

# **Chiral recognition by dissolution DNP NMR spectroscopy of $^{13}\text{C}$ -labeled DL-methionine**

E. Monteagudo,<sup>a</sup> A. Virgili,<sup>b</sup> T. Parella<sup>a</sup> and M. Pérez-Trujillo\*<sup>a</sup>

\* To whom correspondence should be addressed

<sup>a</sup> Servei de Ressonància Magnètica Nuclear, Universitat Autònoma de Barcelona, E-08193 Cerdanyola del Vallès, Barcelona, Spain

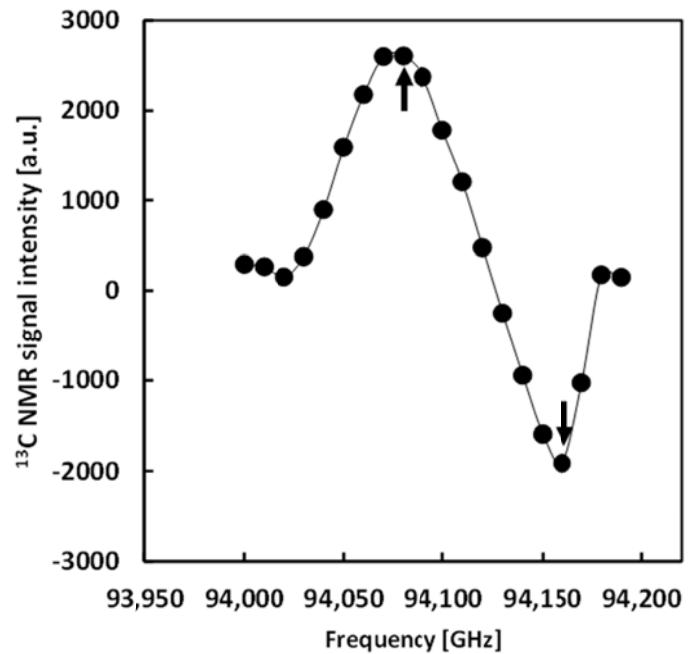
<sup>b</sup> Departament de Química, Facultat de Ciències i Biociències, Universitat Autònoma de Barcelona, E-08193 Cerdanyola del Vallès, Barcelona, Spain

Supporting information includes:

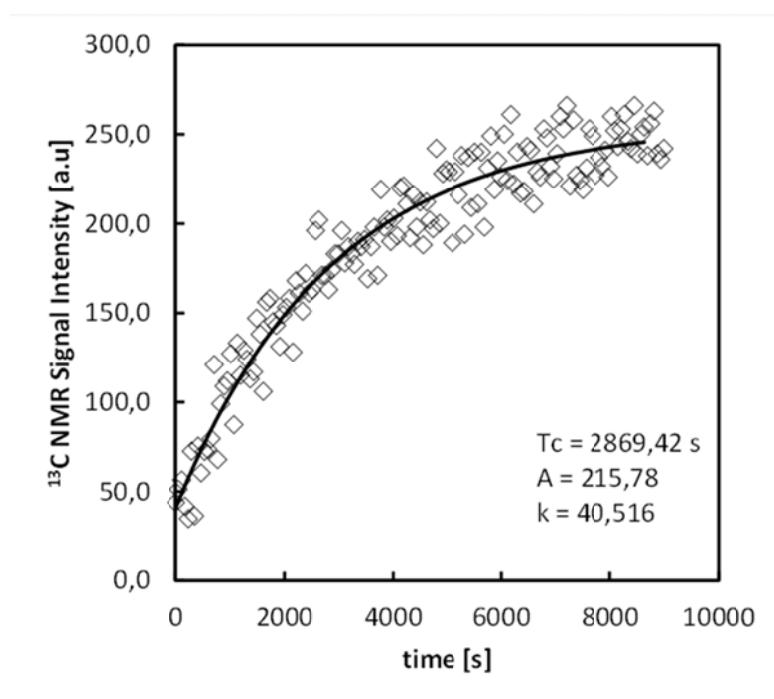
## **Results**

- Curve for the determination of the optimal  $^{13}\text{C}$  microwave irradiation frequency (Figure S1).
- Solid-state  $^{13}\text{C}$  polarization build-up curve of DL-[1- $^{13}\text{C}$ ]-methionine (Figure S2).
- $^{13}\text{C}$  NMR spectra of the d-DNP NMR enantiodifferentiated sample at thermal equilibrium acquired with 1scan (Figure S3).

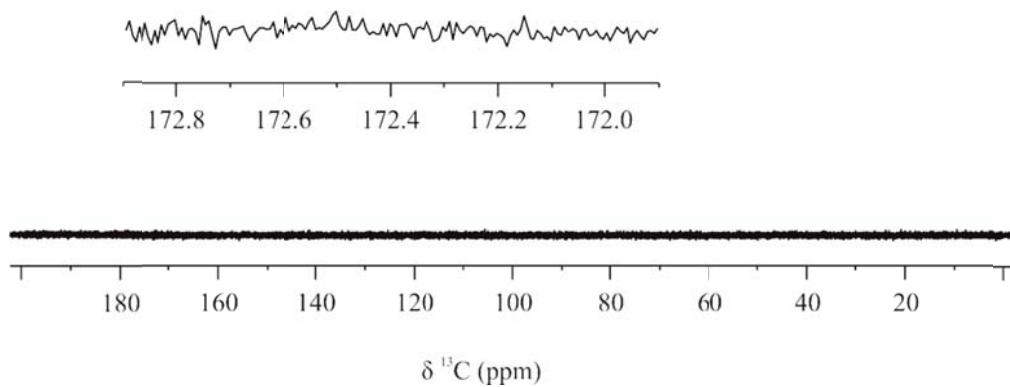
## Results



**Figure S1.** <sup>13</sup>C microwave spectrum of 50 µl (53,3 mg) of [1-<sup>13</sup>C]-pyruvic acid doped with trityl radical OX63 (15 mM) in a matrix of H<sub>2</sub>O:glycerol (1:1). Sample was polarized for 3 min at each different frequency. Arrows indicate the P(+) = 94,078 GHz and P(-) = 94,160 GHz, respectively.



**Figure S2.** Solid-state  $^{13}\text{C}$  polarization build-up curve of DL-[1- $^{13}\text{C}$ ]-methionine (50  $\mu\text{l}$ , 224 mM) with trityl radical OX63 (15 mM) into  $\text{H}_2\text{O}:\text{glycerol}$  (1:1).



**Figure S3.** 150.92 MHz thermal equilibrium single scan <sup>13</sup>C NMR spectrum (expt 1 s) of DL-[1-<sup>13</sup>C]-methionine (2.2 mM) with CSA (-)-18C6H4 (15 eq). The sample contains also trityl radical OX63, glycerol and H<sub>2</sub>O.