

## SUPPORTING INFORMATION:

# Thorough multianalytical characterization and quantification of micro-and nanoplastics from Bracciano Lake's sediments

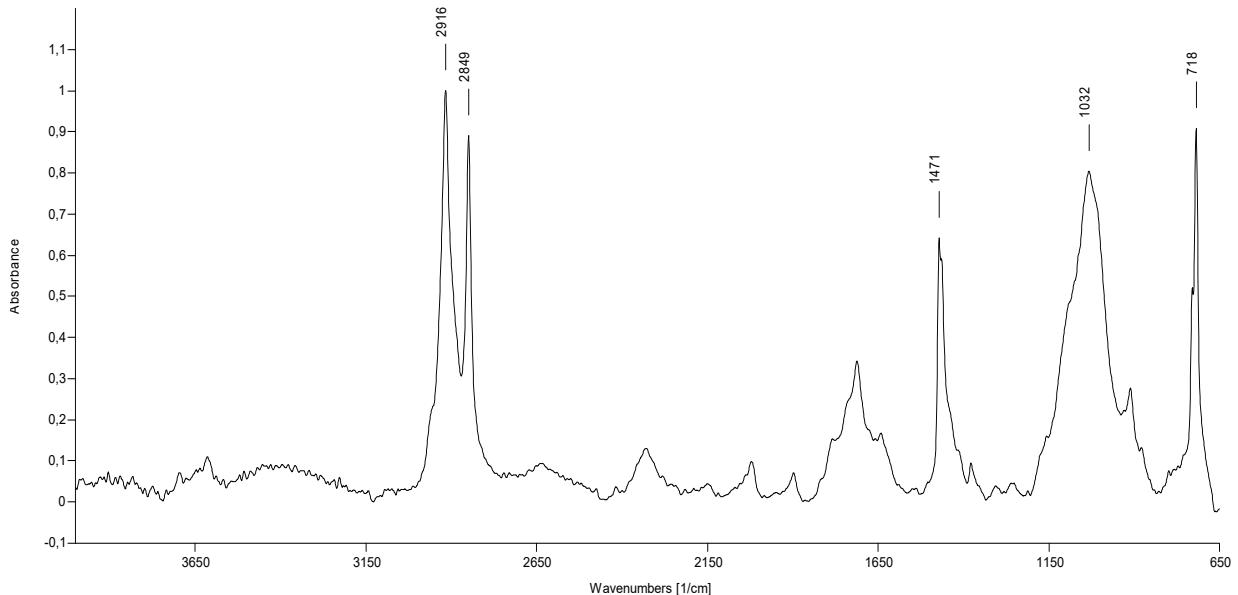
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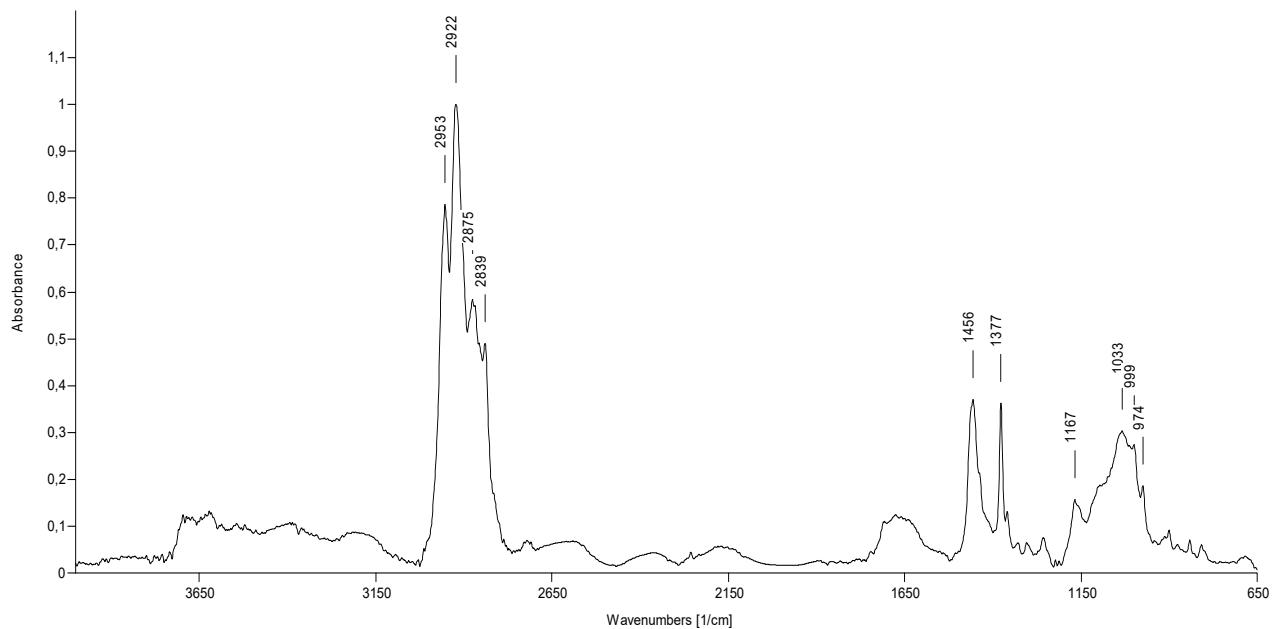
### S1. ATR-FTIR analysis of plastic fragments collected as the sieve fraction between 2 and 5 mm



**Figure S11.** ATR spectrum of the BR1B3-f<sub>1</sub> fragment.

**Table S11.** Characteristic IR absorptions of polyethylene (compare with figure S11)

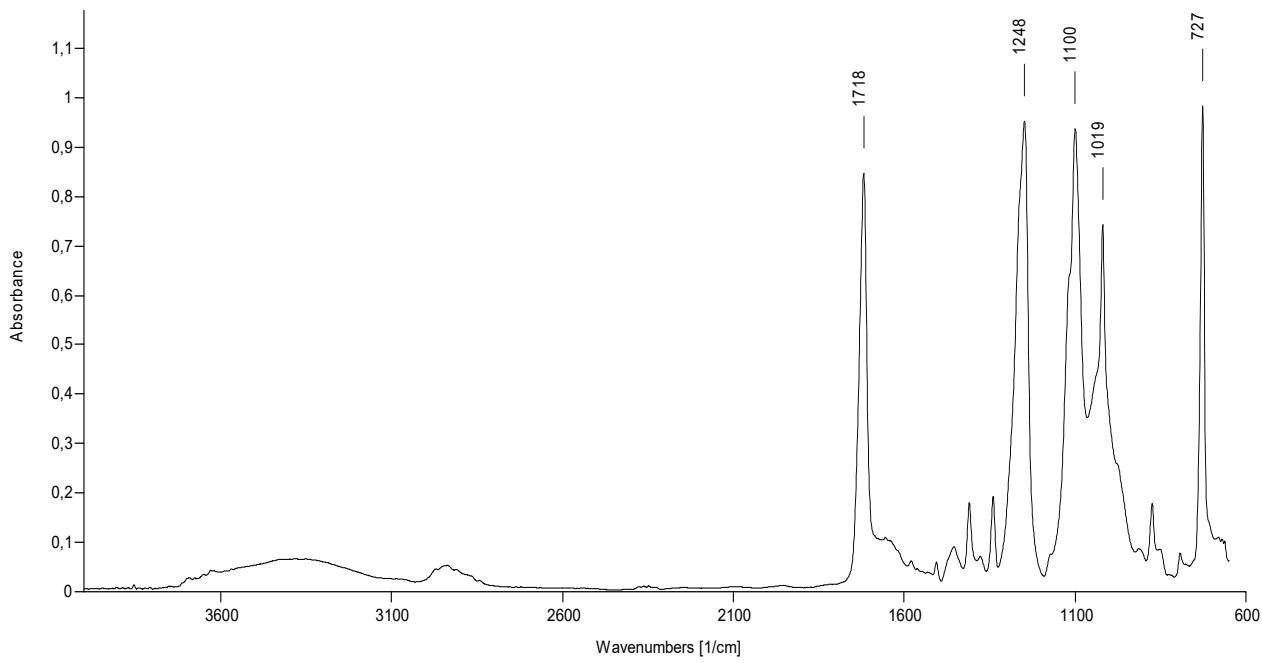
Wavenumber (cm <sup>-1</sup> )		Vibrational mode
Theoretical	Observed (BR1B3-f <sub>1</sub> )	
2915, 2845	2916, 2849	v <sub>s</sub> CH
1472, 1462	1471	δ <sub>s</sub> CH <sub>2</sub>
730, 717	718	ρ CH <sub>2</sub>



