

Quantitative analysis of airborne microplastics by pyrolysis-GC/MS

Part 2: Identification of volatiles and polymers by double-shot method

[Background] In the previous note (PYA3-050E), evolved gas analysis (EGA)-MS measurements of airborne microplastics (AMP) in the atmospheric particulate matter (PM) were carried out to determine the furnace temperature for double-shot measurements. In this note, the double-shot method was used for the analysis of PM samples: thermal desorption (TD)-GC/MS as the first stage for volatile components, followed by Py-GC/MS as the second stage for polymer components.

[Experimental] For double-shot measurements, a GC/MS system equipped with a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the GC inlet was used. A metal capillary column (UA5-30M-0.25F) was used as a separation column and connected to the mass detector via Vent-free GC/MS adapter. Circular portions of PM-collected filters were punched into 4-mm-diameter disks, and three of them were placed in a sample cup, and double-shot measurement was carried out as follows: (1) the sample cup was dropped into the furnace preheated at 100 °C and then the furnace temperature was increased to 300 °C at 30 °C/min. The sample cup was then returned to the standby position, and then GC/MS measurement was started. (2) the second stage was started by dropping the sample cup again into the furnace heated at 600 °C, followed by Py-GC/MS measurement.

[Results] As an example of double-shot results, a TD chromatogram and a pyrogram obtained for the >PM₁₀ fraction of the PM-A sample are shown in Fig. 1. Aliphatic carboxylic acids and phthalates were found in the TD chromatogram. Phthalates are considered to arise from plasticizers contained in AMP, and the detected concentrations of phthalates were found to be in the order of >PM₁₀, PM_{2.5-10}, and PM_{2.5}. From the pyrogram, polyethylene (PE) was identified by the presence of characteristic α -olefin and alkane peaks. Poly(methyl methacrylate) (PMMA) was confirmed by the detection of methyl methacrylate (MMA) monomer. Similarly, the presence of polystyrene (PS) was indicated by the peaks of styrene monomer, dimer, and trimer. In addition, detection of styrene-butadiene (SB) hybrid dimers and SBB hybrid terpolymers provided the evidence for the presence of styrene-butadiene rubber (SBR) copolymer. Furthermore, detection of the isoprene-derived dimers dipentene and 2,4-dimethyl-4-vinylcyclohexene indicated the presence of natural rubber (NR), while the detection of 2,4-dimethyl-1-heptene suggested the presence of polypropylene (PP). Based on these findings obtained by using the double-shot method, the polymers present in the AMP were identified as PE, PP, PS, SBR, PMMA, and NR. The quantitative analysis of PP, PS and SBR is presented in the next technical note (PYA1-185E) using the indicator ions of their characteristic pyrolyzates.

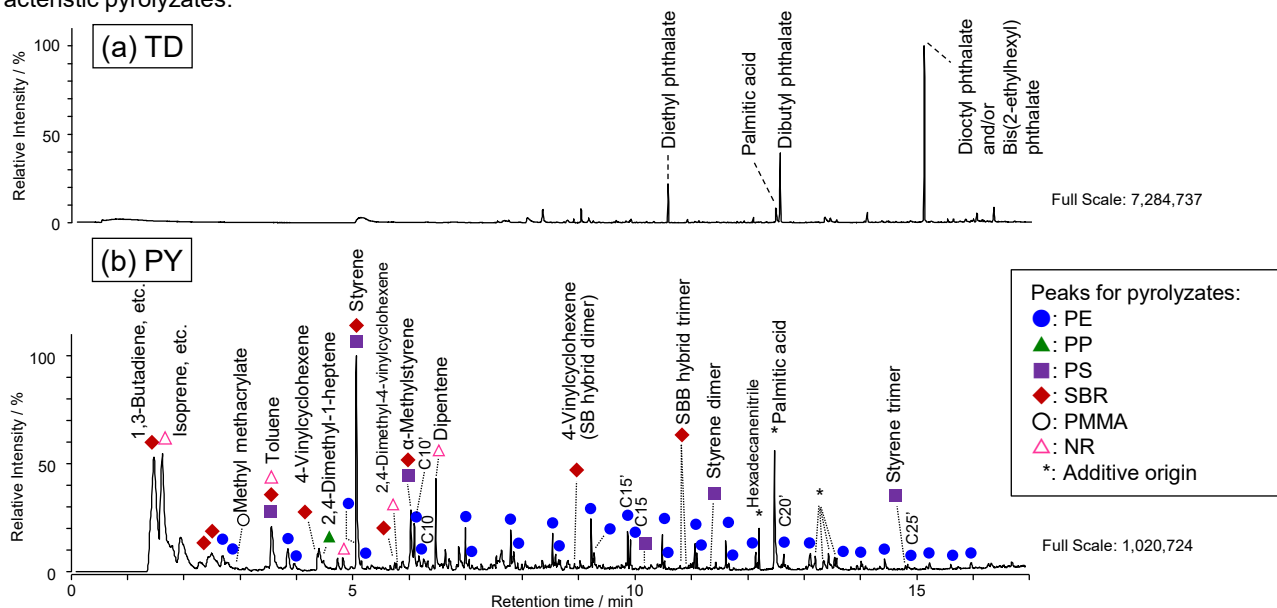


Fig. 1 (a) TD chromatogram and (b) Pyrogram obtained by double-shot TD/Py-GC/MS of >PM₁₀ fraction of PM-A sample

Furnace temp.(TD): 100-30 °C/min-300 °C(3 min), Furnace temp.(Py): 600 °C, Furnace-interface temp.: 300 °C, GC injector temp.: 300 °C, Column flow rate: 1.5 mL/min, Split ratio: 1/10, Separation column: UA⁺-5 (5 % diphenyl-95 % dimethylpolysiloxane; L=30 m, i.d.=0.25 mm, df=0.25 μ m), GC oven temp.: 40 (2 min hold)-20 °C/min-320 °C (5 min hold), GC/MS interface temp.: 250 °C, MS scan range: m/z 29-500, MS scan rate: ca. 3.3 scan/s

1) H. Mizuguchi et al., *J. Anal. Appl. Pyrolysis* 171 (2023) 105946.

Keywords : Environmental sample, Environmental pollutants, Airborne microplastic, MPs, Airborne particles, Particulate, Air sampler

Products used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, Eco-Cup LF, UA⁺-5, Packed GC glass insert, Vent-free GC/MS adapter, F-Search MPs

Applications : Environmental analysis, Trace analysis, General polymer analysis

Related technical notes : PYA3-050E (Part 1), PYA1-185E (Part 3)

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