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

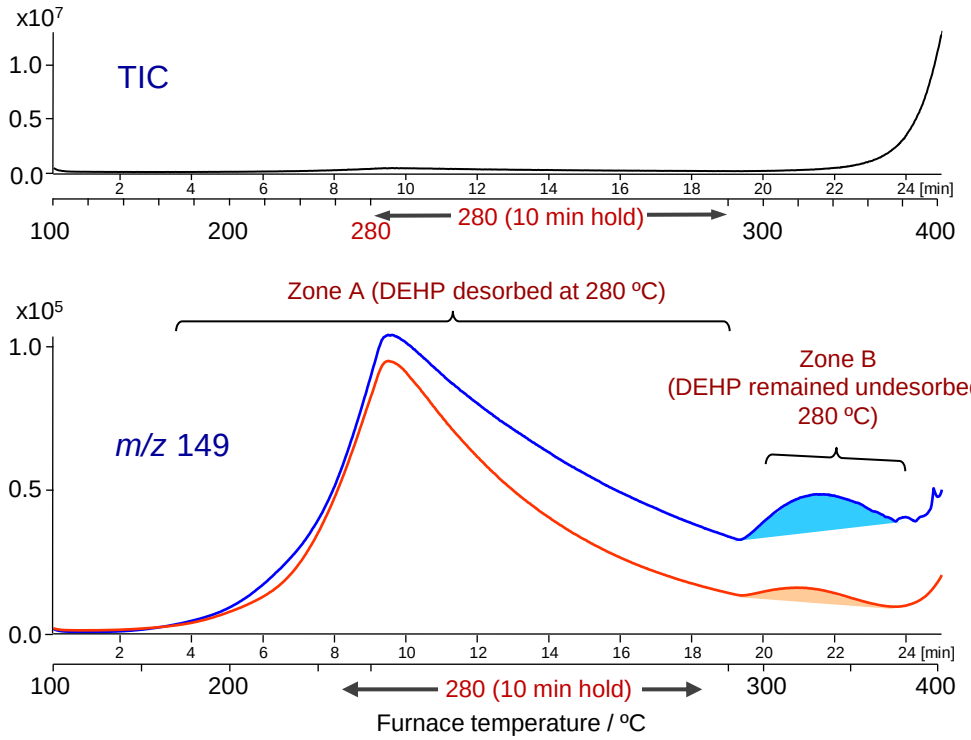
Part 2: Optimization of analytical conditions for thermal desorption GC/MS

[Background] In the previous report (PYA3-043E), the elution temperature of DEHP was examined by varying the sample volume in the range of 1 - 10 µL as a fundamental investigation using evolved gas analysis (EGA)-MS for the determination of DEHP in olive oil. However, when employing TD-GC/MS in SIM mode (*m/z* 149, *m/z* 279), the DEHP peak remained elusive, primarily due to interferences caused by the presence of fat and oil components. To achieve improved separation and effective detection of DEHP, it is necessary to minimize the introduction of interfering components into the separation column. This was achieved by reducing the sample volume and lowering the thermal desorption temperature. In this report, efforts made to optimize the thermal desorption conditions for the detection of DEHP using EGA-MS are detailed.

[Methods] A GC/MS system with a Multi-Shot Pyrolyzer directly interfaced to the GC injector was used. An EGA tube and a vent-free GC/MS adapter were used to connect the GC injector to the MS detector. 3 µL (2.76 mg) or 5 µL (4.6 mg) of commercially available olive oil each spiked with 1 % DEHP was used as a sample for measurements.

[Results] Fig. 1 shows the EGA curve obtained in the programmed temperature mode with a 10 min temperature hold at 280 ºC. The EIC of the ion (*m/z* 149) characteristic to DEHP shows two peaks, most of DEHP desorbs at 280 ºC in Zone A and the DEHP remained in the sample, which was undesorbed at 280 ºC, desorbs at 330 ºC in Zone B. The peak area of DEHP in Zone B relative to the sum of the peak areas of DEHP in Zones A and B was calculated and the percentage of the remained DEHP was calculated and the results are shown in the figure.

By keeping the thermal desorption temperature at 280 ºC for 10 min, over 90% of DEHP was thermally desorbed, while effectively mitigating the volatilization of fat and oil components irrespective of sample volumes. As a result, with a sample volume of 3 µL, over 97% of DEHP was thermally desorbed. Consequently, 3 µL of sample volume and the thermal desorption at 280 ºC with 10-min hold were established as the optimal conditions.



Sample volume	Residual percentage
5 µL	6.5 %
3 µL	2.3 %

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

Sample: Olive oil (3 or 5 µL, 1 % DEHP), Furnace temp.: 100 - 280 ºC (20 ºC/min, 10 min hold) - 400 ºC (20 ºC/min),
GC injector temp.: 300 ºC, EGA tube: UADTM-2.5N (*L*=15 m, i.d.=0.15 mm), Column flow rate: 1 mL/min (He), Split ratio: 1/20,
MS mode: Scan/SIM simultaneous measurement.

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

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 : PYA3-043E (Part 1), PYA1-158E (Part 3)

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