

Development of UA guard column for UA-PBDE column for phthalates analysis Part 3: Quantitative analysis

[Background] Guard columns are widely used whenever samples are known to have large amounts of high boiling compounds. Part 2 of this series (UAT-009E) describes the effectiveness of the UA guard column which keeps the separation efficiency of the UA-PBDE column, with using the UA connector. This note demonstrates that the presence of the guard column and the connector to the separation column does not adversely affect the accuracy or the reproducibility of the data. Examination of the system performance will also provide the analyst with an indication of when to replace the guard column.

[Experimental] 400 samples (0.5 mg each) of powdered poly(vinyl chloride) (PVC) were successively analyzed using thermal desorption-GC/MS (ASTM D7823-018). To examine the effect of the guard column on the UA-PBDE separation efficiency, 0.5 mg polystyrene (PS) sample in dichloromethane containing 500 ppm each of the seven phthalates*¹ specified in IEC62321-8*² was analyzed as a test sample. Quantitative value and reproducibility for the PS sample were determined in three stages, i.e., before the 1st run, after 200 runs and after 400 runs of the PVC samples. Experiments were done repeatedly (n=3).

[Results] The quantitative value (ppm) and its reproducibility (%RSD) vs. the number of assays with and without the guard column are shown in Fig. 1. Without guard column, the quantitative value of BBP decreased from 500 to 400 ppm after 400 runs of the PVC samples (0.5 mg each). The reproducibility also got worse by more than 20 %RSD. BBP is known to decompose when in contact with hot, contaminated surfaces, like that often found in a hot guard column or transfer line. On the other hand, when the guard column was placed between the injection port and the analytical column, no significant change in the quantitative values, even after 400 runs were observed. The reproducibility of BBP was only slightly affected. In practical use, good reproducibility and quantitative values can continuously be obtained by appropriately replacing the guard column with a new one when necessary.

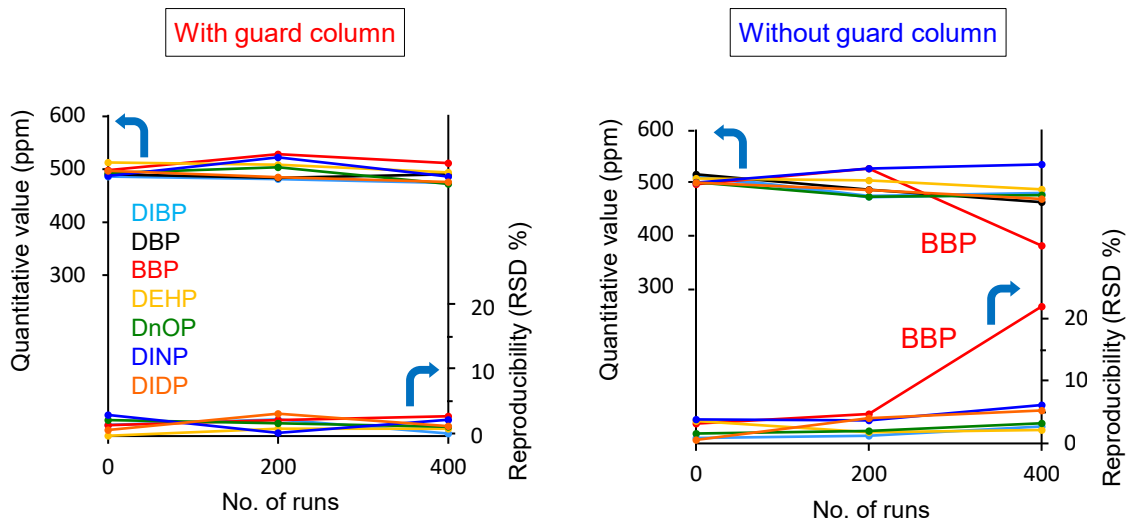


Fig. 1 Quantitative values and reproducibility of phthalates after repetitive analysis of 0.5 mg of PVC

Thermal desorption temp.: 200 °C - 340 °C (20 °C/min, 1 min hold), Split ratio: 1/50, GC oven temp.: 80 °C - 300 °C (20 °C/min, 5 min hold), Separation column: UA-PBDE (polydimethylsiloxane), L=15 m, i.d.=0.25 mm, df=0.05 µm, UA guard column: L=1.5 m, i.d.=0.25 mm, df=0.1 µm, Column flow rate: 1.2 mL/min, MS scan range: m/z 50 - 1000, Sample amount: 0.5 mg of polystyrene containing 500 ppm each of seven phthalates

*1 Di-isobutyl phthalate (DIBP), Di-n-butyl phthalate (DBP), Benzyl butyl phthalate (BBP), Di(2-ethylhexyl)phthalate (DEHP), Di-n-octyl phthalate (DnOP), Di-isononyl phthalate (DINP), Di-isodecyl phthalate (DIDP)

*2 IEC62321-8, Determination of certain substance in electrotechnical products-Part 8: Phthalates in polymers by gas chromatography-mass spectrometry (GC-MS), gas chromatography-mass spectrometry using a pyrolyzer/thermal desorption accessory (Py/TD-GC-MS)

Keywords : Phthalates, Guard column replacement timing, connector, PBDE column

Product used : Multi-Shot Pyrolyzer, UA-PBDE, UA connector, UA guard column Ph

Applications : Polymer analysis, Quality control

Related technical notes : UAT-008E (Part 1), UAT-009E (Part 2)

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