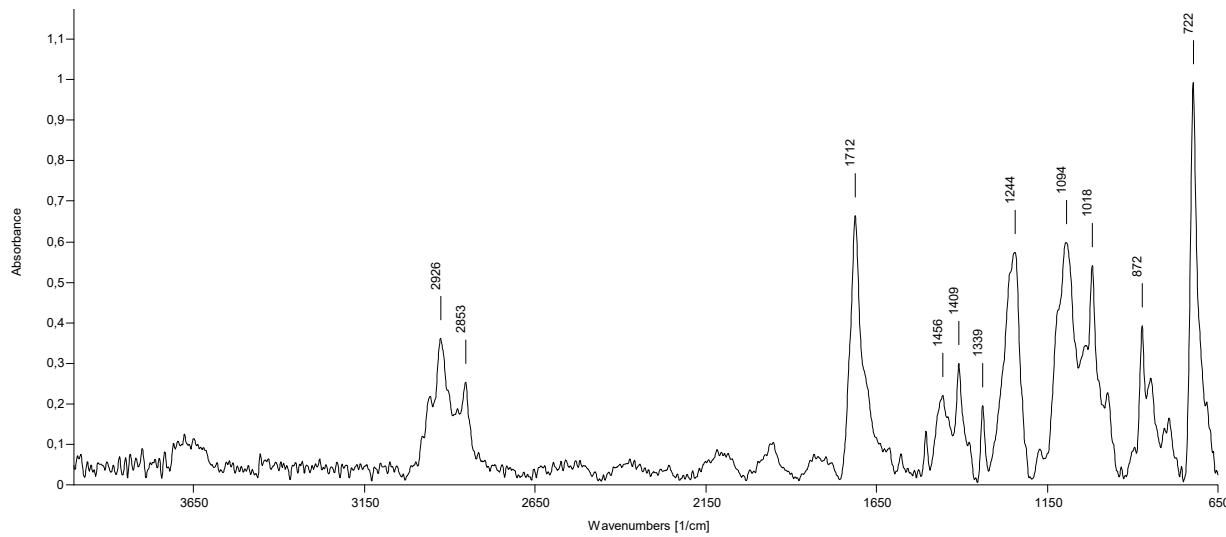
**Figure SI2.** ATR spectrum of the BR2C2-f<sub>3</sub> fragment.

**Table SI2.** Characteristic IR absorptions of polypropylene (compare with figure SI2)

Theoretical	Wavenumber (cm <sup>-1</sup> ) Observed (BR2C2-f <sub>3</sub> )	Vibrational mode
2950, 2915, 2838	2953, 2922, 2875, 2839	$\nu_s$ CH
1455	1456	$\delta_s$ CH <sub>2</sub>
1377	1377	$\delta_s$ CH <sub>3</sub>
1166	1167	$\delta_s$ CH, $\rho$ CH <sub>3</sub> , $\nu_s$ C-C
997	999	$\rho$ CH <sub>3</sub> , $\delta_s$ CH <sub>3</sub> , $\delta_s$ CH
972	974	$\rho$ CH <sub>3</sub> , $\nu_s$ C-C
840	841	$\rho$ CH <sub>2</sub> , $\nu_s$ C-CH <sub>3</sub>
808	809	$\rho$ CH <sub>2</sub> , $\nu_s$ C-C, $\nu_s$ C-CH



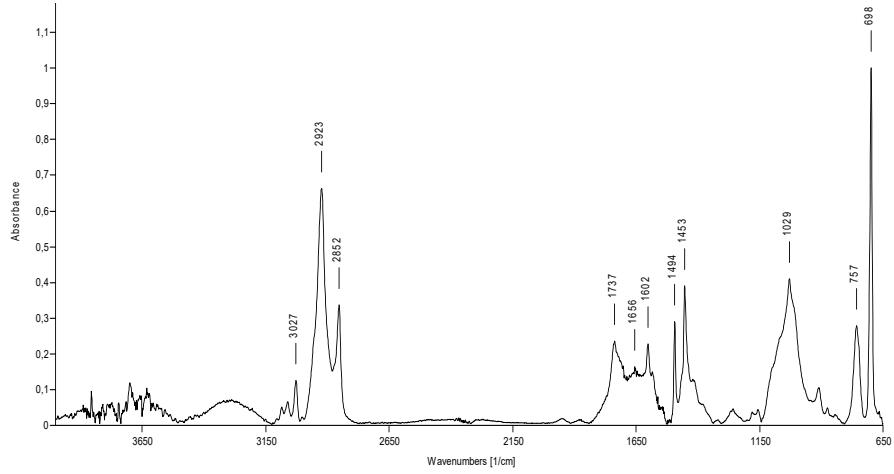
**Figure SI3.** ATR spectrum of the BR2B2-f<sub>1</sub> fragment.



