

Analysis of phthalates in PVC by thermal desorption GC/MS

Part 1: Determination of thermal desorption temperature zone by EGA

[Background] Phthalates are widely used as plasticizers in the plastic industry to enhance product durability. It has been determined that many of the phthalates have a detrimental effect on human health. The phthalates listed in Table 1 are now regulated by the EU (Directive 2005/84/EC) and the US (Consumer Product Safety Improvement Act, Section 108). All the current methodologies for the determination of these six phthalates are based on solvent extraction, filtration and concentration. These methods are cumbersome, time-consuming and often produce data of limited value. This note describes how evolved gas analysis (EGA)-MS is used to determine the thermal zone in which the target compounds evolve as the sample is heated.

[Experimental] The conditions for the EGA-MS analysis are found in the figure below. The Sample consisted of small pieces of a PVC toy taken from ten different locations using a Harris Micropuncher (2 mm in diameter). They were dissolved in 1 mL of THF (50 mg/mL). Six different phthalates were added to the THF solution. 10µL of the THF spiked solution was added to a sample cup; the solvent was evaporated leaving a thin film of the sample on the surface of the cup (ca. 0.5mg).

[Results] Fig. 1, Extracted ion chromatograms (EICs) are used to determine the thermal zone for each of the phthalates and the hydrogen chloride that is generated as PVC is heated. Using average mass spectrum, the first peak is identified as DINCH (1,2 cyclohexane dicarboxylic acid di-isononyl ester). It is critical to ensure that the ions used to monitor the six phthalates can be differentiated from ions produced by non-target additives, like DINCH. The EICs clearly show that the temperature range in which all six phthalates are thermally desorbed is 100 to 350°C. (The quantitative determination of phthalates in the toy sample by TD-GC/MS is described in Technical Note PYA1-064E.)

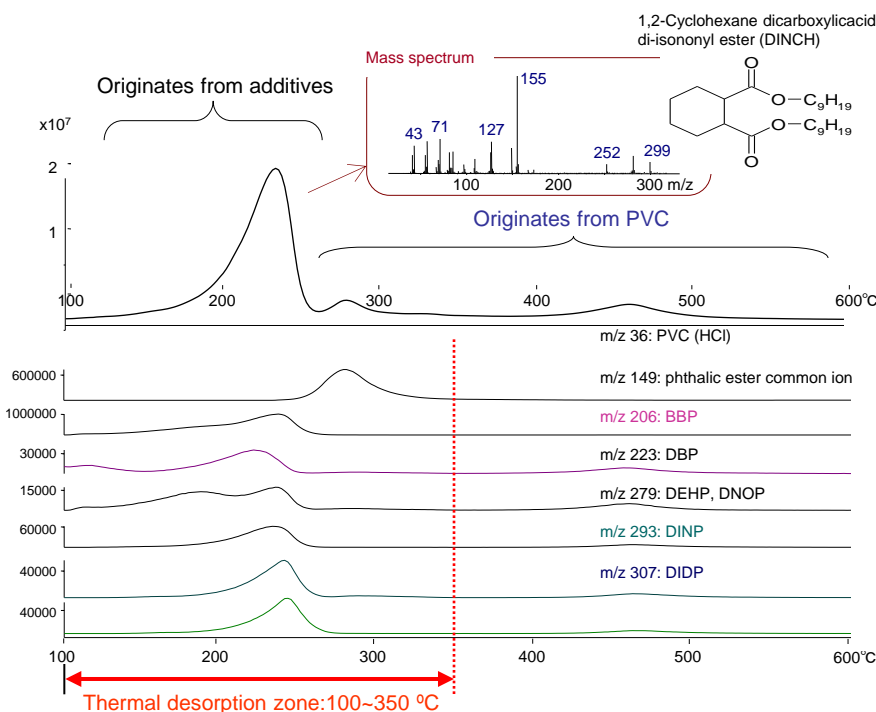


Table 1. Use restricted phthalates (0.1% upper limit by Directive 2005/84/EC)

Di(2-ethylhexyl)phthalate (DEHP)
Dibutylphthalate (DBP)
Butylbenzylphthalate (BBP)
Diisononylphthalate (DINP)
Diisodecylphthalate (DIDP)
Di(n-octyl)phthalate (DNOP)

Fig. 1 TIC thermogram and extracted mass chromatograms of PVC, to which 1% of each phthalate was added

Pyrolyzer: 100 – 600°C (20°C/min), GC oven temp.: 300°C, EGA tube: UADTM-2.5N (L=2.5 m, i.d.=0.15 mm), Column flow rate: 1 mL/min; He, Split ratio: 1/20, Inj. port temp.: 320°C

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Keywords : Restricted phthalates, plastic toy, thermal desorption temperature, EGA, thermogram

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

Applications : Restricted phthalates, plasticizer

Related technical notes : PYA1-064E

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