

Supporting Information

Multipronged Approach to Profiling Metabolites in *Beta Vulgaris* Dried Pulp Extract Using Chromatography, NMR, and other Spectroscopy Methods

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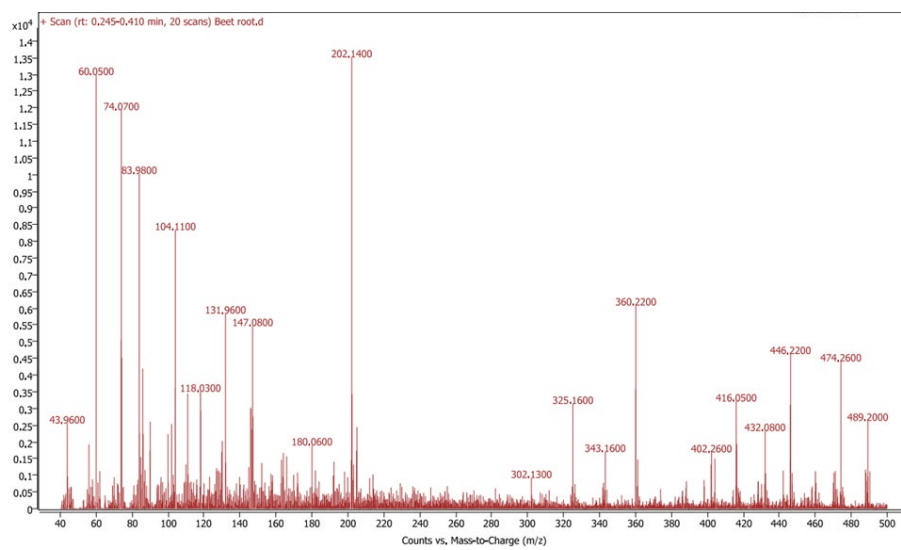


Figure S1. The MS fragmentation spectrum of the compounds and metabolites in the aqueous extract of beetroot powder in positive polarity mode.

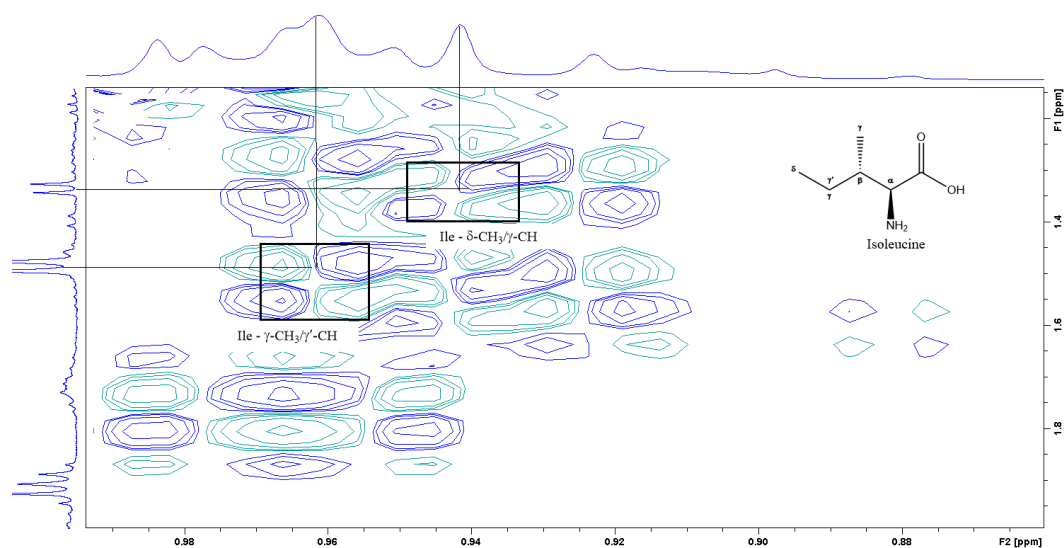


Figure S2. A section of the COSY spectrum showing spin coupling in the amino acid Isoleucine.

