NOAH: Experimental setup and processing

In a nutshell

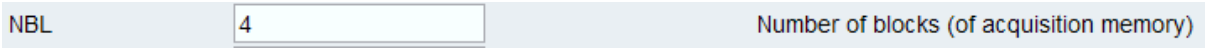
- Install the au-programs, pulse programs and parameter sets in your corresponding user libraries. Note: there are different sets of au-programs for TopSpin-3 and TopSpin-4 users.
- Use the provided parameter sets as a starting point for setting up the NOAH experiments.
- Adjust the required experimental parameters (prosol, sw, o1, td, etc) as usual.
- Use the *xaup* command for processing. The spectra are saved in X001, X002, X003… experiments where X is the original experiment number.

Important: make sure you set the cnst10 to the swC/swH ratio and cnst20 to swN/swH ratio, as appropriate. These constants are used in the NOAH pulse sequences to scale the sw in F1 as required.

Details

The NOAH experiment is set up as a single experiment in a manner similar to conventional 2D heteronuclear ^{13}C (^{15}N) techniques, but with the following specific requirements:

- The number of memory blocks used for data acquisition must be defined with the acquisition parameter *NBL*. This must be set equal to the number of NOAH modules in the sequence eg NBL=4 for a NOAH-4 experiment.



- The TD1 data size must be increased (scaled) according to the number of NOAH modules in the sequence. Thus, a NOAH-4 requiring 256 t_1 points for each NOAH module dictates that TD1 be set to 256*4 = 1024 points.
- Since only a single F1 frequency domain is explicitly defined (typically for the ^{13}C dimension) the scaling constants for the F1 spectral widths for all other F1 dimensions (^{15}N and/or ^{1}H) must be set for the correct definition of these frequencies in the final data sets.
    - *cnst10* defines the ^{13}C:^{1}H spectral width (as Hz) ratio
    - *cnst20* defines the ^{13}C:^{15}N spectral width (as Hz) ratio (if ^{15}N experiments are used)

For ^{13}C HSQC experiments, the optional multiplicity editing may be activated by setting the *zg* options to **–DEDIT**. If left blank, no editing will be included.

Data processing makes use of the following AU processing scripts:

- **spiltx**: splits the single NOAH data set into *NBL* individual data sets for manual processing as separate experiments. For a NOAH experiment acquired in EXPNO ***X***, **splitx** will produce data sets numbered ***X001***, ***X002***, ***X00N*** … etc according to the number *N* of NOAH modules.
- **splitx_au**: this splits the data set as above, and then automatically processes the derived data sets (for its use see the requirements detailed below for NOAH within automation)

- **fixF1**: corrects the F1 chemical shift axis for ^{1}H (i.e. homonuclear experiments). Uses *cnst10* as input.
- **fixF1n**: corrects the F1 chemical shift axis for ^{15}N. Uses *cnst20* as input.

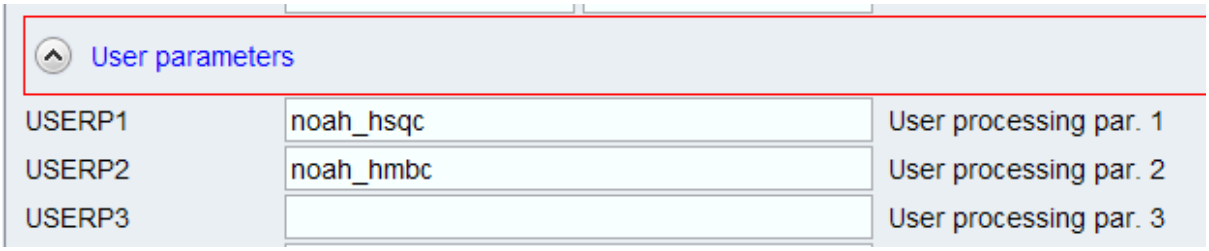
For use in automation, additional processing scripts are required, as discussed below.

Using NOAH within automation

It is possible to configure NOAH parameter sets to acquire and then fully process the resulting separate 2D data sets within ICON-NMR. Some additional settings are required to achieve this:

- The processing AU programme (ProcPar *AUNMP*) must be defined to use the **splitx_au** script.

- The appropriate AU scripts needed for processing each individual experiment must be defined as the ProcPar User parameters *USERP1… USERPN* for each of the *N* NOAH experiments. These must be defined in the order that matches the sequence of NOAH modules.



These AU scripts are executed for each individual experiment by the **splitx_au** programme to yield the final, fully processed spectra. They include the appropriate **fixF1** routines for the correction of the chemical shift axis of homonuclear experiments.

The following AU scripts are available for the different types of experiment modules:

-**noah_hmqc**: phase-sensitive processing for (^{15}N) HMQC
-**noah_hsqc**: phase-sensitive processing for HSQC
-**noah_hmbc**: magnitude-mode processing for HMBC
-**noah_cosy**: magnitude-mode processing for homonuclear experiments (e.g. COSY / NOESY)
-**noah_noesy**: phase-sensitive processing for homonuclear experiments (e.g. NOESY)

Reference:

Ē. Kupče and T. Claridge, Angew. Chem. Int. Ed. Eng., vol.56 (39), pp. 11779-11783 (2017).

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