

Supporting Information

Extraction and Pyrolysis-GC-MS analysis of Polyethylene in samples with medium to high lipid content

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Text S1- Final optimised method for extraction of PE in lipid rich matrices

A 5% enzyme solution was prepared from the active ingredients of CREON® 10,000 capsules, purchased over the counter from a pharmacy chain in Brisbane, Australia. The contents of the capsules were emptied and ground in a ceramic mortar and pestle to a fine powder. One gram of the powder was dissolved in 20 mL of MilliQ, immediately adjusting the pH to between 7 and 8 with a 1M sodium carbonate solution. The enzyme solution was sonicated for 15 min and the pH of the final solution readjusted to between 7 and 8 if necessary. The solution was filtered through a 1 µm pore size glass fibre filter, GFF (13mm, ProSciTech Ptd Ltd, Kirwan, Queensland) and the final prepared solution used immediately. The solution was prepared fresh and used immediately for each batch of samples.

A sample mass of 0.5 g of freeze-dried material was reconstituted by adding 2 mL of MilliQ water and 1 mL of 5% bile salts in MilliQ water solution, and the sample vortexed to mix thoroughly and form an emulsion. The sample pH was adjusted to 10 with 1M sodium carbonate solution and samples heated to 37 °C for 5 minutes in an Thermoline Orbital incubator shaker (Thermoline Scientific, Wetherill Park, NSW). After samples were at temperature, 3 mL of the 5% enzyme solution was added and the samples vortexed to mix and incubated at 37 °C with gentle rotation (100 rpm) for 1 hour.

The digested samples were loaded into 10 mL ASE cells containing hydromatrix (ThermoFisher Scientific, Waltham, MA), precleaned with DCM, to fill the void volume. ASE cells were then extracted with 1) methanol at 60 °C and 1500 psi with a 5 min static-time for one extraction cycle 2) hexane:isopropanol (3:2, v/v) at 60 °C and 1500 psi with a 5 min static-time for one extraction cycle. The samples in the ASE cells were then spiked with 40 µg of d₅-PS internal standard and extracted with the original ASE extraction method, DCM at 180 °C and 1500 psi with a 5 min static-time for two extraction cycles. Following extraction, an 80 µl aliquot of the DCM extract was transferred to a pyrolysis cup (Eco-Cup LF, Frontier Laboratories, Japan). The sample cups were covered with aluminium foil and the solvent allowed to evaporate in a laboratory fume cabinet before analysis with Pyr-GCMS.

Supplementary Table 1. Details of pyrolysis products monitored for polyethylene (PE) and deuterated polystyrene (d₅-PS) internal standard including instrument detection limits (IDL). Quantification ions are indicated in bold text

