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Updating IUPAC spectroscopy recommendations and data standards

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International standards need to keep pace with the innovation in analytical equipment and practices. For example, many of the advances in nuclear magnetic resonance (NMR) spectroscopy reported in this journal in recent years have yet to find themselves mirrored by updates in the respective Recommendations of the International Union of Pure and Applied Chemistry (IUPAC), nor in the many and varied proprietary recommended reporting formats of the different peer-reviewed primary scientific journals. Not that every innovation needs to be "standardised": with the speed of many developments it is important to find the right balance between reacting to real movements in a field and enshrining a short-lived fad in a IUPAC Recommendation.

Background

IUPAC Recommendations in the field of nuclear magnetic resonance (NMR) spectroscopy fall into two main categories. First, there are the international data standards related to NMR in the series of JCAMP-DX data formats[1–10] and, second, the Recommendations on nomenclature and presentation of NMR data such as those by Robin Harris *et al.* in 2001 and 2008 and earlier.[11–14]

These Recommendations deal with standardising practises across a whole field which often become such common usage that practitioners can forget or not even realise why they should carry out a very important task in a particular way. The 2001 Recommendations, for example, standardised the use of the reporting of NMR chemical shifts of all nuclei relative to the ^{1}H resonance of tetramethylsilane (TMS) in dilute (<1%) solution in chloroform. They redefine The Chemical Shift δ to better avoid ambiguities in previous definitions and deal with some of the practicalities of using a unified scale: a critical pre-requisite for generating reference databases. They also published very useful tables of the various spin ½, quadrupolar and lanthanide/actinide elements with reference compounds drawn for various literature sources. The focus was very much on solution-state NMR with only a short section on magic angle spinning and solid-state NMR spectroscopy.

In 2008, recent work on the effects of temperature on the TMS signal and more importantly different solvents were addressed and new recommendations published. These included a reference table of the changes in the ^{1}H chemical shift in TMS with various common solvents. This table quoted figures mostly to two decimal places and sometimes only one significant figure. Reporting and referencing of chemical shifts in solids based on high-resolution MAS experiments were dealt with in much more detail.

The December 2013 ACS guidelines for publishing spectra[16] include:

- Field strength should be noted for each spectrum, not as a comment in the general experimental section.

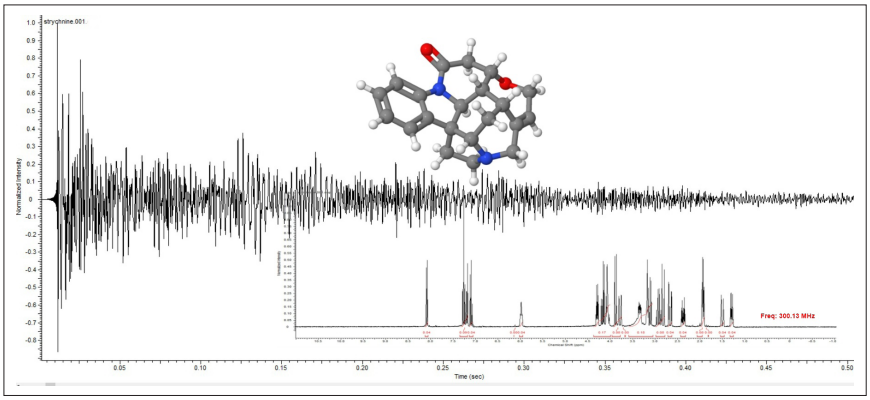

Figure 1. Free induction decay (FID) and ^{1}H NMR spectrum of strychnine, processed using ACD/Labs free software.[15]

- The largest peak in the ^{1}H NMR spectrum should normally arise from the compound, not the solvent.
- All peaks in the ^{1}H NMR spectrum should be integrated.
- Chemical shift values should be included.
- The solvent peak should be clearly labelled on the spectrum.
- All peaks should be visible on the spectrum. Insets are encouraged to show expanded regions.
- At minimum, the spectral window should be –1 ppm to 9 ppm for ^{1}H NMR and –10 ppm to 180 ppm for ^{13}C NMR.

Fortunately for us, the vendors do not stand still and with much higher field strengths becoming available to us (e.g. Reference 17) the precision with which we are able to measure chemical shifts has somewhat outgrown that reported in the existing suite of IUPAC Recommendations.

NMR data file converters

In general, data collected from spectrometers are in the form of free induction decays (FIDs) and if a spectrum is stored at all it is in addition to the FID and not as the sole piece of information. The design of the experimental parameters to collect the data and the processing software to display a spectrum are complex and can vary depending on the instrument manufacturer. Storing a spectrum can therefore be useful for archiving and data exchange between sites with different equipment.

File converters have been created by various vendors[15,18] (for example, see Figure 1) to deal with the lack of up-to-date, simple, well-documented file exchange formats.[19–21] In addition, some spectroscopy software packages have the ability to read a variety of native formats.

NMR data transfer standards

There is currently interest from IUPAC in updating the JCAMP-DX NMR data standards. This is especially relevant for the multi-dimensional NMR standard which was implemented essentially from the distributed final draft when the responsible subcommittee moved to supporting the AnIML initiative at ASTM.

The most recent standard for 1-D NMR is the JCAMP-DX version 5.01 recommendation of 1999.[6] This was released with changes needed to cope with Y2K issues and for conformity with good laboratory practices (GLP), amongst other things.

Volunteers sought!

The draft JCAMP-DX version 6 that dealt with multi-dimensional NMR was released for comment but never finalised. According to the AniML team, there is still no timeframe for development of protocols for *n*D NMR. In the meantime the decision to complete and publish the version 6 standard seems appropriate, but this would require updating with input from both instrument companies and researchers with experience in handling *n*D NMR spectra.

Clearly, volunteers with experience in handling *n*D NMR data and willing to help update the draft for publication will need to be recruited. If you think you can contribute, please contact the authors.

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