. Due to their separation from other signals in the spectrum and strong signal intensity the following methyl singlets were selected for quantitation of the paclitaxel sample Table 4 and Fig. S4 methyl group H 3 18 1.796 ppm, br s, 3H, methyl group H 3 19 1.511 ppm, s, 3H, and acetyl methyl group H 3 10OAc 2.235 ppm, s, 3H. We determined the T 1 relaxation times for all methyl groups selected in order to evaluate potential impact on the integral values measured. Besides relaxation, integration procedure has crucial influence on the method’s accuracy for these lowconcentration samples. We removed inaccuracies usually introduced by manual integration by using the builtin integration routine. Sample loss during recovery and transfer into Shigemi microcell assembly may explain the difference between these two methods. We further investigated the method robustness

by introducing small changes in sample height with 3 mm Shigemi microcell assembly. The sample length dictated by the coil length was 21 mm; thus, the plunger was held in place by Teflon tape. Changes in positioning of the plunger can be introduced while handling the assembly, even unknowingly. Reproducibility of the method was assessed by performing measurements in triplicate for all of the calibration samples Table 2 . Switching from a regular NMR tube to a Shigemi microcell assembly did not to affect the reproducibility Table 3 . A method for 1 H qNMR measurements was developed based on the residual protic solvent as internal standard in the same reduced diameter NMR tubes.

The method was able to accurately detect lowmicrogram quantities of DMSOsoluble material, with potential to expand to other solventmatched Shigemi microcell assemblies. Accurate quantification of samples of isolated natural products can be used as an early selection tool prior to pursuing full data acquisition on the 900 MHz platform. For protonated carbons SNR is between 100 and 140. Supplementary Material Supplemental Click here to view. 195K, docx Acknowledgments The authors would like to extend our thanks to Dr Benjamin Ramirez Director of the Center for Structural Biology NMR Facility at the University of Illinois at Chicago for generous access to the instrumentation. Support for the ultrahigh field NMR Facility comes from NIH P41 grant GM68944 Dr Peter G. W. Gettins. This research project is supported by NIH grants RO1GM075856 and PO1CA125066. Footnotes Supporting information Additional supporting information may be found in the online version of this article at the publisher’s web site. Martin GE, Hadden CE. Ernst RR, Bodenhausen G, Wokaun A. Principles of Nuclear Magnetic Resonance in One and Two Dimensions. Rabenstein DL, Keire DA. In Modern NMR Techniques and Their Application in Chemistry. O’Neill IK, Pringuer MA, Prosser HJ. Farrant RD, Hollerton JC, Lynn SM, Provera S, Sidebottom PJ, Upton RJ. Wani MC, Taylor HL, Wall ME, Coggon P, McPhail AT. Krunic A, Mo SY, Orjala J. SMASH NMR Conference; Santa Fe, NM. Martin GE, Hilton BD, Blinov KA. Liu Y, Green MD, Marques R, Pereira T, Helmy R, Thomas Williamson R, Bermel W, Martin GE.