Damien Jeannerat, University of Geneva, Switzerland

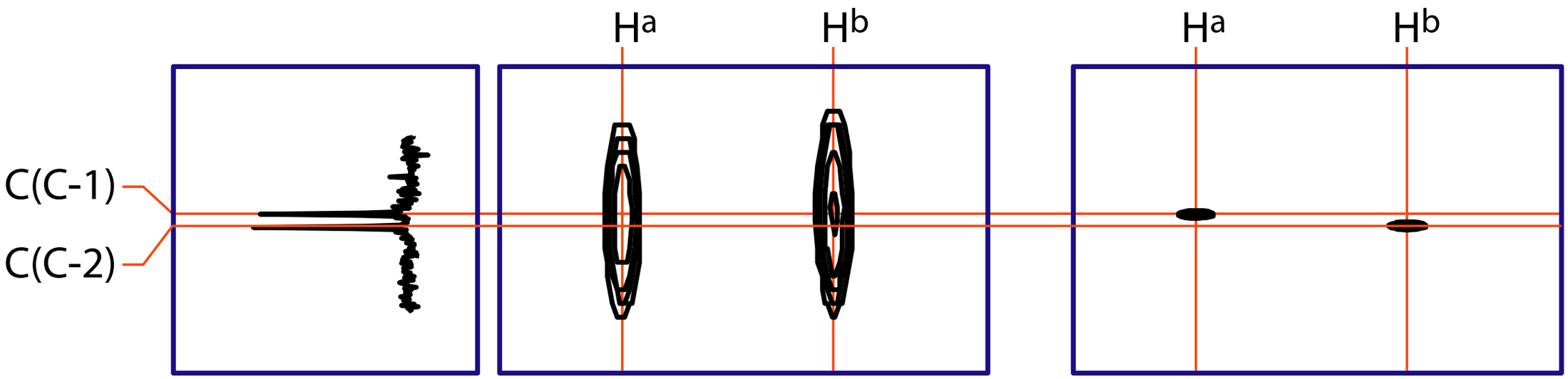
Repliement spectral de la dimention ^{13}C. Un puissant outil d'étude de mélanges complexes de petites molécules utilisants des expériences basées sur l'HSQC

Spectral Aliasing of the ^{13}C dimension. A powerful tool to Study Mixtures of Small Molecules Using HSQC-Based experiments

Do not hesitate to contact:
damien.jeannera@unige.ch

High resolution in indirect dimensions

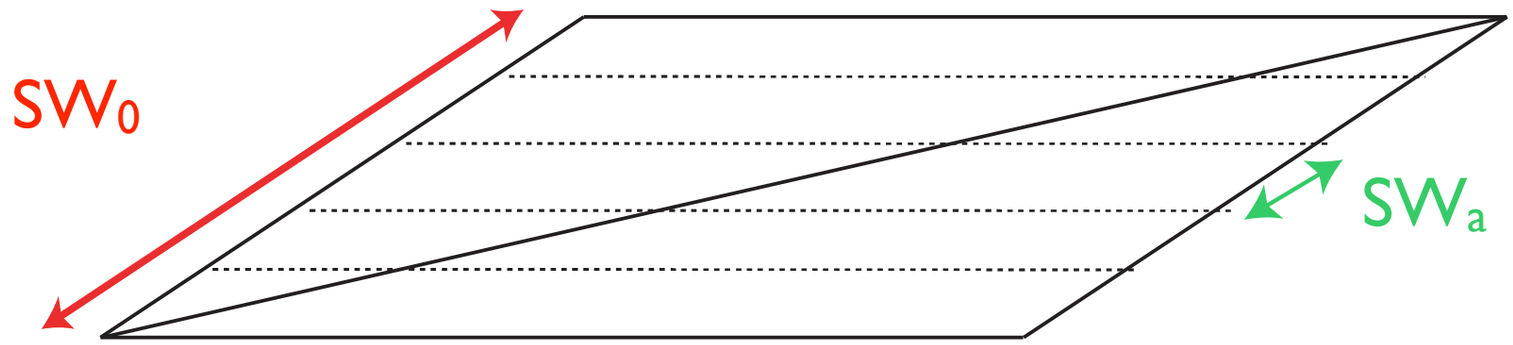
Number of t_1 increments: 128 pt 3200 pt



Acquisition time : 8 min

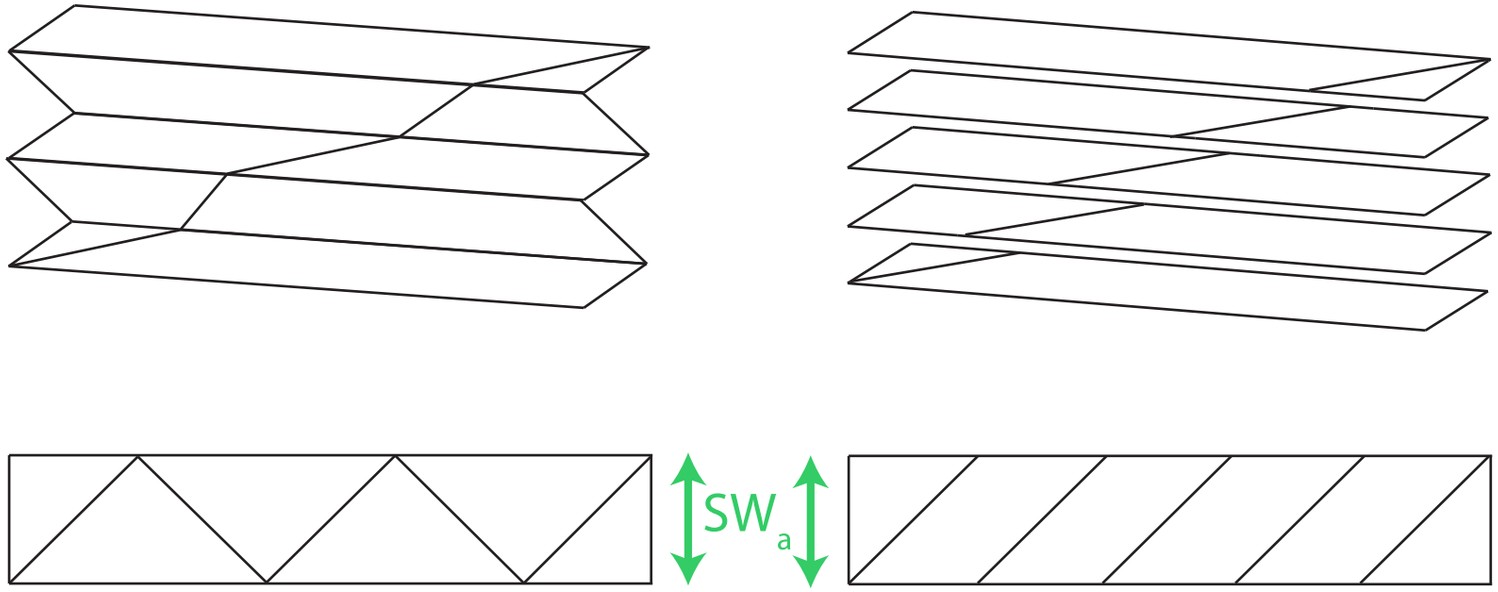
Acquisition time : 3 h 33 min !

