**Supporting Information**

**for**

# Selective 1D TOCSY NMR Experiments for a Rapid Identification of Minor Components in the Lipid Fraction of Milk and Dairy Products: Towards Spin Chromatography?

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**FIGURE CAPTIONS**

**Figure S1.** (a) 500 MHz $^{1}$H NMR spectrum of 20 mM solution of the (9-trans, 11-trans) 18:2 conjugated linoleic acid in $CDCl_3$ (T= 298 K, acquisition time= 4.3 s, relaxation delay= 5 s, number of scans= 256, experimental time ~25 min). (b)-(e) selective 1D TOCSY spectra of the above solution using a mixing time of $\tau_m$ = 33 ms (b), 70 ms (c), 200 ms (d), and 400 ms (e). The asterisk denotes the selected target resonance which was excited. For (b)-(e) the magnetization transfer network is illustrated.

**Figure S2.** (a) 500 MHz $^{1}$H NMR spectrum of the lipid fraction of a lyophilized milk sample in $CDCl_3$ (T= 298 K, number of scans= 256, acquisition time= 4.3 s, relaxation delay= 5 s, total experimental time ~ 25 min). The insert shows x512 magnification of the spectrum in order to display resonances from the 18:2 CLA and other minor species. (b) – (d) 1D TOCSY spectra of the above solution with $\tau_m$ = 400 ms (total experimental time ~25 min). The asterisks denote the selected target resonances which were excited.

**Figure S3**. (a) Selected region of the 1D $^{1}$H NMR spectrum of the solution of Figure 2(a). The asterisks denote the position of the selected target resonances which were excited. (b) and (c) selective 1D TOCSY spectra of the above solution. In (b) $\tau_m$= 400 ms was used for magnetization transfer from H11 to H9 of the (9-cis, 11-trans) 18:2 CLA. In (c) $\tau_m$= 70 ms was used in order to optimize magnetization transfer from H10, 11 to H9,12 of the (9-trans, 11-trans) 18:2 CLA.

**Figure S4.** Selected regions of 500 MHz (a) 2D TOCSY spectrum of the lipid fraction of a lyophilized cheese sample in $CDCl_3$ (32 repetitions of 512 increments, total experimental time 5h 5 min); (b) (A) and (B) are the extracted column and row, respectively, on the H11 resonance of

the (9-cis, 11-trans) 18:2 CLA isomer of the 2D TOCSY spectrum as displayed in (a); (C) 1D TOCSY of the same solution as in (a) with the selective pulse on the H11 resonance of the (9-cis, 11-trans) 18:2 CLA isomer is illustrated for comparative reasons (T= 298 K, number of scans = 256, acquisition time = 4.3 s, relaxation delay 5 s, total experiment time ~25 min).

**Figure S5.** 1D TOCSY quantitation procedure: (a) 1D TOCSY spectrum of the milk sample of Figure 5 and Figure S2 with the selective pulse on the H9 resonance of caproleic acid ($\tau_m$= 200 ms); (b) and (c) successive additions of 1.00 and 0.98 mM (in tube) of caproleic acid, respectively ($\tau_m$= 200 ms).