**Figure SI41.** ATR spectrum of the BR2C2-f<sub>1</sub> fragment.

**Table SI3.** Characteristic IR absorptions of polyethylene terephthalate (compare with figure SI3 and SI4)

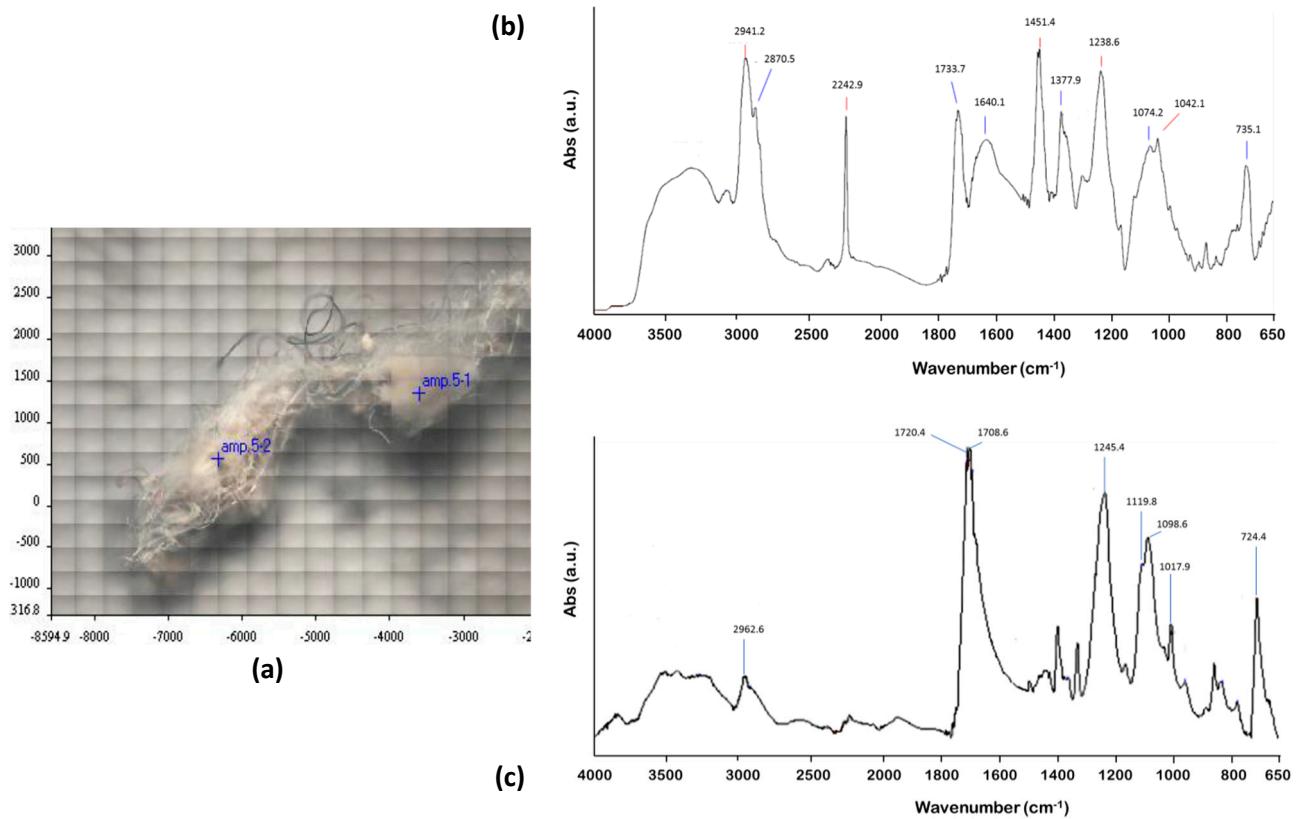
Theoretical	Wavenumber (cm <sup>-1</sup> ) Observed (BR2B2-f <sub>1</sub> and BR2C2-f <sub>1</sub> )	Vibrational mode
2925, 2850	2926, 2853	$\nu_{as}$ and $\nu_s$ of CH <sub>2</sub>
1720	1718-1712	$\nu_s$ C=O
1250	1248-1244	$\nu_s$ C(=O)-O
1175	1173 (weak)	1,4-disubstituted ring C-C stretching
1120	1118 (shoulder)	$\nu_s$ C-O, trans-conformer
1020	1018-1019	1,4-disubstituted ring C-C stretching
870	872	ring C-C stretching
730	722-727	$\delta_{out-of-plane}$ of ring C-H and ester C=O



**Figure S15.** ATR spectrum of the BR1C3-f<sub>2</sub> fragment.

**Table S14.** Characteristic IR absorptions of polystyrene (compare with figure S15)

Wavenumber (cm <sup>-1</sup> )		Vibrational mode
Theoretical	Observed (BR1C3-f <sub>2</sub> )	
3024	3027	v <sub>s</sub> aromatic CH
2847	2852	v <sub>s</sub> CH
1601, 1492	1602, 1494	v <sub>s</sub> aromatic ring
1451	1453	δ <sub>s</sub> CH <sub>2</sub>
1027	1029	δ <sub>s</sub> aromatic CH
694	698	δ <sub>s</sub> aromatic CH out of plane

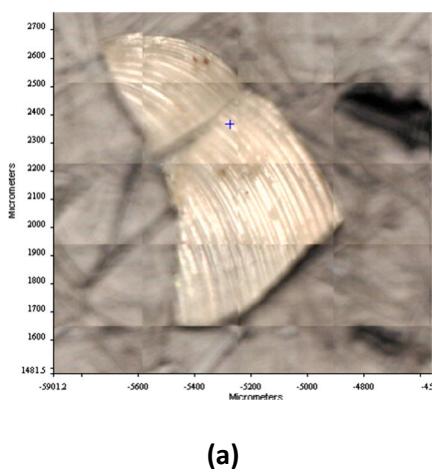


**Figure SI6.** **a)** Optical microscopy image showing the spots of the surface of fragment BR1C3-f<sub>4</sub> analysed by micro-ATR; **b)** micro-ATR FTIR of spot 5-1 (isolated black fiber); **c)** micro-ATR FTIR of spot 5-2 (white fibers).

The spectra reported in **Figure SI6** indicate that the aggregate of microfibers is based on PET (spectrum SI6-c) the most common synthetic polyester textile fiber (as reported in **Table SI3.**) and on a minor amount of acrylic fibers (spectrum SI6-b), as shown by the nitrile peak at  $2242.9\text{ cm}^{-1}$  typical of acrylonitrile copolymers.

## S2. ATR analysis of natural and plastic fragments isolated in the passing fraction (size < 2 mm)

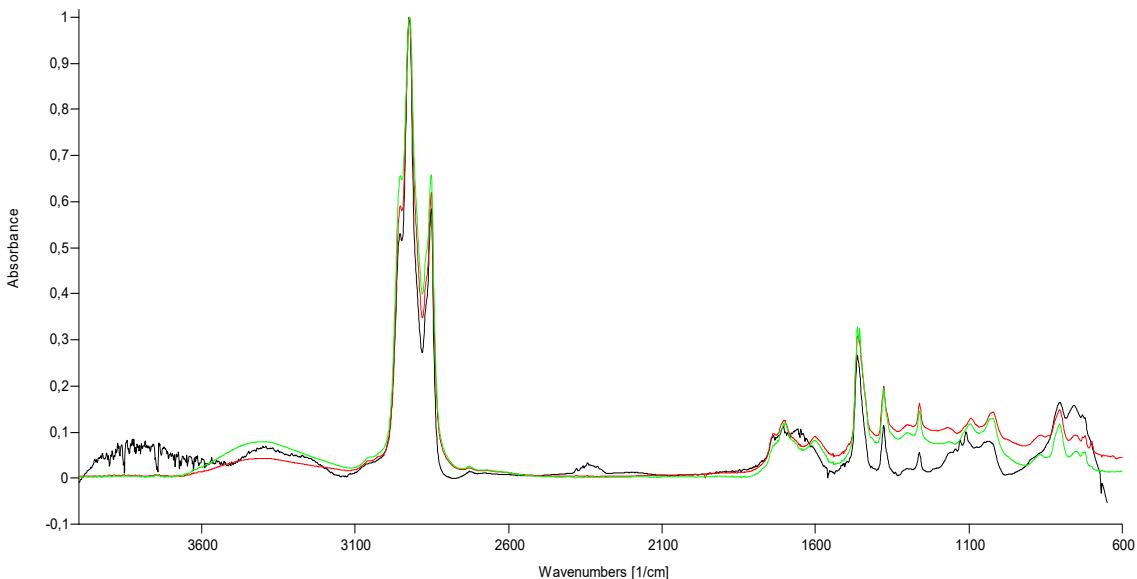
In **Figure SI7(b)** the peaks at  $1411$ ,  $843$ , and  $711\text{ cm}^{-1}$  can be assigned to calcium carbonate. In addition, the absorption bands at  $3289\text{ cm}^{-1}$  (O-H and/or N-H stretching modes),  $1656$  and  $1536\text{ cm}^{-1}$  (Amide-I N-C=O stretching and Amide-II N-H deformation) are attributable to proteins, such as conchiolin in natural sea shell. Thus, the spectrum may be assigned to sea shell.



