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Fast and Quantitative 2D NMR for metabolomic applications within MetaboHUB

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Fast and Quantitative 2D NMR for metabolomic applications within the MetaboHUB Infrastructure



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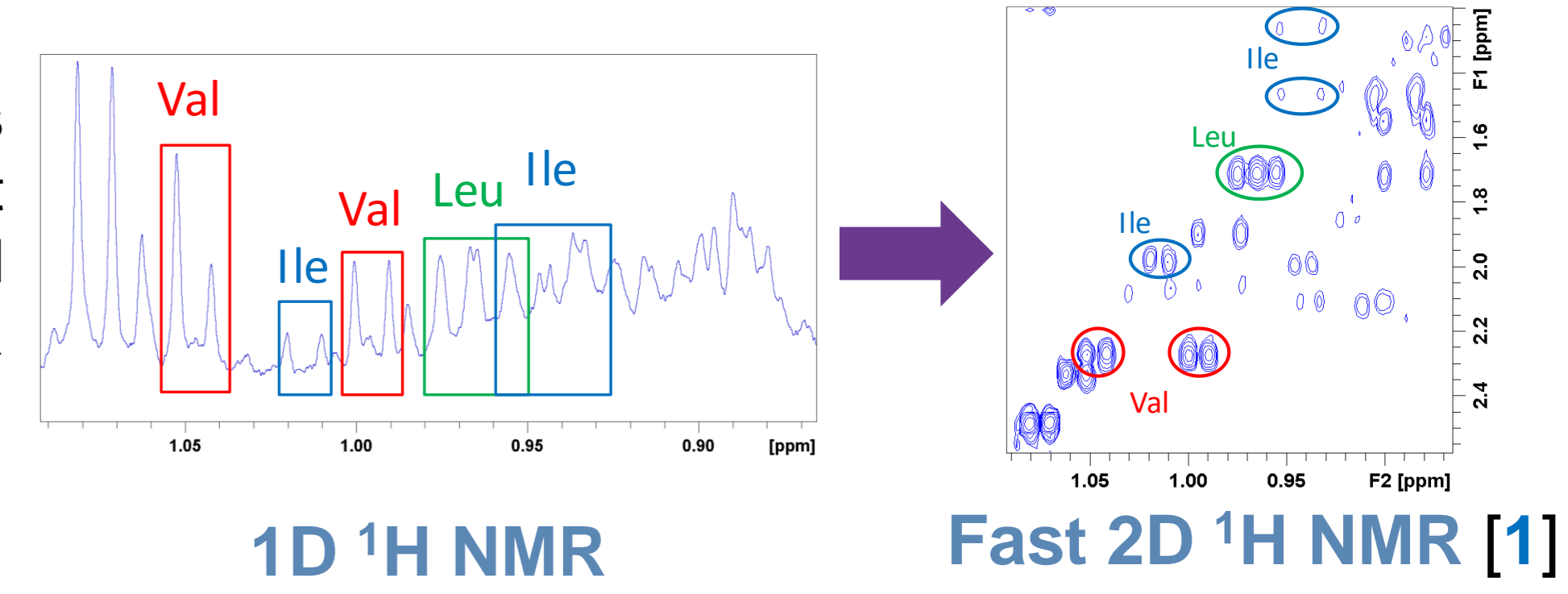