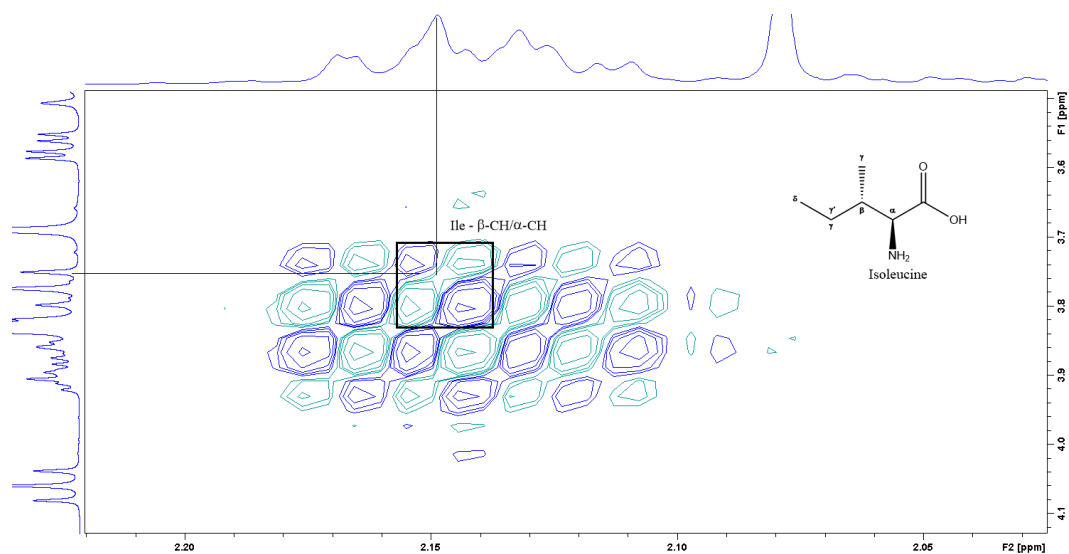


Figure S3. A section of the COSY spectrum showing the amino acid Isoleucine.