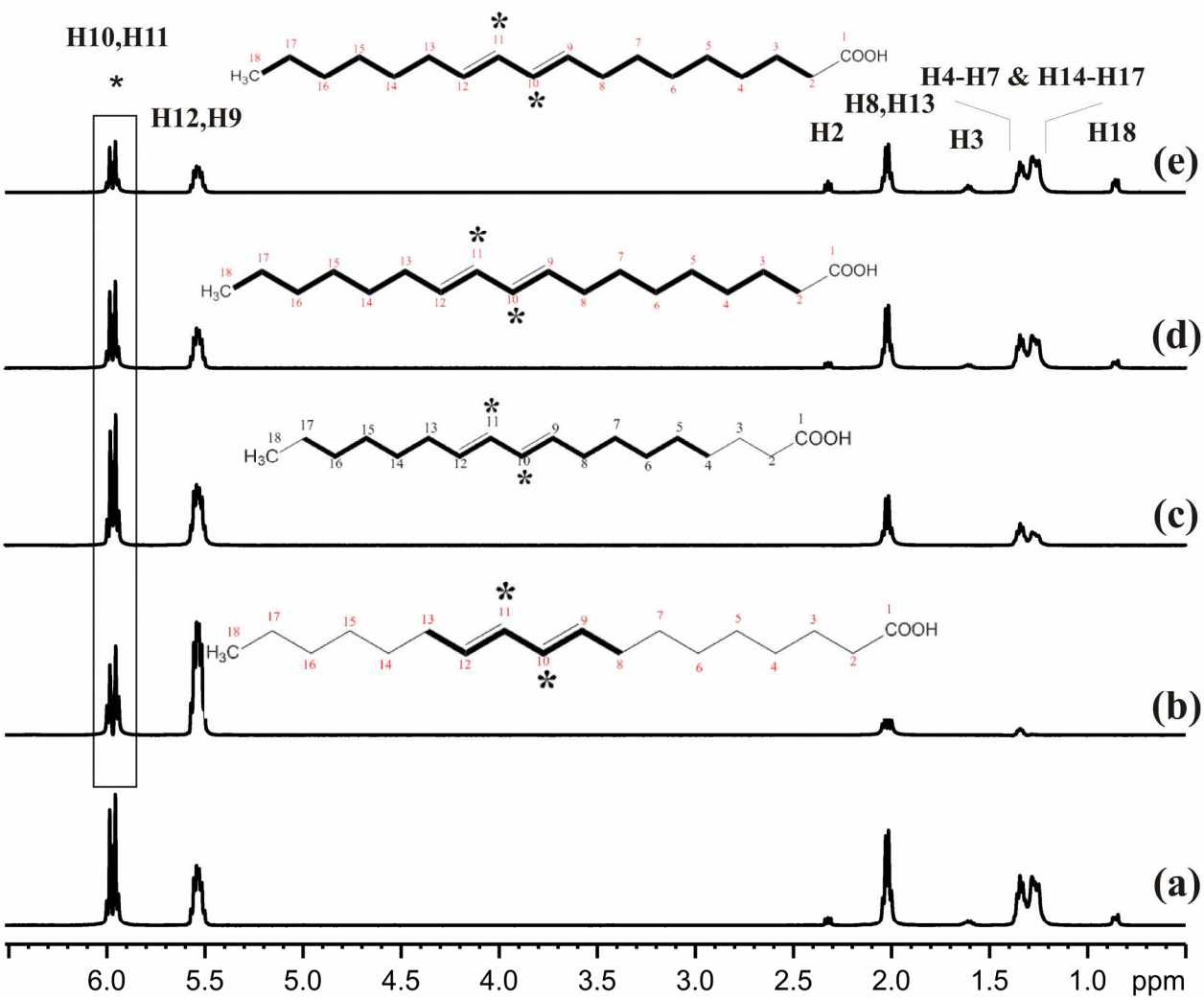
H10,H11
H12,H9
H2
H8,H13
H3
H4-H7 & H14-H17
H18
(e)
(d)
(c)
(b)
(a)
COOH
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S1