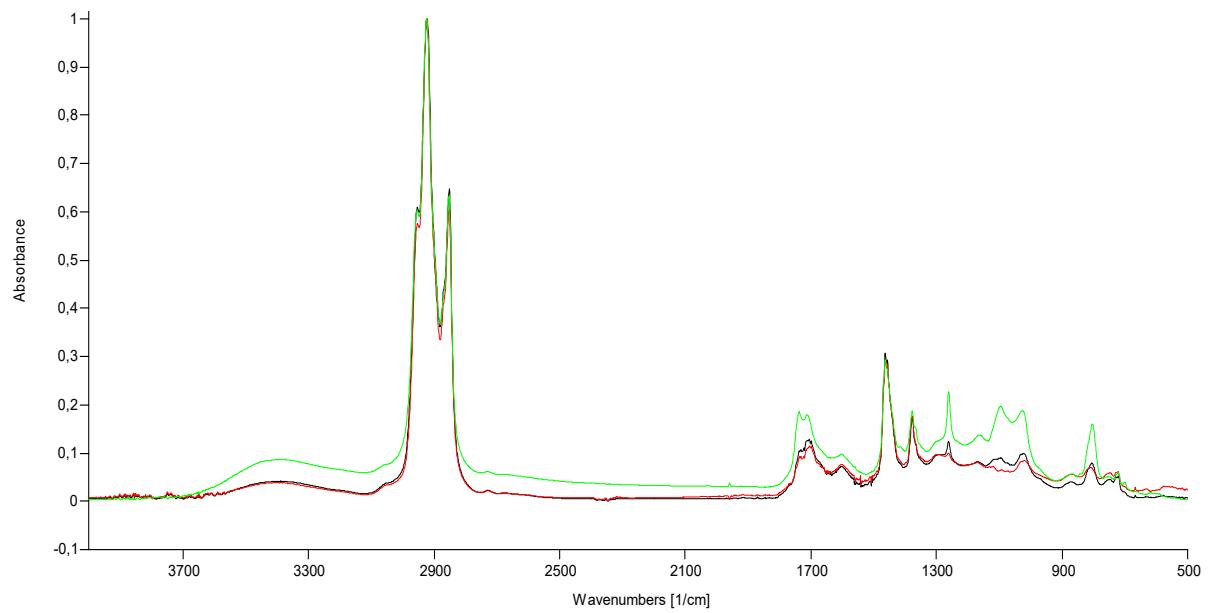
(b)

**Figure SI7.** Biogenic fragment from BR1C3: **a)** optical microscopy image; **b)** micro-ATR FTIR.

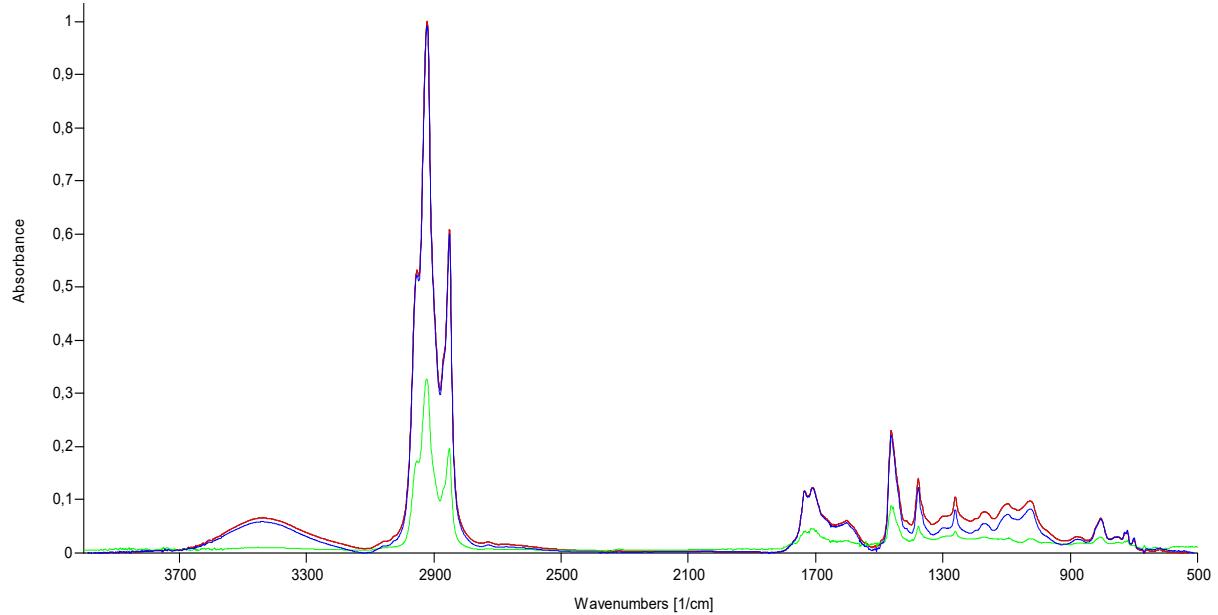
## S3-Spectroscopic analysis of dichloromethane-extractable fractions



