Supporting Information

Quantitative analysis of selected plastics in high commercial value Australian seafood by Pyrolysis Gas Chromatography Mass Spectrometry

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Chemicals and reference materials

Polystyrene, poly (methyl methacrylate) and polyvinylchloride were purchased from Sigma-Aldrich and low density polyethylene (LDPE) from Thermo Fischer Scientific. Polyethylene terephthalate (PET) was kindly provided by the Norwegian Institute for Water Research (NIVA) in Oslo, Norway and polypropylene (PP) was donated by a plastic manufacturer from Melbourne, Australia.

Liquid/gas chromatography grade dichloromethane (DCM) was purchased from Merck (Darmstadt, Germany). Hydromatrix was purchased from Agilent Technologies (Santa Clara, CA, USA). Potassium hydroxide (KOH) in pellet form was purchased from Sigma-Aldrich (221473 – 2.5KG) and ultra-pure water (18.2 MΩ cm$^{-1}$) was obtained from a Milli-Q filtration unit (Merck Millipore, MA, USA).

Table S 1 Plastic standards used for data base library matching and mass calibration. NS – non specified

| Polymer standard                                      | Abbreviation | Size (µm) | Source of supply            |
|-------------------------------------------------------|--------------|-----------|----------------------------|
| Polystyrene (327794-1G) (powder form)                 | PS           | NS        | Sigma- Aldrich             |
| Poly (methyl methacrylate) (81489 – 500 MG)            | PMMA         | NS        | Sigma- Aldrich             |
| Low Density Polyethylene (A10239) (powder form)        | LDPE         | 500       | Thermo Fischer Scientific  |
| Polyethylene terephthalate (pellet form)               | PET          | 20-110 µm | NIVA                      |
| Polyvinylchloride (powder form) (182621-25G)          | PVC          | NS        | Sigma- Aldrich             |
| Polypropylene, isotactic (EP341R) (pellet form)        | PP           | 20-120 µm | Lyondellbasell (VIC)       |
Extraction of plastic standards using an Accelerated Solvent Extraction (ASE) method

All samples were extracted by a Dionex ASE-350 system. High temperature Viton-O-rings (Dionex 056325) were used in the end-caps, glass fibre filters (Dionex 056781) were used in the cell base and Hydromatrix sorbent was used to cover the sample, reduce solvent and prevent floatation.

To assess extraction efficiency, between 20 and 50 mg of each selected plastic was weighed and placed inside of an ASE cell (34 mL) previously covered with a filter. Five replicates were performed for each type of plastic (PS, PE, PET, PMMA, PP and PVC) in addition to three controls (cells filled with only Hydromatrix). Following extraction the recovered mass of each polymer was determined gravimetrically by evaporating the liquid in the collection bottles to dryness in a fume hood under a gentle stream of nitrogen (40 °C). Analysis was performed on the dried residues to quantify the mass of polymer recovered by weight (0.01 g of precision).

Table S 2 ASE conditions for the selected plastics

| Parameter                  | Optimized extraction parameters |
|----------------------------|---------------------------------|
| Extraction solvent         | Dichloromethane                 |
| Extraction temperature (°C)| 180                             |
| Static time (s)            | 5                               |
| Cycles                     | 3                               |
| Rinse volume (%)           | 80                              |
| Purge time (s)             | 75                              |
| System rinse volume (ml)   | 9                               |
| Heating time (min)         | 9                               |
| Pressure (psi)             | 1500                            |
Pyrolysis Gas Chromatography/ Mass Spectrometry (Py-GC/MS) analysis

Py-GC/MS was performed using a Multi-shot Pyrolyzer (EGA/PY-3030D) equipped with an auto-shot sampler (AS-1020E) (Frontier Laboratories Ltd., Fukushima Japan) coupled to a Shimadzu GC/MS-QP2010 plus. The MS was operated in electron ionisation (EI) mode and compounds separated on a Frontier Laboratories Ultra ALLOY -5 capillary column (30 m x 0.25 mm x 0.25 µm) with helium as the carrier gas.