Spectral aliasing



TPPI

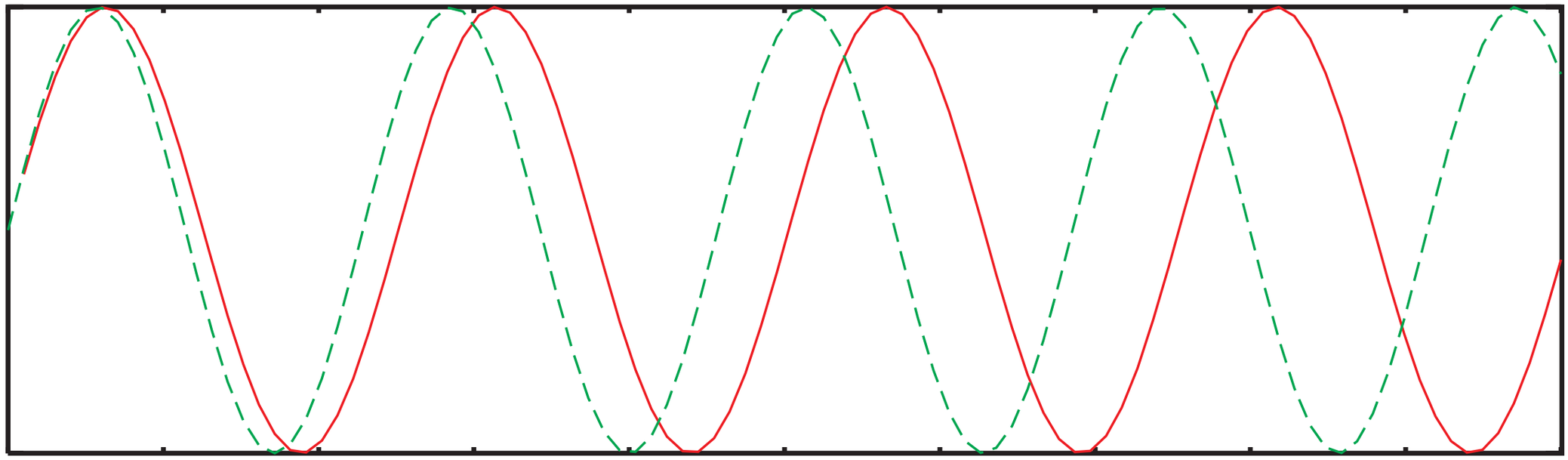
States
Echo/Antiecho
States-TPPI

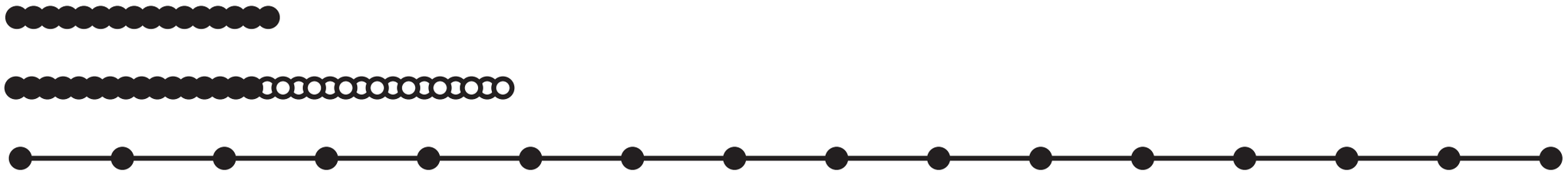


$$\nu_a = \mathrm{mod}(\nu_0 + SW_a/2 - CF, SW_a) - SW_a/2 + CF$$

$$\nu_0 = \nu_a \pm n\, SW_a$$

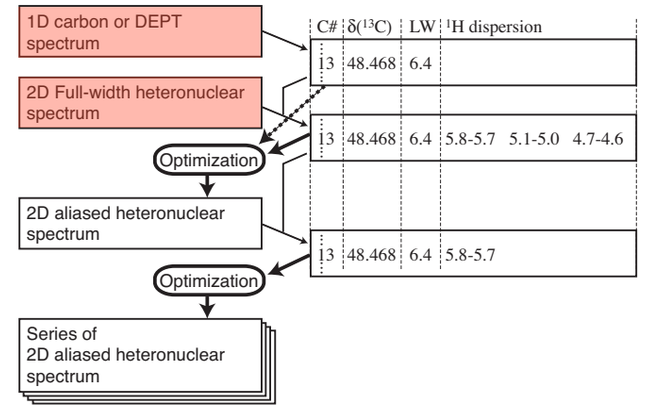
Spectral aliasing



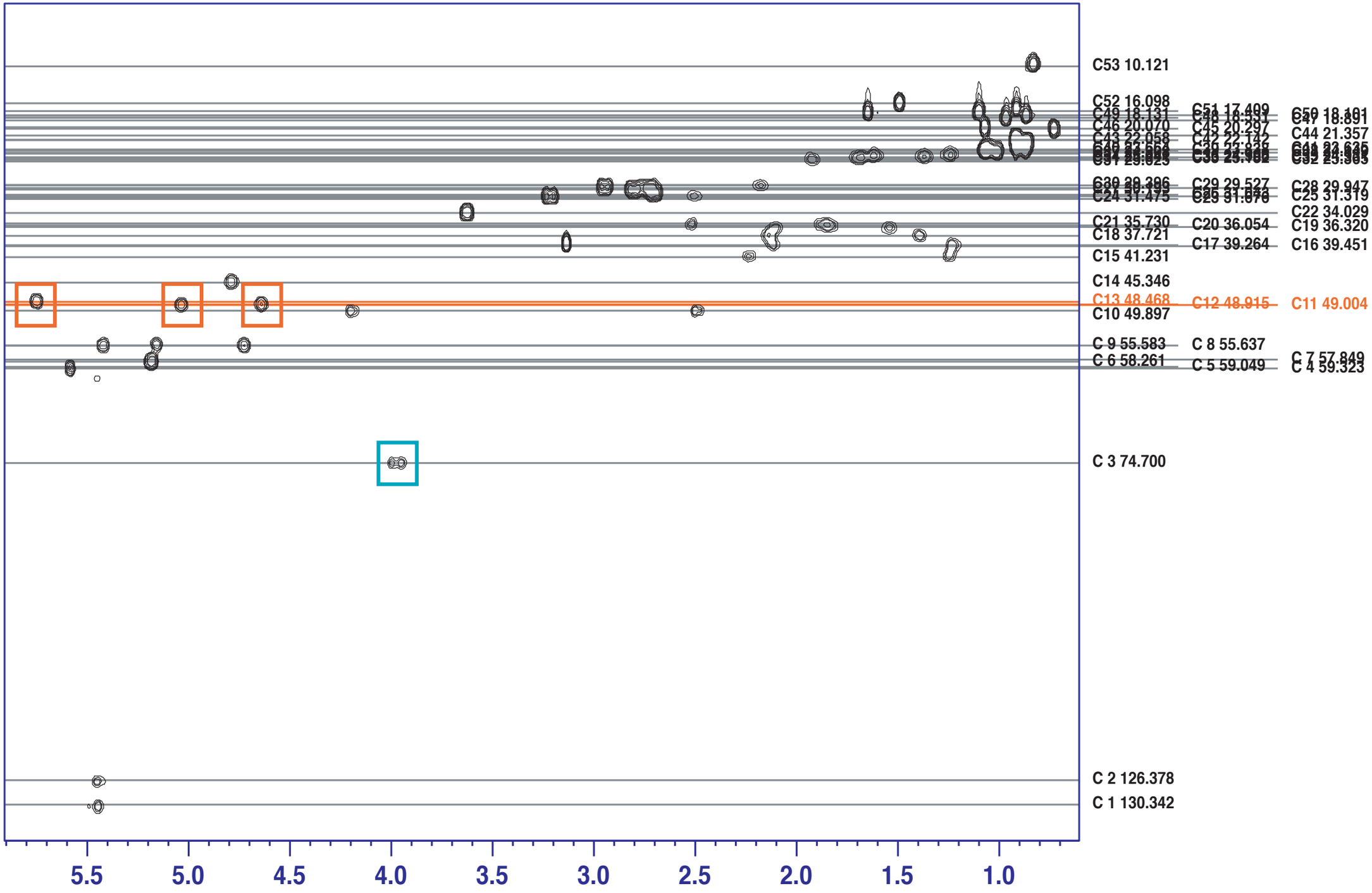


$$\mathrm{DW} = \frac{1}{2\mathrm{SW}}$$

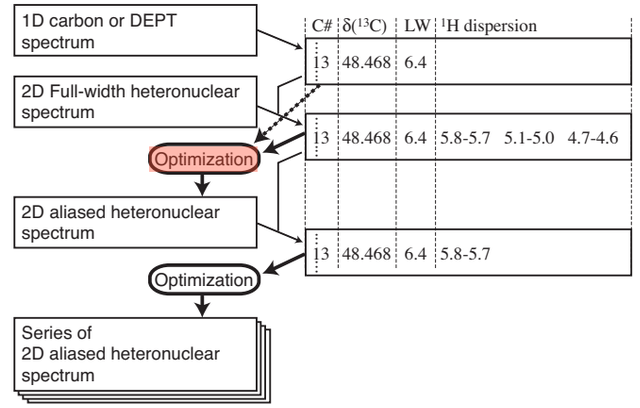
Computer-optimized spectral aliasing



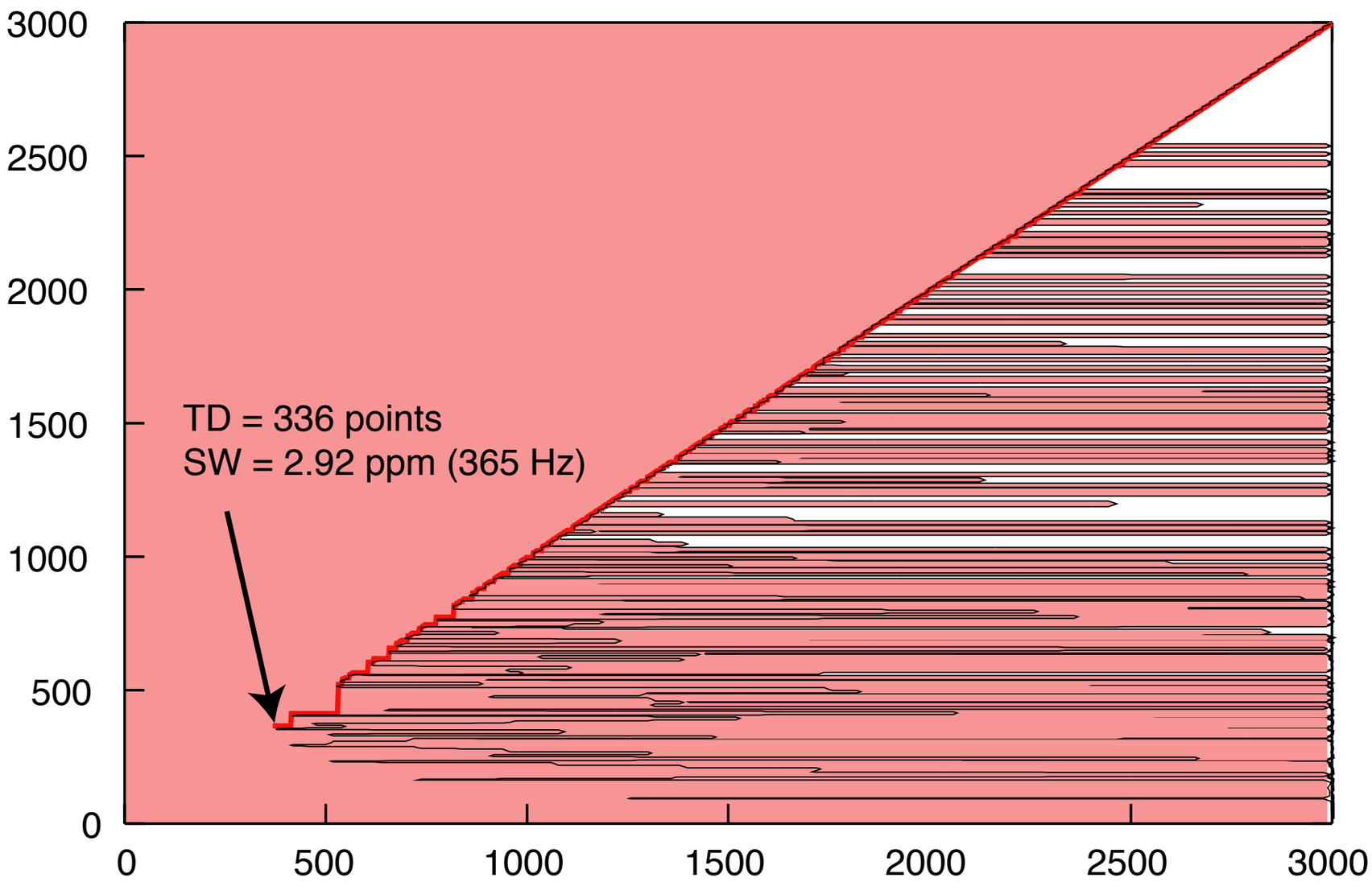
Cyclosporin A $C_{62}H_{111}N_{11}O_{12}$