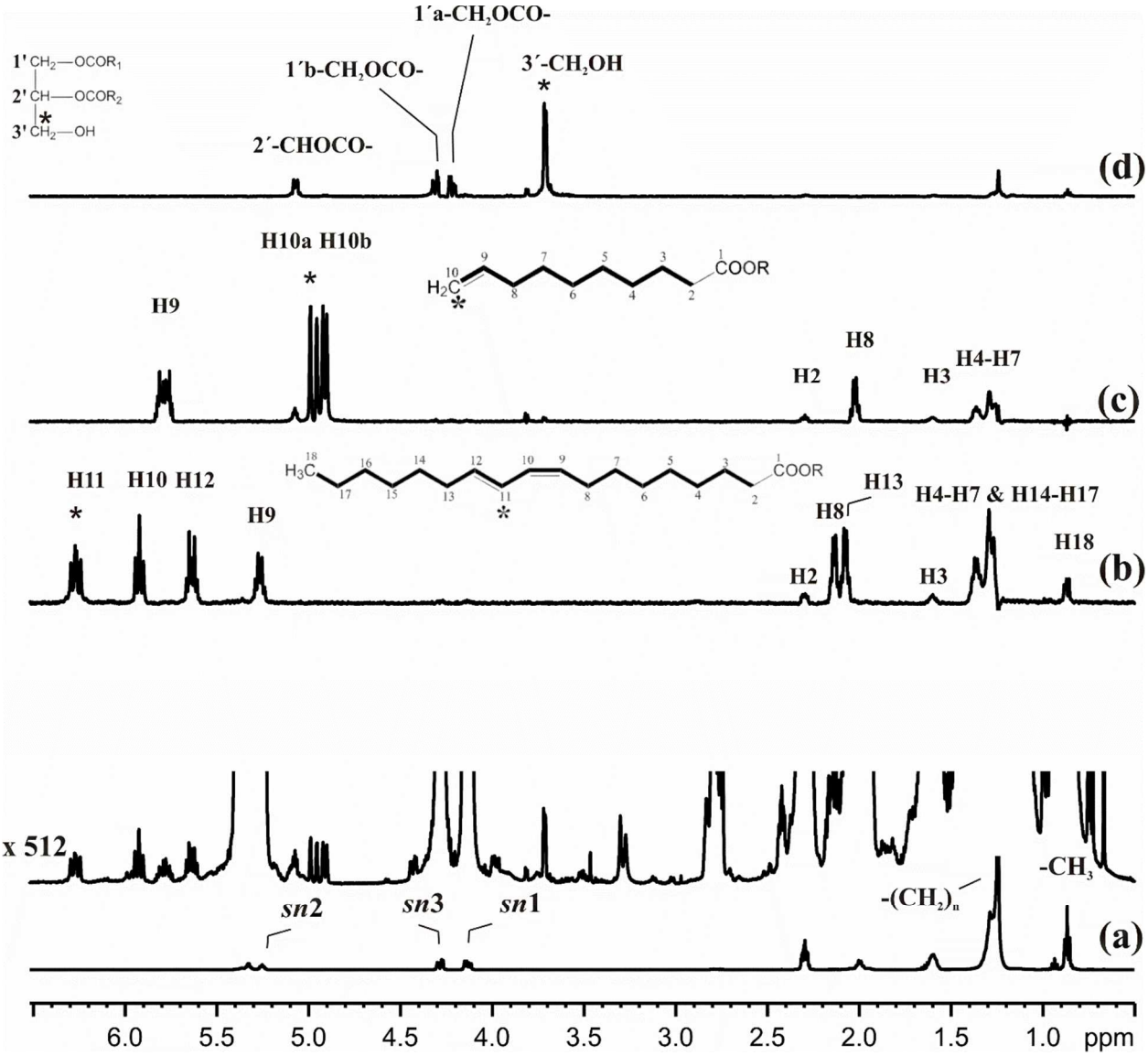
1´a-CH2OCO-
1´b-CH2OCO-
3´-CH2OH
2´-CHOCO-
(d)
H10a H10b
H9
H8
H2
H3
H4-H7
(c)
H11
H10 H12
H9
H13
H8
H4-H7 & H14-H17
H18
H2
H3
(b)
x 512
sn2
sn3
sn1
-(CH2)n
-CH3
(a)
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S2