The initial oven program was set at 40 °C for 2 min, then increased to 320 °C at 20 °C/min and held for 14 min. Data was acquired in full scan mode (mass range 40 to 600 m/z) with a scanning rate of 2000 Hz. Library search of Shimadzu was used for peak identification of the pyrolyzates together with Kovats retention index (RI) data.

Table S 3  Conditions for Single shot Pyrolysis-GC/MS measurements

| Apparatus                          | Parameters                                    | Settings                                                   |
|------------------------------------|-----------------------------------------------|------------------------------------------------------------|
| Micro-furnace Pyrolyzer (Single-shot analysis) EGA/PY-3030D | Temperature program                           | 70 °C (2 min.) > 320 °C (12.5 min ) at 20 °C min⁻¹          |
|                                    | Pyrolysis temperature                         | 650 °C                                                     |
|                                    | Interface temperature                         | 300 °C                                                     |
|                                    | Pyrolysis time                                | 0.20 minutes (12 seconds)                                  |
| Gas Chromatograph                  | Column                                        | Ultra-Alloy® 5 capillary column (30 m, 0.25 mm I.D., 0.25 µm film thickness) (Frontier Lab) |
|                                    | Injector port temperature                     | 300 °C                                                     |
|                                    | Column oven temperature program                | 40 °C (2 min) → (20 °C /min) → 320 °C (14 min)             |
|                                    | Injector mode                                 | Split (5:1)                                                |
|                                    | Carrier gas                                   | Helium, 52.1 cm/s, constant linear velocity               |
| Mass Spectrometer                  | Ion source temperature                        | 250 °C                                                     |
|                                    | Ionization energy                             | Electron ionization (EI) ); 70 eV                         |
|                                    | Scan range                                    | 40 to 600 m/z                                              |
Table S 4 Selected indicator ions of pyrolysis products of different polymers at 650 °C; M/z masses highlighted in **bold** were used for quantification: polystyrene (PS), polypropylene (PP), polyethylene terephthalate (PET), poly-(methyl methacrylate) (PMMA), polyethylene (PE) and polyvinyl chloride (PVC)

