Electronic supplementary information for *Analytical Methods*

^{13}C isotopomics of triacylglycerols using NMR with polarization transfer techniques

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NMR spectrometry experiments

For quantitative ^{13}C NMR, oils (403.2 mg) were dissolved in chloroform-d (630.0 mg) and the resulting solutions transferred into 5 mm NMR tubes. For each sample, 6 ^{13}C INEPT (Insensitive Nuclei Enhanced by Polarization Transfer) spectra were recorded using 11.7 T Bruker Avance-III spectrometer equipped with a 5 mm o.d. dual cryoprobe ^{13}C/^{1}H tuned at the recording frequency of 125.76 MHz for ^{13}C. The temperature of the probe was set at 293 K. The acquisition parameters for ^{13}C NMR spectral were as follows: ^{13}C and ^{1}H offsets were set at the middle of the frequency range (92.5 ppm for the ^{13}C and 3 ppm for the ^{1}H), pulse width 10 µs for the 90° ^{1}H and 11 µs for the 90° ^{13}C, 16 scans with a repletion delay of 24 s were recorded in order to have a signal-to-noise ratio higher than 600 on the C2 of glycerol. τ_1 was adjusted to 2.704 ms, and the refocusing period τ_2 was adjusted to 1.409 ms. Adiabatic full passage pulses were generated using Mathcad 8 (MathSoft, Inc.). They were designed with a cosine amplitude modulation of the RF field (ω_1^{max} = 157.1 kHz or 93.89 kHz for ^{13}C or ^{1}H, respectively) and an offset independent adiabaticity (OIA) by optimizing the frequency sweep ΔF (ΔF = 39 kHz or 17 kHz for ^{13}C or ^{1}H, respectively). For inversion pulses, adiabatic full passage pulses were used. For refocusing pulses, composite adiabatic pulses were used. ^{1}H decoupling was performed using adiabatic full passage RF pulses with cosine square amplitude modulation (ν_2^{max} = 17.6 kHz) and offset independent adiabaticity with optimized frequency sweep (ΔF = 14 kHz).

NMR data processing and analysis

FIDs were zero-filled to 128 K and submitted to an exponential multiplication inducing a line broadening of 1.5 Hz before Fourier transform. The ^{13}C NMR spectra were manually phased. An automatic polynomial baseline correction (n = 5) was applied to the resulting spectra. The curve fitting was carried out in accordance with a Lorentzian mathematical model using PERCH Software (PERCH NMR Software, University of Kuopio, Finland) and 97 peak areas were obtained for each sample.

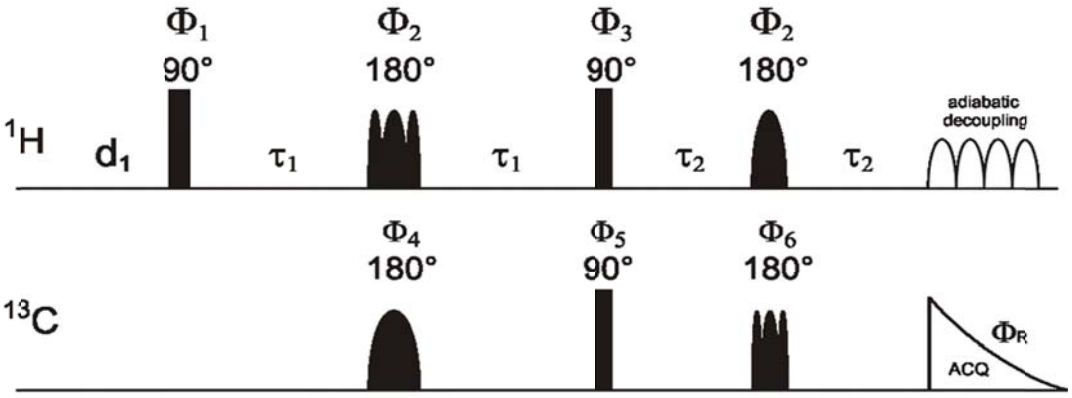
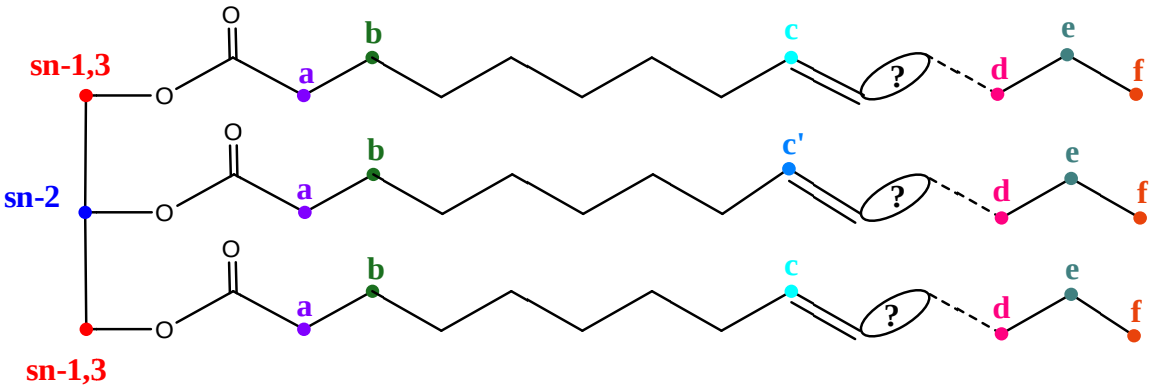