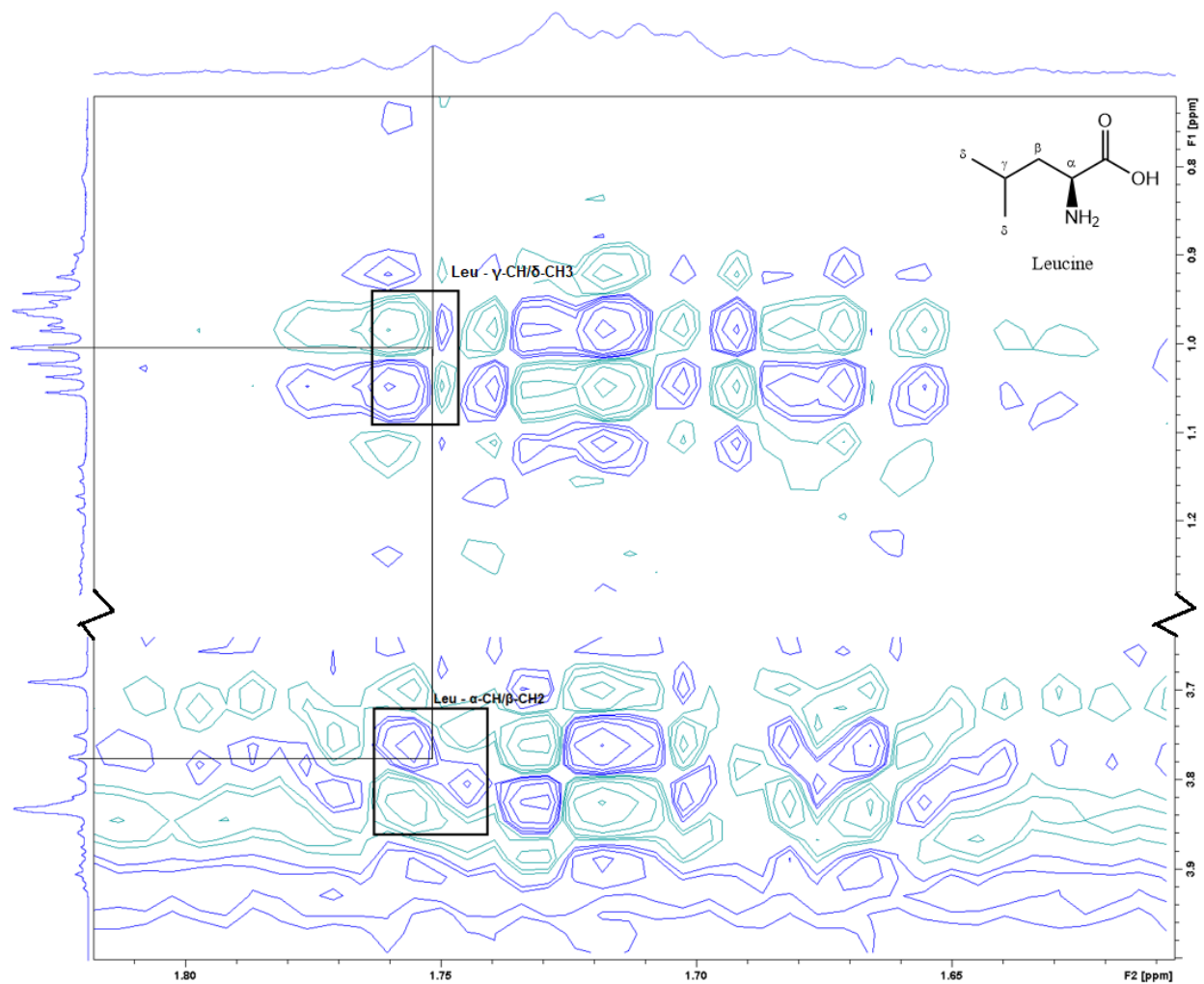


Figure S4. COSY spectrum showing the amino acid Leucine (D₂O-salt, 400 MHz).

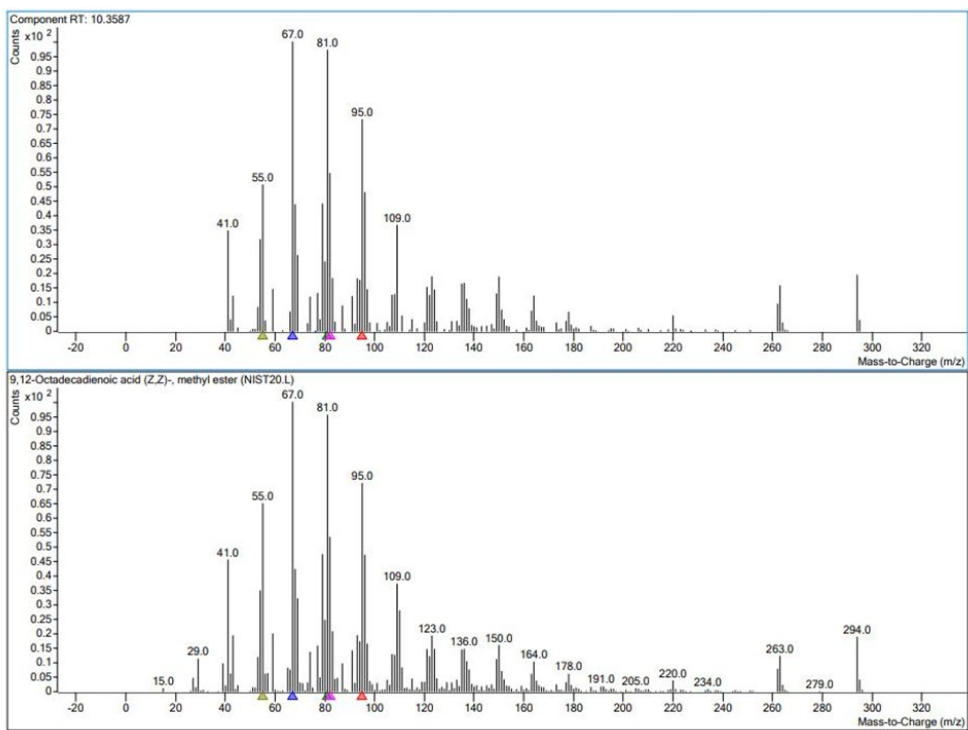


Figure S5. MS spectra of 9,12-Octadecadienoic acid, methyl ester (RT = 15.419 min.) experiment spectrum overlaid with the reference library spectrum.