| Polymer | Characteristic decomposition product(s) | Indicator ions (m/z ratio) | Retention time (min) |
|---------|----------------------------------------|----------------------------|----------------------|
| PS      | Styrene                                | 104, 78                    | 5.835                |
|         | **Styrene dimer: 3-butene-1,3-diyl dibenzene** | 91, **130**, 193, 208      | **12.875**           |
|         | Styrene trimer: 5-hexene-1,3,5-triyltribenzene | 91, 117, 194, 312          | 16.765               |
| PP      | n-pentane                              | 55, 72                     | 2.105                |
|         | 2-methyl-1-pentene                     | 56, 69, 84                 | 2.235                |
|         | 2,4, 6-dimethyl-1-heptene              | 70, 83, **126**            | **5.15**             |
| PET     | benzene                                | 78, 52                     | 2.825                |
|         | Vinyl benzoate                         | 105, 77, 148, 51           | 8.42                 |
|         | BENZOIC ACID                           | 105, 122, 77               | 9.095                |
|         | diphenyl                               | 154, 131, 76               | 10.525               |
|         | divinil terephthalate                  | 175, 104                   | 11.77                |
|         | 4-(vinyloxycarbonyl) benzoic acid      | 149, 121                   | 12.105               |
|         | Ethan-1,2-diyl dibenzoate              | 105, 77, 227               | 15.27                |
|         | 2- (benzoyloxy)ethy1 vinyl terephthalate | 297, 149                   | 17.445               |
|         | Ethan-1,2-diyl divinyl diterphthalate   | 364, 325, 296, 219         | 20.13                |
|         | Bis(2-(benzoyloxy)ethyl) terephthalate  | 105, 297, 149              | 29                   |
| PMMA    | Methyl acrylate                        | 55, 85                     | 2.4                  |
|         | **Methyl methacrylate**                | 69, **100**, 89             | **3.405**            |
|         | (Z)-trimethyl 4,6-dimethylhept-2-ene-2,4,6-tricarboxylate | 121, 149                  | 13.425               |
|         | (Z)-dimethyl 2,4-dimethylpent-2-enedioate | 67, 95, 127, 111, 154      | 9.065                |
|         | dimethyl 2, 2-dimethyl-4-methylenepentanedioate | 81, 101, 109, 125, 140    | 9.45                 |
|         | C11H18O4                               | 81, 95, 109, 123           | 10.055               |
| PE      | 1-Nonene (C9)                          | 83, 97, 111                | 5.815                |
|         | 1, 9-decadiene (C10)                   | 67, 81, 95, 110,123        | 6.895                |
|         | **1-decene (C10)**                     | **83**, 97, 111, 140       | **6.97**             |
|         | n-decane (C10)                         | 71, 85, 98, 113, 142      | 7.06                 |
|         | 1-undecene (C11)                       | 83, 97,111,126, 152       | 7.97                 |
|         | **3-tetradecene (nitr) or 1-dodecene (C12)** | **83**, 97, 111, 125      | **8.87**             |
|         | 1-tridecane (C13)                      | 83, 97, 111, 125           | 9.695                |
|         | **1-tetradecene (C14)**                | **83**, 97, 111,125,140   | **10.465**           |
|         | 1 pentadecene (C15)                    | 83, 97, 111                | 11.185               |
|         | 1-hexadecene (C16)                     | -                         | 11.865               |
|         | 1-eicosene (C20)                       | 83, 97, 111                | 14.255               |
|         | C17                                     | 83, 97                     | 12.51                |
|         | 1-heptene (C7)                         | 70, 83, 98                 | 3.175                |
|         | C8                                      | -                         | 4.575                |
|         | C6                                      | -                         | 2.27                 |
| PVC     | Benzene                                 | 78, 52                     | 2.855                |
|         | Toluene                                 | 91, 65                     | 4.165                |
|         | Indene                                  | 116, 119                   | 7.640                |
|         | Styrene                                 | 104, 78                    | 5.835                |
**Multivariate calibration curve for PE**

To generate a multivariate calibration curve for quantification of PE in different matrices, we used a least square approach, where the algorithm minimizes the sum of squares of the distance between each point and the line. The final calibration curve with an adjusted $R^2$ of 0.99 included four coefficients and the intercept term. These four coefficients consisted of three associated to each independent variable (i.e. peak intensity) and the interaction between 1-decene and 1-dodecene. For the validation of this multivariate calibration curve, we employed leave one out approach, where during each iteration one of the points in the curve will be removed while generating a new curve using the remaining points $^{1,2}$. This process was repeated in order to have each point the calibration curve removed at least once.
Table S 5 Recovery of plastic standards after ASE with DCM at 180 °C