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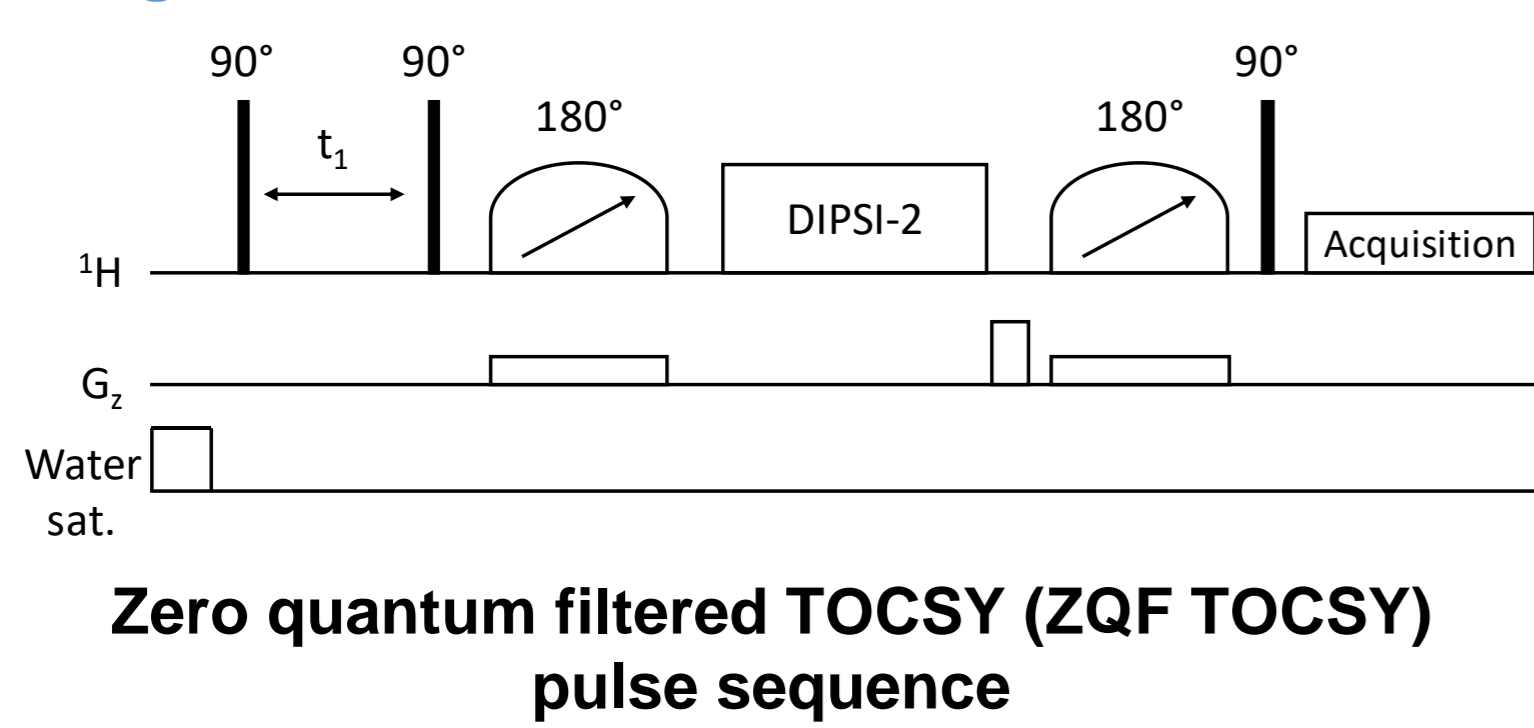
- 1: Nantes Université, CNRS, CEISAM, UMR 6230. MetaboHUB-Grand-Ouest
- 2: TBI, Université de Toulouse, CNRS, INRAE, INSA, MetaboHUB - MetaToul, National Infrastructure of Metabolomics and Fluxomics.
- 3: Toxalim (Research Centre in Food Toxicology), Toulouse University, INRAE UMR 1331, ENVT, INP-Purpan, UPS, MetaToul-AXIOM Platform, National Infrastructure of Metabolomics and Fluxomics: MetaboHUB, INRAE.
- 4: Univ. Bordeaux, Bordeaux INP, INRAE, OENO, UMR 1366, ISVV, Bordeaux Metabolome - MetaboHUB.
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- 8: Université de Tours, INSERM, Imaging Brain & Neuropsychiatry iBrain U1253.
- 9: Université Clermont Auvergne, Clermont Auvergne INP, CNRS, Institut de Chimie de Clermont-Ferrand. Université Clermont Auvergne, INRAE, UNH, Plateforme d'Exploration du Métabolisme, MetaboHUB Clermont

