

Development of UA guard column for UA-PBDE column for phthalates analysis Part 2: Effectiveness of UA guard column

[Background] The development and the structure of the UA connector and the UA guard column used for UA-PBDE column are described in Part 1 (UAT-008E). This note describes the effectiveness of the UA guard column for the multi-sample analysis of polyvinyl chloride (PVC).

[Experimental] The effectiveness of the UA guard column was evaluated by comparing the results obtained with and without the UA guard column connected to the UA-PBDE column. For the measurement, 1 µL of a dichloromethane solution containing 500 ppm each of the seven phthalates*¹ specified in IEC62321-8*² was placed in a sample cup and analyzed after the solvent was evaporated. For the evaluation of the guard column, the phthalates mixture samples were analyzed by thermal desorption (TD)-GC/MS before and after 400 successive runs of 0.5 mg of powdered PVC, after subsequent 200 successive runs of 5.0 mg of the PVC, and finally after removal of the guard column (UA-PBDE column alone).

[Results] After 400 runs of 0.5 mg of PVC, with the guard column installed, the chromatogram stayed unchanged as shown in Fig. 1. On the other hand, without the guard column the peak area of BBP decreased and the peak shape became abnormal regardless of the presence or absence of the guard column. After 200 successive analysis of 5.0 mg of PVC, the peak shape became abnormal (tailing peaks) regardless of the presence or absence of the guard column; however, when the guard column was removed, the chromatogram obtained with the separation column alone was equivalent to that obtained before the tests. This clearly shows that the performance of the separation column was fully protected by the guard column. The effects on the quantitative values are described in UAT-010E (Part 3).

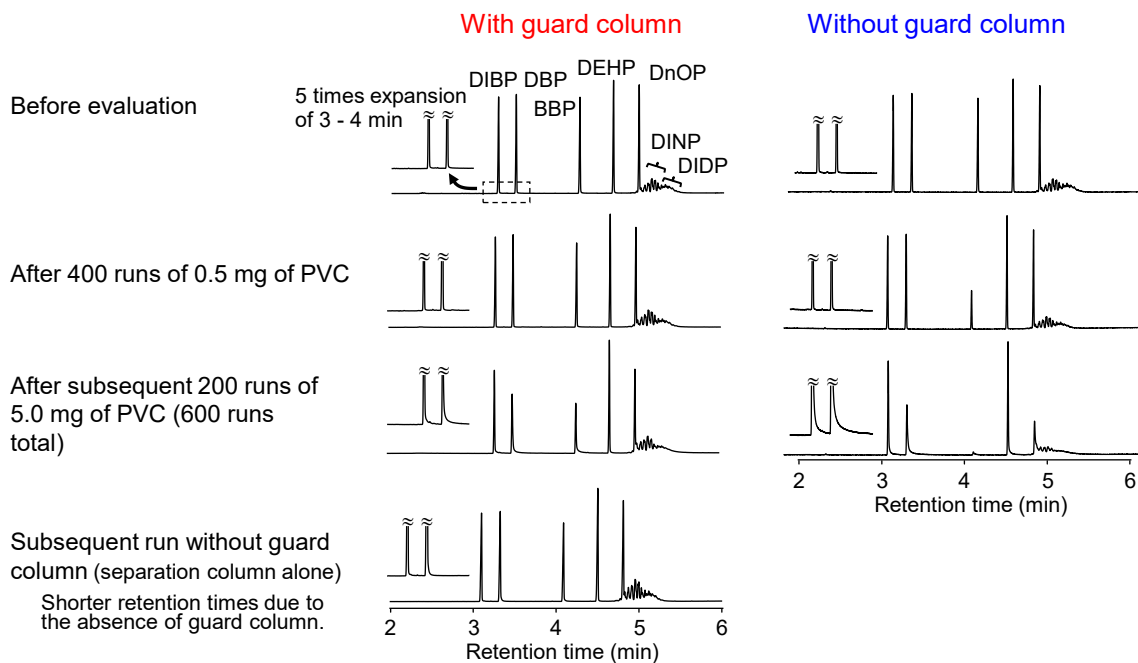


Fig. 1 TD-GC/MS chromatograms of phthalates with and without guard column

Thermal desorption temp.: 200 °C - 340 °C (20 °C/min, 1 min hold), Split ratio: 1/50, GC oven temp.: 80 °C - 300 °C (40 °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 µg each of the 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, Effectiveness of guard column, Connector, PBDE column

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

Applications : Polymer analysis, Quality control

Related technical notes : UAT-008E (Part 1), UAT-010E (Part 3)

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