| Polymer type | Spike amount (mg) | Recovered amount (mg) | (%) Recovery | Average recovery mean ± STD (%) | RSD (%) |
|--------------|-------------------|-----------------------|--------------|---------------------------------|---------|
|              | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 |                      |            |                    |
| PMMA         | 45.4  | 50.1  | 44.4  | 47.6  | 44.9  | 97.7 ± 3.3 | 3.4   |
|              | 42.5  | 48.8  | 42.4  | 48.2  | 45.2  | 98.2 ± 13.4| 13.7  |
|              | 93.6  | 97.4  | 95.5  | 101.3 | 100.7 | 92.8 ± 8.8 | 9.4   |
| PS           | 29.5  | 22.7  | 20.8  | 25.8  | 21.8  | 94.2 ± 5.3 | 5.6   |
|              | 31.7  | 25.4  | 18.6  | 20.5  | 22.4  | 90.8 ± 7.6 | 8.4   |
|              | 107.5 | 111.9 | 89.4  | 79.5  | 102.8 | 96.9 ± 7.5 | 8.7   |
| PET          | 22.6  | 50.8  | 43.6  | 36.7  | 45.9  | 92.8 ± 8.8 | 9.4   |
|              | 20.3  | 47.3  | 40.2  | 30.2  | 48.9  | 92.8 ± 8.8 | 9.4   |
|              | 89.8  | 93.1  | 92.2  | 82.3  | 106.5 | 92.8 ± 8.8 | 9.4   |
| PP           | 37.4  | 43.6  | 45.4  | 43.4  | 41.1  | 92.8 ± 8.8 | 9.4   |
|              | 35.1  | 39.9  | 43.4  | 38.2  | 42.0  | 92.8 ± 8.8 | 9.4   |
|              | 93.9  | 91.5  | 95.6  | 88.0  | 102.2 | 92.8 ± 8.8 | 9.4   |
| PE           | 45.1  | 41.7  | 45.6  | 37.8  | 44.6  | 90.8 ± 7.6 | 8.4   |
|              | 35.3  | 41.1  | 42.4  | 34.3  | 41.6  | 90.8 ± 7.6 | 8.4   |
|              | 78.2  | 98.6  | 93.0  | 90.7  | 93.3  | 90.8 ± 7.6 | 8.4   |
| PVC          | 45.5  | 40.3  | 37.4  | 28.5  | 39.2  | 86.8 ± 7.5 | 8.7   |
|              | 38.9  | 36.6  | 31.6  | 21.9  | 38.0  | 86.8 ± 7.5 | 8.7   |
|              | 85.4  | 90.7  | 84.4  | 76.7  | 96.9  | 86.8 ± 7.5 | 8.7   |

Replicate (Rep) 1, 2, 3, 4 and 5 represent the number of experiments for each polymer standard; blue numbers represent the percentage of recovery of each trial; average recovery represents the mean recovery from the five replicates; STD: standard deviation; RSD: relative standard deviation (%); poly-(methyl methacrylate) (PMMA), polystyrene (PS), polyethylene terephthalate (PET), polypropylene (PP), polyethylene (PE) and polyvinyl chloride (PVC)
Table S 6 Recovery of spiked oyster samples after ASE with DCM at 180 ºC

| Polymer type | Spike amount (mg) | Recovered amount (mg) | (%) Recovery | Average recovery mean ± STD (%) | RSD (%) |
|--------------|------------------|-----------------------|--------------|----------------------------------|---------|
|              | Rep. 1          | Rep. 2                | Rep. 3       | Rep. 4                            | Rep. 5   |
| PS           | 14.01           | 14.95                 | 16.23        | 15.06                             | 17.49    | 14.29               | 83.1 ± 17.9 | 21.6 |
|              | 17.06           | 10.87                 | 13.69        | 11.19                             | 13.42    | 9.82                |             |      |
|              | 121.77          | 72.71                 | 84.35        | 74.30                             | 76.72    | 68.719              |             |      |
| PE           | 19.45           | 21.20                 | 18.58        | 18.51                             | 18.37    | 18.82               | 85.7 ± 6.5  | 7.6  |
|              | 19.01           | 16.28                 | 15.55        | 15.69                             | 16.37    | 15.40               |             |      |
|              | 97.74           | 76.79                 | 83.69        | 84.76                             | 89.11    | 81.83               |             |      |
| PP           | 21.57           | 18.17                 | 19.59        | 18.58                             | 18.47    | 18.76               | 78.3 ± 18.5 | 23.7 |
|              | 14.28           | 13.58                 | 14.09        | 12.01                             | 21.96    | 13.76               |             |      |
|              | 66.20           | 74.74                 | 71.92        | 64.64                             | 118.90   | 73.35               |             |      |
| PVC          | 22.05           | 19.32                 | 20.55        | 18.97                             | 21.48    | 21.43               | 86.5 ± 13.1 | 15.1 |
|              | 17.69           | 14.41                 | 17.44        | 17.02                             | 24.36    | 16.29               |             |      |
|              | 80.23           | 74.59                 | 84.87        | 89.72                             | 113.41   | 76.01               |             |      |
| PMMA         | 15.92           | 15.64                 | 16.71        | 15.90                             | 15.93    | 15.37               | 100.7 ± 14.5 | 14.4 |
|              | 17.98           | 13.15                 | 15.07        | 18.90                             | 13.56    | 17.34               |             |      |
|              | 112.94          | 84.08                 | 90.19        | 118.87                            | 85.12    | 112.82              |             |      |
| PET          | 25.95           | 21.39                 | 22.94        | 27.46                             | 24.24    | 25.85               | 32.1 ± 11   | 34.3 |
|              | 12.96           | 5.67                  | 6.16         | 6.69                              | 10.77    | 5.27                |             |      |
|              | 49.94           | 26.51                 | 26.85        | 24.36                             | 44.43    | 20.39               |             |      |

