Easy Work Flow for Selective Composite Excitation Experiments

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Introduction

The development of DPFGSE based 1D selective excitation experiments as analogues for full 2-dimensional methods has provided simplicity and time savings for extracting specific structural information for drugs and natural products. With isotope filtering it is also possible to screen mixtures through double resonance. In this poster we will illustrate the power of selective excitation using model compounds as alternatives to detailed 2 dimensional experiments and show how experiment design can greatly simplify the applications of complex selective excitation experiments by non-expert NMR users.

Objective

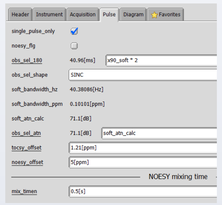
An objective of this poster is to show how pulse-program design which employs flags to control options in the actual experiment affords a very compact and easy to navigate user experience. All needed items such as excitation band information is built in a single location rather than requiring the user to transfer information into multiple experiments to conduct multi-composite selective excitation experiments such as STEPNOESY[1] or FESTA[2].

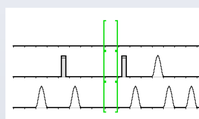
JEOL's Delta pulse programing is unique in that a single text file defines both the pulse sequence events as well as providing a flexible interface for user interactions. Virtually anything imagined can be easily constructed with as much detail or alternatively as much simplicity desired. We have found it very convenient to provide a single pulse program with options to turn different elements such as TOCSY or NOESY off or on. All entries for key parameters such as multiple selective excitation points are in a single job file so as information is obtained in a setup step it can be easily transferred back into the same job and resubmitted for acquisition in the next or final acquisition stages.

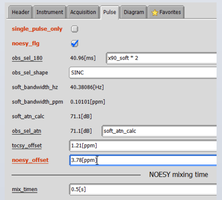
STEPNOESY and SRI-FESTA present different challenges both from a pulse programing perspective and also for the user interface. Parameters such as type of selective excitation pulse can be selected by the user through a drop down menu. Changing such a decision as experience is gained with an experiment is simple: select another shape and resubmit. If desired the addition or removal of a single character can change a menu to a fixed choice for basic users.

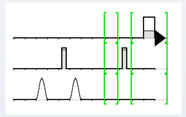
The STEPNOESY example was acquired with ECZ400 console and ROYAL probe. The SRI-FESTA example acquired with ECZ-500R and ROYAL HFX probe.

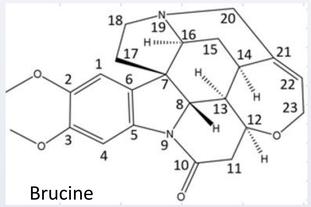
STEPNOESY Example



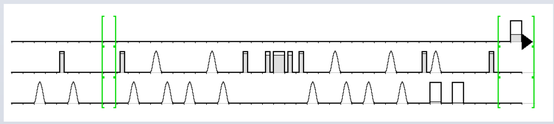


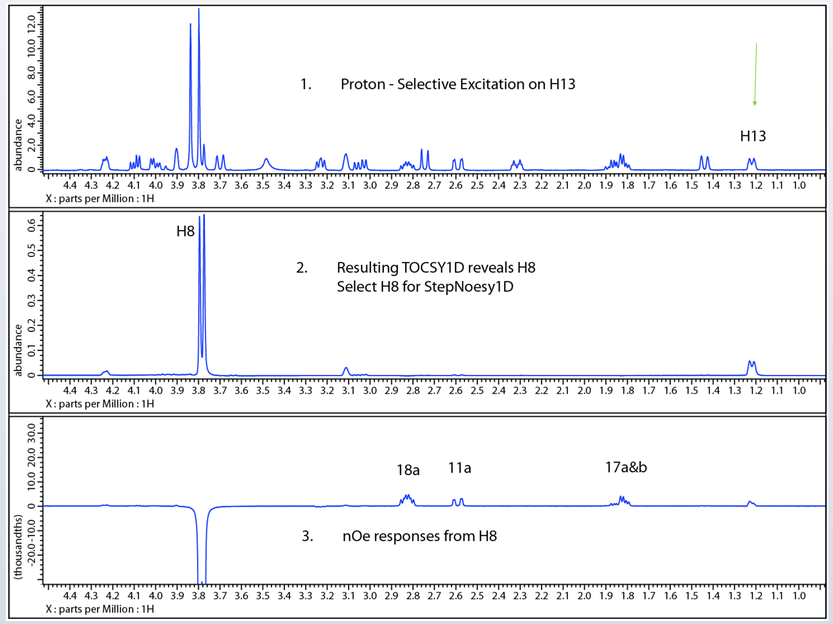






The obscured H-8 resonance is revealed for nOe selection by the initial selective TOCSY step at H-13.

