

A novel SEXY approach to conquer poor resolution in Solid-State NMR using Saturation Exchange Spectroscopy

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Introduction

- Solid-state NMR provides information on the structure and dynamics of various biomolecules at atomic resolution¹.
- **Spectral resolution** limits the amount of site-specific information extractable from an NMR spectrum².
- **Saturation exchange spectroscopy (SEXY)** is a novel approach being developed to allow information to be extracted from **extensively broadened peaks**.

How will SEXY conquer poor resolution?

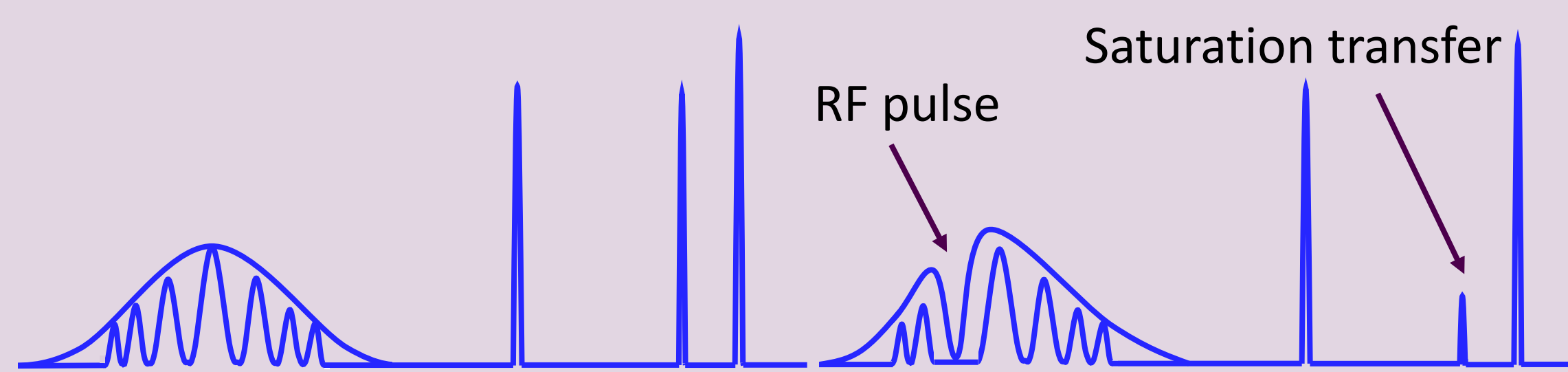


Figure 1. A radio frequency (RF) pulse **selectively saturates** a broadened peak. Saturation is transferred to additional peaks through **dipolar coupling** (through space interactions).

Methods: Proof of Concept

MLF was packed into a 1.3 mm triple-resonance probe. All spectra were obtained at a ¹H Larmor frequency of 600 MHz and a magic angle spinning frequency of 60 KHz.

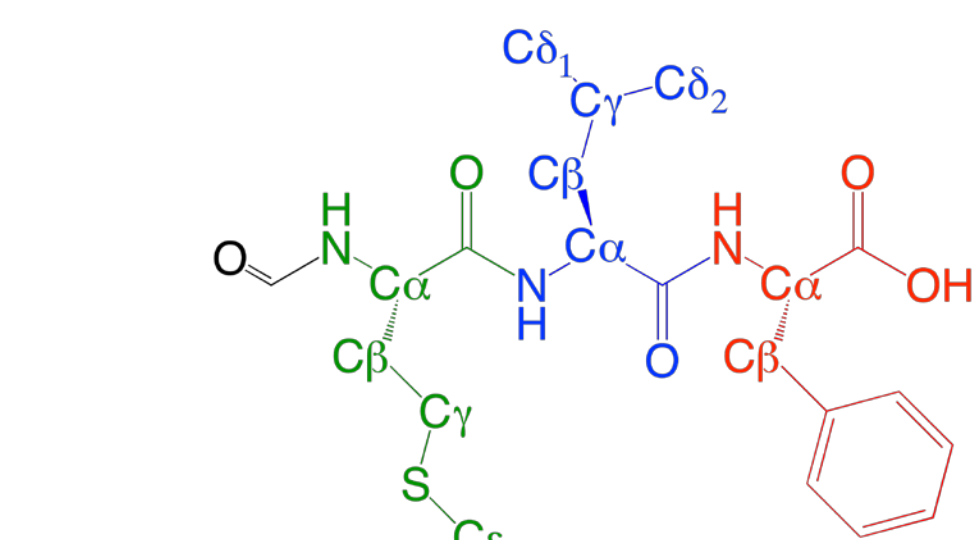


Figure 2. Fully labelled N-formyl-Met-Leu-Phe-OH (MLF).