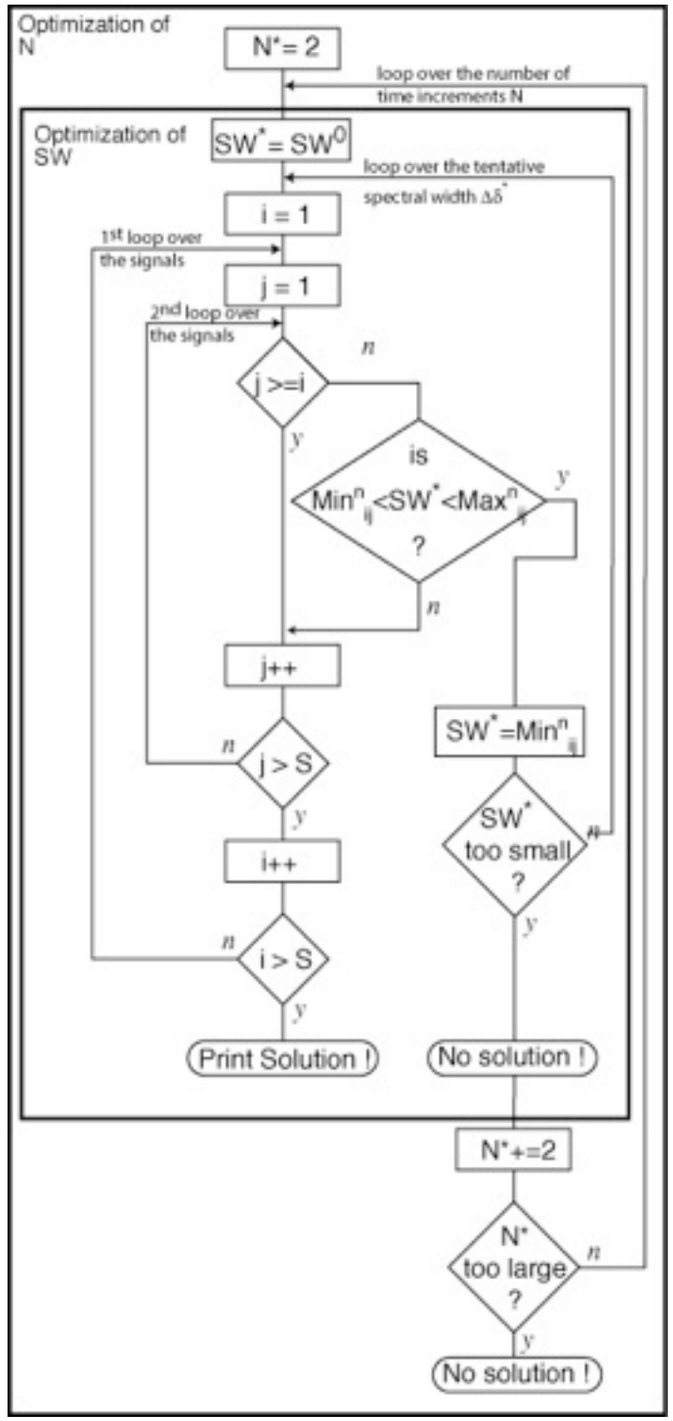


Computer-optimized spectral aliasing

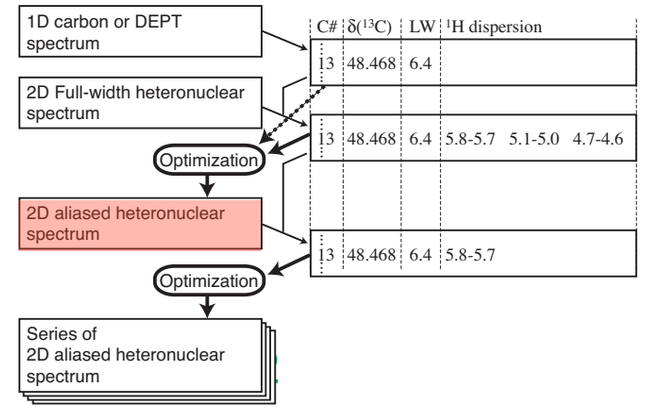


SWa

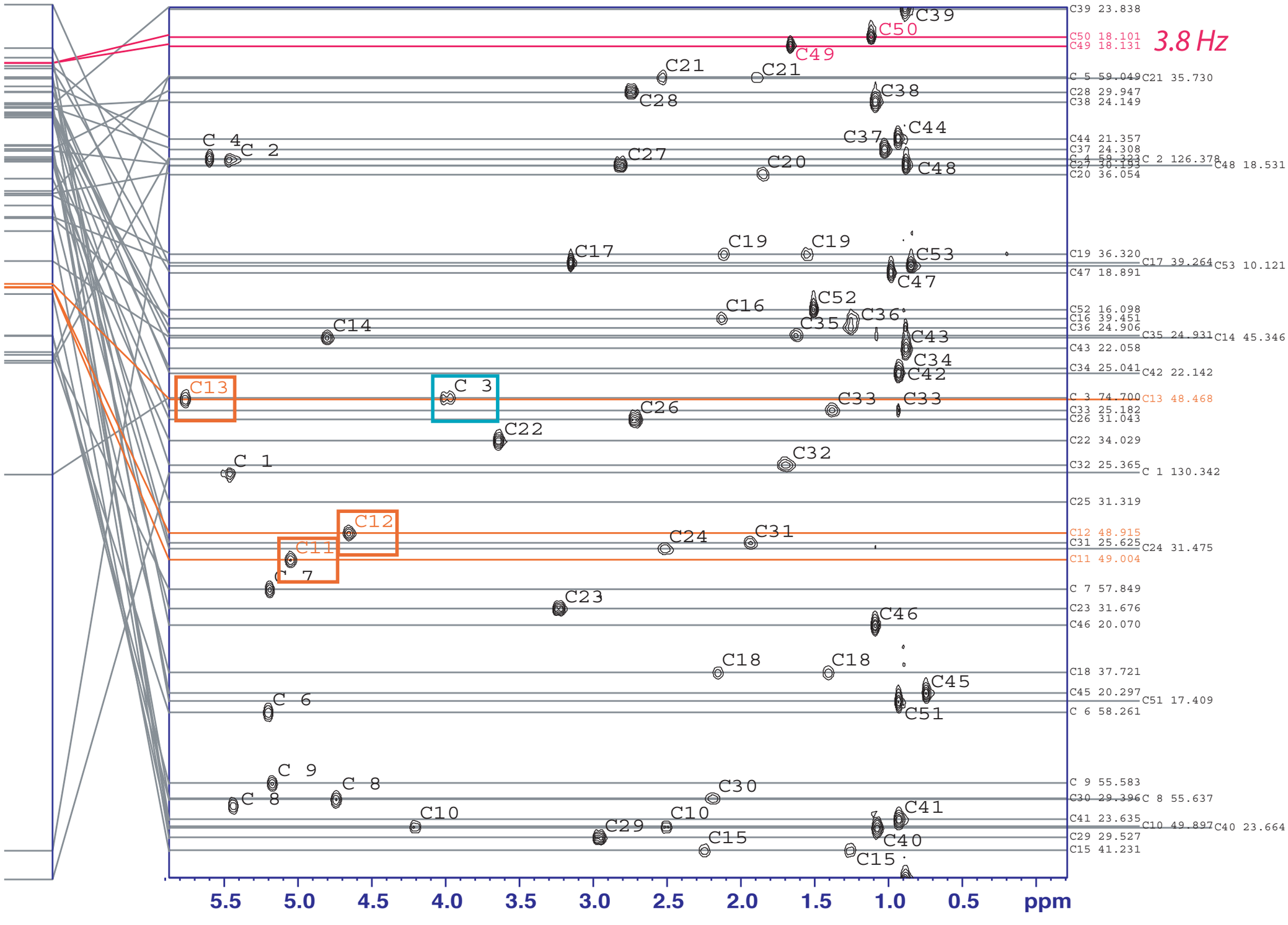




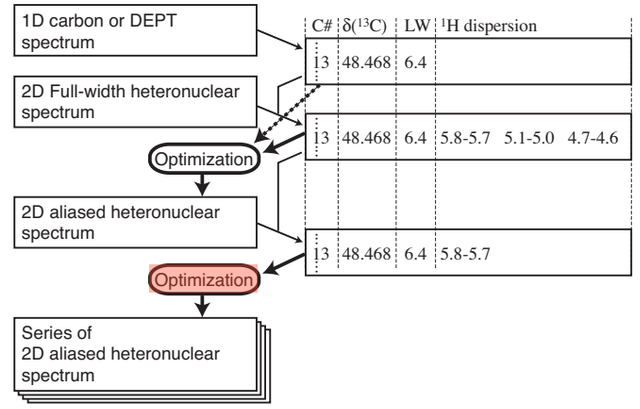
Computer-optimized spectral aliasing



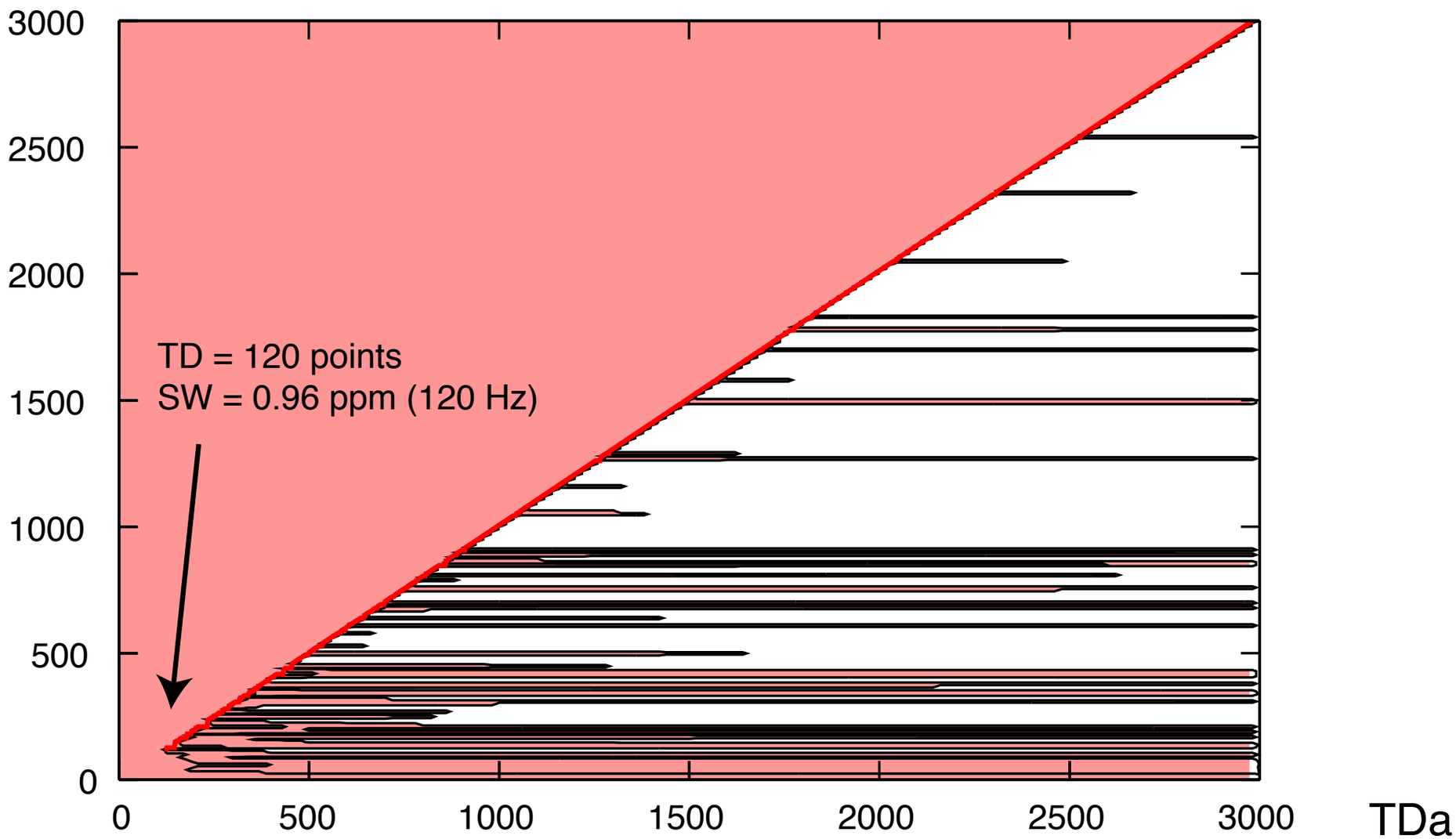
Cyclosporin A $C_{62}H_{111}N_{11}O_{12}$ SWa= 2.92 ppm TD = 336 pt. Max. t_1 = 456.9 ms



Computer-optimized spectral aliasing

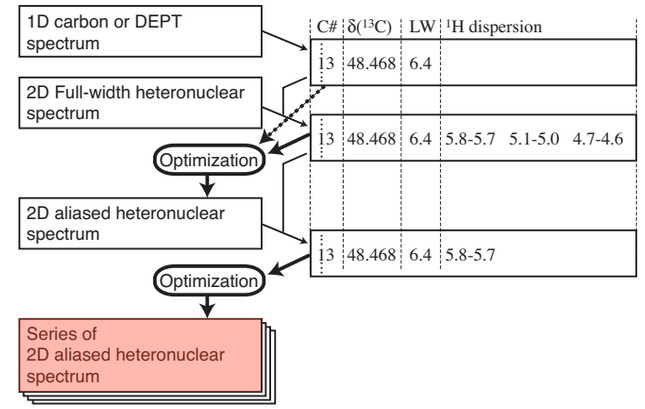


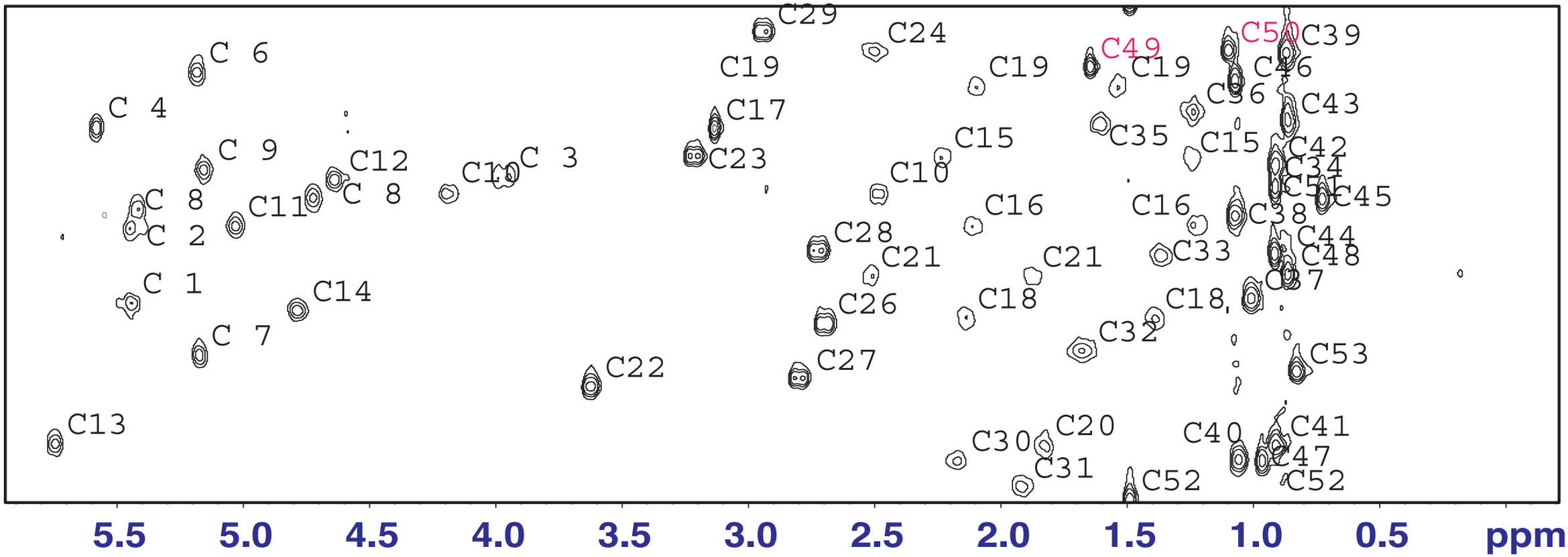
SWa



D. Jeannerat, *J.Magn. Reson.* 186, p112,

Computer-optimized spectral aliasing





Cyclosporin A

$C_{62}H_{111}N_{11}O_{12}$

$SW_a = 0.96$ ppm

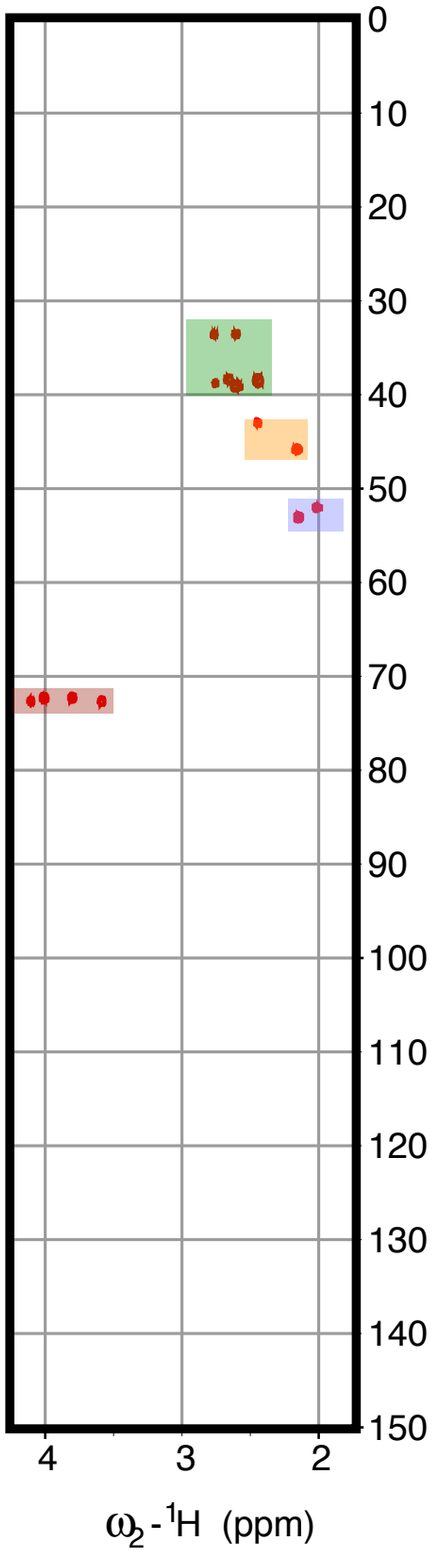
TD=120 pt.