Context

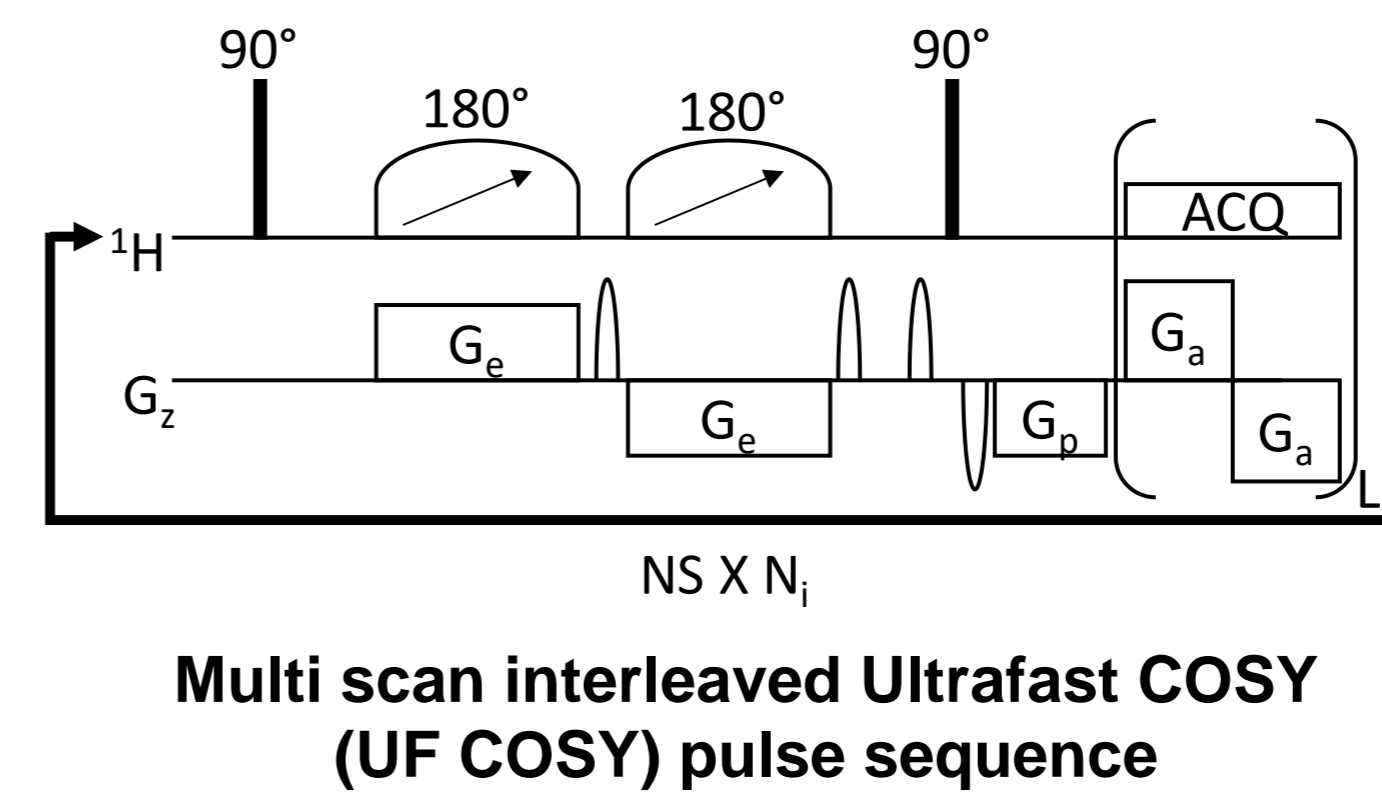
NMR-based metabolomic studies are mostly performed with well-established protocols based on proton 1D liquid-state NMR. However, conventional 1D ¹H NMR is hampered by overlapping signals which are typically encountered in biological samples (biofluids or extracts), thus impairing the identification of biomarkers. As depicted on the figure below, it has been shown that advanced fast and quantitative 2D NMR methods can overcome this issue while staying compatible with high-throughput and quantitative needs of metabolomics [1, 2, 3]. Hence, two fast NMR methods, involving Non-Uniform Sampling and ultrafast NMR were optimized and implemented across seven NMR metabolomic platforms, all involved in the MetaboHUB infrastructure.