Replicate (Rep) 1, 2, 3, 4, 5 and 6 represent the number of experiments for each polymer standard; blue numbers represent the percentage of recovery of each trial; average recovery represents the mean recovery from the five replicates; STD: standard deviation RSD: relative standard deviation (%); polystyrene (PS), polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), poly-(methyl methacrylate) (PMMA) and polyethylene terephthalate (PET)
Table S 7 PS standard (1 mg ml\(^{-1}\)) peak areas and correspondent mass (n = 10)

| Number of runs | Peak area (styrene dimer: 3-butene-1,3-diyl dibenzene) | Total mass (µg) | Mean ± STD | RSD (%) |
|----------------|---------------------------------------------------------|-----------------|------------|---------|
| 1              | 1045825                                                | 3454            |            |         |
| 2              | 1009723                                                | 3335            |            |         |
| 3              | 1399269                                                | 4619            |            |         |
| 4              | 1180607                                                | 3898            |            |         |
| 5              | 1255677                                                | 4146            |            |         |
| 6              | 1406879                                                | 4644            |            |         |
| 7              | 985000                                                 | 3253            |            |         |
| 8              | 1149959                                                | 3797            |            |         |
| 9              | 1428720                                                | 4716            |            |         |
| 10             | 1692921                                                | 5587            | 4145 ± 707 | 17      |

Table S 8 Calibration functions for selected plastics. *results are based on ions for the plastic as presented in Table S4:

| Plastic type | Indicator ions                  | Linear range (µg) | Calibration functions | Linearity \(R^2\) * |
|--------------|---------------------------------|-------------------|-----------------------|---------------------|
| PS           | 3-butene-1,3-diyl dibenzene     | 0.02 - 10         | \(Y = 189548 \times X - 1580.9\) | 0.99                |
| PE           | 1-decene (C\(10\))             | 0.02 - 10         | \(Y = 99469 \times X - 10677\) | 0.98                |
| PVC          | Benzene                         | 0.02 - 10         | \(Y = 3825975.5 \times X - 594914.38\) | 0.98                |
| PP           | 2,4, 6-dimethyl-1-heptene       | 0.02 - 10         | \(Y = 63821 \times X - 118.72\) | 0.98                |
| PMMA         | Methyl methacrylate             | 0.02 - 10         | \(Y = 30793 \times X - 7518.4\) | 0.99                |

Polystyrene (PS), polyethylene (PE), polyvinyl chloride (PVC) polypropylene (PP) and poly-(methyl methacrylate) (PMMA)
Table S 9 LOD and LOQ (in µg per g of tissue)

| Sample  | Plastic | LOD (µg/g tissue) | LOQ (µg/g tissue) |
|---------|---------|------------------|-------------------|
| Oysters | PS      | 0.70             | 0.96              |
|         | PE      | 0.90             | 9.41              |
|         | PVC     | 1.04             | 10.93             |
|         | PP      | 0.83             | 2.45              |
|         | PMMA    | 0.95             | 24.29             |
| Prawns  | PS      | 0.70             | 0.96              |
|         | PE      | 8.05             | 9.35              |
|         | PVC     | 10.82            | 10.87             |
|         | PP      | 0.85             | 2.44              |
|         | PMMA    | 19.22            | 24.15             |
| Squid   | PS      | 0.54             | 0.75              |
|         | PE      | 0.69             | 7.28              |
|         | PVC     | 0.81             | 8.46              |
|         | PP      | 0.64             | 1.90              |
|         | PMMA    | 0.74             | 18.80             |
| Sardines| PS      | 0.51             | 0.70              |
|         | PE      | 5.85             | 6.79              |
|         | PVC     | 7.86             | 7.90              |
|         | PP      | 0.62             | 1.77              |
|         | PMMA    | 13.96            | 17.54             |
| Crabs   | PS      | 0.05             | 0.07              |
|         | PE      | 0.62             | 0.72              |
|         | PVC     | 0.83             | 0.83              |
|         | PP      | 0.07             | 0.19              |
|         | PMMA    | 1.48             | 1.86              |
Table S 10 Mass (µg) of each plastic found in the procedural blanks (n=10) measured by Py-GC/MS

