

Quantitative analysis of phthalate Bis(2 - Ethylhexyl) Phthalate (DEHP) in heat resistant PVC sheath using ASTM D7823-14 (thermal desorption-GC/MS)

[Background] Phthalate esters are added to PVC formulations to improve the processability and the performance of the PVC. Recently many of these phthalates have been found to adversely affect human health and their use has been restricted or in some cases banned. Bis(2-ethylhexyl) phthalate (DEHP) is among the first six compounds that the EU is phasing out under its REACH program. There are more than 17 analytical methods currently being used to measure DEHP in PVC. With two exceptions, the analysis begins with a cumbersome, time consuming liquid/ liquid extraction. This work uses a new ASTM Method: D-7823-14 which utilizes thermal desorption (TD)-GC/MS. The method is simple to use and has proven to yield superior precision and accuracy values when compared to those obtained using solvent extraction. This note describes the quantitative TD-GC/MS analysis of DEHP in the outside sheath of commercial-grade cable.

[Experimental] TD-GC/MS system consisted of a Multi-Shot pyrolyzer (EGA/PY-3030D Frontier Labs) interfaced directly to a GC/MS split injection port. 500 µg of a PVC thin film was placed in an inert SS sample cup. The TD was done as described in the ASTM method. The concentration of DEHP was calculated using an absolute area calibration curve method (*m/z*: 279). The ASTM method requires that quantization be done using standard addition because of the possible interference from other plasticizers (e.g., DINCH) in the PVC. In this case the plasticizers were trimellitate and adipates and there was no interference with the DEHP ions.

[Results] A typical total ion chromatogram (TIC) and extracted ion chromatograms (EICs) of the PVC sample are shown in Fig.1. *m/z*: 149 and 279 are used to confirm the presence of DEHP. Two positional isomers of DEHP (regioisomers), tris(2-ethylhexyl) trimellitate (TOTM) and TOTM anhydride are also present. Quantification was performed using the calibration curve shown in Fig.2. The concentration of DEHP in this PVC sample was found to be 245 ppm (n=3, RSD=0.7%).

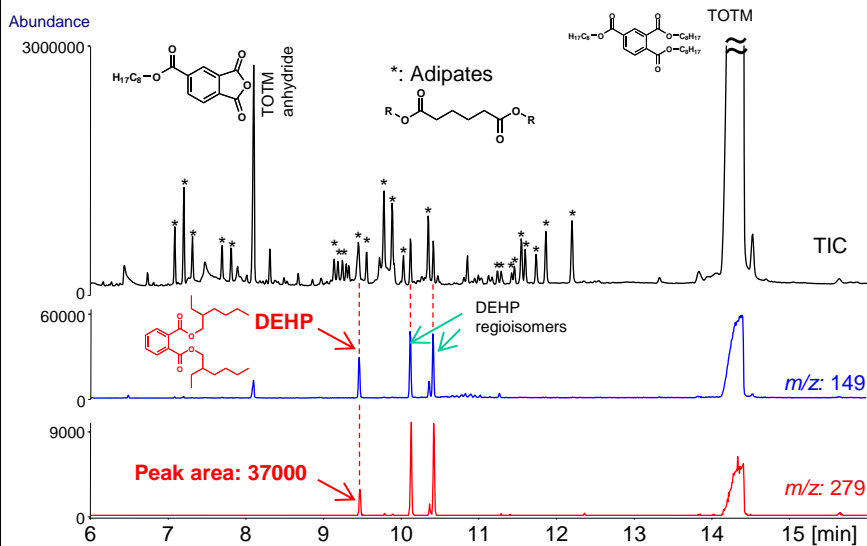


Fig.1 TD-GC/MS chromatogram and extracted ion chromatograms of heat resistant PVC

TD temp.: 100 - 320°C (20 °C/min, 1 min hold), GC oven temp.: 80 (2 min) – 200°C (40 °C/min) – 320°C (15 °C/min, 3 min hold)
 Separation column: Ultra ALLOY+5 (5 % diphenyl 95 % dimethylpolysiloxane, L=30 m, i.d.=0.25 mm, df=0.25 µm)
 Column flow rate: 1.2 mL/min He, Split ratio: 1/50, Sample wt: ca. 500 µg

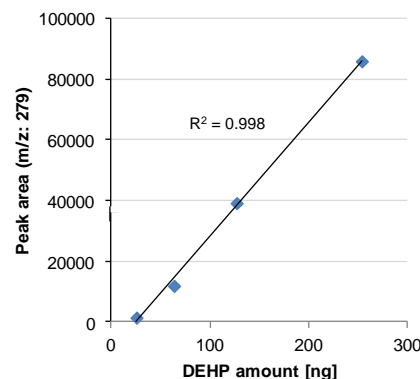


Fig.2 Calibration curve of DEHP

Ref.:
 "A fast, easy and 'green' thermal desorption-GC/MS method for the analysis of phthalate esters in PVC", D. Randle, R. Freeman, A. Hosaka and C. Watanabe, Frontier Technical Brief.
 "Rapid and simple determination of phthalates in plastic toys by a thermal desorption-GC/MS method", T. Yuzawa, C. Watanabe, R.R. Freeman, S. Tsuge, *Anal.Sci.* 25 (2009) 1057–1058.

Keywords : Restricted phthalates, DEHP, plasticizer, additive, quantitative analysis, thermal desorption

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

Applications : Restricted phthalates, plasticizer

Related technical notes : PYA1-063E, PYA1-064E, PYA1-074E

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