Results 1: Finding Saturation Frequencies

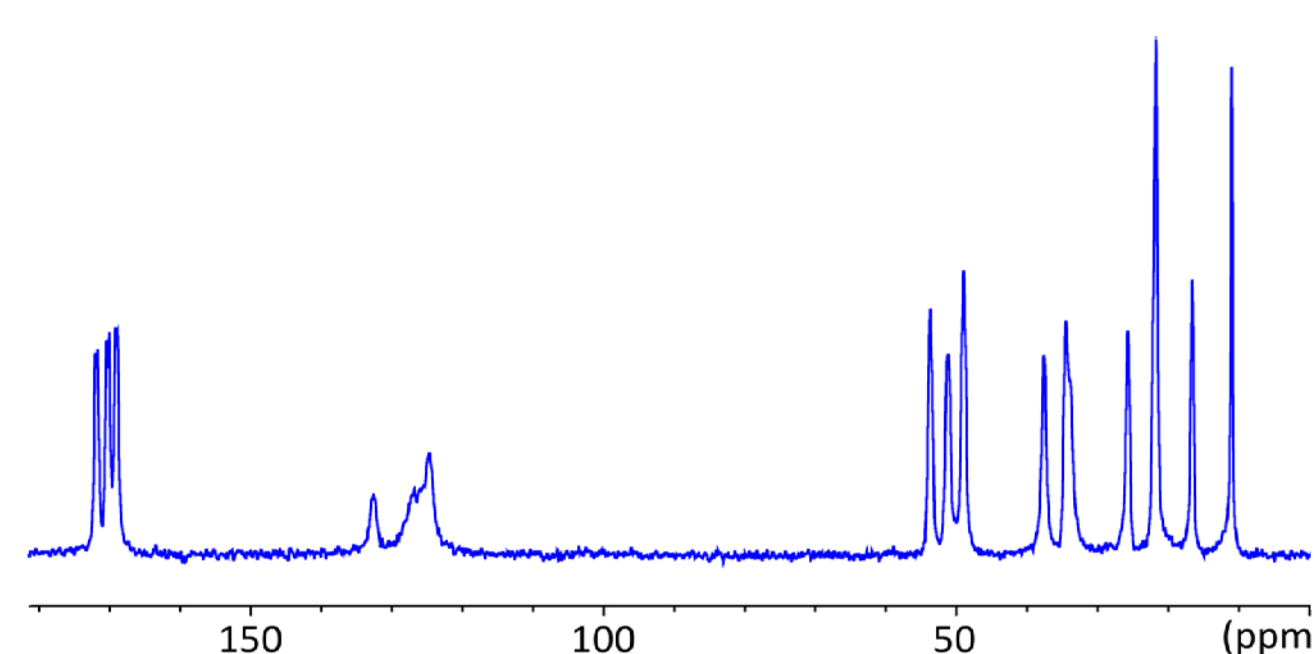


Figure 3. A 1D ¹³C spectrum of MLF with no saturation was used to obtain a list of RF pulse frequencies.

Results 2: MLF Saturation

What does a SEXY 2D spectrum look like?