Fig. S1. Adiabatic refocused INEPT (Insensitive Nuclei Enhanced by Polarization Transfer) sequence with ^{1}H and ^{13}C 180° adiabatic composite refocusing pulses and adiabatic full passage inversion pulses.



sn-1,3: C1,3 of glycerol backbone
sn-2: C2 of glycerol backbone
a: C2 of fatty acids
b: C3 of fatty acids
c: C9 of linoleic acid at glycerol sn-1,3
c': C9 of linoleic acid at glycerol sn-2
d: Cω3 of fatty acids
e: Cω2 of fatty acids
f: Cω1 of fatty acids

Fig. S2. Designation of different carbons in a triacylglycerol molecule

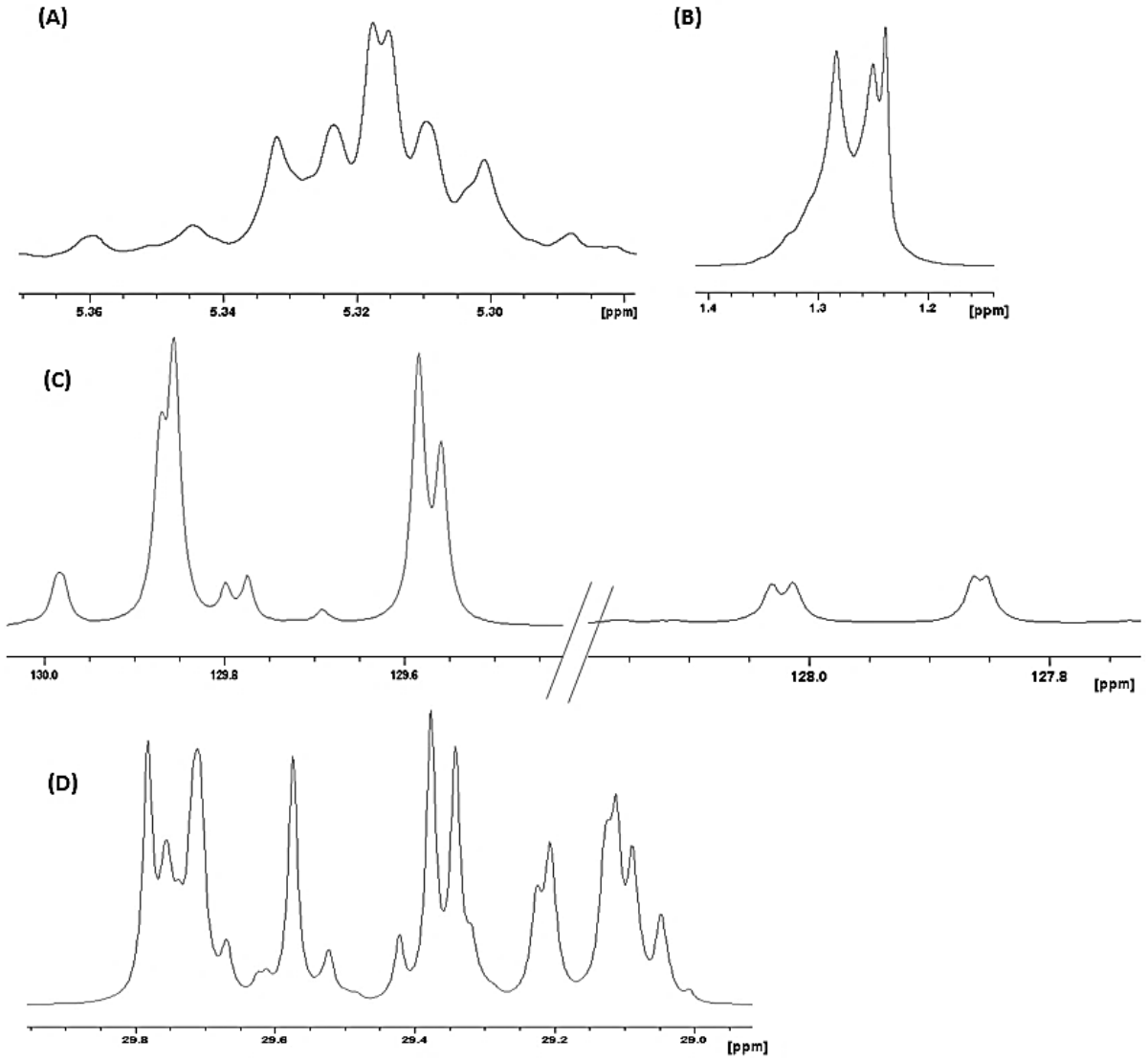


Fig. S3. Comparison of different regions of the ^{1}H and ^{13}C NMR spectra of olive oil: olefinic (A) and aliphatic (B) regions of the ^{1}H NMR spectrum; olefinic (C) and aliphatic (D) regions of the ^{13}C NMR spectrum.