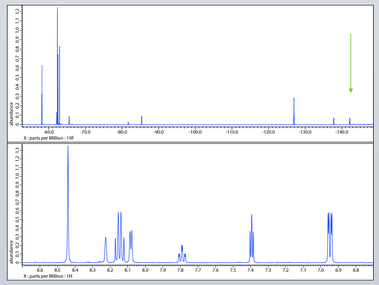


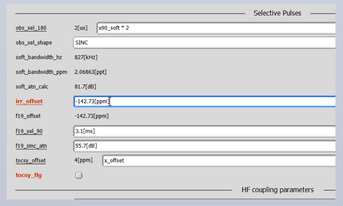


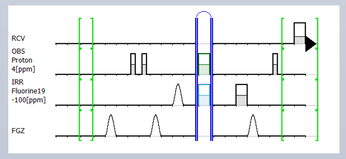
SRI-FESTA Example

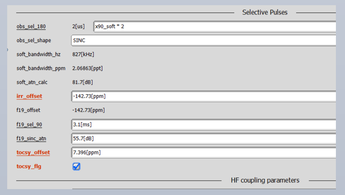
^{19}F (top) and ^{1}H spectra from a mixture of 5 di-fluoro-pyridine isomers.

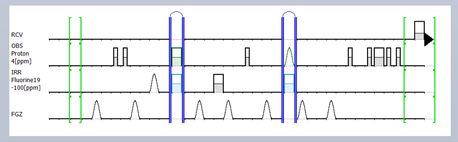
In this example we will use the ^{19}F near -142ppm for the selective inept isotope filter. After the selective INEPT is complete the ^{1}H at 7.396ppm is revealed as target for the subsequent TOCSY addition.

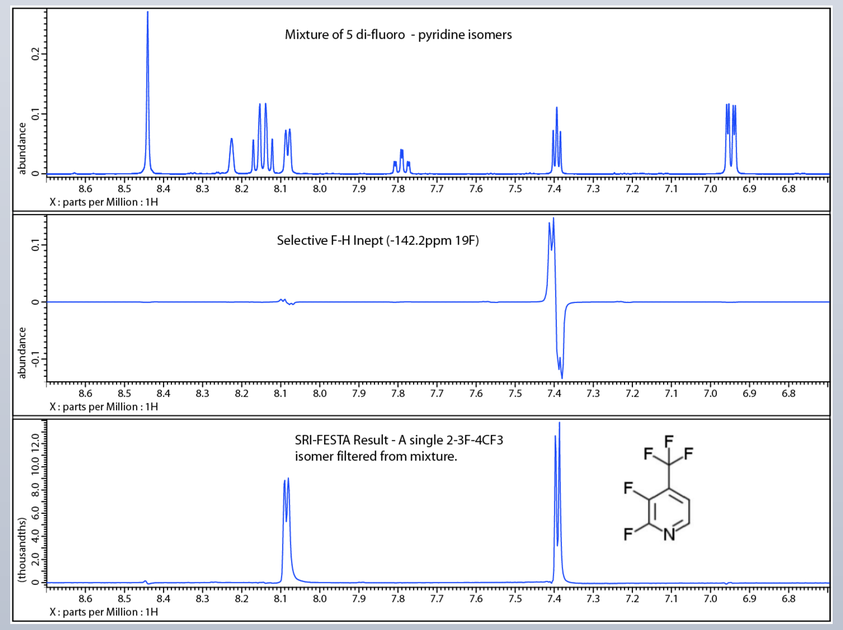












References

1. STEPNOESY Reference: Extending the limits of selective 1D NOESY with an improved selective TOCSY edited preparation function. Haitao Hu, Scott A. Bradley, Krish Krishnamurthy. J. Magn. Reson. Vol 171, Issue 2, Dec 2004 p 201-206.

2.SRI-FESTA : L. Castañar, et al. SMASH 2017 Presentation "Simplifying complexity: a new NMR approach for the analyis of mixtures containing fluorinated species"

Acknowledgements

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