

Preparation and evaluation of calibration standard for microplastic (MP) analysis using SiO₂ as a diluent

Part 1: Investigation of inorganic diluents for MP analysis

[Background] In recent years, demand for the analysis of microplastics (MPs) in the environment has increased. However, analytical methods and standard samples have not been established in this area. MPs in environmental samples generally contain multiple polymer species. Pyrolysis (Py)-GC/MS has the advantage of the ability of mass-based quantification for polymer mixtures in addition to qualitative analysis. Therefore, calibration standards were prepared for the analysis of environmental MPs by Py-GC/MS. An inorganic diluent in the calibration standard was used to reduce the weighing error of trace polymers, and the standard was prepared by homogeneous mixing of 12 types of polymers. In this note, six commonly used inorganic compounds were selected (to be used as diluents) in order to reduce weighing errors of trace polymers, and their effects on the pyrolysis of polymers were investigated.

[Experimental] To a sample cup containing 30 µg of polyurethane (PU), 2 mg of one of the six candidate inorganic compounds used as diluents (Fig. 2) was added and was subjected to Py-GC/MS measurement.

[Results] Fig. 1 shows the hydrolysis reaction of 4,4'-diphenylmethane diisocyanate (MDI) to form 4,4'-methylenedianiline (MDA), the major pyrolyzate of PU, and Fig. 2 shows the extracted ion chromatograms (EICs) for the ions of MDA and MDI. Without diluent, the MDI peak was clearly observed, and no MDA was detected. The MDI peak disappeared, and MDA was detected when α-Al₂O₃, γ-Al₂O₃, TiO₂, CaCO₃, or SiO₂ without deactivation treatment was present. These diluents seemed to adsorb and hydrolyze MDI. On the other hand, in the presence of deactivated SiO₂, the MDI peak was clearly visible as well as those without diluent, while no MDA was detected. It was also shown that deactivated SiO₂ had no effect on the pyrolysis of 11 polymers other than PU. In the next note (PYA1-144E), pyrograms of the MP calibration standard (MPs-SiO₂) with deactivated SiO₂ used as a diluent are presented.

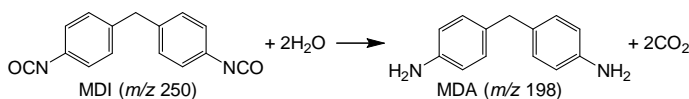


Fig. 1 Hydrolysis of MDI.

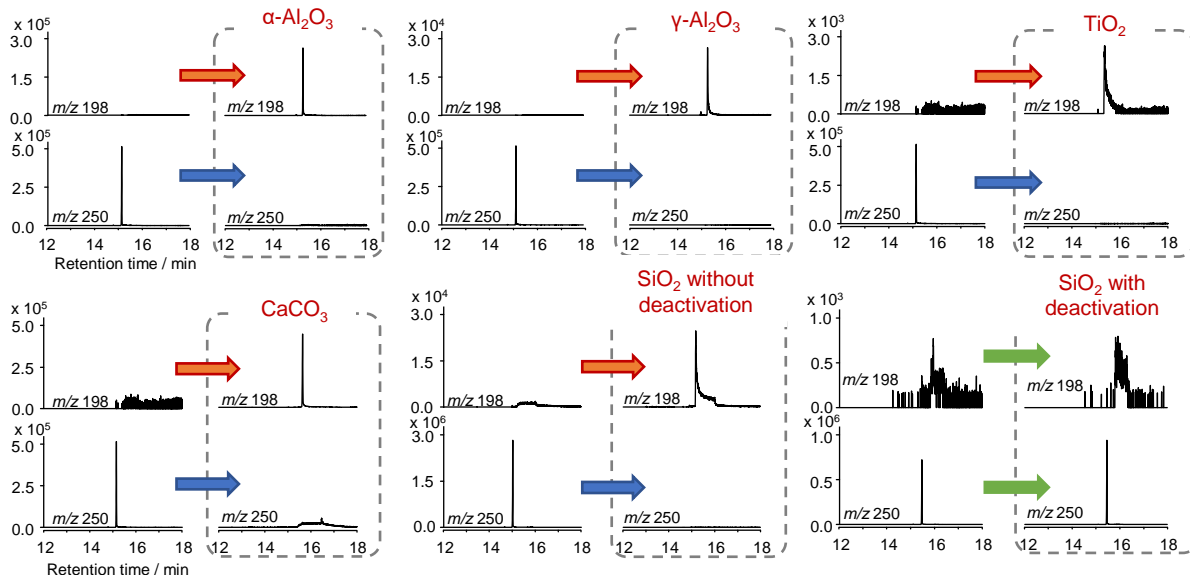


Fig. 2 EICs of mixtures of PU and inorganic compounds.

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Keywords : Microplastics, Calibration standard, Reference material, Diluent

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, Auto-Shot Sampler, Eco-Cup LF, Packed GC glass insert, UAMP column kit, Vent-free GC/MS adapter, F-Search MPs

Applications : Environmental Analysis, Trace analysis, General polymer analysis

Related technical notes : PYA1-144E (Part 2), PYA1-145E (Part 3), PYA1-146E, PYA1-147E, PYA1-148E (MPs-CaCO₃)

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