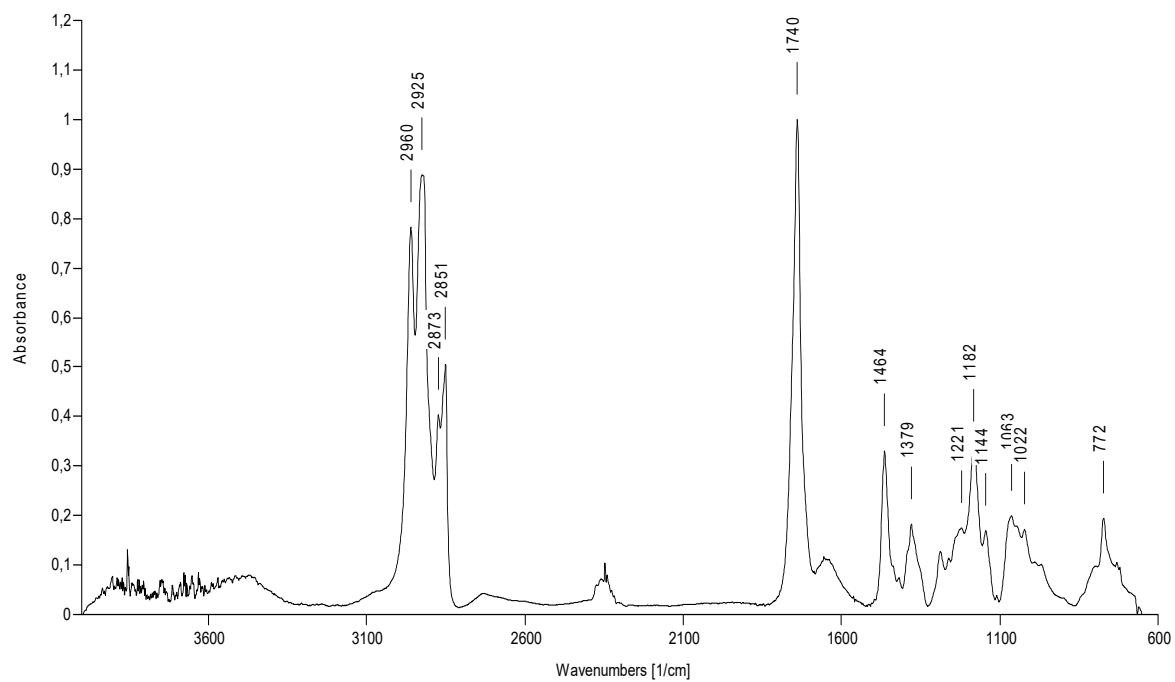
**Figure SI8.** FT-IR spectra of BR1A1 (green line), BR1B1 (red line) and BR1C1 (black line) extracts in  $\text{CH}_2\text{Cl}_2$ .



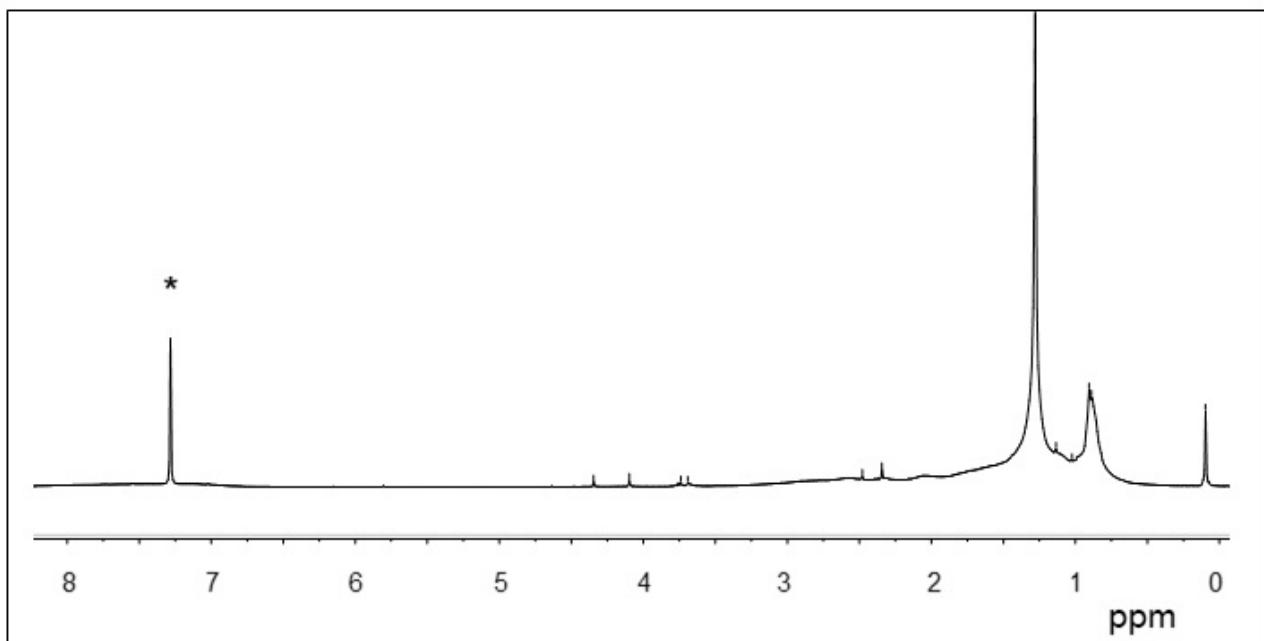
**Figure SI9.** FT-IR spectra of BR1A2 (red line), BR1B2 (black line) and BR1C2 (green line) extracts in dichloromethane.



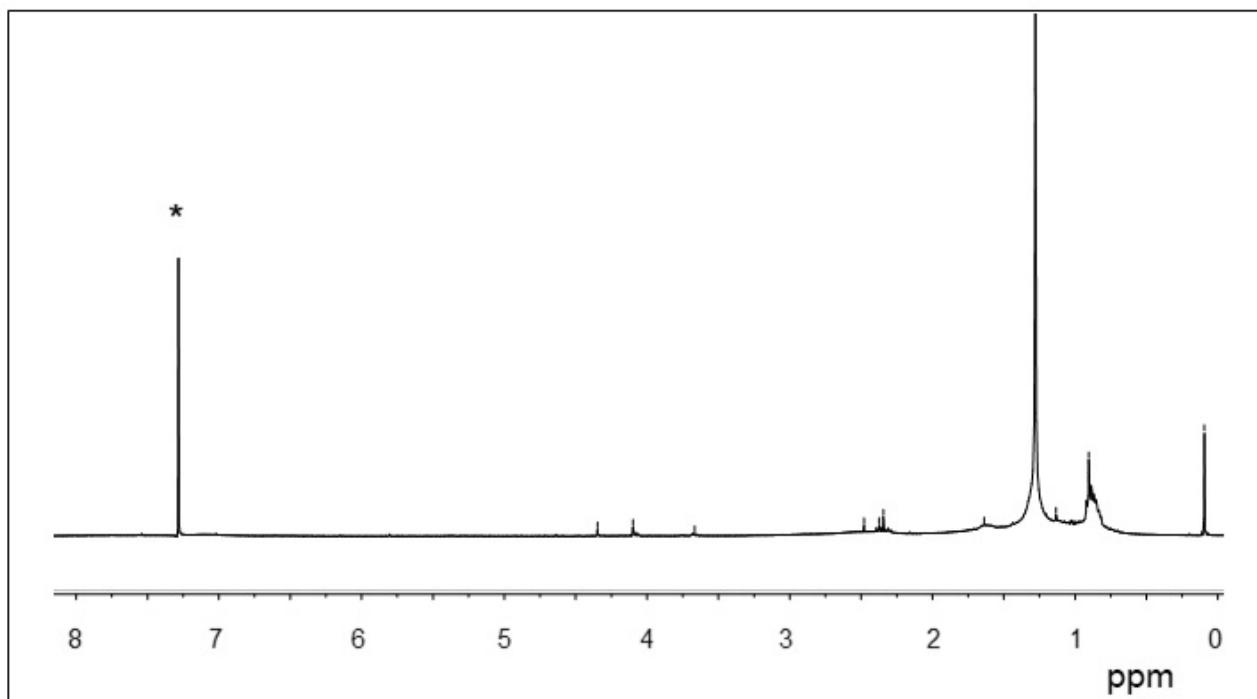
**Figure SI10.** FT-IR spectra of BR1A3 (red line) and BR1B3 (blue line) and BR1C3 (green line) extracts in dichloromethane.