Optimizations

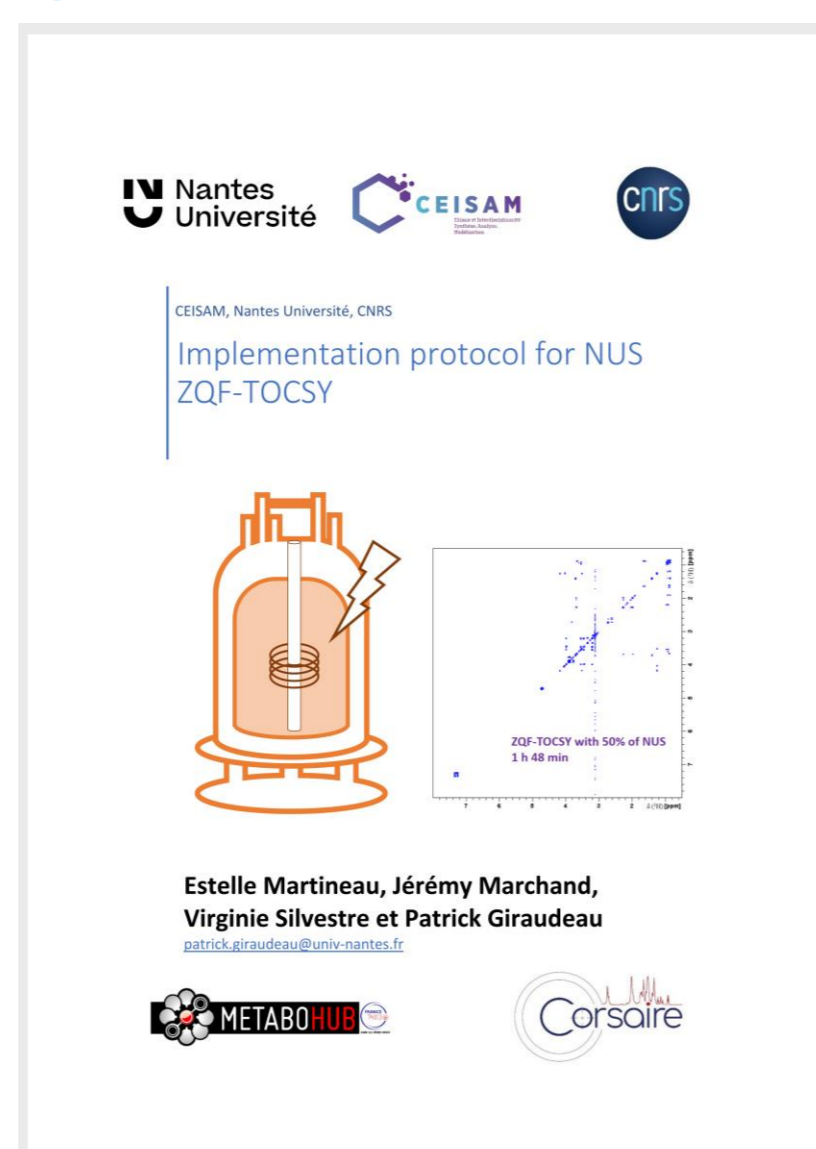


- Calibration of the adiabatic pulses
- Calibration of the gradient for zero quantum suppression [4]
- Non Uniform Sampling (NUS) optimization
- Processing optimization



- Calibration of the gradients and chirp pulses for spatial encoding
- Optimization of the interleaved scans
- Optimization of spectral width
- Optimization of the spectral window
- Optimization of apodization

