Preparation and evaluation of calibration standard for microplastic (MP) analysis using $CaCO_3$ as a diluent
Part 1: Reactions of polymer pyrolyzates in the presence of $CaCO_3$ as a catalyst

[Background] Previous notes (PYA1-143E through PYA1-145E) reported the characterization of a calibration standard for microplastic (MP) analysis. The standard is called MPs-SiO2 which is a homogeneous mixture composed of 12 types of polymers and diluent SiO_2 powders. However, with MPs-SiO2, MDI, a pyrolyzate of PU, reacts with pyrolyzates of PET and nylon, and this causes MDI peak to disappear. For stable detection of pyrolyzates of the 12 polymers including PU, the effect of $CaCO_3$ used as both a diluent and a catalyst on the reactions of pyrolyzates was investigated, and the MP analysis was examined by employing the MP calibration standard (MPs-CaCO3) by which reactive pyrolyzates are converted to stable compounds.[1,2] In this report, the reactions of pyrolyzates of PET, PC, and PU using $CaCO_3$ as a catalyst in the determination of characteristic pyrolyzates for the analysis of the MP calibration standard are described.

[Experimental] PET (16 µg), PC (2 µg), and PU (30 µg) with and without $CaCO_3$ (4 mg) were each put in a sample cup (Eco-Cup LF) and subjected to Py-GC/MS measurements.

[Results] Fig. 1 shows pyrograms of PET, PC, and PU, and the reaction schemes for polymer pyrolysis and pyrolyzates catalyzed by $CaCO_3$. (a) PET: benzoic acid (BA) is converted to benzophenone (BP). (b) PC: the pyrolysis of bisphenol A (BPA), the main product, is accelerated and the amount of 4-isopropenylphenol (IPP) formed is increased. (c) PU: through the hydrolysis of 4,4'-diphenylmethane diisocyanate (MDI), 4,4'-methylenedianiline (MDA) is formed.

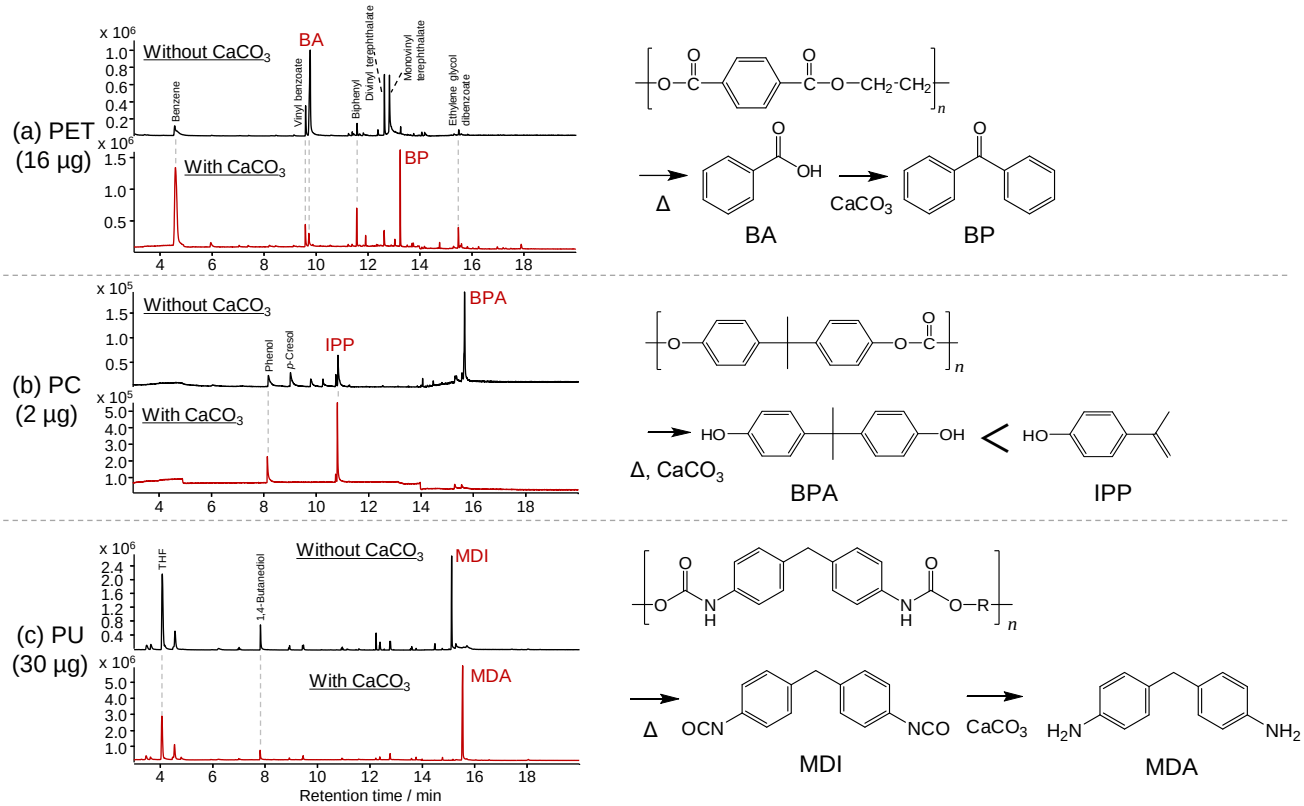

Fig. 1 Pyrograms of PET, PC, and PU (left) and reaction schemes of pyrolyzates catalyzed by $CaCO_3$ (right).

1) R. A. Hites and K. Biemann, *J. Am. Chem. Soc.* 94 (1972) 16, 5772–5777.
2) T. Ishimura *et al.*, *J. Anal. Appl. Pyrolysis* 157 (2021) 105188.

Keywords : Microplastic, Calibration standard material, Reference material, Diluent

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, Auto-Shot Sampler, Eco-Cup LF, GC glass insert with filler, UAMP column kit, Vent-free GC/MS adapter, F-Search MPs 2.0

Applications : Environmental analysis, Trace analysis, General polymer analysis

Related technical notes : PYA1-147E (Part 2), PYA1-148E (Part 3), PYA1-143E, PYA1-144E, PYA1-145E (MPs-SiO2)

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