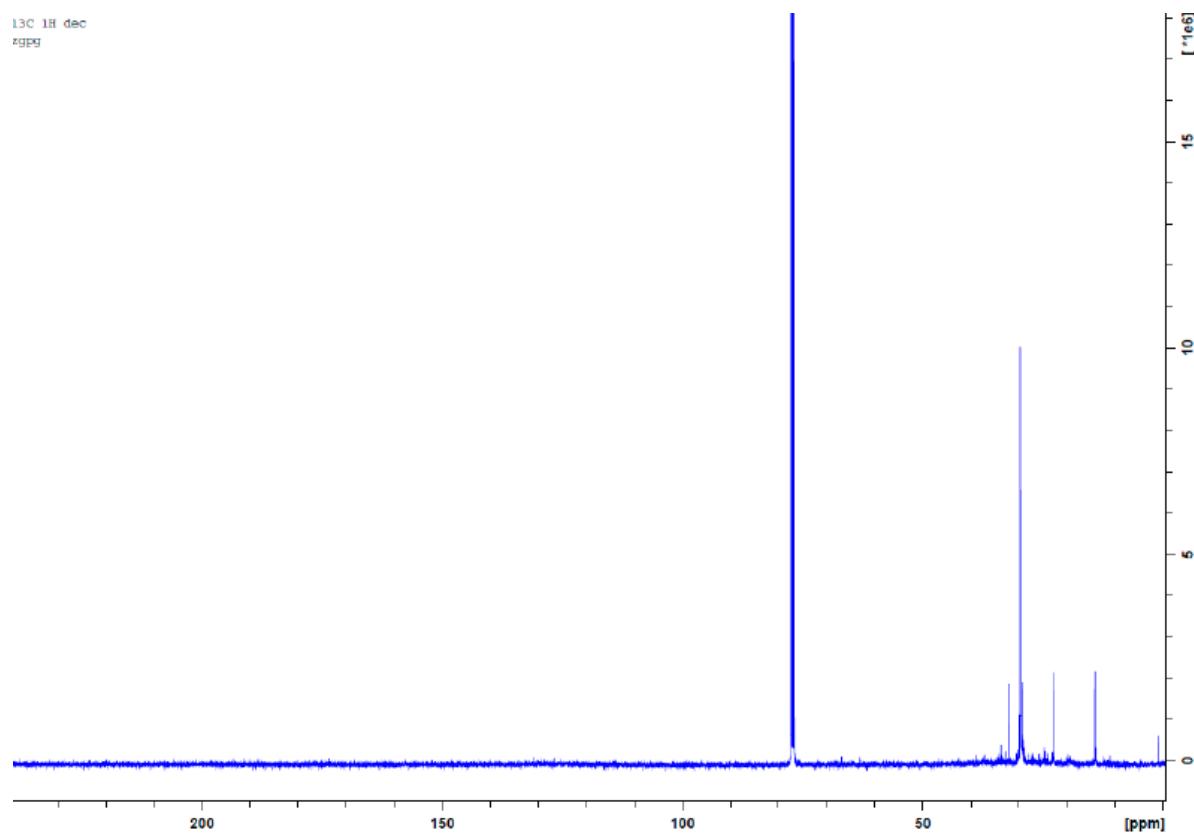
**Figure SI11.** FT-IR spectrum of the BR2B3 sample extracted in dichloromethane



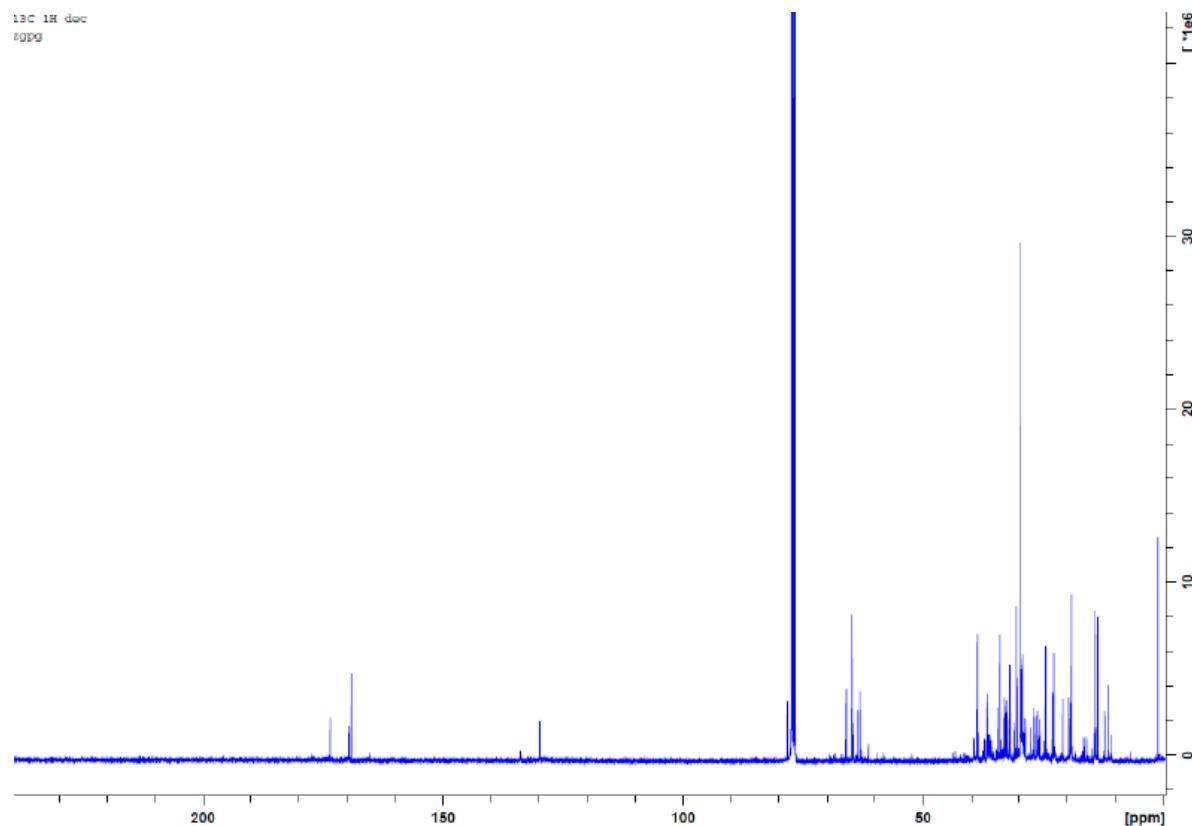
**Figure SI12.** <sup>1</sup>H-NMR spectrum of the BR1B1 sample extracted in dichloromethane.



