Natural Product Reports

SUPPORTING INFORMATION

# Dissemination of Original NMR Data Enhances Reproducibility and Integrity in Chemical Research

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## SI-1. The NMR Free Induction Decay (FID): Generation and Information Content

In modern NMR, the measured signal arises from the current induced in a coil surrounding the sample, produced by radio frequency excited nuclear spins of the molecules. This Free Induction Decay (FID) comprises the oscillating signals for each individual magnetic moment arising from the precessing magnetic dipoles that lose phase coherence and decay over time to their non-excited state (relaxation). FIDs represent a superposition of decaying sinusoids in the time domain, and this data can be converted into the frequency domain by a Fourier Transformation (FT), yielding an NMR spectrum.

The frequencies, numbers, and amplitudes of the spectral lines of each nucleus depend on its chemical environment: electronic, geometric, and spatial. The highly sensitive interaction of the individual nuclear spins within the molecular spin system(s) creates a unique pattern that can be uni-determinant for the underlying chemical structure, notably in the context of the micro environment of the sample. This applies generally to all FT-NMR experiments, regardless of observed nucleus and dimensionality, as the most widely used 2D NMR data are mere assemblies of FIDs acquired with different experimental parameters.[1]

## SI-2. FID Post-acquisition Processing

Regardless of the experiment (detected nuclei, dimensionality), the FID contains all the information present in an NMR spectrum. In the case of the classical 1D $^1$H NMR experiment this information consists of the chemical shift ($\delta$), signal intensity, coupling constants ($J$), line width ($\omega_{1/2}$), phase, and line shape. In fact, each point of the FID has information on each of the data points of the spectrum. Post-acquisition processing allows transformation of the FID into an interpretable form and gives access to its informational richness. FID treatments such as zero-filling, apodization functions, phasing, and baseline correction may optimize the quality and/or enhance certain characteristics of the spectrum.[1]

Such FID processing may be performed by various third party software that offer both automated and hands-on operation, yielding NMR spectra for visual interpretation. Advanced extraction of the NMR spin parameters up to the level of quantum mechanical (QM)-based NMR full-spin analysis requires additional tools.[2]

Despite the availability of inverse FT, a processed spectrum cannot recreate the *original* FID as some information contained in the originally acquired is irretrievably lost during most post-acquisition processing. This emphasizes the special value of the original FID data, including 2D serial files. At the same time, it should be pointed out that post-acquisition processing is a powerful step in the entire NMR workflow, which empowers the analyst to extract the maximum of information from the data. Thus, the optimal combination consists of the preservation of original data and their well-documented processing.

## SI-3. FID Archiving as a Means of Enhancing Structural Correctness

While it is difficult to determine the effect that FID archiving would have had in the past on mitigating misassigned structures, Nicolaou's[3] report with 50+ tabulated and discussed cases and numerous citations indicate the dimension of the issue. In addition to limitations pertaining to the general accessibility of legacy raw NMR data, the ability to assess past examples also depends on data quality, as it impacts usability for making accurate distinctions. Conversely, for the purpose of moving forward with the proposed FID archiving, producing quality data must also be emphasized. The case of aquatolide is exemplary as raw data from the original 1989 work were unavailable and published figures inconclusive for assessing the congruence with the re-isolated material, yet confirming the (need for) revision was possible because FIDs were available.[4]

## References

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