

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

Part 3: Determination of DEHP using thermal desorption GC/MS

[Background] In the previous report (PYA3-044E), the optimization of the thermal desorption (TD) conditions for quantification of DEHP in olive oil was reported. This report builds upon the previous study by implementing the optimized conditions for the determination of DEHP in olive oil using TD-GC/MS.

[Experimental] A GC/MS system with a Multi-Shot Pyrolyzer directly interfaced to the GC injector was used. A separation column UA+-5 and a vent-free GC/MS adapter were used to connect the GC injector to the MS detector. The sample was commercially available olive oil, and 3 μ L (2.76 mg) of it was put in a sample cup and introduced into the pyrolyzer furnace for TD-GC/MS measurements. The furnace temperature was programmed as 100 - (60 $^{\circ}$ C/min) - 280 $^{\circ}$ C (10 min hold). The MS was operated in the SIM mode (ions: m/z 149, 279).

[Result] TD chromatograms of olive oil obtained using the SIM mode is shown in Fig. 1. The peak for DEHP was found to be well-resolved without interferences from other components. The DEHP concentration in the olive oil was determined to be 0.379 ppm by the absolute calibration method and 0.367 ppm by the standard addition method. The detection limit was 0.064 ppm, corresponding to 1/20 of the regulatory limit of 1.5 ppm. These results demonstrate that TD-GC/MS was quite useful to determine the DEHP concentration in edible oils.

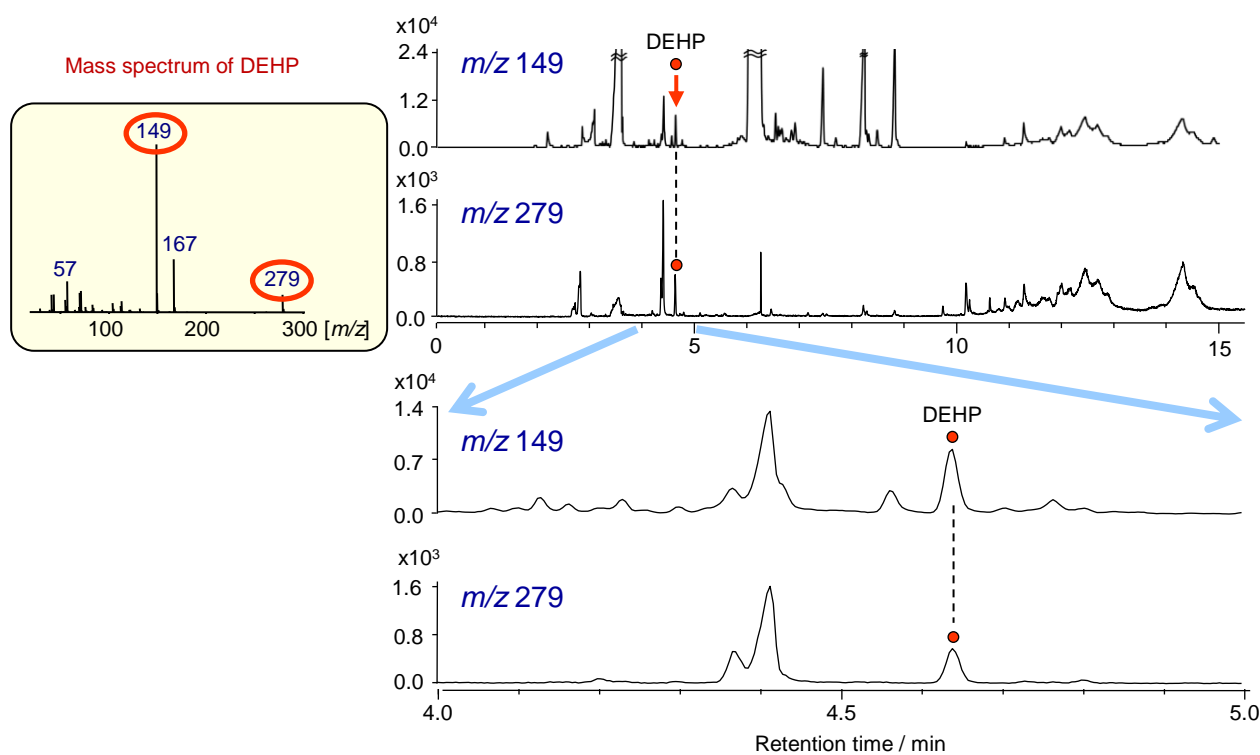


Fig. 1 TD chromatograms of olive oil obtained in the SIM mode.

Furnace temp.: 100 - 60 $^{\circ}$ C/min - 280 $^{\circ}$ C (10 min hold), Furnace-GC interface temp.: 300 $^{\circ}$ C, GC injector temp.: 300 $^{\circ}$ C,
 Separation column: UA+-5 (L=15 m, i.d.=0.25 mm, df=0.25 μ m),
 GC oven temp.: 80 $^{\circ}$ C - 40 $^{\circ}$ C/min - 200 $^{\circ}$ C - 10 $^{\circ}$ C/min - 260 $^{\circ}$ C - 40 $^{\circ}$ C/min - 320 $^{\circ}$ C (5 min hold),
 Column flow rate: 1.0 mL/min (He), Split ratio: 1/20, Sample amount: 3 μ L (2.76 mg).

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

Products used : Multi-Shot Pyrolyzer, UA+-5, Eco-Cup G, Vent-free GC/MS adapter

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

Related technical notes : [PYA1-069E](#), [PYA3-043E \(Part 1\)](#), [PYA3-044E \(Part 2\)](#)

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