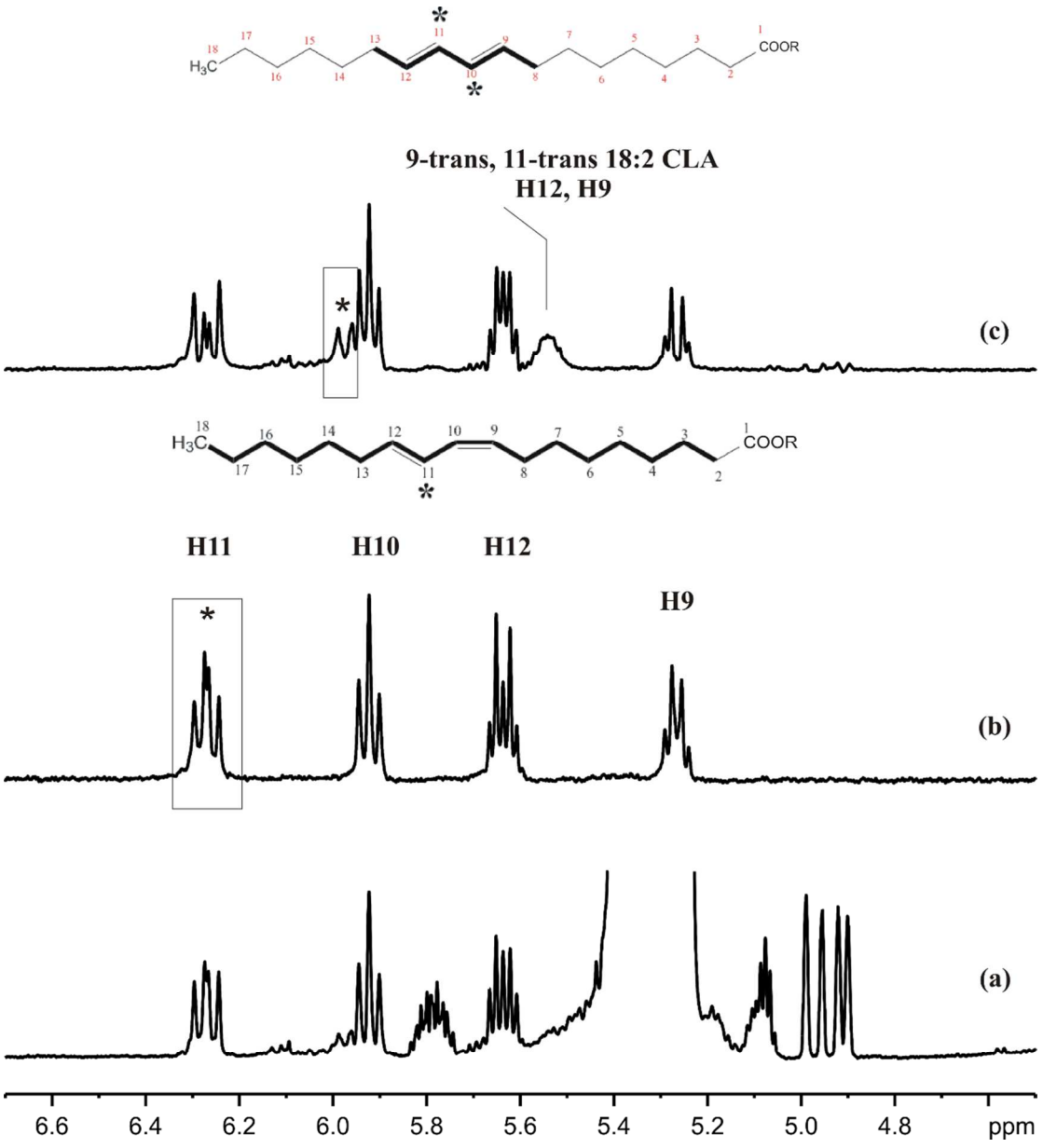
9-trans, 11-trans 18:2 CLA
H12, H9
(c)
H11
H10
H12
H9
(b)
(a)
6.6
6.4
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
ppm