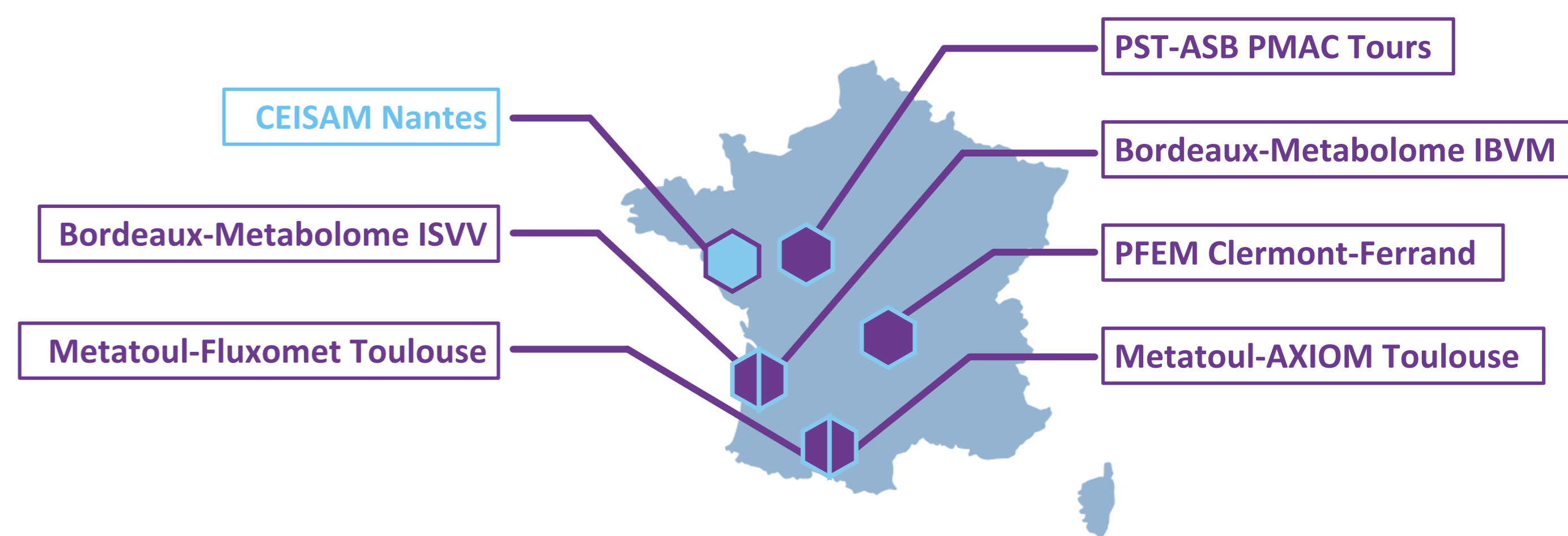
Protocols and Implementation



Full protocols for implementation of Fast 2D ¹H NMR methods available here :



The NMR groups within MetaboHUB :

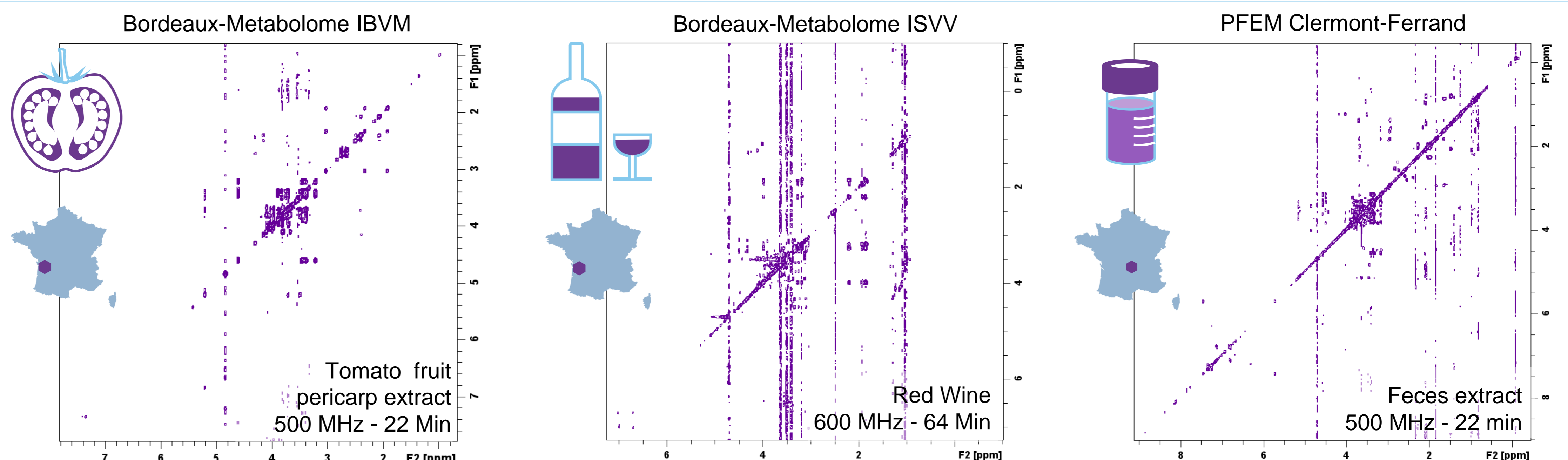


The two fast 2D NMR methods were optimized in Nantes and then implemented on site in the other six NMR facilities within MetaboHUB, in an harmonized way.