| KOH filters | PS  | PE  | PVC | PP  | PMMA |
|-------------|-----|-----|-----|-----|------|
| 1           | -   | 0.2 | 0.2 | 1.4 | -    |
| 2           | -   | 0.1 | -   | -   | -    |
| 3           | -   | -   | -   | -   | -    |
| 4           | -   | 0.1 | -   | -   | -    |
| 5           | -   | -   | -   | -   | -    |
| 6           | -   | -   | -   | -   | -    |
| 7           | -   | 0.1 | -   | -   | -    |
| 8           | -   | -   | -   | -   | -    |
| 9           | -   | -   | -   | -   | -    |
| 10          | -   | 0.1 | -   | -   | -    |
| Sum         | -   | 0.6 | 0.2 | 1.5 | -    |
| Average     | -   | 0.1 | -   | 0.1 | -    |
| Std         | -   | 0.1 | -   | 0.4 | -    |
Table S 11 Concentration of PS, PE, PVC, PP and PMMA (µg/g tissue) measured in seafood (each sample was analysed in triplicate); < LOQ: below limit of quantification; when a value was <LOQ, ½ LOD was used;

| Sample ID | Concentration (µg g⁻¹) | | |
|-----------|------------------------|---|---|---|---|
| **Oysters** Crassostrea gigas | **ΣPlastic** (µg g⁻¹) | PS | PE | PVC | PP | PMMA |
| 1 | - | - | 23.55 | - | - | 23.55 |
| 2 | - | - | 13.40 | - | - | 13.40 |
| 3 | - | - | < LOQ | - | - | 0.52 |
| 4 | - | - | < LOQ | - | - | 0.52 |
| 5 | - | - | 14.32 | - | - | 14.32 |
| 6 | - | - | 14.82 | < LOQ | - | 15.23 |
| 7 | - | - | 17.60 | < LOQ | - | 18.01 |
| 8 | - | - | - | - | - | - |
| 9 | - | - | - | < LOQ | - | 0.41 |
| 10 | - | - | 16.18 | < LOQ | - | 16.59 |
| **Prawns** Penaeus esculentus | | PS | PE | PVC | PP | PMMA |
| 1 | - | - | 11.52 | < LOQ | - | 11.95 |
| 2 | - | - | 15.93 | 10.46 | - | 26.39 |
| 3 | - | - | - | - | - | - |
| 4 | - | - | - | < LOQ | - | 0.43 |
| 5 | - | - | - | - | - | - |
| 6 | - | - | - | 3.53 | - | 3.53 |
| 7 | - | - | - | 7.24 | - | 7.24 |
| 8 | - | - | - | 15.43 | - | 15.43 |
| 9 | - | - | - | 2.92 | - | 2.92 |
| 10 | - | - | - | 4.50 | - | 4.50 |
| **Squid** Nototodarus gouldi | | PS | PE | PVC | PP | PMMA |
| 1 | - | - | 10.91 | < LOQ | - | 11.23 |
| 2 | - | - | - | - | - | - |
| 3 | - | - | - | < LOQ | - | 0.32 |
| 4 | - | - | - | - | - | - |
| 5 | - | - | - | 23.85 | - | 23.85 |
| 6 | - | - | - | - | - | - |
| 7 | - | - | - | - | - | - |
| 8 | - | - | - | - | - | - |
| 9 | - | - | - | - | - | - |
| 10 | - | - | - | - | - | - |
|          | Crabs                          | PS  | PE  | PVC | PP   | PMMA |
|----------|-------------------------------|-----|-----|-----|------|------|
|          | Portunus armatus              |     |     |     |      |      |
| 1        |                               | 8.07| -   | 29.05| 10.27| 4.53 |
| 2        |                               | 2.86| -   | 1.18 | 11.82 | < LOQ |
| 3        |                               | 4.98| -   | 5.39 | 19.30 | -    |
| 4        |                               | 0.58| -   | 1.15 | 25.76 | -    |
| 5        |                               | 0.69| -   | 20.53| 14.74 | -    |
| 6        |                               | 0.28| -   | 3.68 | 2.55  | -    |
| 7        |                               | 0.57| -   | 3.32 | 14.29 | -    |
| 8        |                               | 6.80| -   | 39.31| 3.26  | 2.43 |
| 9        |                               | 0.86| -   | 18.61| 7.25  | -    |
