Phthalates analysis of bottled water using hollow fiber solid phase microextraction / thermal desorption gas chromatography

[Background] Phthalates (PAEs) and their metabolic and degradation products are considered endocrine disruptors that damage human genes, cause teratogenesis, affect fertility such as precocious puberty, and increase risk for cardiac and other diseases. Because PAEs are not covalently bonded to the polymer chain, they can be easily released from food packaging materials and transferred to water or foods in direct contact with the packages. This note describes the analysis of PAEs in bottled water using hollow fiber solid phase microextraction and thermal desorption gas chromatography (TD-GC).

[Experimental] 10 bottled water samples were commercially obtained.

Hollow fiber solid phase microextraction: a polysulfone (PSF) hollow fiber (*L*=1 cm) was used as an extraction element. First, 4 mL of bottled water was put in a 20 mL vial, and NaCl was added to make its concentration as 0.15 mol/L. Next, PSF extraction element was immersed in the water sample. Solid-phase extraction was done at 50 ºC for 30 minutes at a stirring speed of 300 rpm (Fig. 1). After extraction, the PSF extraction element was picked up by tweezers and dried on a filter paper.

TD-GC: A Double-Shot Pyrolyzer (PY-2020iD) was directly interfaced to the injector of a GC instrument equipped with a flame ionization detector (FID) (Fig. 1). The dried PSF element was placed in a sample cup and then introduced into the pyrolyzer furnace heated at 300 ºC under a nitrogen carrier gas. A DB-5 (*L*=30 m, i.d.=0.25 mm, df.=0.25 µm) was used as a separation column, and the column flow rate was 1.0 mL/min. The GC oven was temperature programmed; heated from 50 ºC to 200 ºC (10 min hold) at a ramp rate of 10 ºC/min, and then to 300 ºC (10 min hold) at the same ramp rate.

[Results] The chromatograms of a water sample and a PAEs-spiked sample are shown in Figs. 2 (a) and (b), respectively. Calibration curves for PAEs obtained by TD-GC showed a good linearity in the concentration range of 2 to 1000 µg/L. The correlation coefficients (*r*) were found to be *r*>0.99 except dimethoxyethyl phthalate (*r*>0.98). The relative standard deviations (RSDs) of the peak areas were 9.5% (*n*=6) or less. In addition, high enrichment factors (280-9930 times) were obtained for 10 phthalates, and analysis with the spiked samples yielded recovery rates of 87.0 to 117.7%.

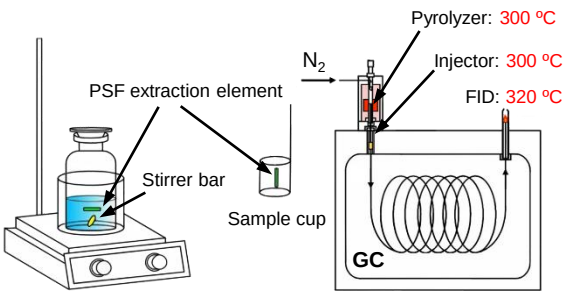

Fig. 1 Schematic diagram of hollow fiber membrane solid phase microextraction / TD-GC.

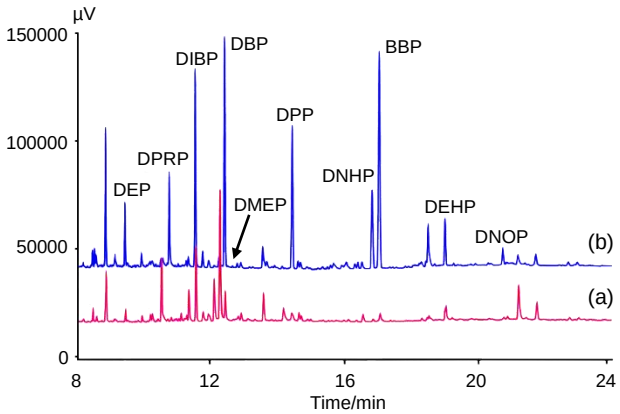

Fig. 2 Chromatograms of phthalates in bottled water
(a) Bottled water, (b) Spiked sample (PAE's concentration: 50 µg/L each*).

* DEP: diethyl phthalate, DPRP: dipropyl phthalate, DIBP: diisobutyl phthalate, DBP: dibutyl phthalate, DMEP: dimethoxyethyl phthalate, DPP: dipentyl phthalate, DNHP: di-*n*-hexyl phthalate, BBP: benzyl butyl phthalate, DEHP: di-2-ethylhexyl phthalate, DNOP: di-*n*-octyl phthalate.

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Keywords : Hollow fiber membrane, Solid-phase microextraction, Phthalates, Thermal desorption gas chromatography

Product used : Multi-Functional Pyrolyzer

Applications : Analysis of phthalates in bottled water

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