**Figure SI13.** <sup>1</sup>H-NMR spectrum of the BR1C3 sample extracted in dichloromethane.

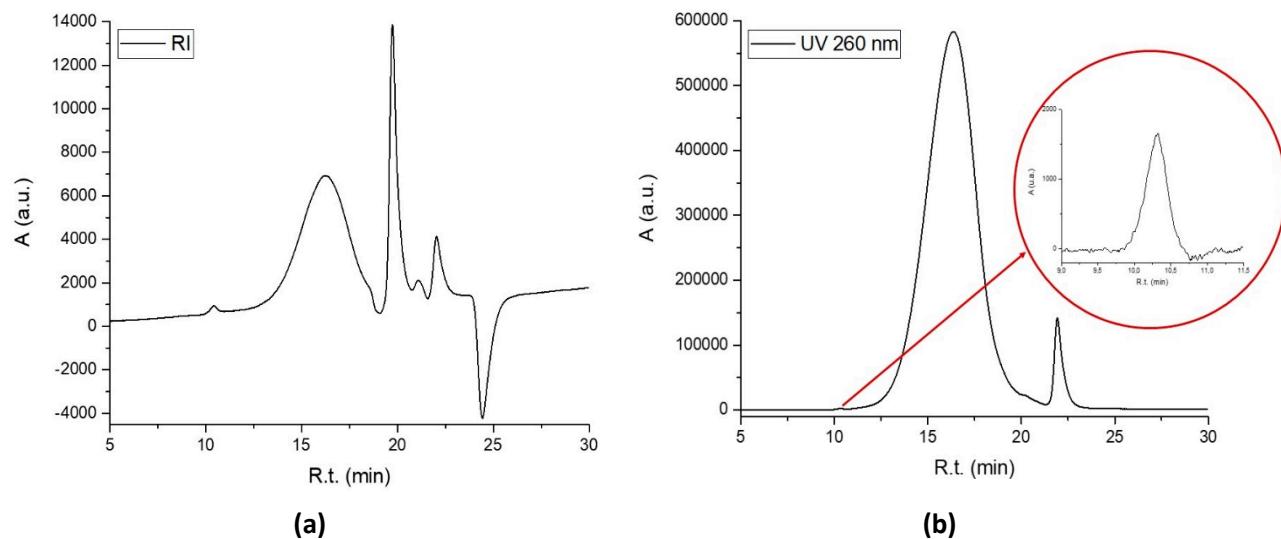


**Figure SI14.** <sup>13</sup>C-NMR spectrum of the BR1B3 sample extracted in dichloromethane.



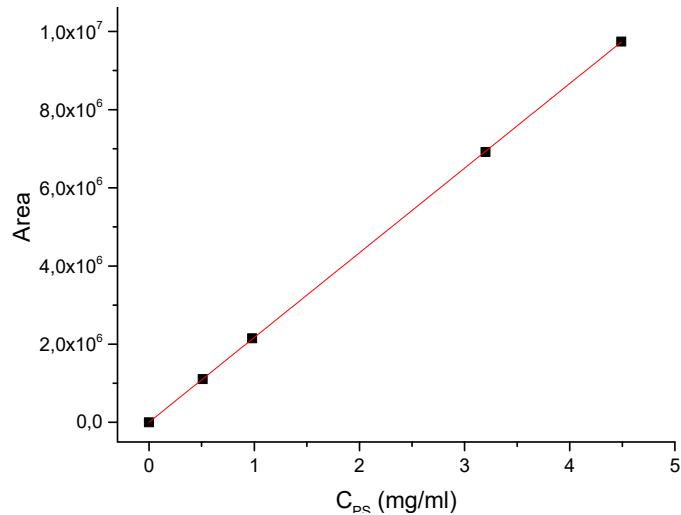
**Figure SI15.**  $^{13}\text{C}$ -NMR spectrum of the BR2B3 sample extracted in dichloromethane.

#### S4. Analysis of DCM extracts by Size Exclusion Chromatography (SEC)



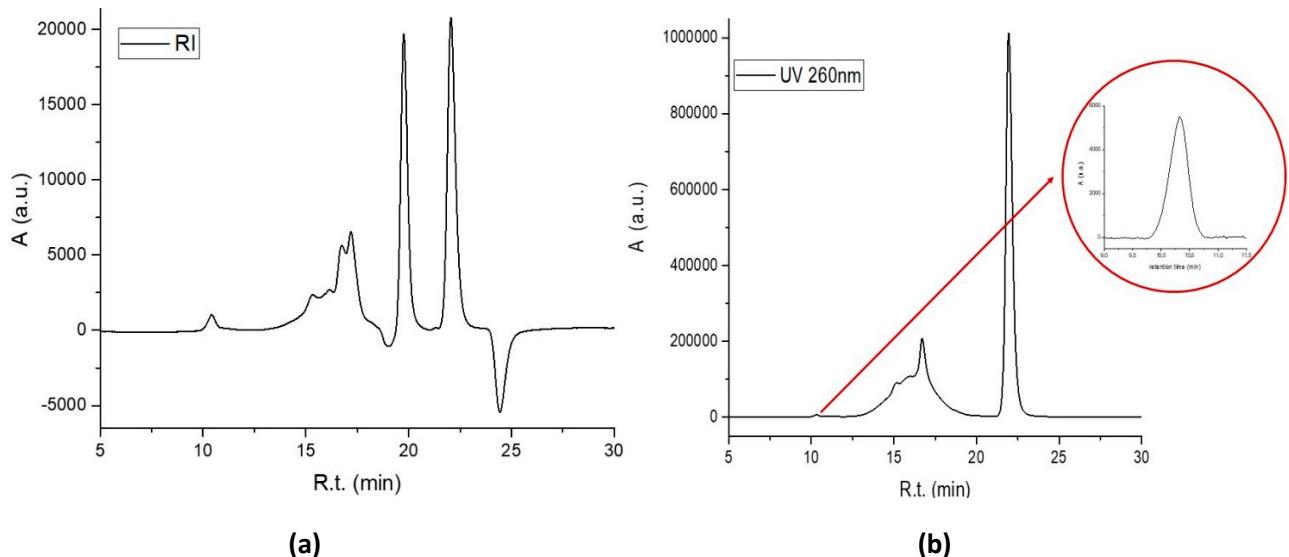
**Figure SI16.** SEC profiles as recorded by (a) Refractive index and (b) UV ( $\lambda=260\text{ nm}$ ) detectors from DCM extracts of BR1A1 sample.

**Figure SI17** shows the linear interpolation of the experimental data obtained by injecting three replicates of containing 0.5-4.5 mg / ml of PS dissolved in chloroform; the injection volume in the column is 50  $\mu$ L.