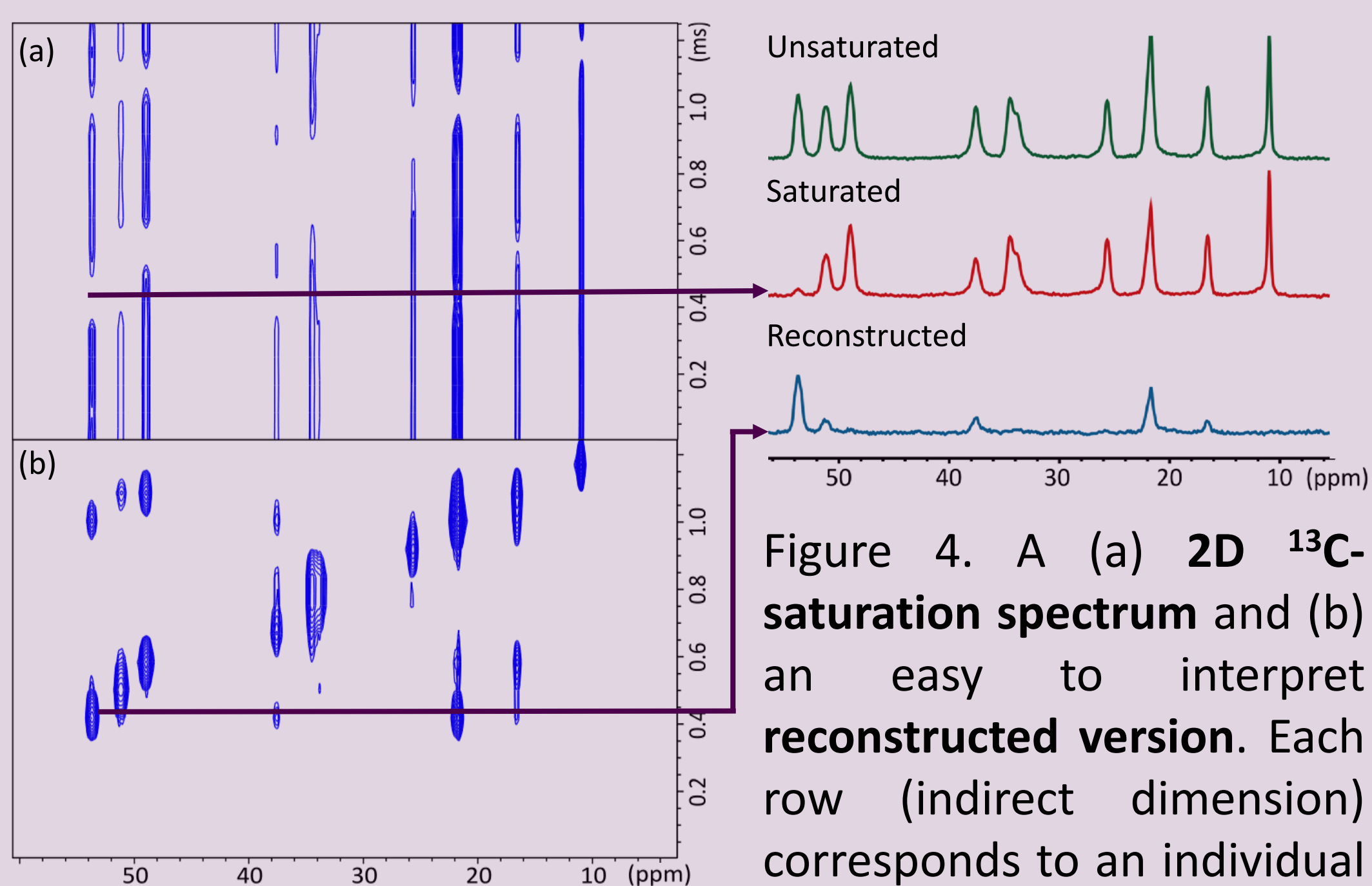


Figure 4. A (a) **2D ¹³C-saturation spectrum** and (b) an easy to interpret **reconstructed version**. Each row (indirect dimension) corresponds to an individual RF pulse.