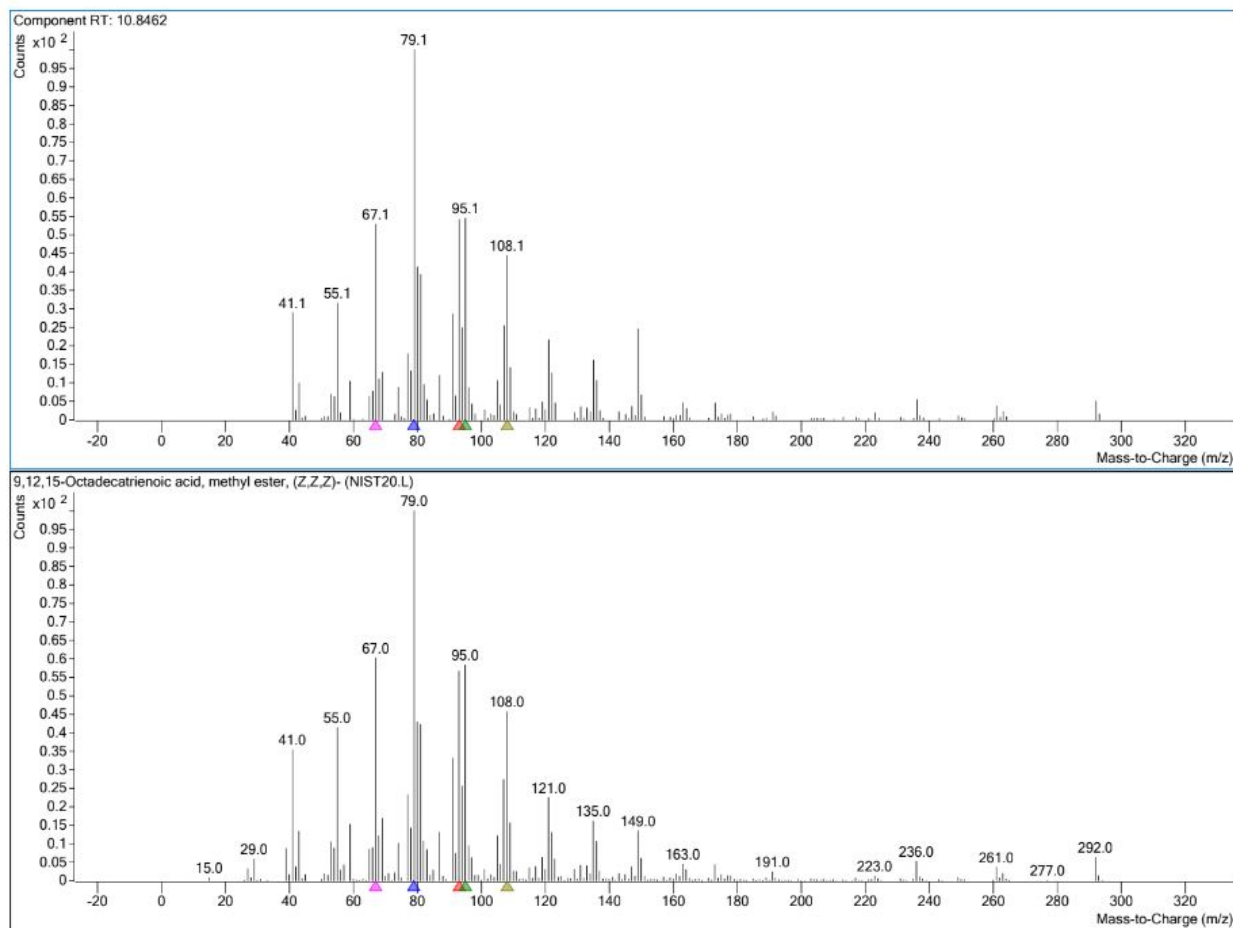


Figure S6. MS spectra of 9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z) (RT = 10.846 min.) experiment spectrum overlaid with the reference NIST library spectrum.

