



**HAL**  
open science

## Solid state NMR and diatoms: probing the interfaces

Sylvie Masse, Guillaume Laurent, T. Coradin

► **To cite this version:**

Sylvie Masse, Guillaume Laurent, T. Coradin. Solid state NMR and diatoms: probing the interfaces. Biom mineralization short course, Dec 2014, Paris, France. . hal-01138970

**HAL Id: hal-01138970**

**<https://hal.sorbonne-universite.fr/hal-01138970>**

Submitted on 3 Apr 2015

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

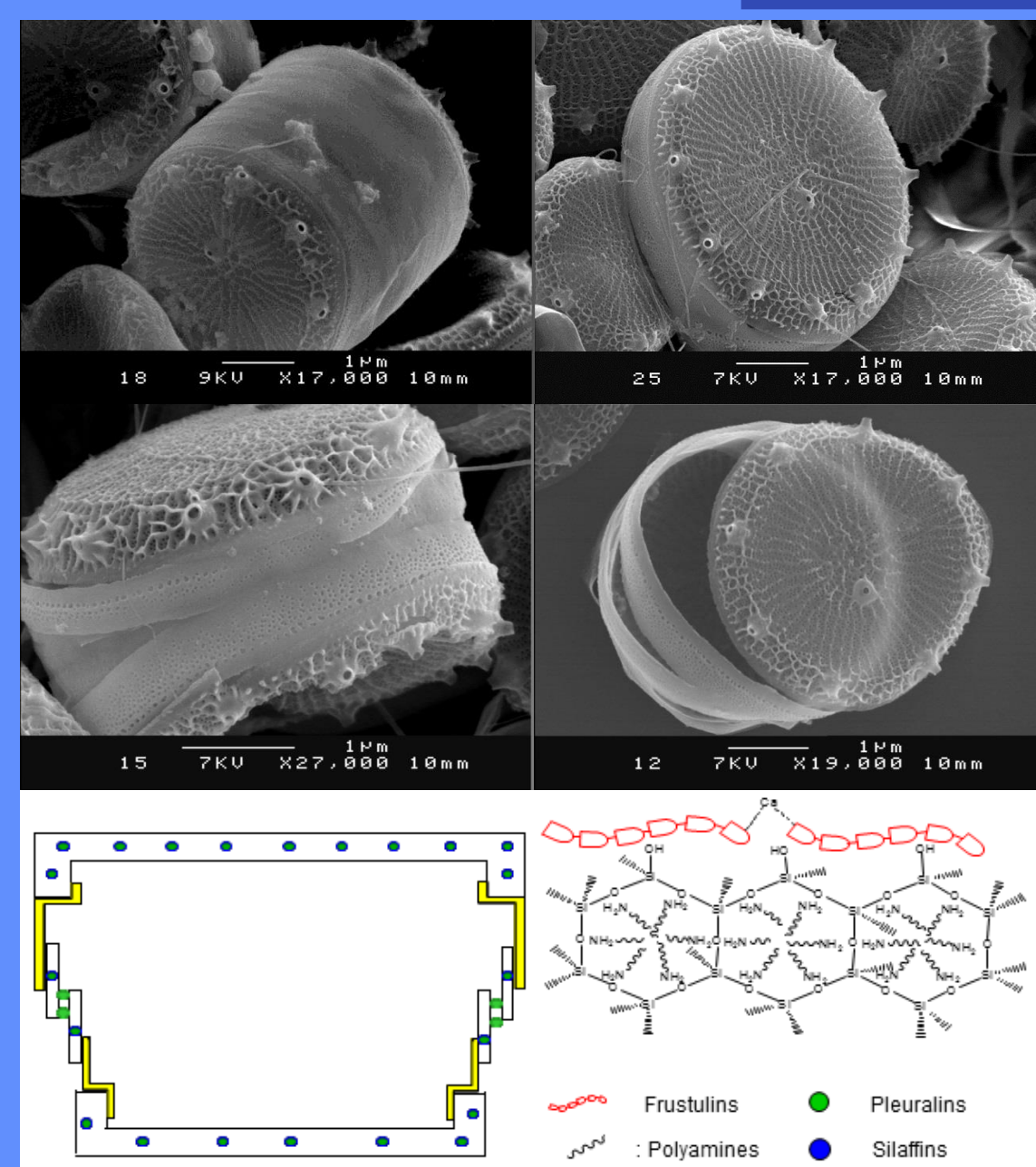


Distributed under a Creative Commons Attribution - NonCommercial - NoDerivatives 4.0 International License