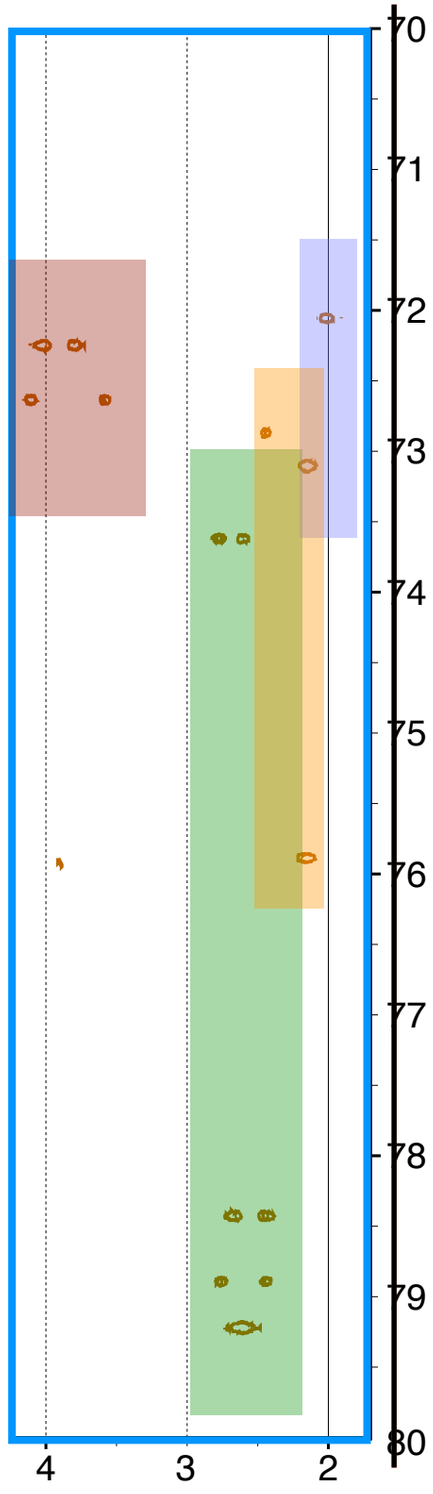
Max. $t_1 = 499.5$ ms

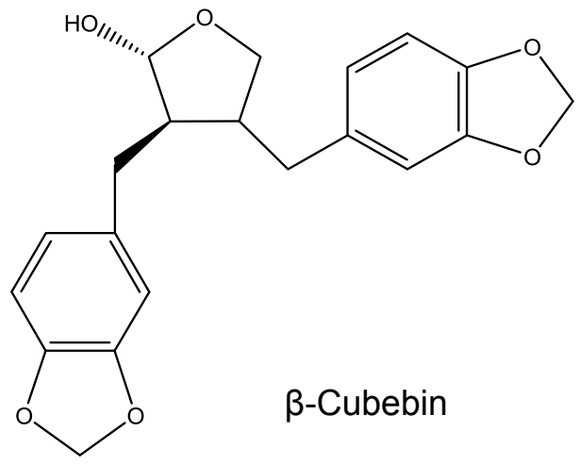
Factor = 125.9

D. Jeannerat, *J.Magn. Reson.* 186, p112,

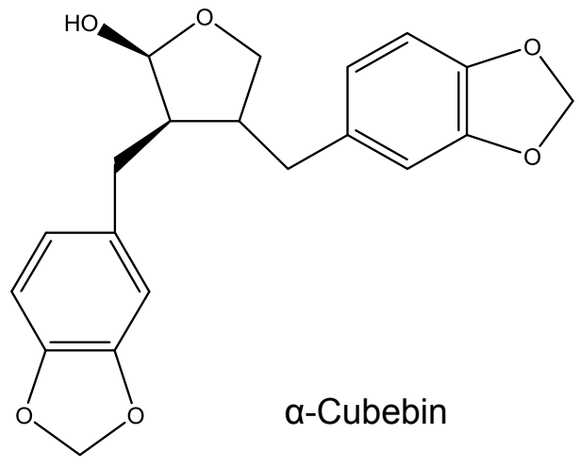
Combining full and 10-ppm spectra





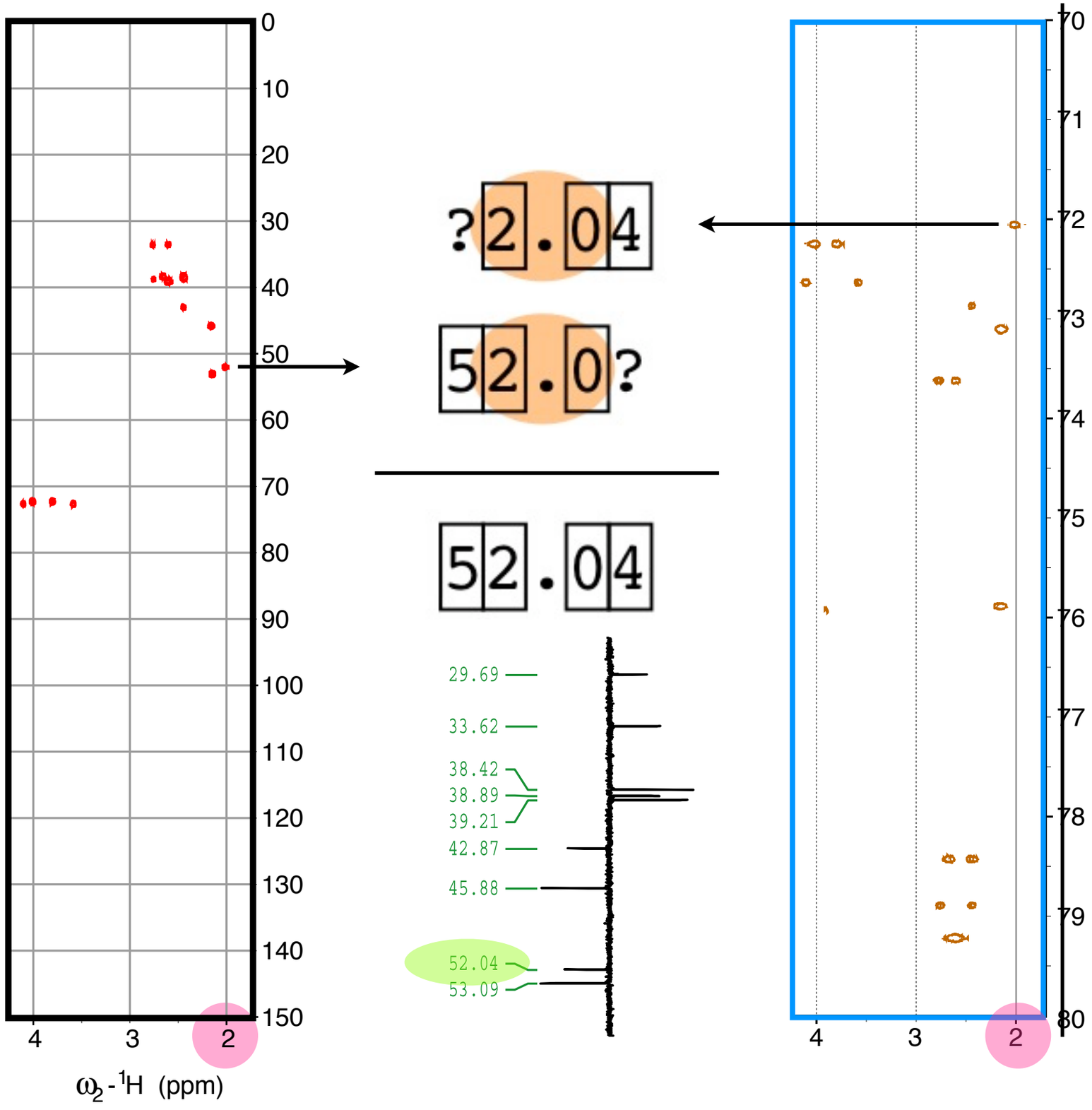


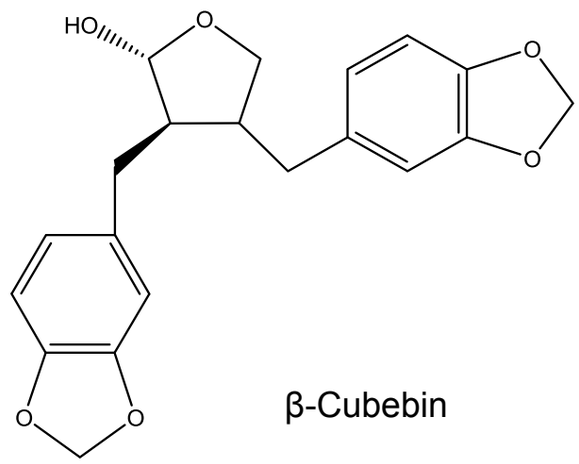
β-Cubebin



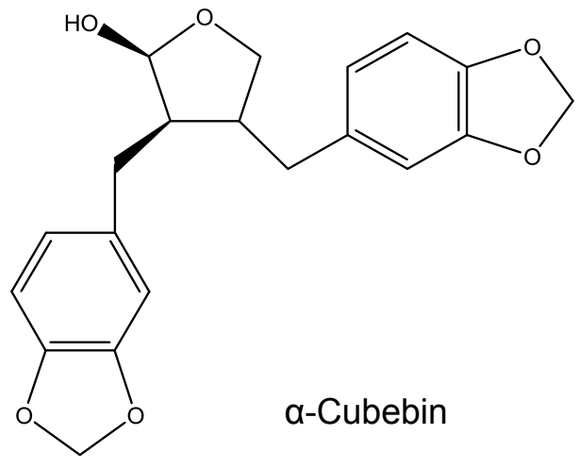
α-Cubebin

Combining full and 10-ppm spectra





β-Cubebin



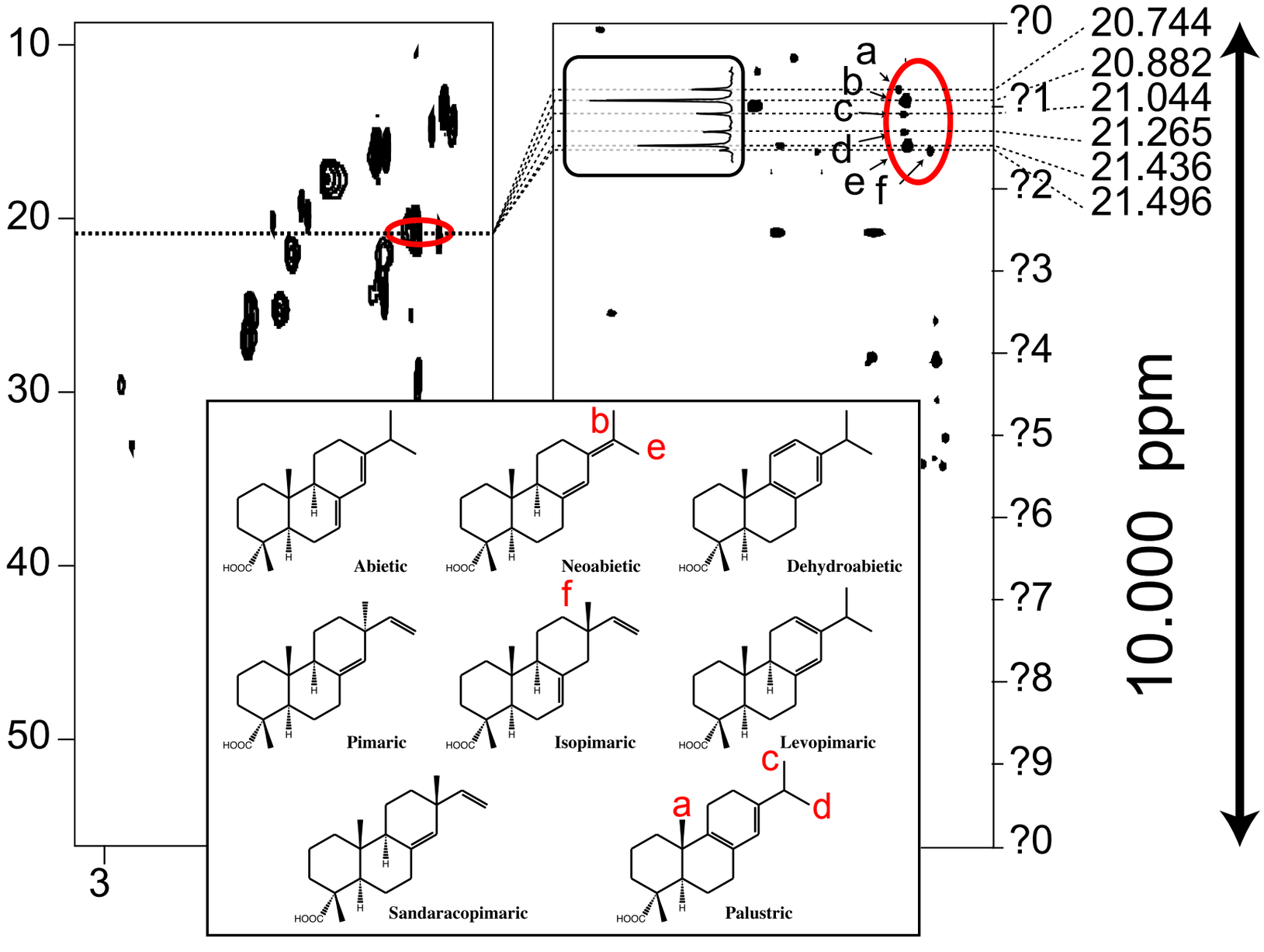
α-Cubebin

Combining full and 10-ppm spectra

COMMUNICATION www.rsc.org/chemcomm | ChemComm

High-precision heteronuclear 2D NMR experiments using 10-ppm spectral window to resolve carbon overlap†

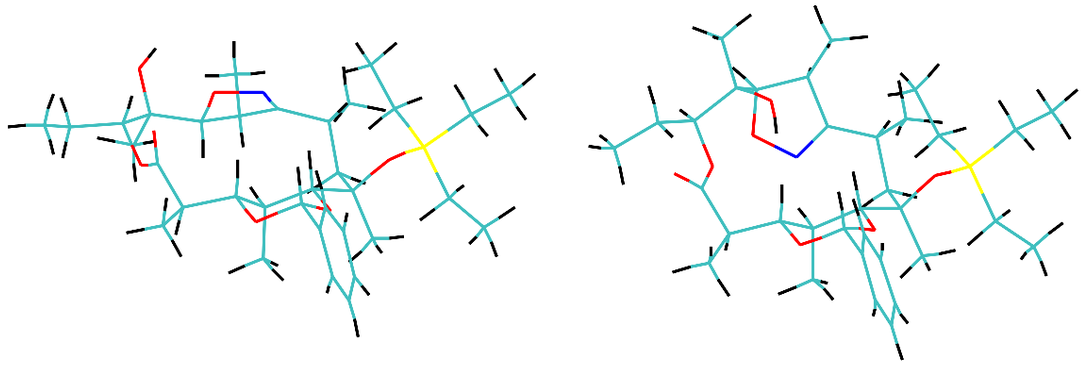