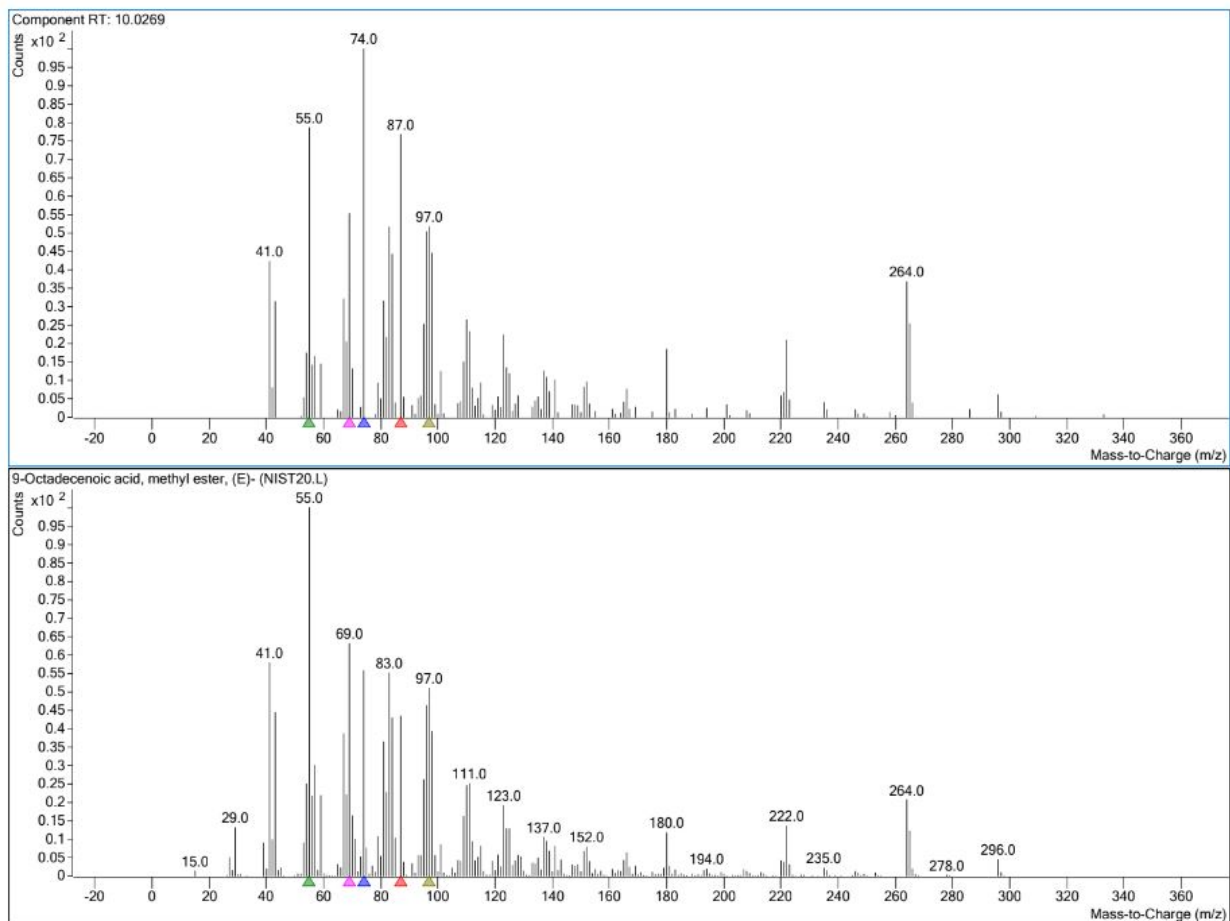


Figure S7. MS spectra of 9-Octadecenoic acid, methyl ester, (E) (RT = 10.027 min.) experiment spectrum overlaid with the reference NIST library spectrum.