## INTRODUCTION

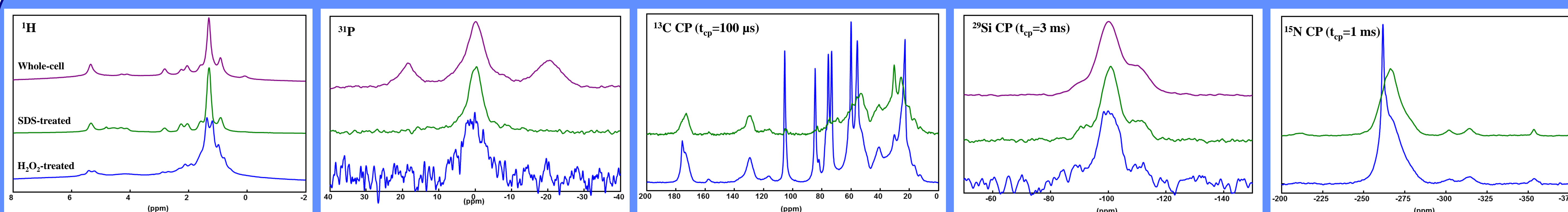


Partnership :  
V. MARTIN-JEZEQUEL, B. TESSON  
Fac. de Sciences et Techniques  
ISOMER, NANTES



Gyromagnetic ratio $ \gamma $		$\frac{\gamma}{\text{NA}}$		Relative sensitivity $\searrow$	
$I$	$\gamma$ (10 <sup>6</sup> )	$\gamma$ (10 <sup>6</sup> )	$\gamma$ (10 <sup>6</sup> )	$\gamma$ (10 <sup>6</sup> )	$\gamma$ (10 <sup>6</sup> )
<sup>1</sup> H	100%	100%	100%	100%	100%
<sup>31</sup> P	100%	100%	100%	100%	100%
<sup>13</sup> C	11%	11%	11%	11%	11%
<sup>29</sup> Si	4.7%	4.7%	4.7%	4.7%	4.7%
<sup>15</sup> N	0.36%	0.36%	0.36%	0.36%	0.36%