Bruno Vitorge,[a] Stefan Bieri,[b] Munir Humam,[b] Philippe Christen,[b] Kurt Hostettmann,[b] Orlando Muñoz,[c] Sandra Loss[d] and Damien Jeannerat*[a]



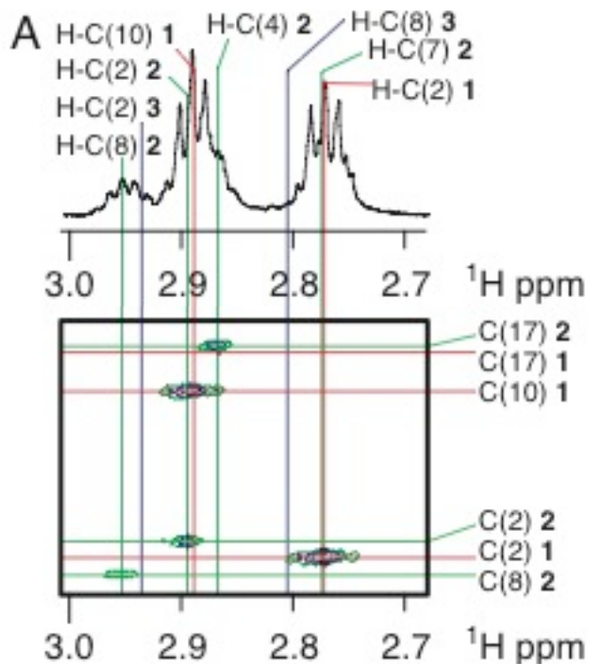
Bruno Vitorge

Applications

Combinations
- 2D HSQC-TOCSY
- 2D HSQC-NOESY



Series
- selective-TOCSY-HSQC

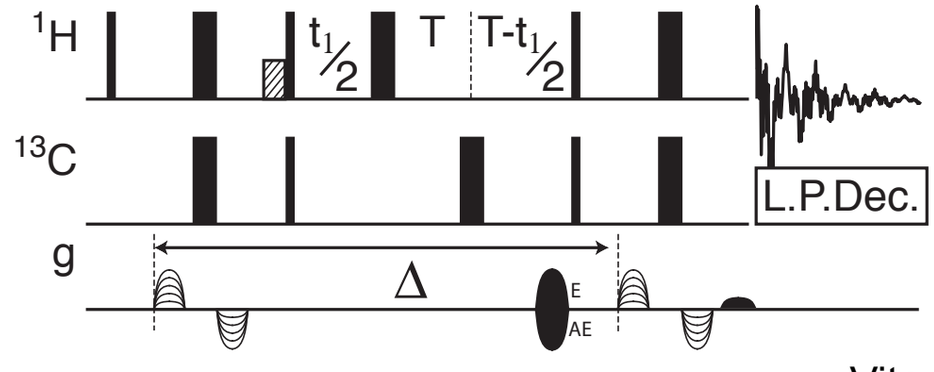


2D-aliasing
- 3D HSQC-TOCSY
- 3D HSQC-COSY

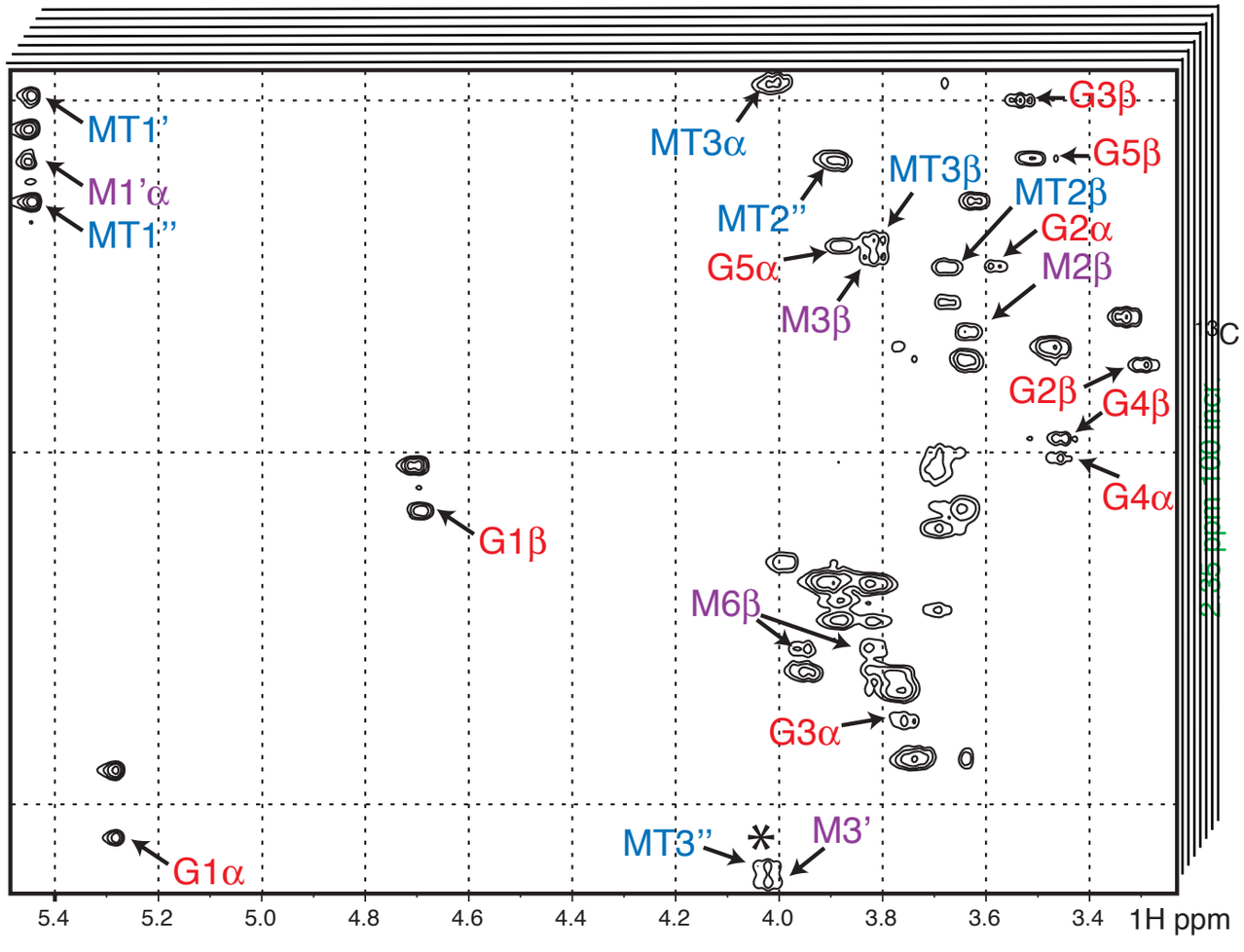
D. Mury, *et al. Magn. Reson. in Chem. (2009),* ***47****, 909*

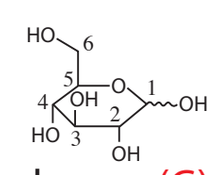
Diffusion measurements

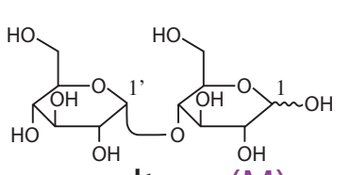
CT-HSQC-IDOSY

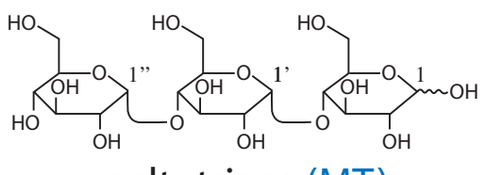


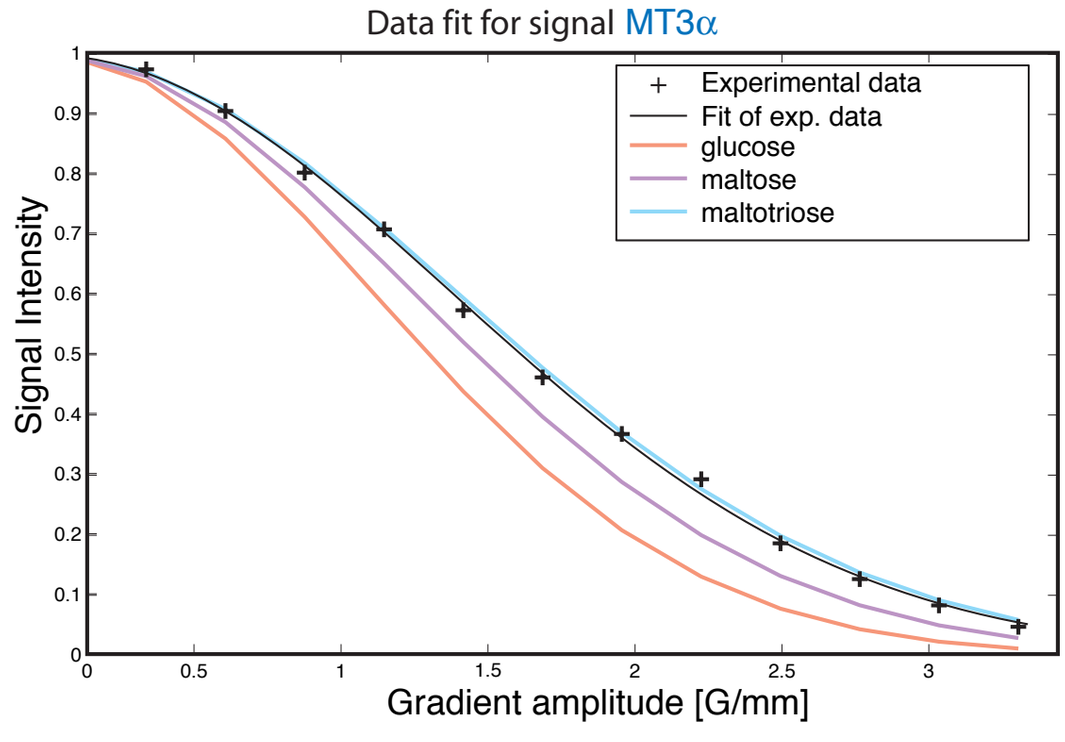
Vitorge *et al.* Anal. Chem. **2006**, *78*, 5601-5606



