

Analysis of phthalates in PVC by thermal desorption GC/MS

Part 2: Effect of sample form on reproducibility

[Background] It has been reported (PYT-018) that the reproducibility depends on the sample form (fine power/thin film). This technical note illustrates how sample form influences reproducibility when performing TD-GC/MS.

[Experimental] The TD-GC/MS system consists of a double-shot pyrolyzer interfaced directly to the split/splitless injection port of a GC/MS. Solid samples were milled to 45 mesh, and thin films were prepared by solvent casting (adding a liquid to the sample cup and then evaporating the solvent as described in technical note PYA1-063E). The thermal desorption for the phthalates of interest was 100-350°C, as described in PYA1-063E. The concentration of the phthalates in the sample were calculated using an absolute area calibration.

[Results] Fig. 1 shows a TIC chromatogram obtained by TD-GC/MS. DINCH, a non-phthalate plasticizer, was identified as the major component in the 17-18 minute retention window. Compounds having fragment ions m/z 149, 223, 279 were found at 11 and 16 min. Based on the mass spectra and retention times, these peaks are identified as DBP and DEHP. The concentration of the two phthalates is ca. 50 and ca. 300 ppm, respectively. The reproducibility ($n=5$) of the DEHP concentration is 5% RSD for the powder, and 1% for the thin film. The difference between the two can be attributed to the lack of homogeneity of the solid sample. This simple example clearly shows that TD-GC/MS is a viable technique and that a thin films yield the best analytical precision (i.e., reproducibility).

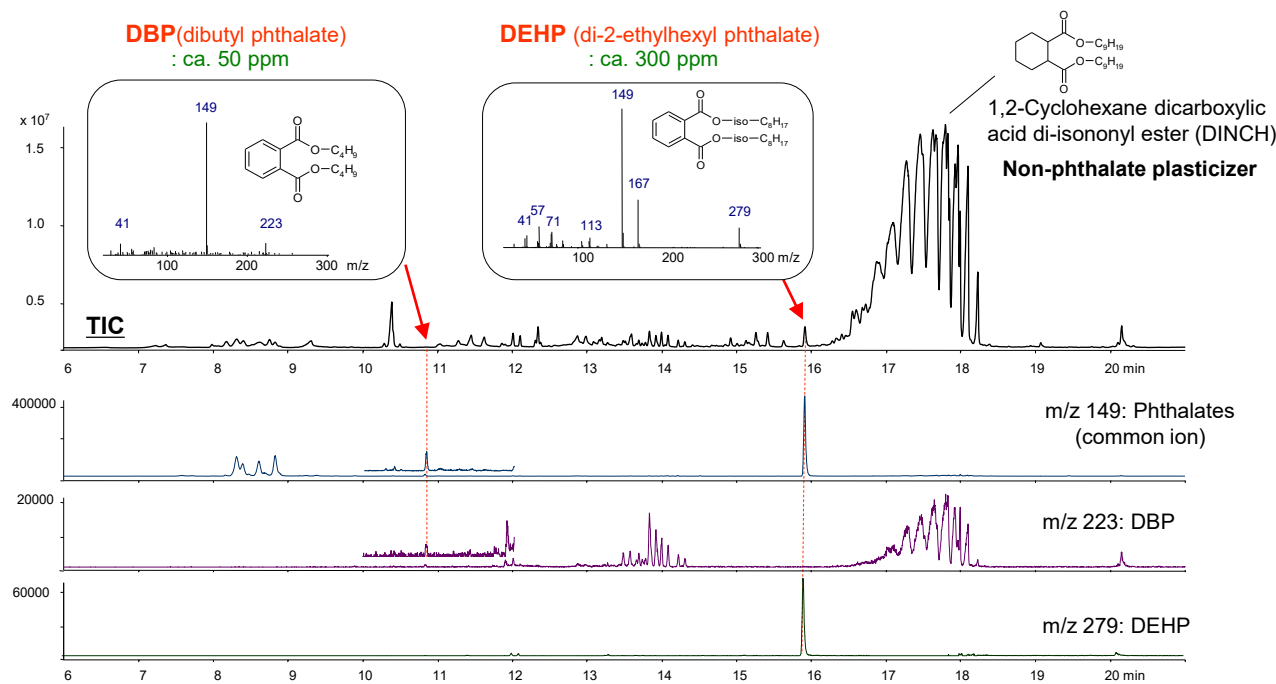


Fig. 1 TIC and extracted ion chromatograms of a PVC sample obtained by TD-GC/MS

Thermal desorption temp: 100 - 350°C (40 °C/min, 3 min), GC oven temp: 80 - 320°C (10 °C/min, 6 min), Separation column*: Ultra ALLOY-1 (polydimethylsiloxane, $L=30$ m, i.d.= 0.25 mm, $df=0.05$ μ m), Column flow rate: 1 mL/min He, Split ratio: 1/20

* For separation column, please use Ultra ALLOY-5 (5% diphenyl 95% dimethylpolysiloxane, $L=30$ m, i.d.=0.25 mm, $df=0.25$ μ m).

Ref: T. Yuzawa, *et al.*, *Anal. Sci.*, **25** (2009),1057

Keywords : Phthalates, Simple TD-GC/MS quantitative analysis, Reproducibility, Sample form (power, thin film) DBP, DEHP, Toy

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

Applications : Additive analysis, Simple quantitative analysis, Sample form

Related technical notes : PYA1-063E, PYT-018E

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