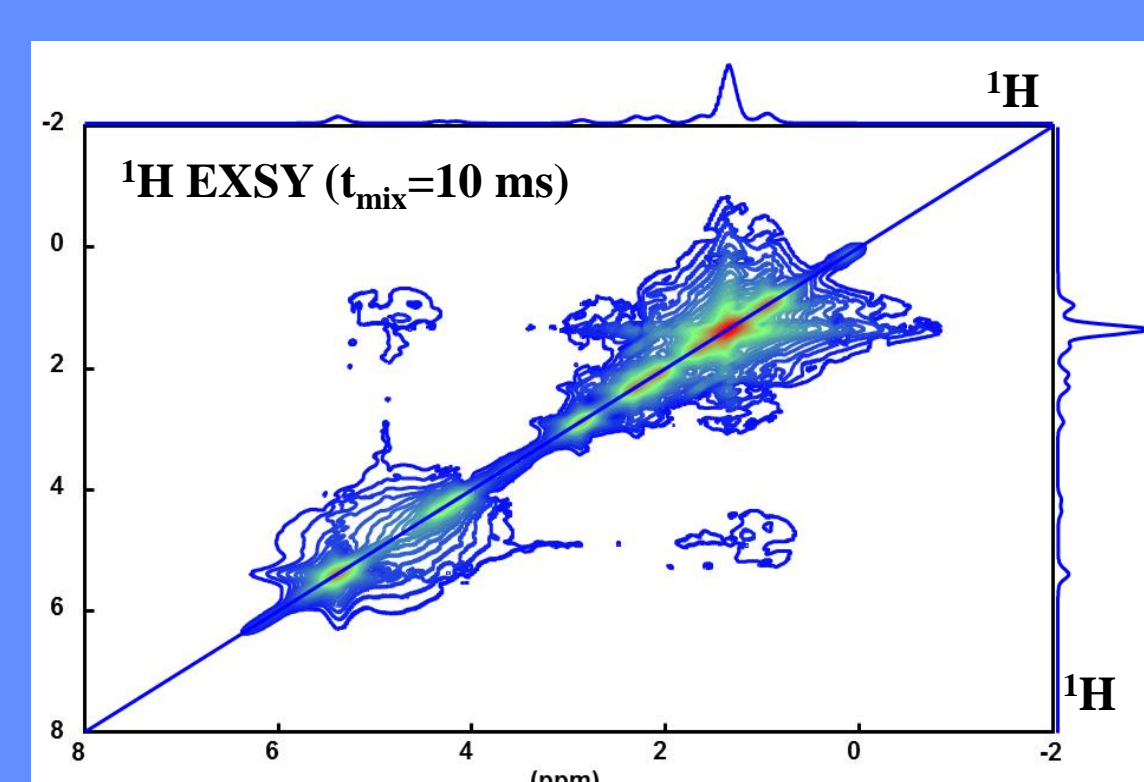
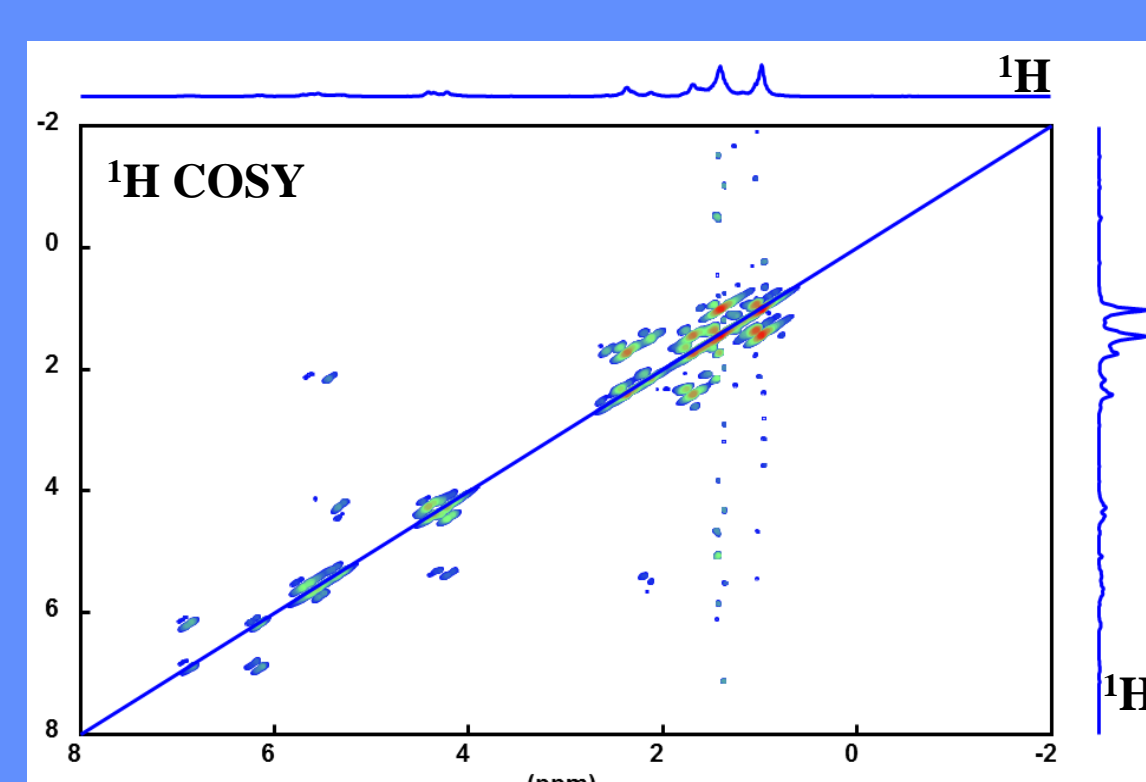
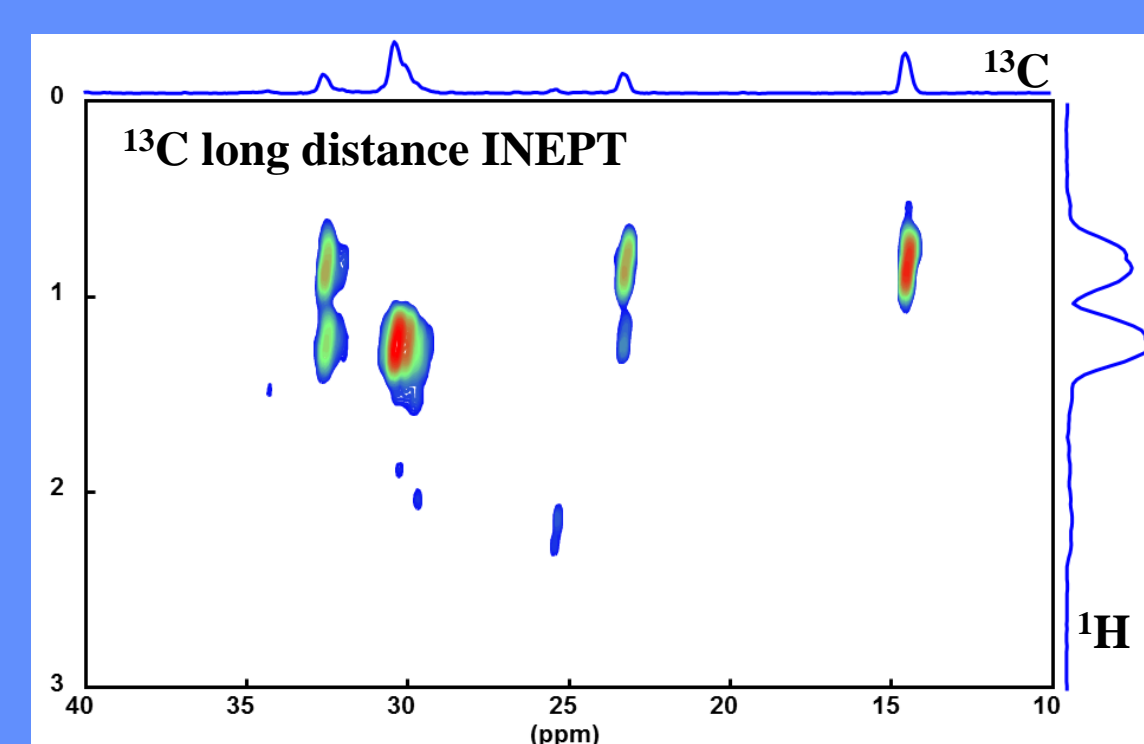