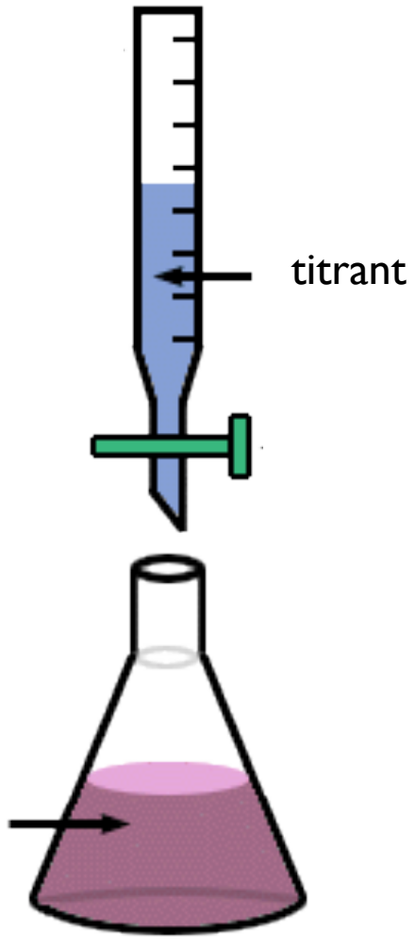




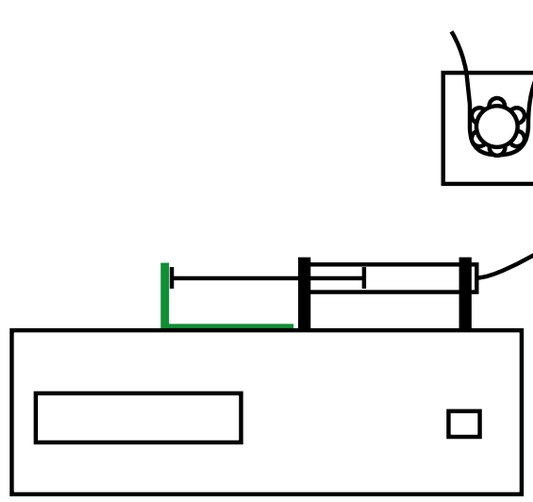


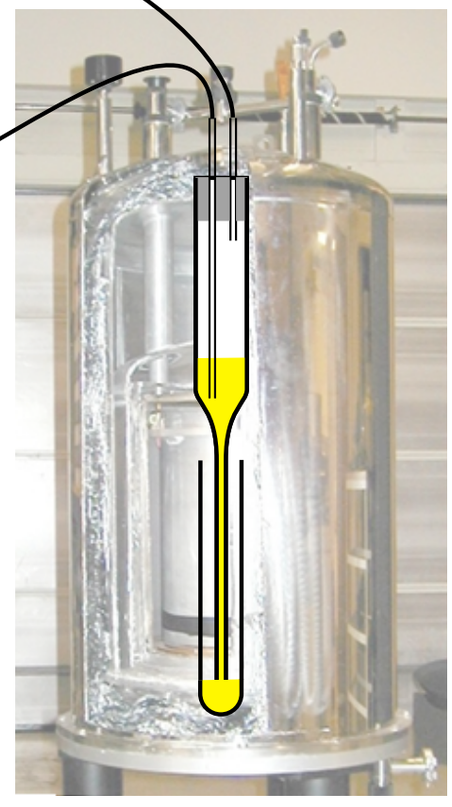


NMR titrations



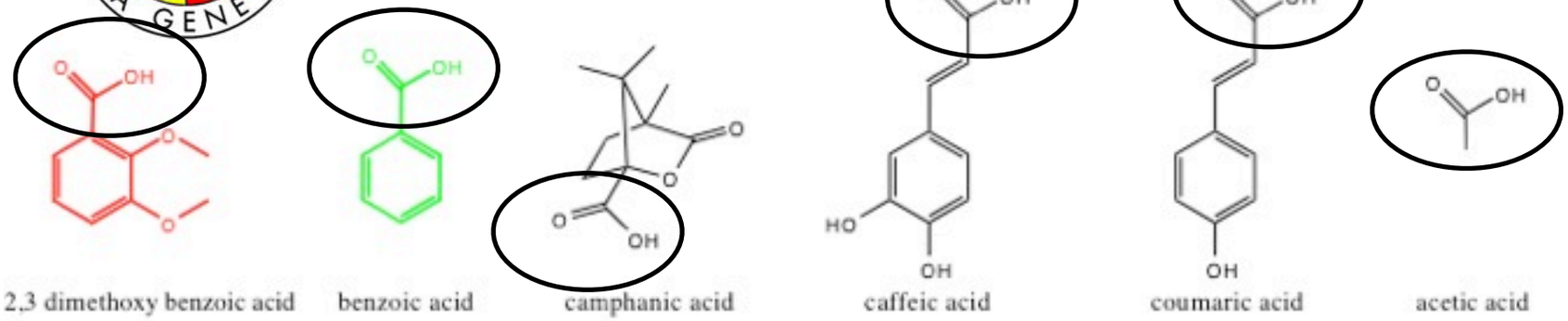
Mixture of compounds



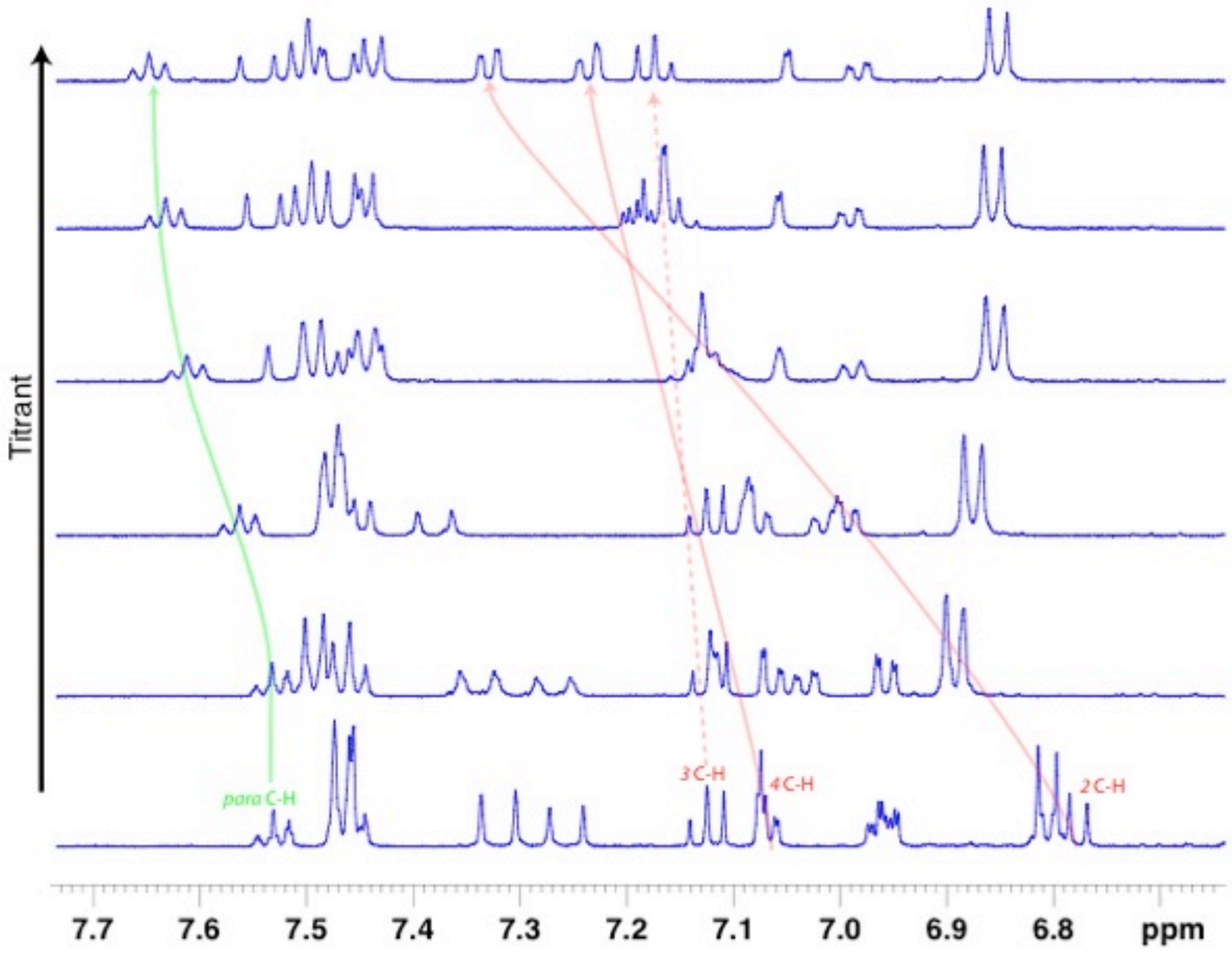


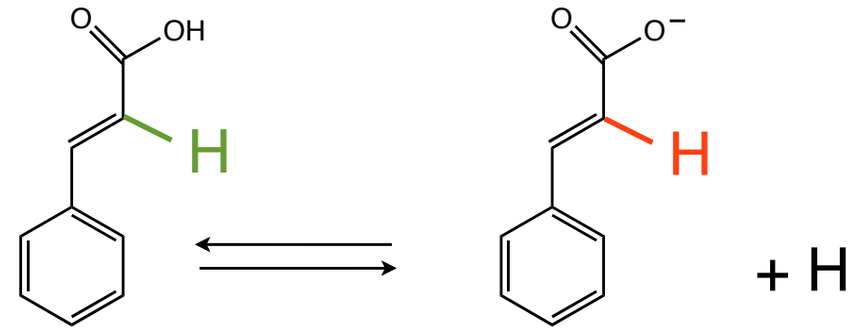
Rupali Shivapurkar

NMR titrations



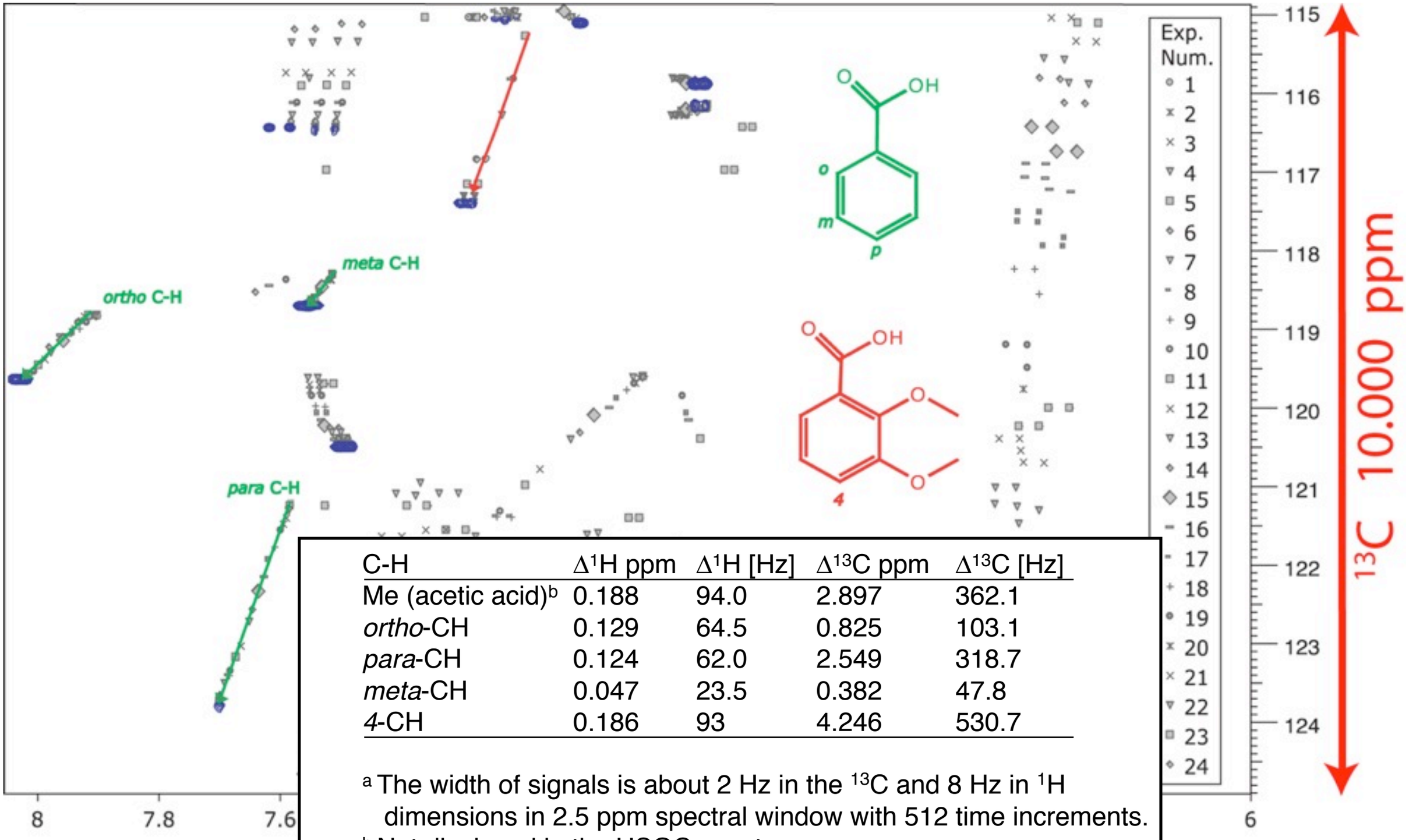
2,3 dimethoxy benzoic acid | benzoic acid | camphanic acid | caffeic acid | coumaric acid | acetic acid





