

Differentiation of DOTP and DNOP in analysis of restricted phthalate esters using thermal desorption GC/MS

[Background] Thermal desorption (TD)-GC/MS is a simple analytical method for the determination of restricted phthalates in polymers. ASTM D7823-13* utilizes TD-GC/MS to determine six restricted phthalates in PVC. D7823-13 uses retention times and mass spectra for the identification of phthalates; therefore, attention must be given to similar compounds having similar retention indices and mass spectra. Miss-identification will result in a false positive and an elevated concentration for the target phthalate. One such example is the possible miss-identification of di-(n-octyl) phthalate (DNOP, CAS: 117-84-0) and bis (2-ethylhexyl) terephthalate (DOTP, CAS: 6422-86-2). Both have m/z 149 and 279 - ions that are used to identify and quantitate DNOP. This note shows that these two phthalates are differentiated using the standard GC method described in ASTM 7823-13..

[Experimental] 70 µg of PVC containing tens of % of DINCH® and hundreds of ppm of DNOP and DOTP was placed in a sample cup, and analyzed using the conditions described in ASTM D7823-13. A Multi-Shot Pyrolyzer (EGA/PY-3030D) was directly interfaced to the injection port of the GC/MS system.

[Results] DNOP and DOTP co-elute with DINCH (see Fig. 1) which precludes the positive identification and quantitation of either phthalate using the TIC. The mass spectra of both DNOP and DOTP have 149 and 279 ions – see Fig. 2, and therefore extracted ions chromatograms (EIC) cannot be used to differential DNOP and DOTP. On the other hand, m/z 261 is a significant ion in the DOTP mass spectrum. As demonstrated here, the retention time and the presence or absence of m/z 261 ion can be used to differentiate between DNOP and DOTP.

* Frontier Lab original technical brief (<http://www.frontier-lab.com/techinfo/phthalate/index.html>)

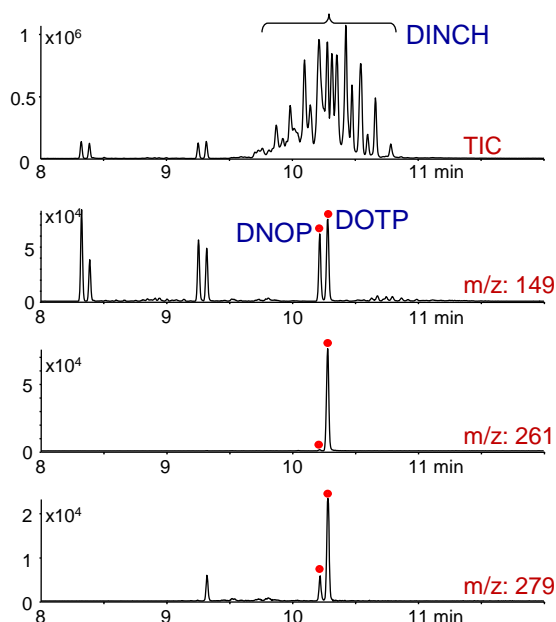


Fig. 1 Chromatograms of a PVC which contains various plasticizers obtained by TD-GC/MS

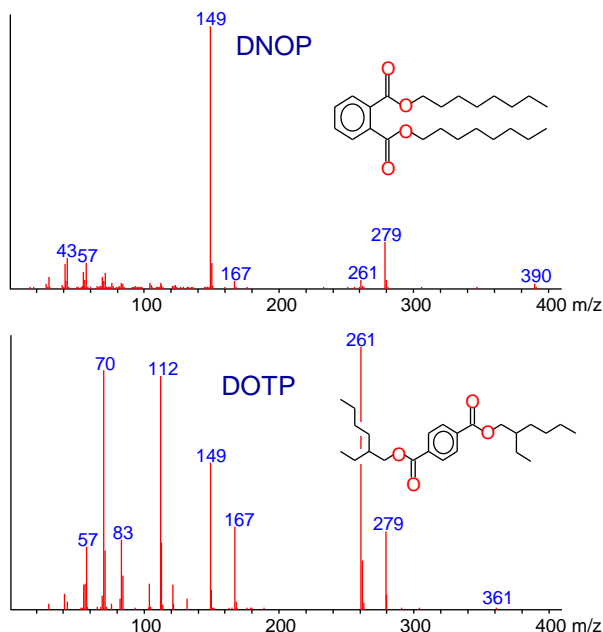


Fig. 2 Mass spectra of DNOP and DOTP

TD: 100-320°C (20°C/min, 5min hold),
 GC oven: 80-200°C (50°C/min)-320°C (15°C/min, 2min hold)
 Separation column: Ultra ALLOY+5 (5% diphenyl 95% dimethylpolysiloxane)
 L=30 m, i.d.=0.25 mm, df=0.25 µm, split ratio: 1/100, sample wt.: approx. 70 µg

Keywords : ASTM, phthalates, DINCH, DOTP, DNOP, PVC

Products used : Multi-functional pyrolyzer, Auto-Shot Sampler, Vent-free GC/MS adapter, UA+-5

Applications : Phthalates in consumer products

Related technical notes : PYA1-063E, PYA1-064E, PYA1-068E, PYA1-069E

Please forward your inquiries via our web page or send us a fax message.

R&D and manufactured by :
Frontier Laboratories Ltd.

Phone: (81)24-935-5100 Fax: (81)24-935-5102
<http://www.frontier-lab.com/>