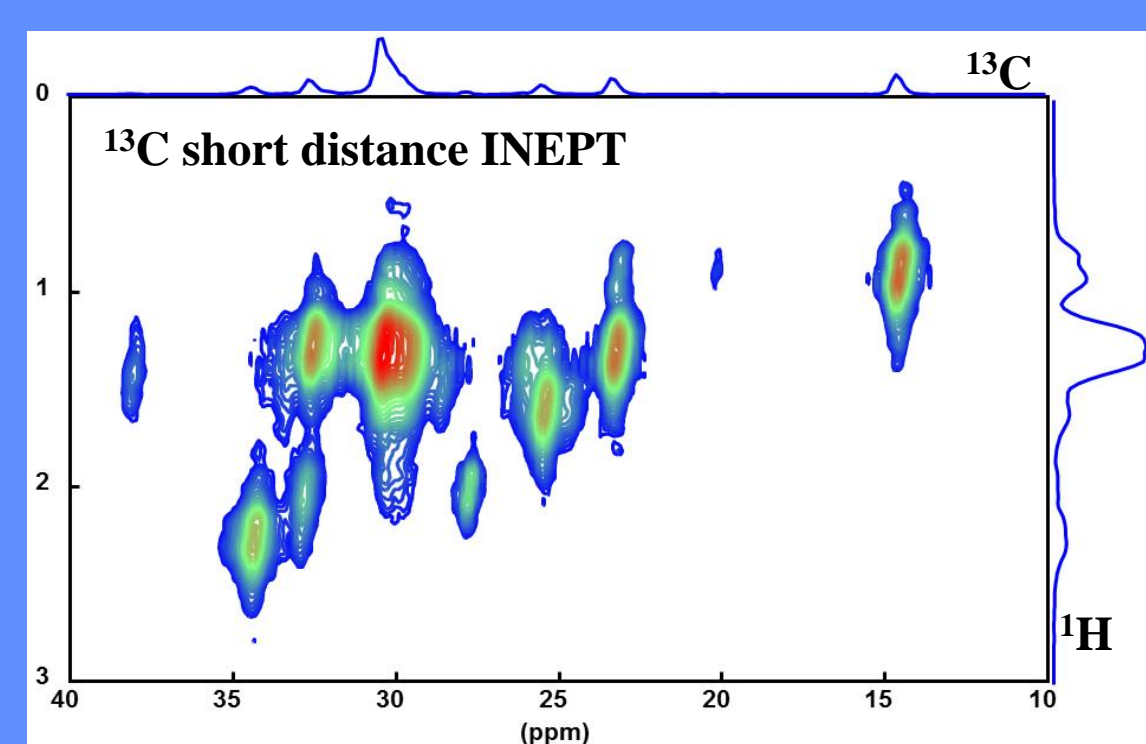
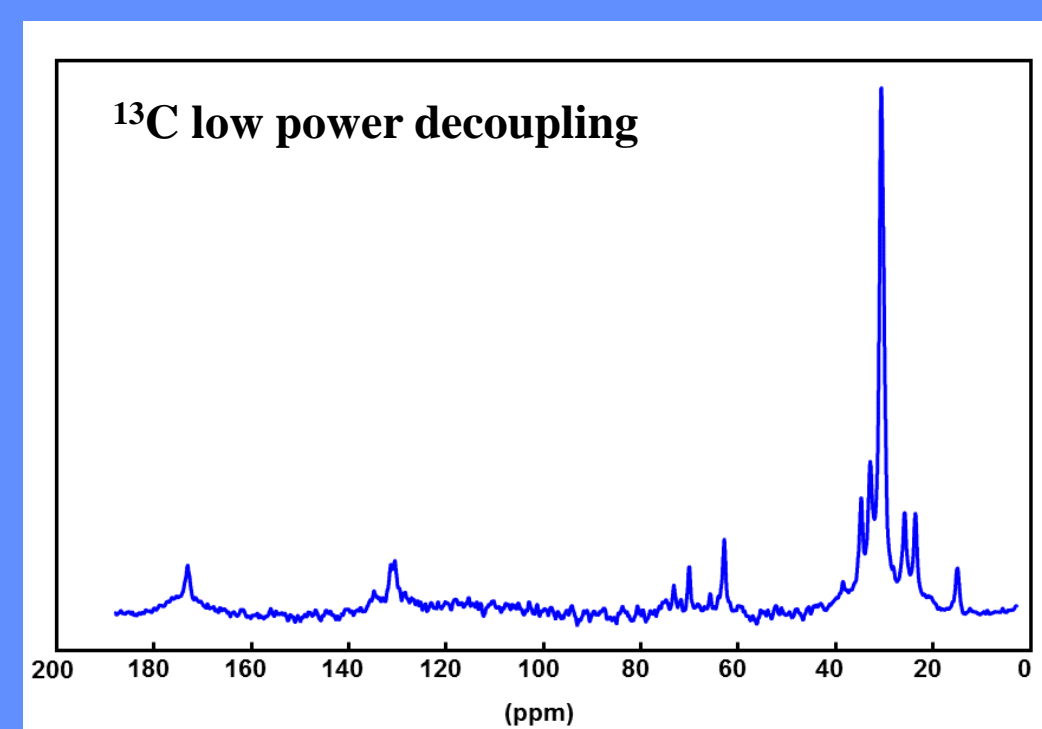
## CHEMICAL TREATMENT