Applications

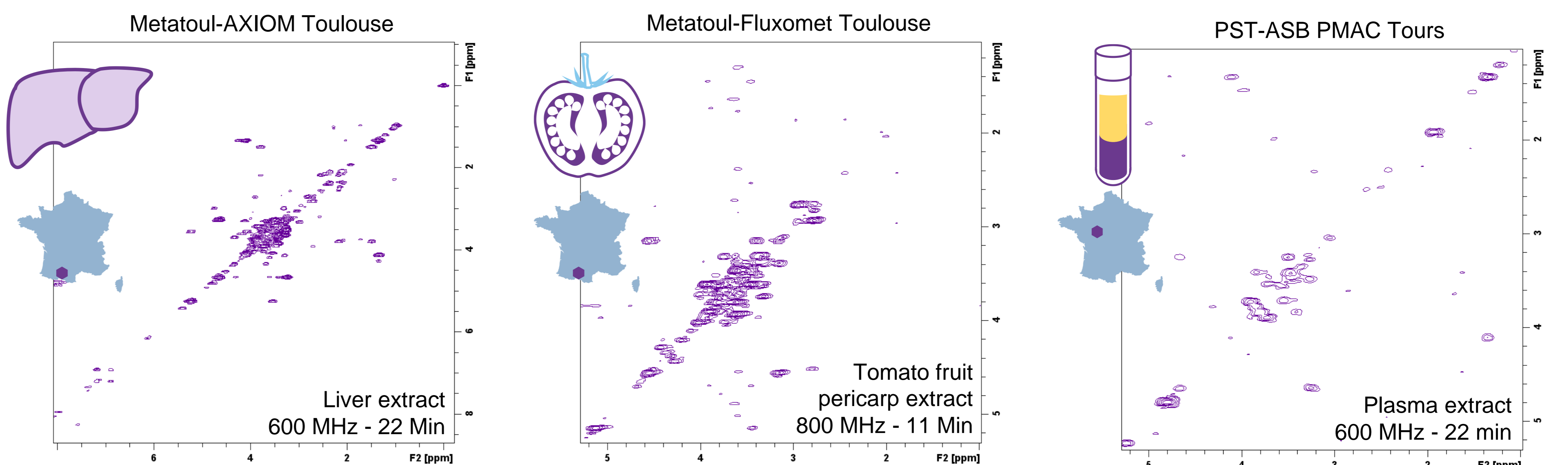
ZQF TOCSY with Non Uniform Sampling Acquisition [4]

After implementation, the ZQF TOCSY has been applied to a variety of new matrices, each one being specific to a research question of interest of each involved group. All spectra were recorded with 50% NUS



Ultrafast COSY [5]

In a similar fashion, UF COSY has been applied to a variety of new applications, giving access to high-throughput 2D NMR metabolomic datasets.



Towards interlaboratory studies

The implementation of innovative and fast 2D ¹H NMR methods in an harmonized way also gives new perspectives for collaborative projects. Following a previous study involving 1D ¹H NMR and associated processing workflows [6], these tools pave the way towards the first interlaboratory study involving fast 2D NMR metabolomic data.

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2. J. Marchand, E. Martineau, Y. Guitton, B. Le Bizec, G. Dervilly-Pinel, P. Giraudeau (2018). *Metabolomics*, **14**, 60

3. C. Lhoste, B. Lorandiel, C. Praud, A. Marchand, R. Mishra, A. Dey, A. Bernard, J.-N. Dumez, P. Giraudeau. (2022). *Prog. Nucl. Magn. Reson. Spectrosc.*, **130-131**, 1-46

4. M. J. Thrippleton, J. Keeler (2003). *Angew. Chem. Int. Ed.*, **42**, 3938-3941

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