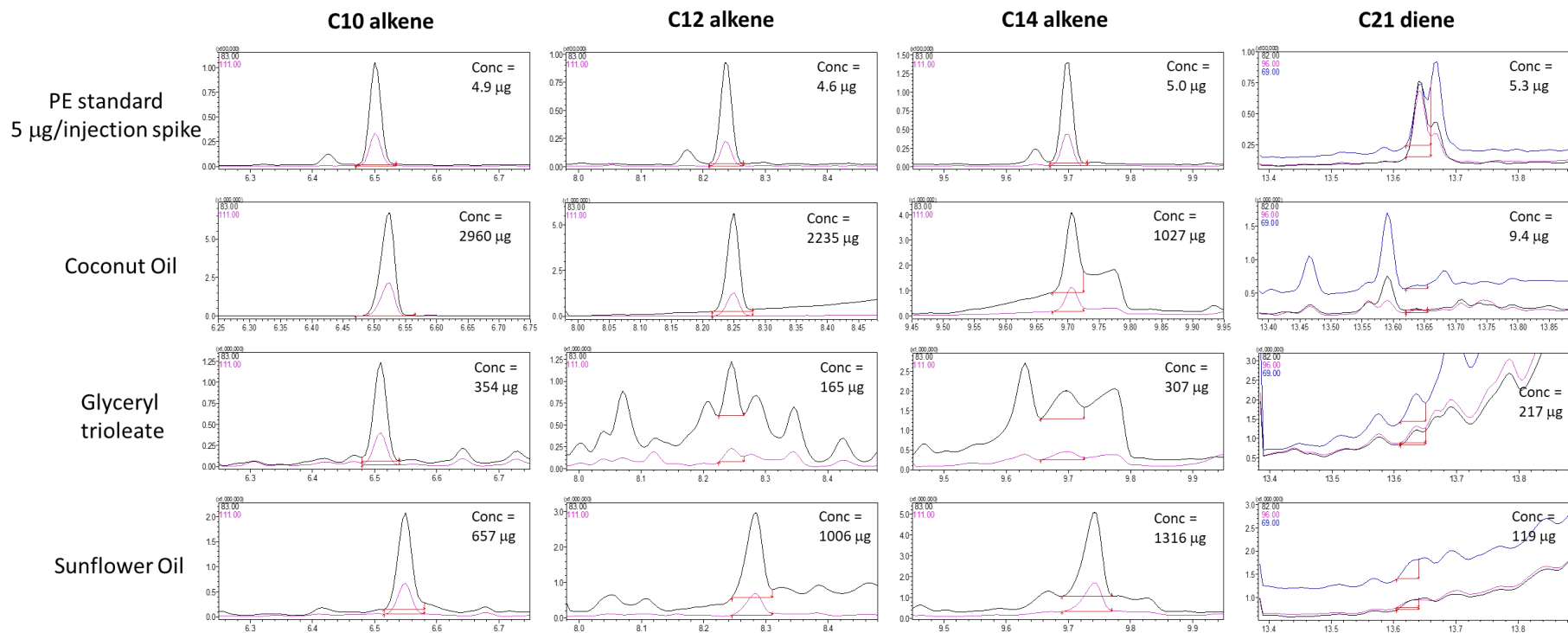
Polymer	Pyrolysis product	Retention time (min)	Ions monitored (m/z)	IDL (µg/injection)
Polyethylene	1-Decene (C10)	6.43	83, 111	0.03
	1-Dodecene (C12)	8.15	83, 111	0.02
	1-Tetradecene (C14)	9.61	83, 111	0.02
	1,20-Heneicosadiene (C21)	13.55	82, 96, 69	0.1
d ₅ -Polystyrene	d ₅ -styrene monomer	5.37	82, 107, 108, 109	---

Supplementary Table 2. Calculated concentrations of PE from the 4 different pyrolysis products from analysis of three triacylglycerols, basmati rice and a PE analytical standard, extracted using the old ASE method