Whole-cell, SDS-treated and H<sub>2</sub>O<sub>2</sub>-treated samples were isotopically enriched with <sup>29</sup>Si, <sup>13</sup>C/<sup>29</sup>Si/<sup>15</sup>N and <sup>13</sup>C/<sup>15</sup>N, respectively. While SDS is used to clean the frustule, H<sub>2</sub>O<sub>2</sub> treatment seems to be much more aggressive, probably leading to partial dissolution-recrystallization.

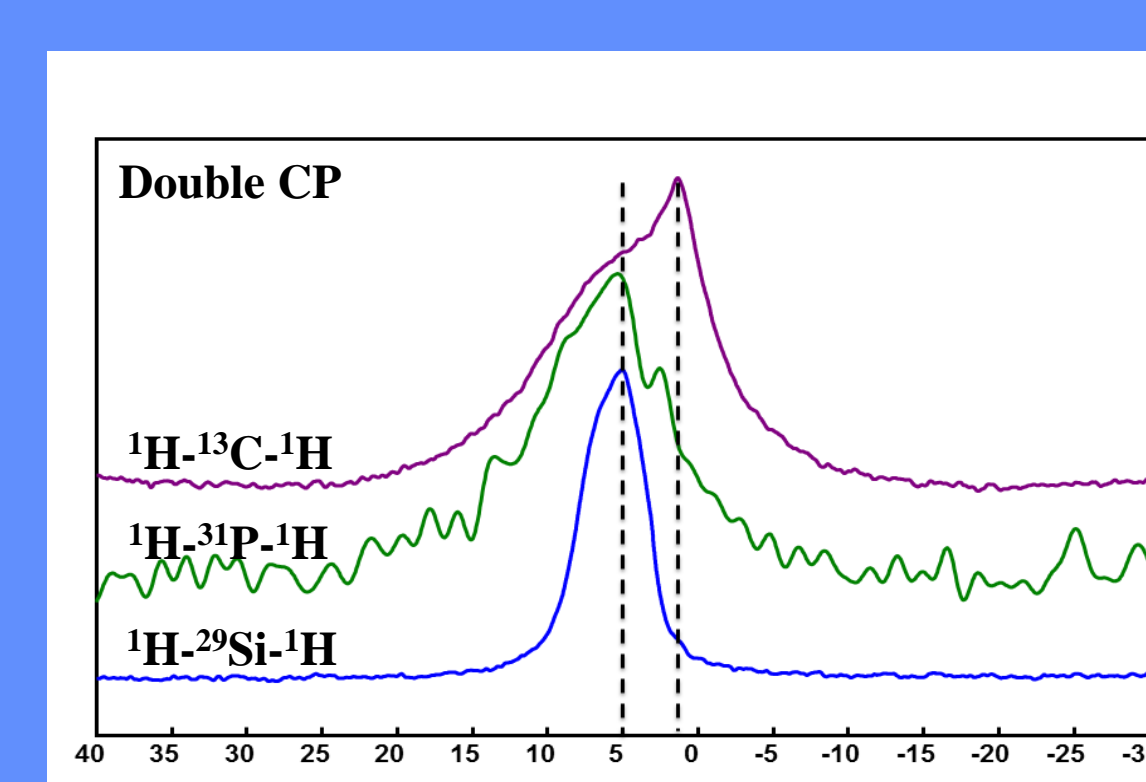