Figure S3

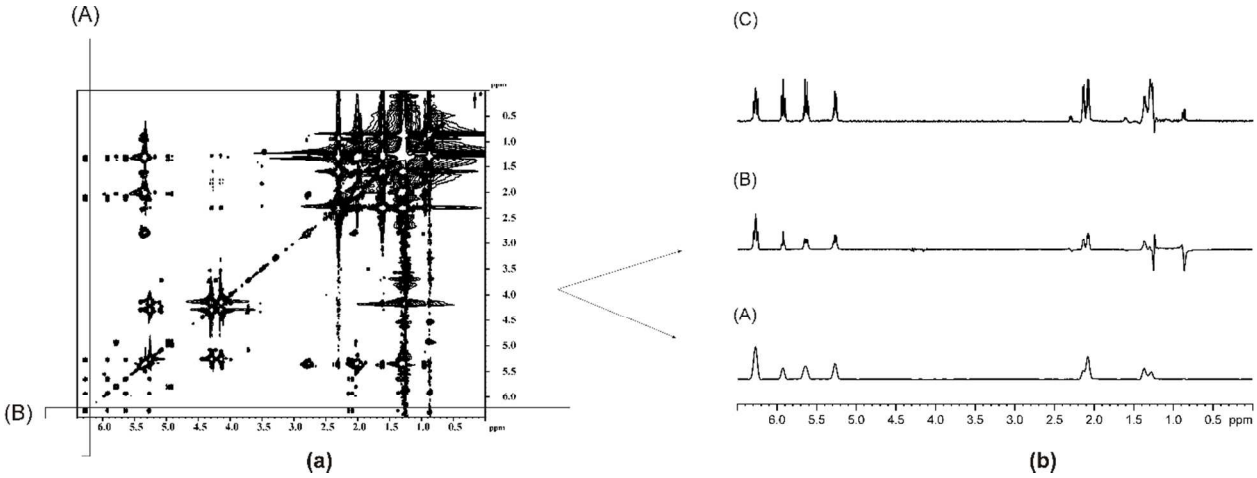


Figure S4

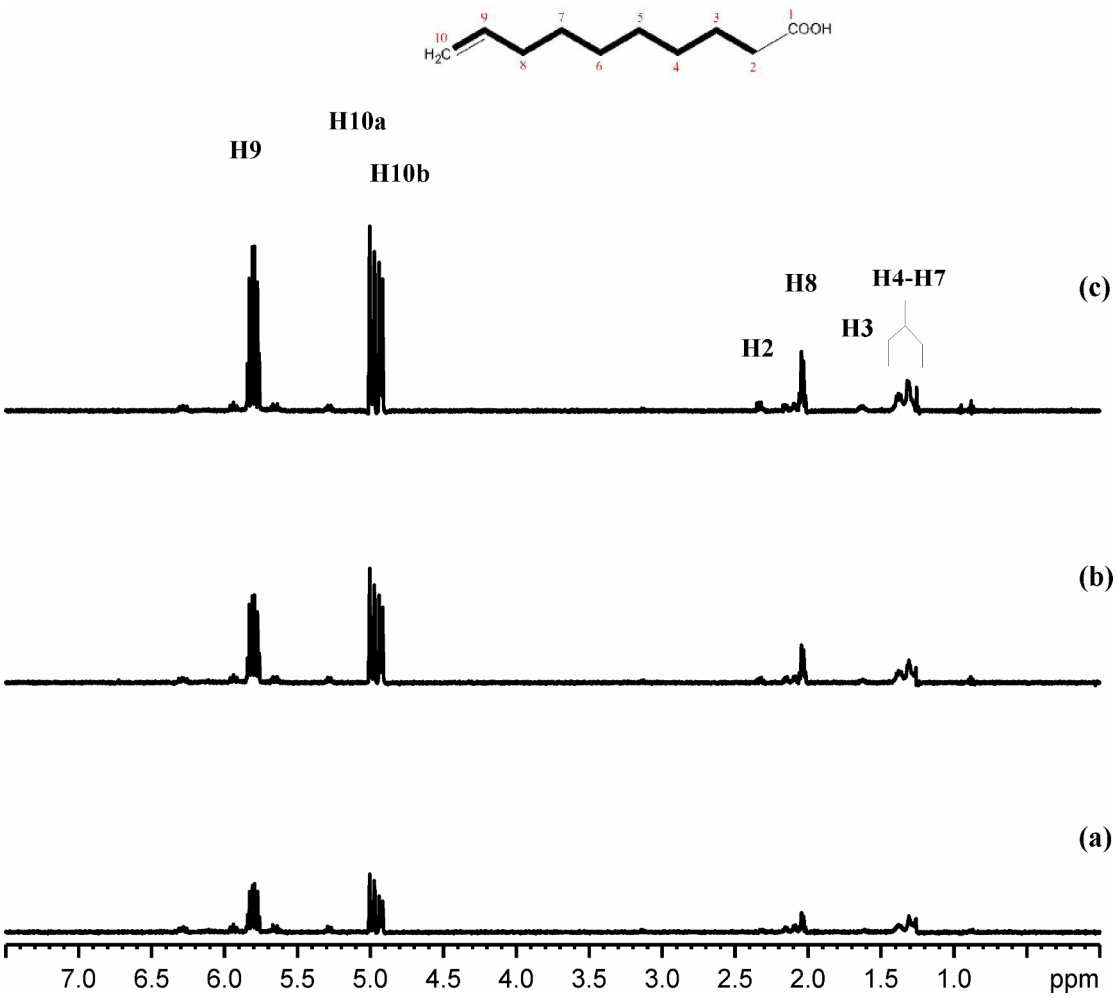
H9
H10a
H10b
H8
H4-H7
H3
H2
(c)
(b)
(a)
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S5