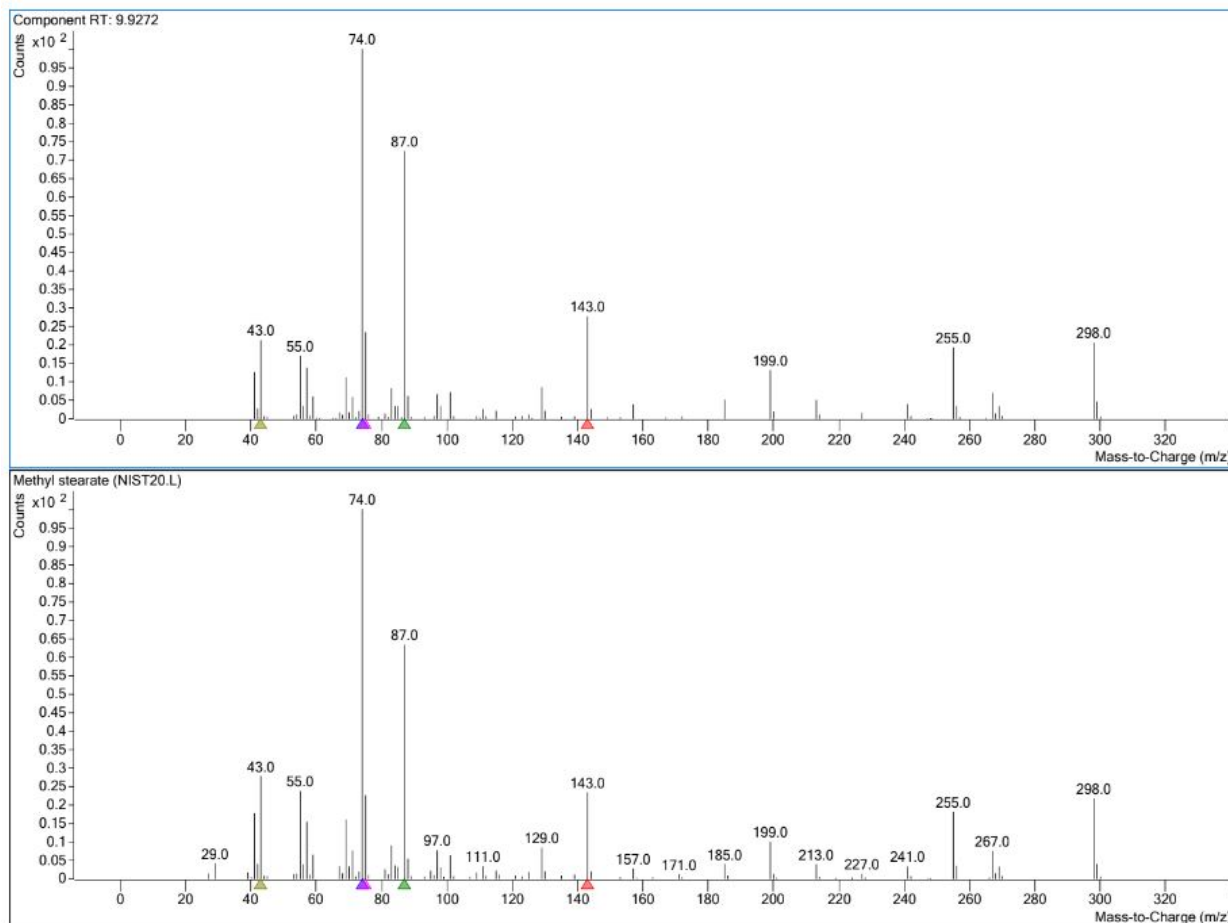


Figure S8. MS spectra of methyl stearate (RT = 9.927 min.) experiment spectrum overlaid with the NIST reference library spectrum.