Rupali Shivapurkar

NMR titrations



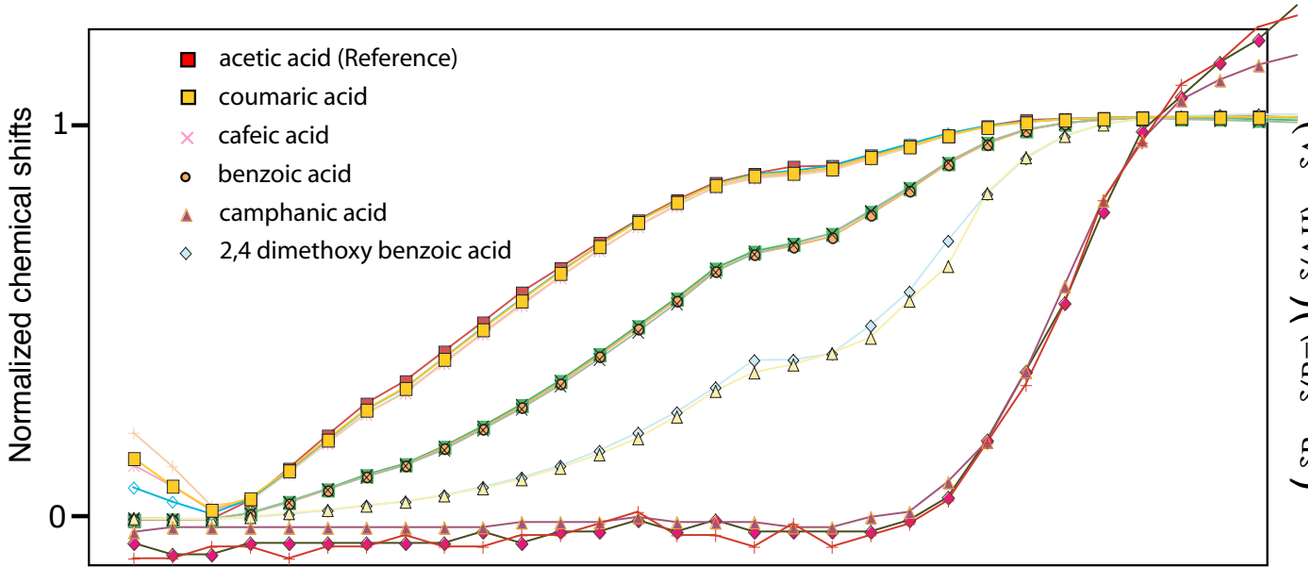
C-H	Δ^1H ppm	Δ^1H [Hz]	Δ^{13}C ppm	Δ^{13}C [Hz]
Me (acetic acid)[b]	0.188	94.0	2.897	362.1
ortho-CH	0.129	64.5	0.825	103.1
para-CH	0.124	62.0	2.549	318.7
meta-CH	0.047	23.5	0.382	47.8
4-CH	0.186	93	4.246	530.7

[a] The width of signals is about 2 Hz in the ^{13}C and 8 Hz in ^{1}H dimensions in 2.5 ppm spectral window with 512 time increments.
[b] Not displayed in the HSQC spectrum.

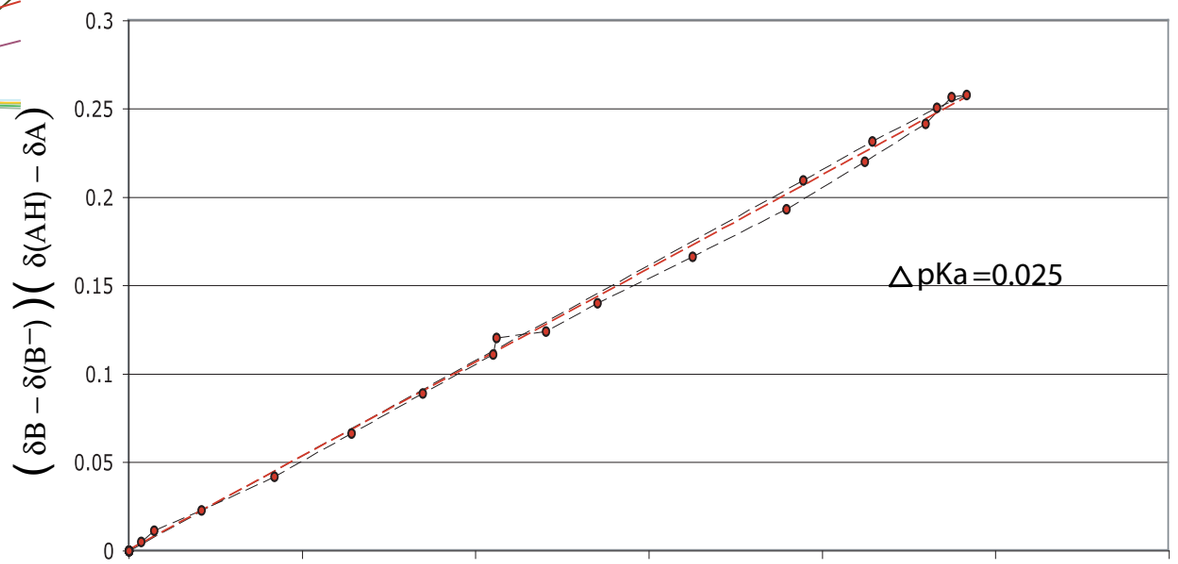
Rupali Shivapurkar

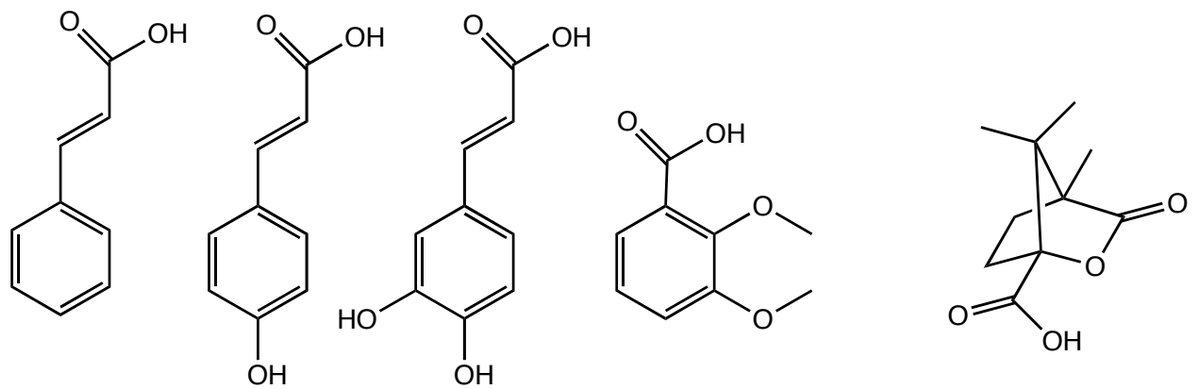
NMR titrations

Normalization of the ^{13}C chemical shifts



Acetic ac / coumaric ac. Δ pKa = 0.025





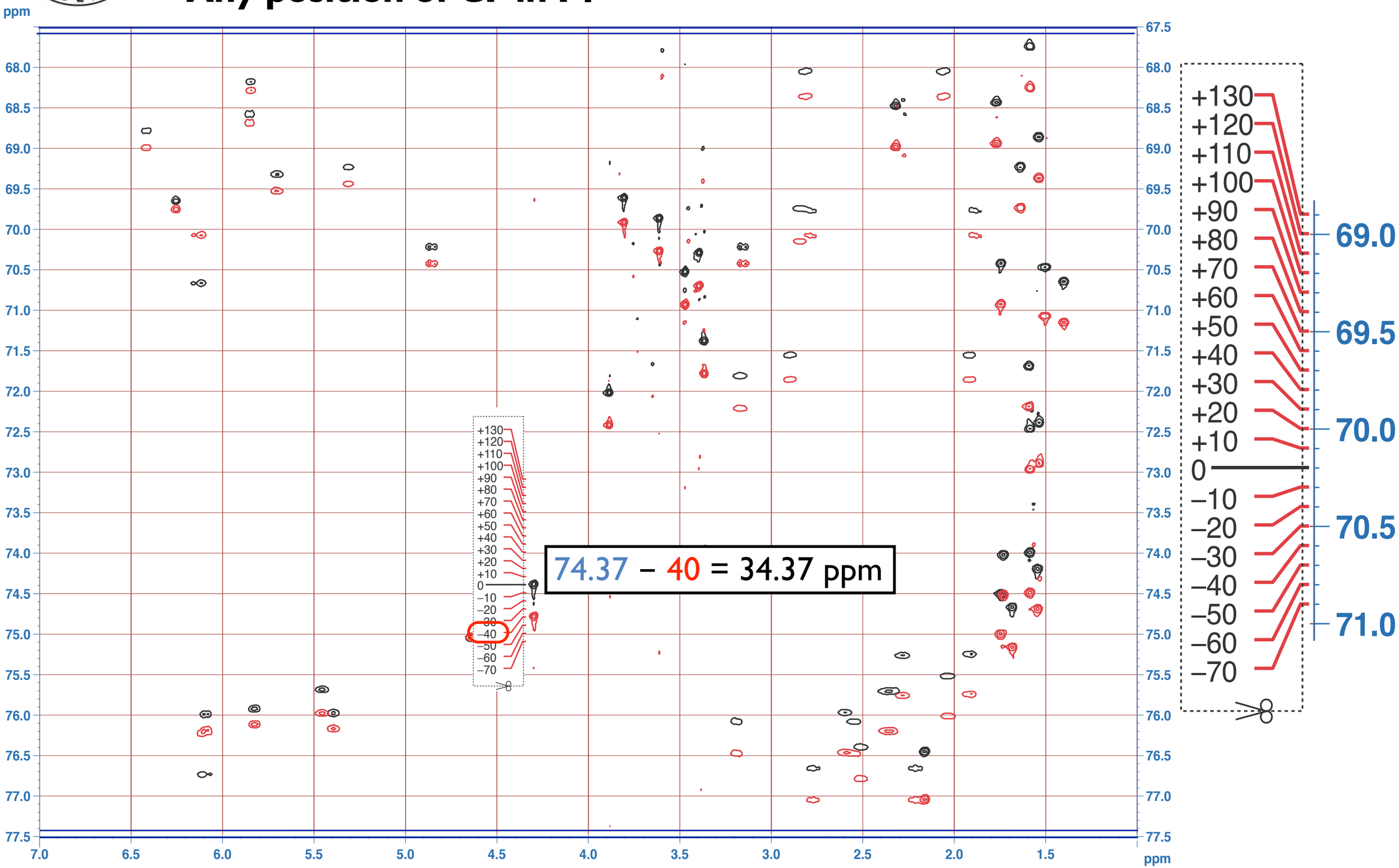
cinnamic acid coumaric acid caffeic acid 2,3 dimethyl benzoic acid camphanic acid

