MARKES international

Application

Released: April 2009
Updated: September 2017

THE QUEEN'S AWARDS FOR ENTERPRISE: INTERNATIONAL TRADE 2015

Application Note 035

Thermal desorption of phthalate plasticisers

Summary

In this Application Note, we demonstrate that Markes' thermal desorbers give quantitative and reproducible results for desorption of high-boiling plasticisers such as bis(2-ethylhexyl) phthalate.

Introduction

Phthalates are commonly used as plasticisers to produce stronger but more flexible polymers with a wider range of uses than the original material. One of the most common plasticisers is bis(2-ethylhexyl) phthalate (DEHP), which is often added to PVC, which is in turn used to make a wide range of containers and construction products. DEHP is a known irritant, and there are also concerns relating to the health effects of long-term exposure.

Problems regarding the manufacture of silicon wafers in cleanroom facilities have also been associated with phthalate plasticisers, with their low volatility resulting in low ambient vapour concentrations that are 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 di-n-butyl phthalate (DBP) and 10 µL bis(2-ethylhexyl) phthalate (DEHP) were dissolved in 100 mL of methanol. A 2 µL aliquot of this solution, containing about 200 ng of each component, was introduced to the sampling end of a quartz wool–Tenax® TA inert-coated stainless steel sorbent tube in 50 mL/min of pure helium.

TD:

Instrument:	UNITY™
Cold trap:	Quartz wool backed up by Tenax TA and Carbopack™ B
Trap low:	–10°C
Trap high:	300°C (10 min)
Prepurge:	1 min
Desorb:	300°C (10 min)
Desorb flow:	25 mL/min
Split flow:	38 mL/min (on during both tube and trap desorb)
Flow path:	200°C
Split ratio:	64:1

GC–MS:

Instrument:	Agilent 6890 GC and Agilent 5973 MS
Column flow:	1.5 mL/min
Oven ramp:	60°C (2 min), 20°C/min to 280°C (5 min)

Results and discussion

Using the unique re-collection facility available on Markes' thermal desorbers, 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.

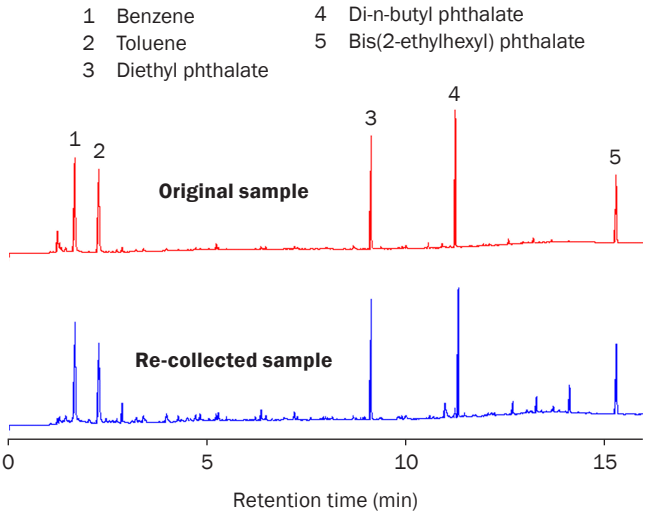

Figure 1: Original analysis of phthalate standard, and repeat analysis following re-collection.

Compound name	Peak area	
	First analysis	Repeat analysis
Benzene	94.5	94.0
Toluene	90.4	95.9
Diethyl phthalate	60.7	64.5
Di-n-butyl phthalate	72.1	69.8
Bis(2-ethylhexyl) phthalate	60.2	60.7

Table 1: Peak areas from the original and repeat runs of the phthalate standard.

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Conclusions

Markes' thermal desorbers, with their short, inert and uniformly heated flow paths, are compatible with quantitative desorption of high-boiling compounds such as phthalates. 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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