

Determination of phthalate in edible oil by thermal desorption GC/MS

Part 1: Thermal desorption temperature of DEHP in evolved gas analysis (EGA)-MS

[Background] Phthalates are plasticizers contained in many plastic products such as polyvinyl chloride (PVC), and their use has been regulated due to their hazardous effects on human health. Bis(2-ethylhexyl) phthalate (DEHP) is easily contaminated in food manufacturing processes and storage containers. The EU regulates DEHP in fatty foods to be less than 1.5 ppm. In this report, the elution temperatures of DEHP in olive oil were examined by varying the sample volume as a first step for the quantitative analysis of DEHP in olive oil by evolved gas analysis (EGA)-MS.

[Experimental] A Multi-Shot Pyrolyzer was directly interfaced to the GC injector of a GC/MS system, and an EGA tube and a vent-free GC/MS adapter were used to connect the GC injector to the MS detector. Commercially available olive oil was used as a sample, and 1 % each of DEHP was added to 1 μ L (0.92 mg) to 10 μ L (9.2 mg) of olive oil and placed in a sample cup with an inner diameter of 3 mm.

[Result] In the EGA curves of olive oil (Fig. 1), the elution temperatures of triglycerides, the main components of olive oil, and DEHP, were able to be observed. From the TIC, triglycerides begin to volatilize around 350 $^{\circ}$ C, and from the EIC (m/z 149), DEHP started to elute from 200 to 380 $^{\circ}$ C. The elution temperature of DEHP shifted to higher temperatures with increased sample volume. This may be due to the lowered thermal conductivity resulting from a decrease in surface area against sample volume as the sample volume increases (Fig. 2). In the next report (PYA3-044E), the thermal desorption temperature is optimized by EGA-MS.

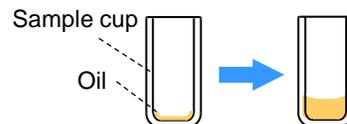


Fig. 2 Change in the ratio of surface area to sample volume (image).

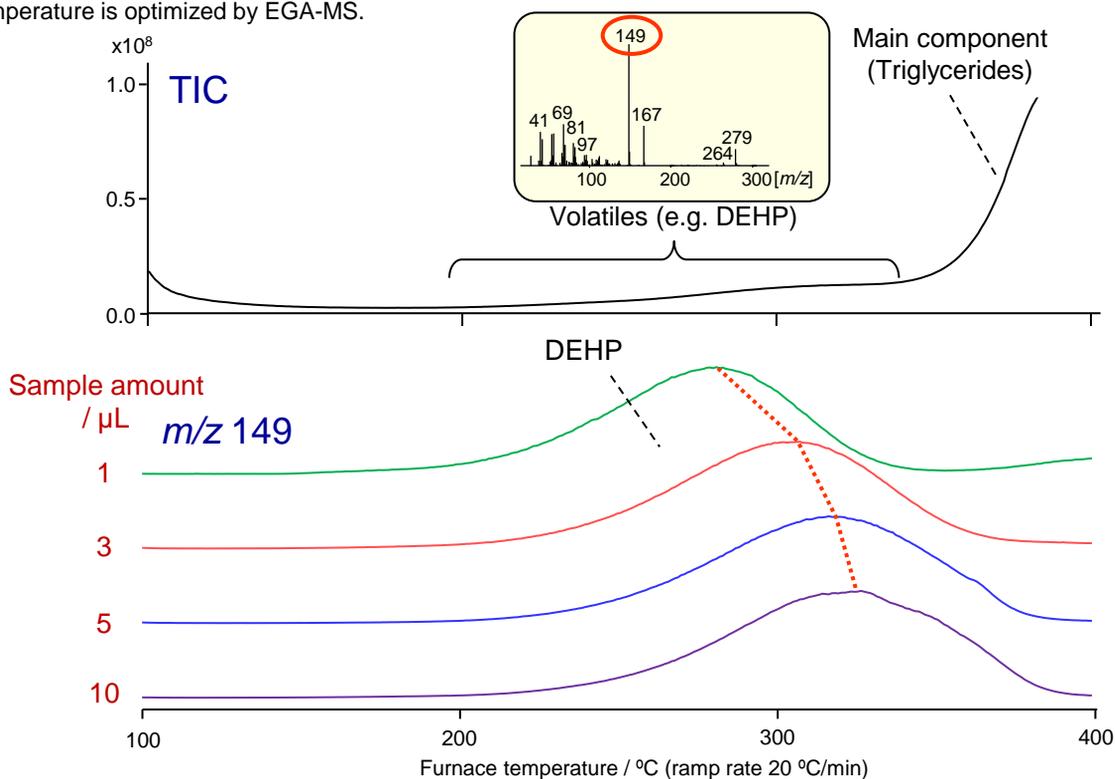


Fig. 1 EGA thermograms of olive oil (contains 1 % DEHP).

Furnace temp.: 100 - 400 $^{\circ}$ C (20 $^{\circ}$ C/min), Furnace-interface temp.: 300 $^{\circ}$ C, GC injector temp.: 300 $^{\circ}$ C, EGA tube: UADTM-2.5N (L=15 m, i.d.=0.15 mm), Column flow: 1 mL/min (He), Split ratio: 1/20, GC oven: 300 $^{\circ}$ C, MS ion source temp.: 230 $^{\circ}$ C, Sample amount: 1 μ L (0.92 mg) - 10 μ L (9.2 mg).

Keywords : Fats and oils, Edible oils, Phthalates, Trace analysis, Regulated substances

Products used : Multi-Shot Pyrolyzer, UADTM-2.5N, Eco-Cup G, Vent-free GC/MS adapter

Applications : Food analysis, Fat and oil analysis, Natural organics analysis, Additives analysis

Related technical notes : [PYA3-044E \(Part 2\)](#), [PYA1-158E \(Part 3\)](#)

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