| 10       |                               | 1.21| 43.18| 29.34| 2.53  | -    |
|          | Sardinops neopilchardus       |     |     |     |      |      |
| 1        |                               | -   | -   | -   | 3.32 | 13.96| 17.28 |
| 2        |                               | -   | -   | -   | -    | -    |
| 3        |                               | < LOQ| -   | -   | 13.44| -    | 13.69 |
| 4        |                               | 0.87| 111.55| -   | 5.38 | -    | 117.80 |
| 5        |                               | 27.30| -   | < LOQ| 59.89| -    | 87.44 |
| 6        |                               | < LOQ| -   | -   | 12.96| -    | 13.21 |
| 7        |                               | 0.85| 70.24| -   | 1.59 | -    | 72.68 |
| 8        |                               | 1.93| -   | -   | 45.54| -    | 47.47 |
| 9        |                               | -   | -   | -   | 1.78 | -    | 1.78 |
| 10       |                               | 102.65| 2352.28| 9.72| -    | 26.76| 2491.41 |
Figure S 1 Experimental design for the extraction of oyster’s. Filters were spiked with 6 different plastics: PS, PE, PET, PMMA, PP and PVC. Six glass fibre filters were used as a “blank” with no plastic added and the other 36 were spiked with selected plastics (as schematized).
Figure S 2 Total Ion Chromatogram (TIC) pyrograms of extracted plastics by ASE. Detailed decomposition products and retention times are summarized in Table S4. Polystyrene (PS), polypropylene (PP), poly-(methyl methacrylate) (PMMA), polyethylene (PE) and polyvinyl chloride (PVC)
Testing the dissolution of plastics in DCM after ASE extraction

After extraction, the samples were immediately placed in the pyrolysis cups (Eco-Cups 80LF, Frontier Labs, Japan) and evaporated to dryness in a fume hood to avoid airborne contamination of microplastics. The solubility of the selected plastics in DCM after ASE was tested by placing the plastic standard solutions in the sample cups at different times after the solvent extraction procedure (every 15 minutes for a total period of 2.5 hours).

Figure S 3 Peak area (in counts per second) of indicator compounds for each selected plastic measured over 2.5 hours. Polyethylene (PE), poly-(methyl methacrylate) (PMMA), polypropylene (PP), Polystyrene (PS) and polyvinyl chloride (PVC)
Relation between peak area of specific indicator compounds and split ratio

For all the indicator ions of selected plastics, areas significantly decreased with the increase of the split ratio ($p < 0.05$, One Way ANOVA) - Figure S4. Assuming linearity, the split ratio was set at 5 ensuring that the selected plastics in our samples were efficiently quantified and saturation of the mass spectrometer was avoided.

Figure S 4 Correlation between peak area (in counts per second) of specific compounds selected for each plastic and split ratio (50:1, 20:1, 10:1 and 5:1); polystyrene (PS), polyethylene (PE), polyvinyl chloride (PVC) polypropylene (PP) and poly-(methyl methacrylate) (PMMA)
Figure S 5 Comparison of a TIC (total ion chromatogram) of a plastic standard mixture with TICs of analysed seafood
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