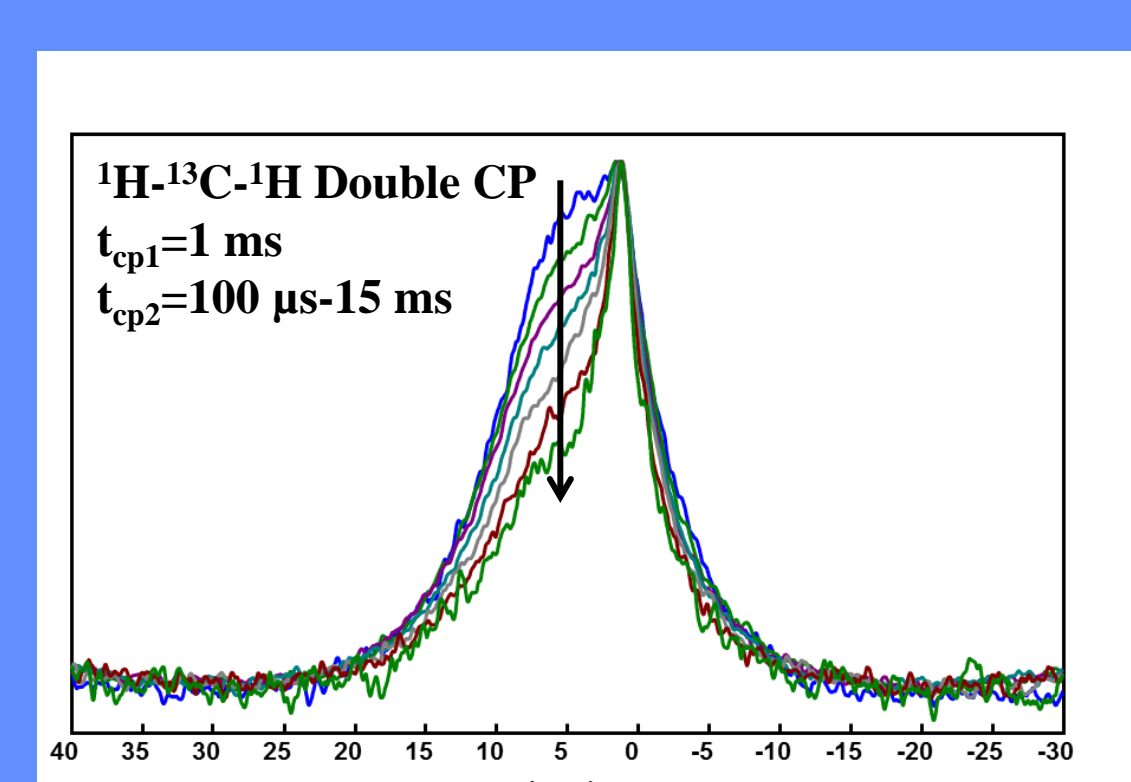
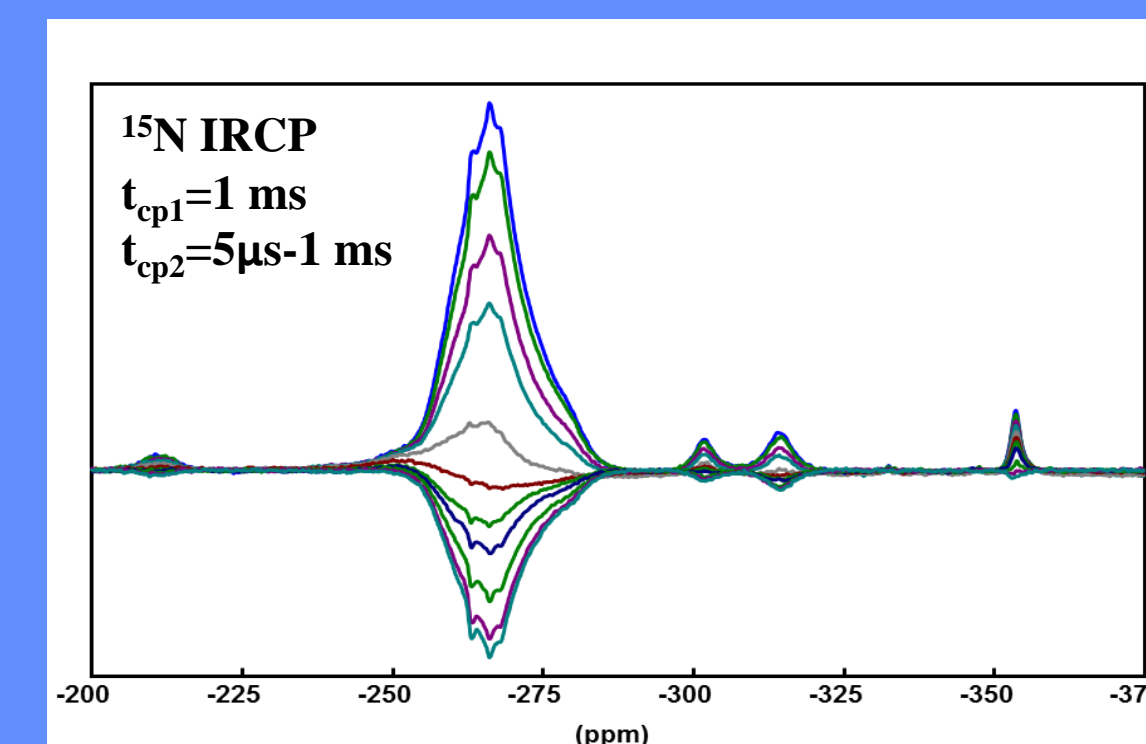
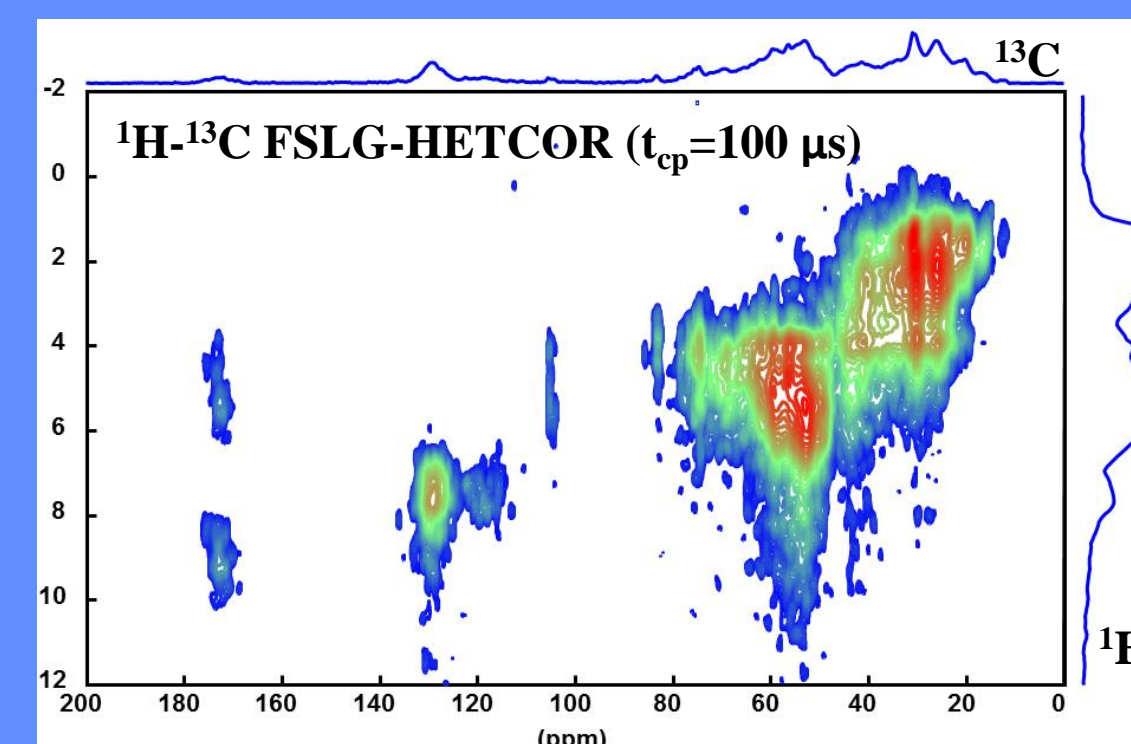
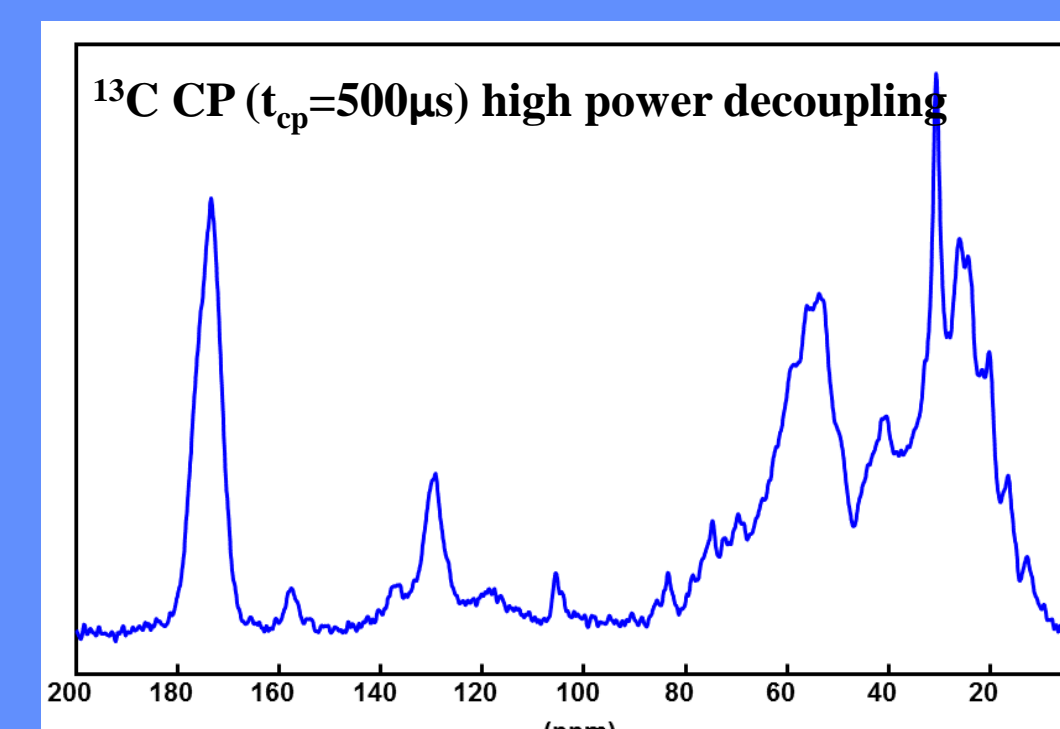
## SDS-TREATED SAMPLE