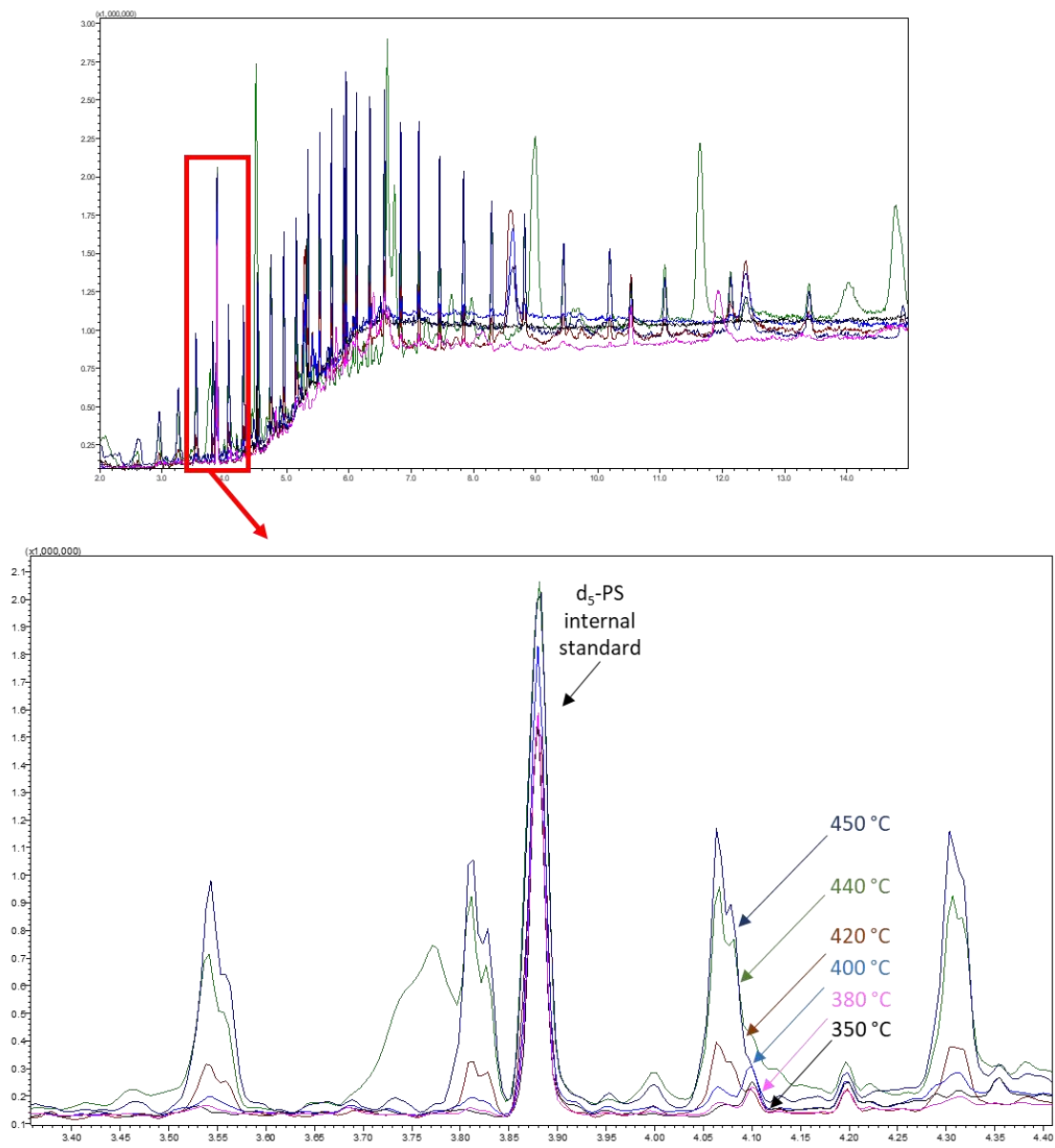
Sample	Concentration (µg/injection)			
	C10 alkene	C12 alkene	C14 alkene	C21 diene
PE only	4.9	4.6	5.0	5.3
<i>Ratio to C10</i>	---	<i>0.94</i>	<i>0.98</i>	<i>1.1</i>
Coconut	2960	2235	1027	9.4
<i>Ratio to C10</i>	---	<i>0.76</i>	<i>0.35</i>	<i>0.003</i>

Glyceryl	354	165	307	217
trioleate	---	0.47	0.87	0.61
<i>Ratio to C10</i>				
Sunflower	657	1006	1316	119
<i>Ratio to C10</i>	---	1.5	2.0	0.18
Basmati Rice	1.5	2.0	2.1	2.4
<i>Ratio to C10</i>	---	1.3	1.4	1.6