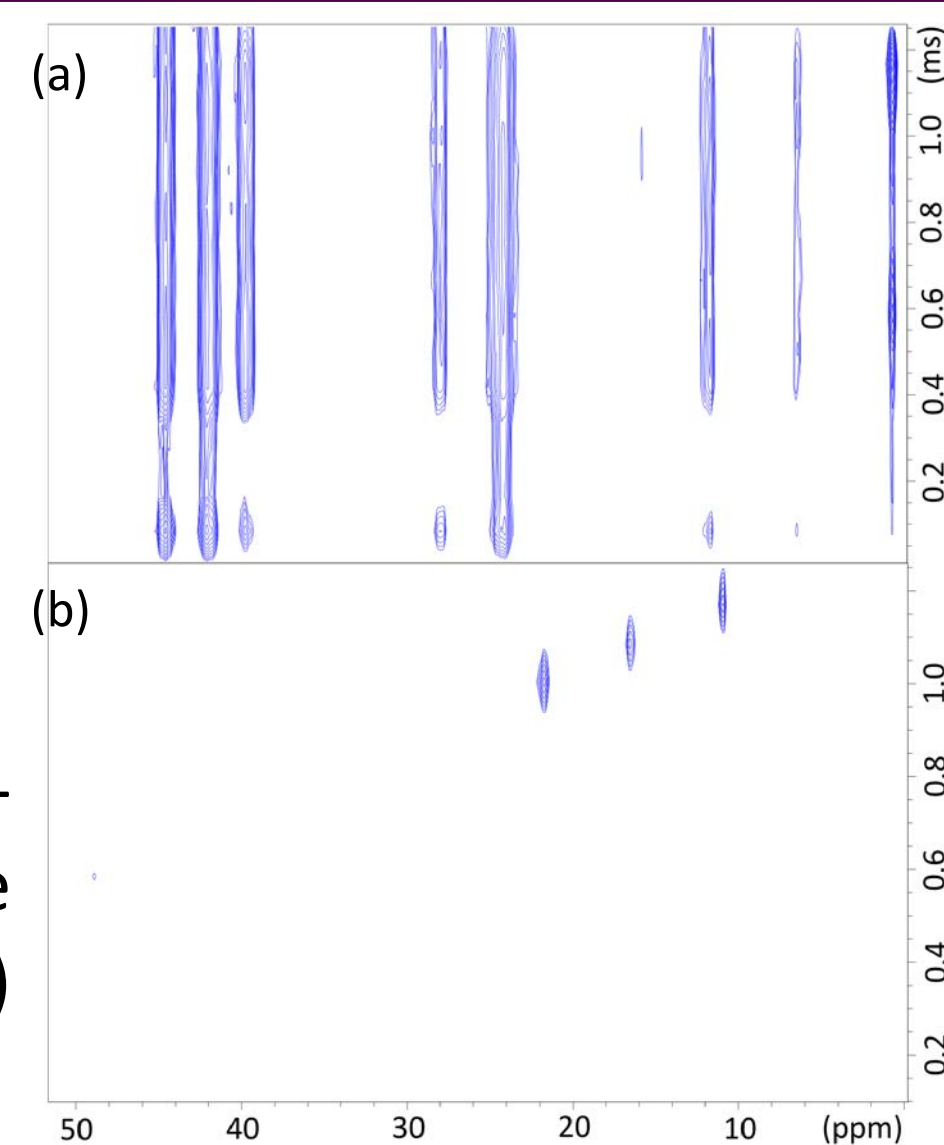
Making and interpreting a reconstructed SEXY 2D spectrum

- 1) Subtract each row of the 2D saturated spectrum (indirect dimension) by a 1D unsaturated spectrum.
- 2) 180° phase correction.
- 3) Peaks along the same row indicate saturation transfer.

How does saturation time affect SEXY?

- Longer saturation times increases the time for saturation transfer.

Figure 5. Reconstructed 2D ¹³C-saturation spectra illustrating the effect of (a) **long** (4.5 s) and (b) **short** (0.01 s) saturation times.



Conclusion and Future Work

- SEXY is a **novel** approach that has presented possible uses in extracting useful information from **extensively broadened peaks**.
- **Saturating MLF** using RF pulses of varying frequencies and novel methods of **reconstructing 2D NMR spectra** have provided **proof of concept**.
- SEXY will now be used to analyse complex systems with inhomogeneous broadening and to study slow motions.

References and Acknowledgments

1. J. Lamley, *et al.*, Am. Chem. Soc., 2014, 136, 16800–16806.
2. S. Ashbrook and S. Sneddon, J. Am. Chem. Soc., 2014, 136, 15440–15456.