### MOBILE SPECIES



Liquid-state-inspired NMR experiments mainly highlight the most mobile species: unsaturated lipids. Nevertheless EXSY exp. shows spin diffusion between two broad protons regions.

### RIGID SPECIES



Solid-state NMR experiments allow to probe another part of the sample: the rigid one. Playing with the selected nuclei, spatial proximities can be assessed.

## CONCLUSION AND REFERENCES

While a lot of work is still needed to fully understand diatoms frustule interface, solid-state NMR appears to be a powerful toolbox. Indeed, this technique is able to selectively probe either mobile or rigid species at a very local scale. Varying methods, species proximity can be checked and connectivity evaluated.

- Tesson, B., Masse, S., Laurent, G., Maquet, J., Livage, J., Martin-Jézéquel, V. and Coradin, T. Contribution of multi-nuclear solid state NMR to the characterization of the Thalassiosira pseudonana diatom cell wall. *Anal Bioanal Chem* 390, 1889–1898 (2008).
- Hatté, C., Hodgins, G., Jull, A. J. T., Bishop, B. & Tesson, B. Marine chronology based on <sup>14</sup>C dating on diatoms proteins. *Marine Chemistry* 109, 143–151 (2008).
- Baccile, N., Laurent, G., Bonhomme, C., Innocenzi, P. & Babonneau, F. Solid-State NMR Characterization of the Surfactant–Silica Interface in Templated Silicas: Acidic versus Basic Conditions. *Chem. Mater.* 19, 1343–1354 (2007).