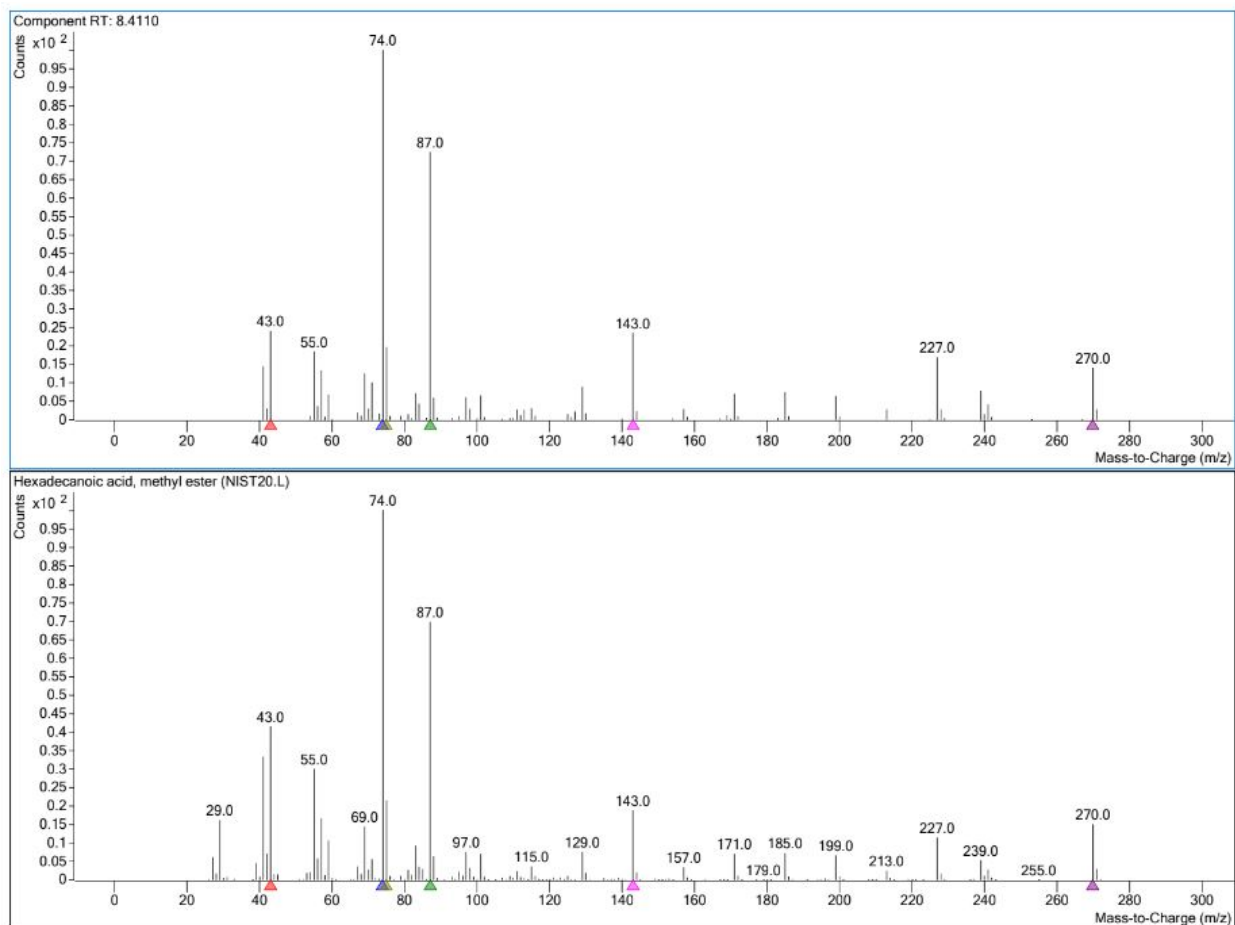


Figure S9. MS spectra of hexadecanoic acid, methyl ester (RT = 8.411 min.) experiment spectrum overlaid with the NIST reference library spectrum.