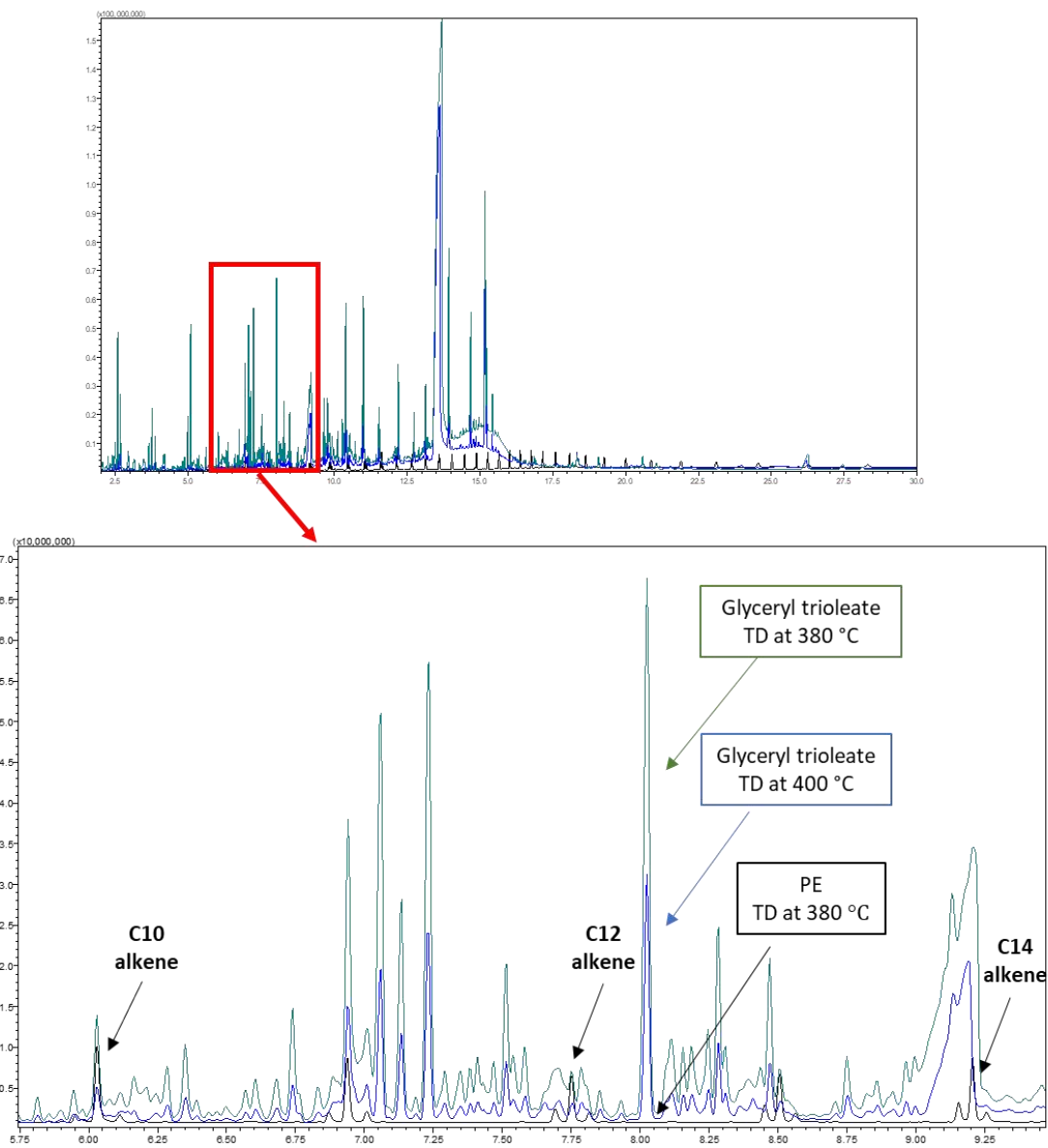
Supplementary Figure 1. Pyrograms of the 4 monitored PE pyrolysis products from three triacylglycerols and a PE analytical standard, extracted using the old ASE method.



Supplementary Figure 2. Thermal desorption pyrograms of PE, analysed with methods reaching maximum temperatures of 350, 380, 400, 420, 440 and 450 °C.



Supplementary Figure 3. Pyrolysis pyrograms of a PE standard and glyceryl trioleate after thermal desorption (TD) at 380 °C and 400 °C



Supplementary Figure 4. Pyrograms of C10 alkene in extracts of hexane:isopropanol (Hex:IPA, 3:2 v/v) and DCM of coconut oil, glyceryl trioleate and sunflower oil, showing the interference peak removed in a Hex:IPA wash for coconut oil and glyceryl trioleate but is remains in DCM extract for sunflower oil.

