

NOAH-NMR Supersequences with Nested Acquisition for Small Molecules

Product used : Nuclear Magnetic Resonance (NMR)

NOAH (NMR by **O**rdered **A**cquisition using ^1H -detection) [1] is a group of nested NMR experiments combining several conventional two-dimensional (2D) NMR pulse sequences, such as COSY, HSQC and HMBC, into one supersequence. Therefore, two or more 2D NMR data can be obtained from a single NOAH experiment. By using a single relaxation delay, the NOAH method significantly reduces the total data collection time and increases the throughput of an NMR instrument in structure elucidation of small organic molecules.

NOAH Supersequence

Figure 1 is a schematic representation of a conventional 2D NMR measurement, and Figure 2 shows the principle of a NOAH supersequence. All conventional 2D NMR sequences contain a recovery delay (relaxation delay) in the order of seconds, which is the longest part of the sequence. If two or more conventional 2D experiments are run in a sequence, each experiment needs to have a recovery delay (Figure 1). In the NOAH supersequence, only a single recovery delay is employed at the very beginning of the nested sequence (Figure 2). As a result, the total data collection time of the NOAH experiment is much shorter in comparison to the classical 2D data collection scheme.

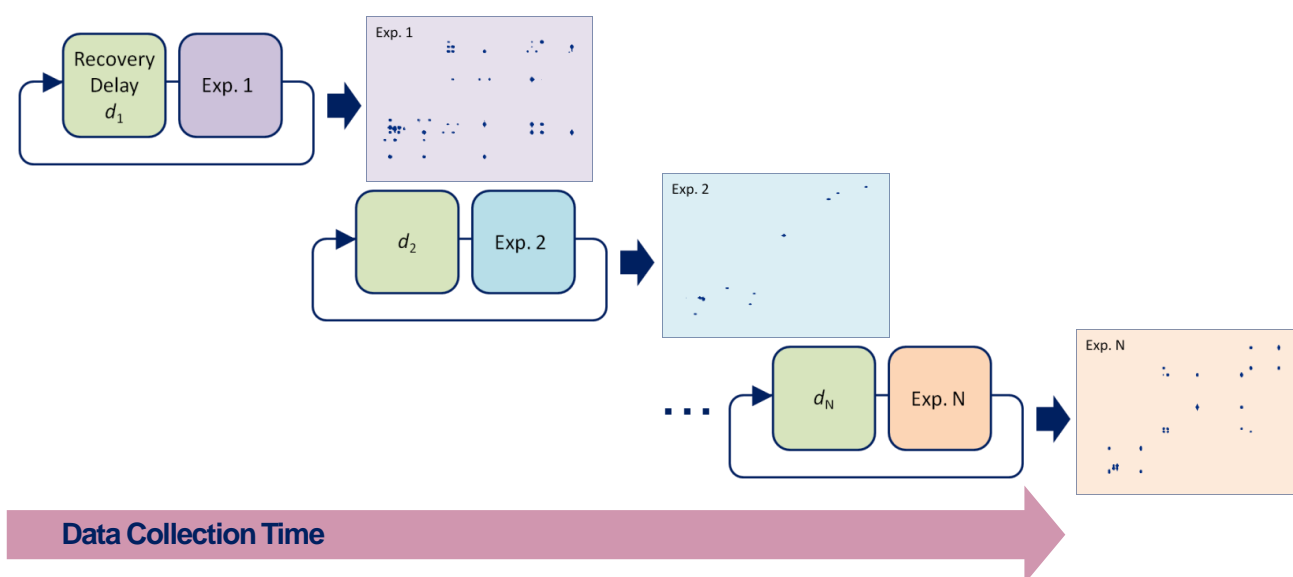


Figure 1. Schematic representation of a series of conventional 2D NMR measurements

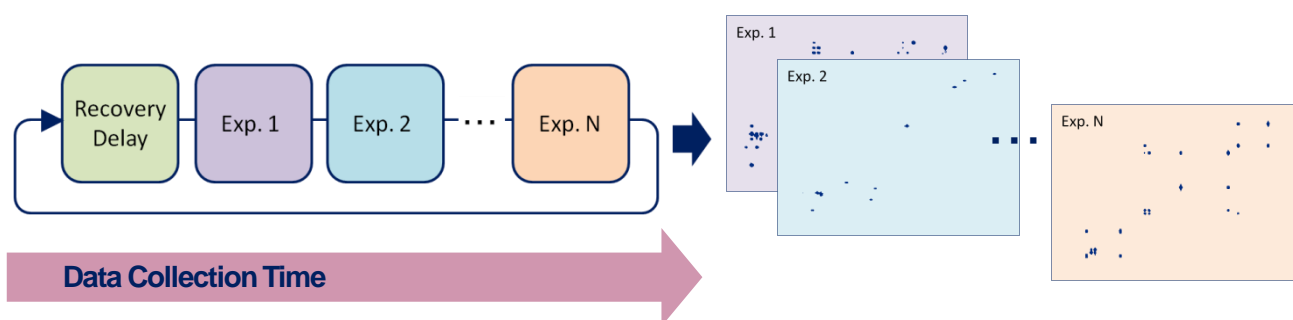


Figure 2. Schematic representation of the NOAH measurement

[1] Ě. Kupče, T. D. W. Claridge, *Angew. Chem. Int. Ed.* 2017, 56, 11779.