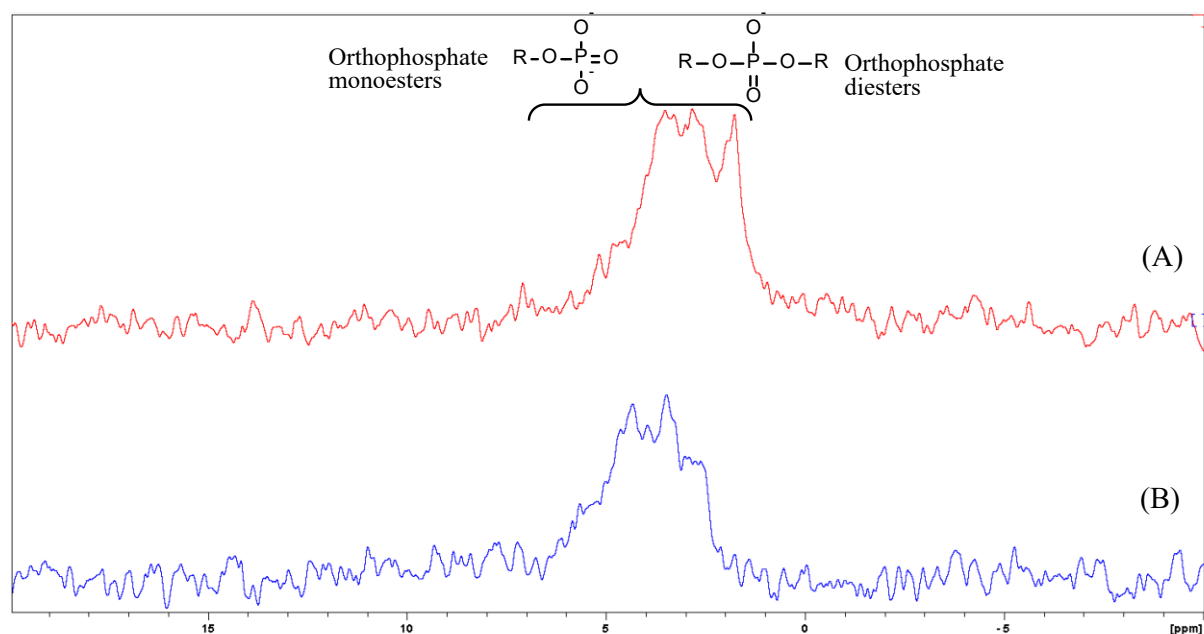


Figure S10. Proton-decoupled ^{31}P NMR spectra of polar extract (A) in D_2O and non-polar extract (B) in CDCl_3 on a 400 MHz spectrometer, $n_s = 10,000$ at 25°C temperature, and 20 Hz line-broadening. Chemical shift is based on the ^{31}P peak of 85% phosphoric acid, which is set at 0 ppm.

$^{13}\text{C}/^1\text{H}$ Atom label (sucrose)	Measured chemical shift, δ (ppm)	Literature chemical shift, δ (ppm) [33]	^1H Atom label (α -D- glucopyranose/ β -D- glucopyranose)	Measured chemical shift, δ (ppm) of (α -D- glucopyranose/ β -D- glucopyranose)	Literature chemical shift, δ (ppm) [34]
C_1/H_1	92.15/5.41	92.15/5.40	H_1	5.25/4.68	5.35/4.74
C_2/H_2	71.13/3.56	71.08/3.55	H_2	3.53/3.39	3.64/3.37
C_3/H_3	72.65/3.77	72.65/3.75	H_3	3.70/3.48	3.81/3.60
C_4/H_4	69.26/3.48	69.26/3.46	H_4	3.56/3.91	3.52/3.92
C_5/H_5	72.09/3.85	72.10/3.82	H_5	3.85/3.51	3.98/3.50
C_6/H_6	60.15/3.81	60.15/3.82			
C'_1/H'_1	61.36/3.68	61.40/3.67			
C'_3/H'_3	76.45/4.22	76.50/4.21			
C'_4/H'_4	74.05/4.06	74.10/4.04			
C'_5/H'_5	81.35/3.90	81.39/3.89			
C'_6/H'_6	62.43/3.83	62.50/3.82			

Table S1. The chemical shift values, δ (ppm) of the ^1H of sucrose and α - and β -D-glucopyranose and the corresponding literature values, and δ (ppm) of ^{13}C of sucrose and the corresponding literature values.

Amino acid	^1H Chemical shifts, δ (ppm)	^1H Chemical shifts, δ (ppm) Literature values[36]
Leucine	δ -CH ₃ -0.978 γ -CH, β -CH ₂ -1.765 α -CH-3.779	δ -CH ₃ -0.948 γ -CH, β -CH ₂ -1.700 α -CH-3.722
Isoleucine	γ -CH ₃ -0.942 δ -CH ₃ -0.962 γ -CH-1.343 γ^1 -CH-1.493 β -CH-2.149 α -CH-3.749	γ -CH ₃ -0.926 δ -CH ₃ -0.997 γ -CH-1.248 γ^1 -CH-1.457 β -CH-1.968 α -CH-3.660
Valine	α -CH-3.689 β -CH-2.284 γ^1 -CH ₃ -1.036 γ -CH ₃ -0.984	α -CH-3.601 β -CH-2.262 γ^1 -CH ₃ -1.029 γ -CH ₃ -0.976

Table S2. ^1H chemical shift of identified amino acids and literature values.

Chemical shift (δ) (ppm)	Assigned H
5.36	9,10,12,13,15,16 – Linolenic acid 9,10,12,13 – Linoleic acid 9,10 – Oleic acid
2.77	11 – Linoleic acid 11,14 – Linolenic acid
2.31	2 – α -CH ₂
2.06	8,17 – Linolenic acid 8,14 – Linoleic acid 8, 11 – Oleic acid
1.60	3 – β -CH ₂
1.26	* - CH ₂ (s) (overlapping)
0.88	18 - CH ₃ (terminal)

Table S3. Chemical shift values and assigned protons in the selective TOCSY spectrum. The asterisk (*) indicates the overlapping CH₂ groups.