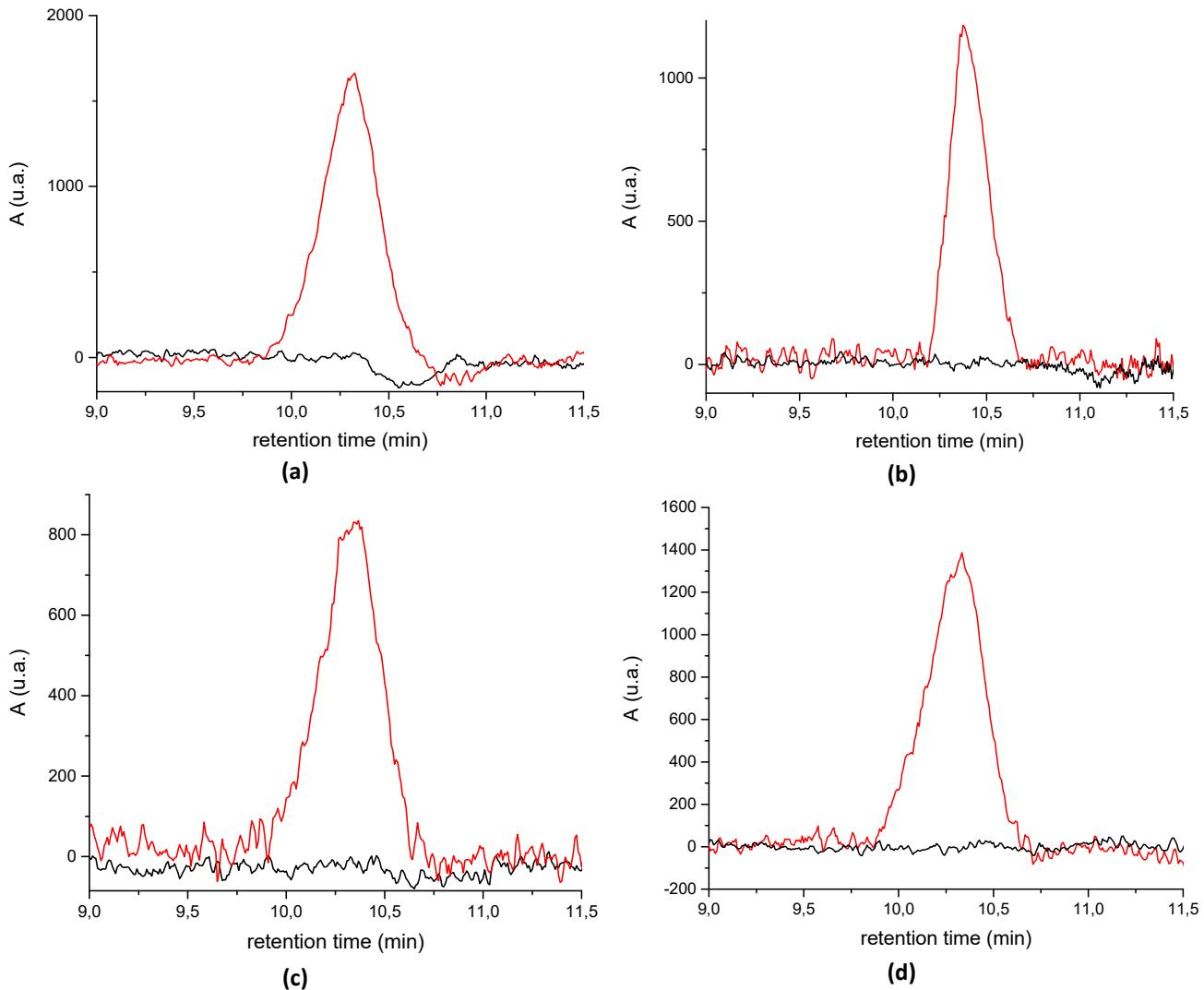


**Figure SI17.** Calibration line recorded with UV detector (260 nm) for SEC analysis.

Equation line:  $A = 2.17E + 6 * C + 6033.36$   $r^2=0.99998$



**Figure SI18.** SEC profiles as recorded by (a) Refractive index, and (b) UV ( $\lambda=260$  nm) detectors from DCM extracts of BR2A2 sample.



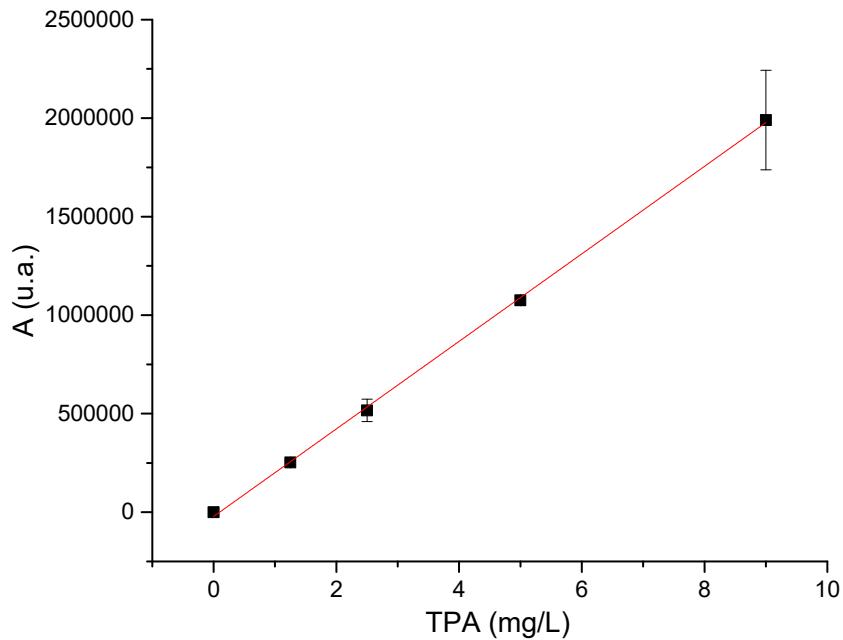
**Figure SI19.** UV 260 nm (red line) and UV 340 nm (black line) SEC profiles of DCM extracts from: a) BR1A1; b) BR1B3; c) BR2A3; d) BR2C2.

## S5. Calibration for HPLC analysis

**Figure SI20** shows the linear interpolation of the experimental data obtained by injecting three replicates of containing 1.25, 2.5, 5 and 9 mg/L of dissolved TPA in NaOH solution submitted to the sample pre-treatment procedure; the injection volume in the column is 50  $\mu$ L.

(a)

(b)



**Figure SI20.** Experimental calibration line for HPLC quantitative analysis of TPA

Equation line:  $A = 2.21E + 8 * C - 21633.7$        $r^2=0.9998$

LOD: 136 µg/kg      LOQ: 453 µg/kg

LOD and LOQ have been calculated as following:

$$\text{LOD} = \left( \frac{\sigma_{\text{sol},A}}{m_{\text{line}}} * 3 \right) * 10^6 \quad \text{LOQ} = \left( \frac{\sigma_{\text{sol},A}}{m_{\text{line}}} * 10 \right) * 10^6$$

where “ $\sigma_{\text{sol},A}$ ” is the standard deviation relevant to the absorbance recorded from multiple injections of the most diluted solutions (1.25 mg/L) of the calibration curve and “ $m$  line” is the slope of the calibration curves; 0.05 % probability level for false positive decisions was considered.