The Example of NOAH-3 Experiment

In Figure 3 (a), there is a NOAH-3 supersequence, that combines HMBC, multiplicity-edited HSQC and COSY. The ^1H - ^{13}C HMBC (b), ^1H - ^{13}C multiplicity-edited HSQC (c) and ^1H - ^1H COSY (d) spectra were collected in 10 minutes. For comparison, it would take nearly 30 minutes to collect the spectra conventionally.

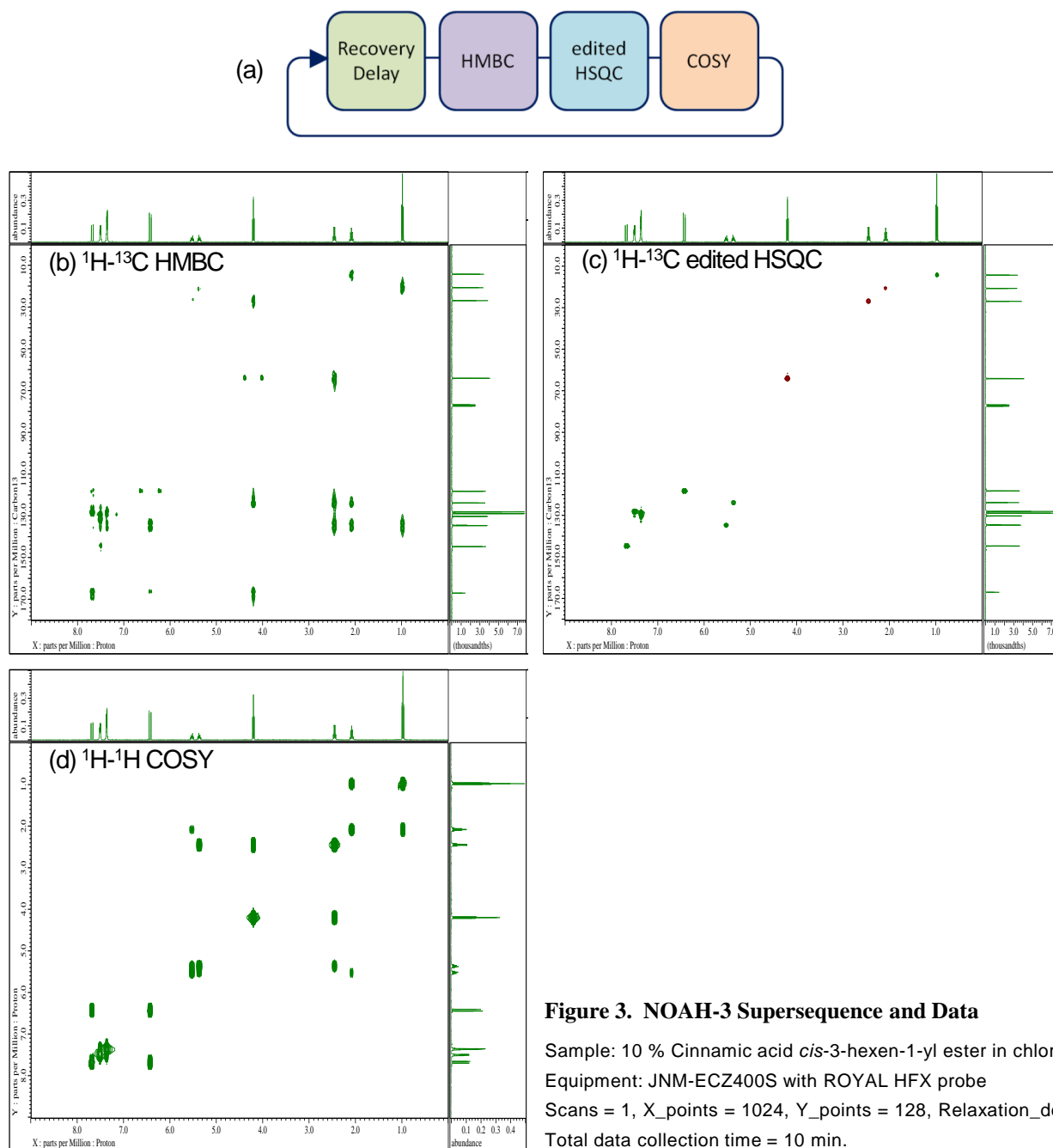


Figure 3. NOAH-3 Supersequence and Data

Sample: 10 % Cinnamic acid *cis*-3-hexen-1-yl ester in chloroform-*d*

Equipment: JNM-ECZ400S with ROYAL HFX probe

Scans = 1, X_points = 1024, Y_points = 128, Relaxation_delay = 1.5 s,

Total data collection time = 10 min.

Copyright © 2019 JEOL Ltd.

Certain products in this brochure are controlled under the "Foreign Exchange and Foreign Trade Law" of Japan in compliance with international security export control. JEOL Ltd. must provide the Japanese Government with "End-user's Statement of Assurance" and "End-use Certificate" in order to obtain the export license needed for export from Japan. If the product to be exported is in this category, the end user will be asked to fill in these certificate forms.

