TDTS 35

Thermal desorption of dioctyl phthalate and other plasticisers

Summary

In this Application Note, we demonstrate that Markes' UNITY™ thermal desorber gives quantitative and reproducible results for desorption of high-boiling plasticisers such as dioctyl phthalate.

Introduction

Organic esters such as diethyl and dioctyl phthalate are commonly used as plasticisers in the polymer industry. Additives like these are used to produce a stronger but more flexible polymer with a wider range of uses than the original material.

One of the most common plasticisers used is dioctyl phthalate (DOP). This colourless and odourless liquid is often added to the multi-purpose plastic PVC, which is in turn used to make a wide range of containers and construction products. DOP is a known irritant affecting the eyes, skin and the respiratory tract. There are also concerns relating to long-term (chronic) exposure, with DOP having been linked with damage to the liver, reproductive system and central nervous system. It is also a suspected carcinogen.

The majority of plastic products containing DOP end up as domestic or industrial waste. However, neither PVC nor DOP degrade quickly, and the plasticiser may therefore present a long-term health hazard.

Problems regarding the manufacture of silicon wafers in 'cleanroom' facilities have also been associated with DOP and related plasticisers. Although DOP is not the only compound known to cause these problems in the vapour phase, it has a boiling point of 405 °C, which means ambient vapour concentrations are very low and hard to detect. The problem is therefore usually first revealed by contamination of the wafers themselves, at substantial cost to the industry.

In this Application Note, we describe TD–GC/MS conditions suitable for monitoring low levels of high-boiling phthalates.

Experimental

Sample:
10 µL benzene, 10 µL toluene, 10 µL diethyl phthalate (DEP), 10 µL dibutyl phthalate (DBP) and 10 µL dioctyl phthalate (DOP) were dissolved in 100 mL of methanol. A sample of this standard solution (2 µL), containing about 200 ng of each component, was introduced to the sampling end of a sorbent tube packed with quartz wool and Tenax® TA sorbent tubes in a 50 mL/min stream of pure helium. Samples were then desorbed and analysed by TD–GC/MS.

TD (UNITY):

Cold trap packing:	Quartz wool backed up by Tenax TA and Carbopack™ B
Trap conditions:	–10 to 300 °C for 10 min
Prepurge:	1 min
Desorb:	300 °C for 10 min
Desorb flow:	25 mL/min
Split flow:	38 mL/min (on during both primary and secondary desorption)
Flow path temp.:	200 °C
Split ratio:	64:1

GC/MS (Agilent 6890 GC and Agilent 5973 MS):

Column flow:	1.5 mL/min
GC temp. program:	60 °C (2 min), 20 °C/min to 280 °C (5 min)
Sample:	2 µL of the standard solution injected onto tube under helium flow (50 mL/min)
Sorbent tube:	Inert coated stainless steel, packed with a plug of quartz wool backed up by a bed of Tenax TA

Results and discussion

Using the unique re-collection facility provided by UNITY, a portion of the original sample was trapped and subsequently analysed, generating reproducible results (Figure 1). This repeat analysis capability eliminates the one-shot limitation of conventional thermal desorption. In this case the overall split ratio was 64:1. The re-collected sample was thus almost identical (64/65) to the original.

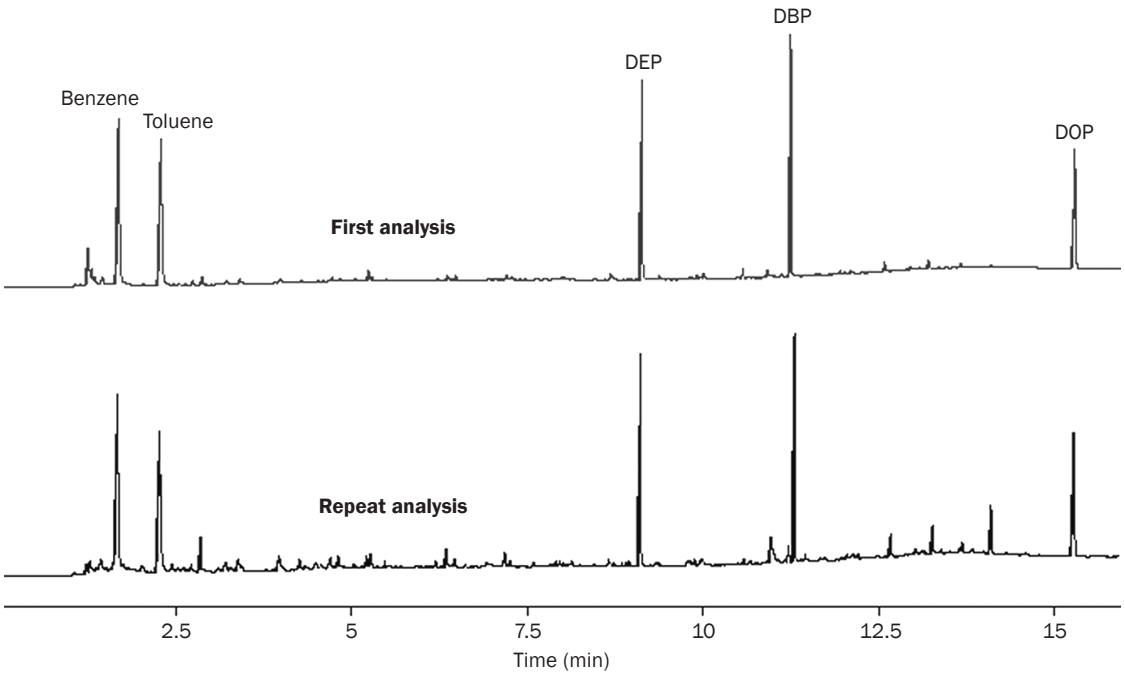

Figure 1: First analysis of plasticiser sample, and repeat analysis following re-collection.

Compound name	Peak area	
	First analysis	Repeat analysis
Benzene	94.5	94.0
Toluene	90.4	95.9
Diethyl phthalate (DEP)	60.7	64.5
Dibutyl phthalate (DBP)	72.1	69.8
Dioctyl phthalate (DOP)	60.2	60.7

Table 1: First analysis of plasticiser sample, and repeat analysis following re-collection.

Conclusions

UNITY, with its short, inert and uniformly heated flow path, is compatible with quantitative desorption of high-boiling compounds such as DOP. System performance has been further verified for this difficult application by the demonstration of re-collection and repeat analysis.

Trademarks

Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.

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