

Determination of volatile phthalate esters using polymer coated sample cups - Part 2 Effect of other polymer coatings

[Background] One of the challenges when analyzing volatile phthalate esters like dimethyl phthalate (DMP) and diethyl phthalate (DEP) is generating data with acceptable reproducibility. Losses due to evaporation prior to analysis result in an inaccurate and imprecise determination. In a previous Technical Note: Part 1 (PYA1-075E), we reported that losses due to evaporation could be mitigated by using glass sample cups whose inner wall was coated with PVC polymer. In this report, we extend our PVC findings to polystyrene (PS) and poly (methyl methacrylate (PMMA).

[Experimental] Glass sample cups (od. 4 mm, id. 3 mm, height 8 mm) were used in this series of tests. Prior to use, the inside surface of the cup was coated with a thin film (2.4 μ m) of plasticizer-free polyvinyl chloride (PVC), polystyrene (PS), or polymethyl methacrylate (PMMA). The coated cups were conditioned in air at 200°C for 60 min. Each cup was spiked with 10 μ L of a dichloromethane solution containing 15 phthalate esters, each at a concentration of 10 ppm. After 200 min. the cup was analyzed using the TD-GC/MS ASTM phthalate method: ASTM D7823. The analytical system was a Multi-Shot Pyrolyzer (EGA/PY-3030D, Frontier Labs.) directly interfaced to the split injector of a GC/MS system.

[Results] Fig. 1 shows the chromatograms of the phthalate analysis using glass sample cups coated with three different polymers. When a polymer-coated, glass sample cup was used, each of the phthalate esters was quantitatively retained. The reproducibility (expressed as the relative standard deviation (n=5) of the peak areas) was 2% which is consistent with values cited in the ASTM method Just as important is that none of the impurities, oligomers or additives associated with these three polymers interfere with the phthalate determinations.

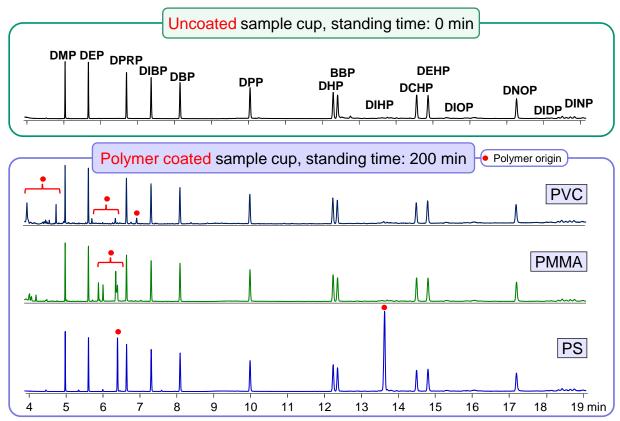


Fig.1 Comparison of uncoated and polymer-coated sample cups in terms of interferences from polymer pyrolyzates (coating thickness: 2.4 µm, standing time: 200 min)

Keywords: Phthalate esters determination, DMP, DEP, TD-GC/MS, Polymer coated sample cups, PVC, PS, PMMA

Products used: Multi-functional pyrolyzer, Auto-Shot Sampler, Vent-free GC/MS adapter, UA+-5

Applications: General polymer analysis, Analysis of additives in toys

Related technical notes: PYA1-063E, PYA1-064E, PYA1-068E, PYA1-069E, PYA1-074E, PYA1-075E

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