4.24 **4.72** **4.70** **3.71** **2.9**

Rupali Shivapurkar

Combining 10 ppm with 9.9 ppm

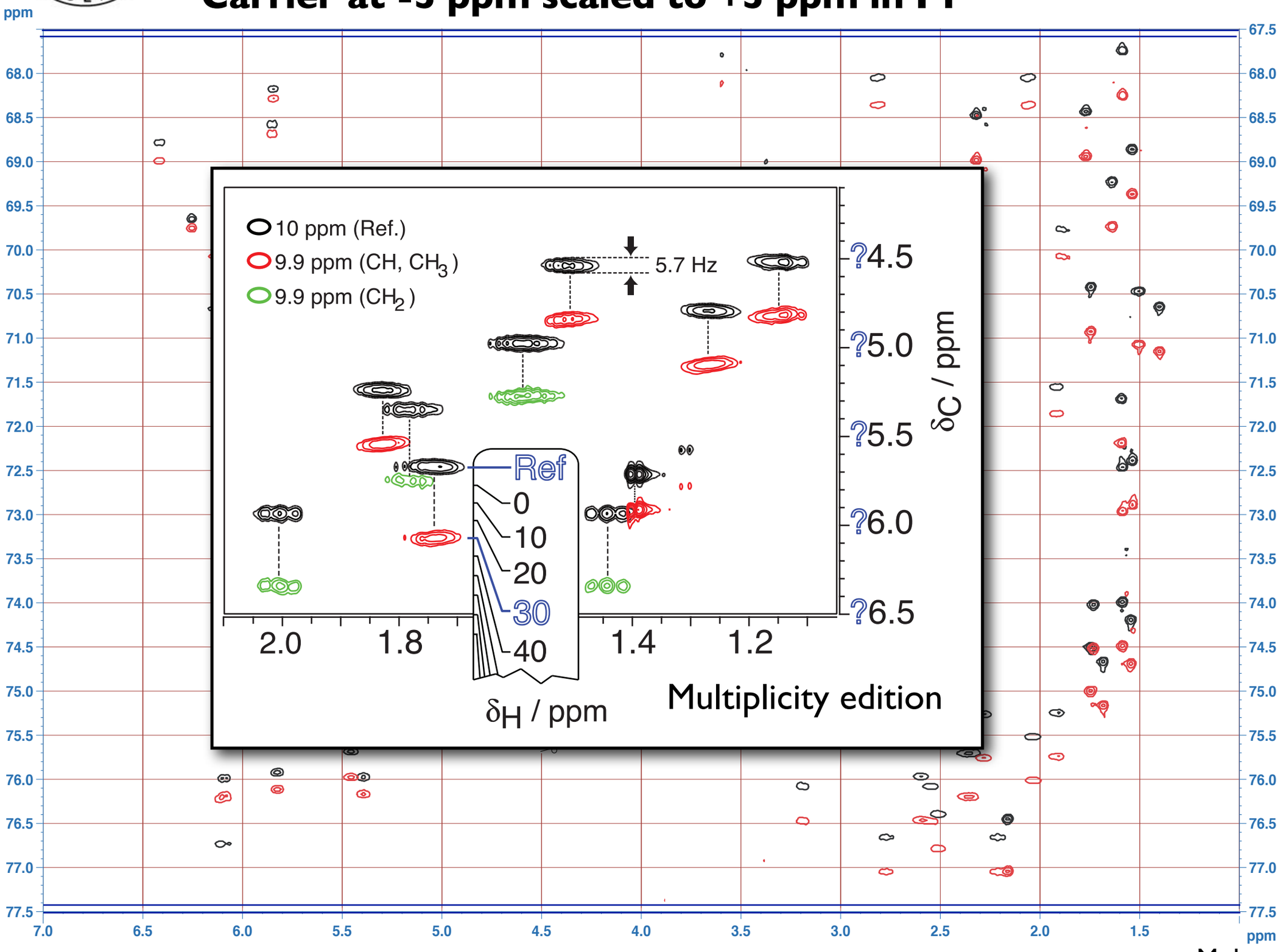
Any position of CF in F1



Mohammadali Foroozandeh

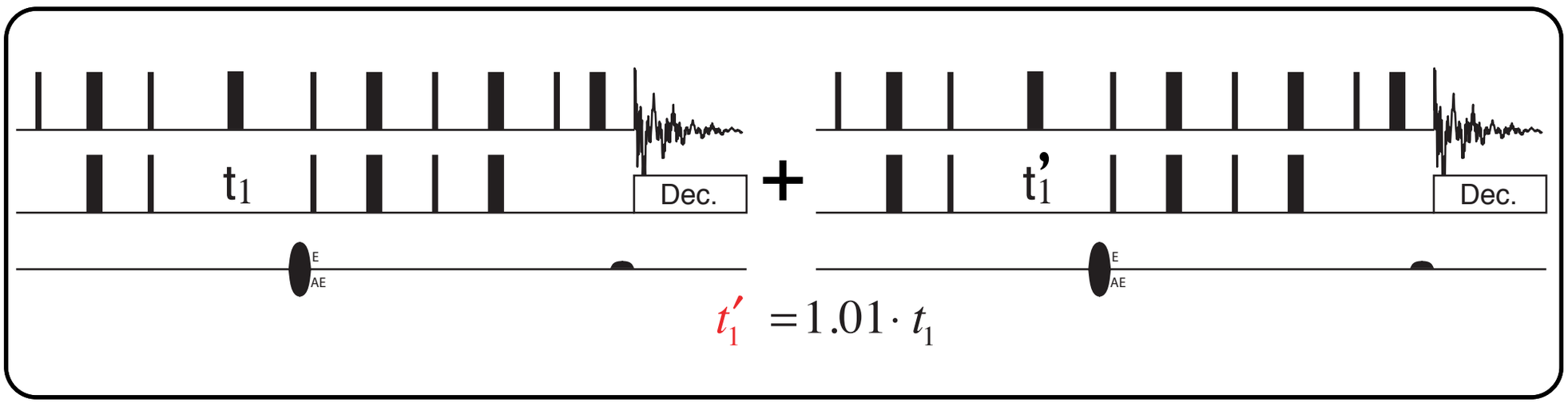
Combining 10 ppm with 9.9 ppm

Carrier at -5 ppm scaled to +5 ppm in F1



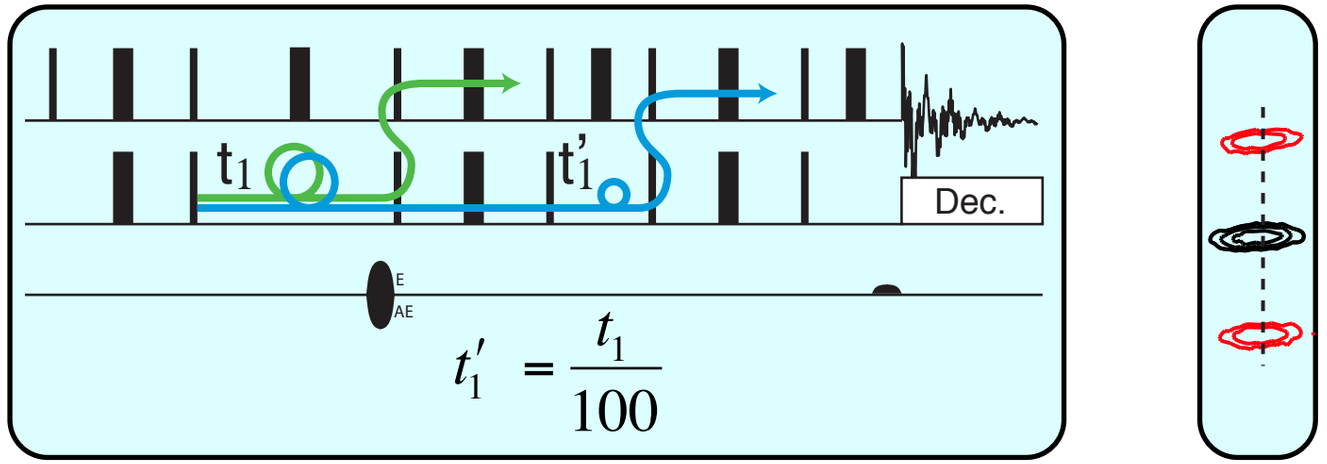
Mohammadali Foroozandeh

Combining 10 ppm with 9.9 ppm



DENA-HSQC

Differential Evolution for Non-ambiguous Aliasing



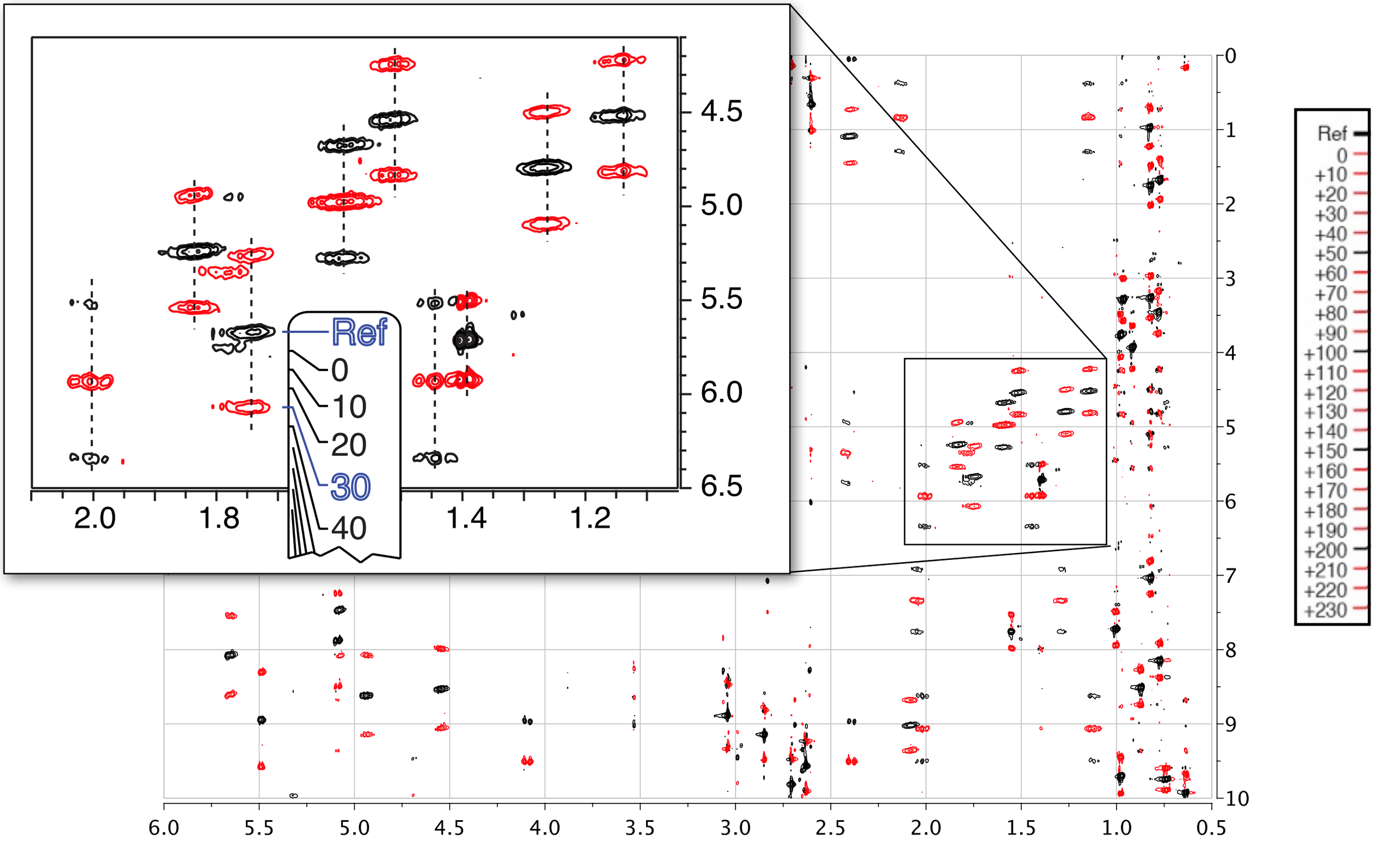
$$\cos(\omega t_1) I_z S_y$$

$$\sin(\omega t_1) I_z S_x$$

$$\cos(\omega t_1)\cos(\omega t_1') = \cos(\omega t_1 + \omega t_1') + \cos(\omega t_1 - \omega t_1')$$

$$\sin(\omega t_1)\cos(\omega t_1') = \sin(\omega t_1 + \omega t_1') + \sin(\omega t_1 - \omega t_1')$$

DENA-HSQC spectra



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Switzerland

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Bruno Vitorge, HSQC-IDOSY, HMBC, 10 ppm experiments

PhD Students

Rupali Shivapurkar, Titration experiments

Mohammadali Foroozandeh, Q3 sign alternation and 10.1 ppm

Technicians

André Pinto

Stéphane Grass

Patrick Romanens

Collaborations

Jean-Marc Nuzillard, *Univ. Champagne-Ardenne, Reim, France*

Philippe Christen, *Phytochemistry, Univ. Geneva, Switzerland*

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