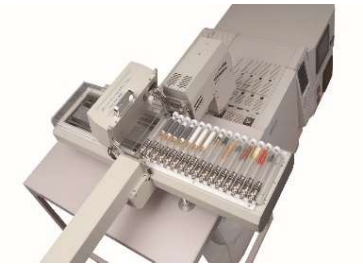
A-048E

Related Product：Thermal Desorption System



Thermal Desorption System
JTD-505Ⅲ

Thermal Desorption-GC/MS Analysis of Phthalic Esters

Keyword:
Thermal Desorption, Polymer Additives, Phthalic Esters

Introduction

There are two ways of quantitative analysis of the four phthalic esters specified in RoHS Directive, one is Semi-quantitative analysis using thermal extraction and the other is Quantitative analysis using solvent extraction. The solvent extraction method is generally better in terms of accuracy but the extraction process itself takes hours. On the other hand, the thermal extraction method has a risk that decomposition products remain in GC/MS as samples are heated to 340°C, resulting in poor analysis.

We found a better way to analyze phthalic esters contained in polymers using Thermal Desorption-GC/MS method.

Experiment

Sample: 0.5mg each of standard sample of Polyethylene, Polyvinyl Chloride and Polypropylene
Thermal Desorption System JTD-505Ⅲ and GC/MS were set as follows:

JTD-505Ⅲ	
Heating Temperature	280°C
Heating Time	15 min.
Trap Temperature	−60°C

GC/MS	
Capillary Column	DB-1, 15 m × 0.25 mm, 0.1 μm
Oven Temperature	80°C (1 min) > 20°C/min > 320°C (2 min)
Interface Temperature	320°C

Total time is 30minutes (15 minutes each for thermal extraction and GC/MS analysis) per sample.

Results

1. PE (Polyethylene)

Sample：PE containing 1,000ppm each of DIBP, DBP, BBP, DEHP, DOP, DINP and DIDP

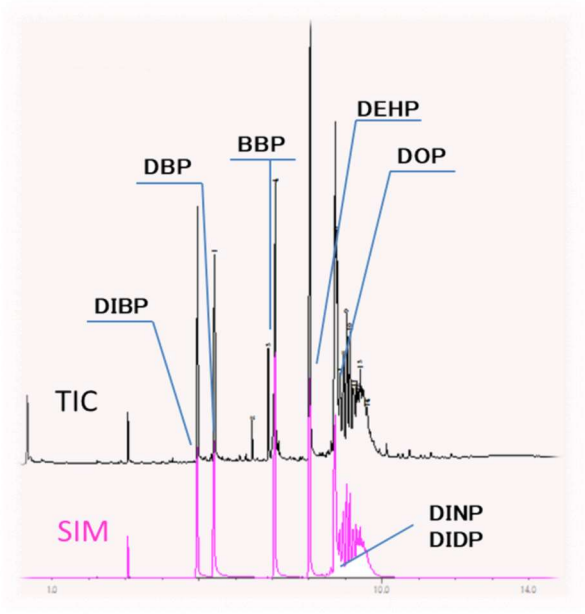


Fig. 1

Fig. 1 The TIC and SIM indicate that PE was not decomposed and all the seven phthalic esters were clearly detected.

The CV value of the peaks were less than 5% for all the phthalic esters. (n=3)

We got the same results with samples containing 100ppm of the additives.

2. PVC (Polyvinylchloride)

Sample：PVC containing 1,000ppm each of DIBP, DBP, BBP, DEHP and DOP

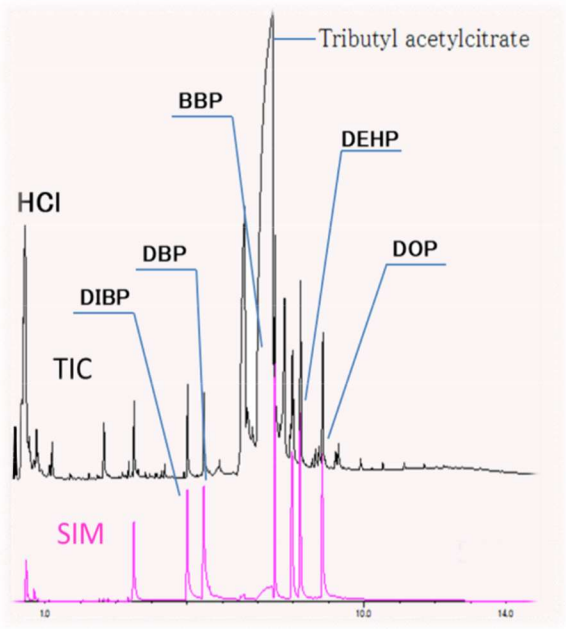


Fig. 2

Fig. 2　As shown on the TIC and SIM, all of the five phthalic esters were clearly detected separately from the peaks of HCL and aromatics, the decomposition products of PVC.

CV values were 4.9% for DIDP, 6.5% for DBP, 12.6% for BBP, 4.9% for DEHP and 3.9% for DOP. (n=3)

The value for BBP was high probably because the peak overlapped with that of Acetyltributylcitrate, the original additive.

3. PP (Polypropylene)

Sample：PP containing 1,000ppm each of DBP, BBP and DEHP

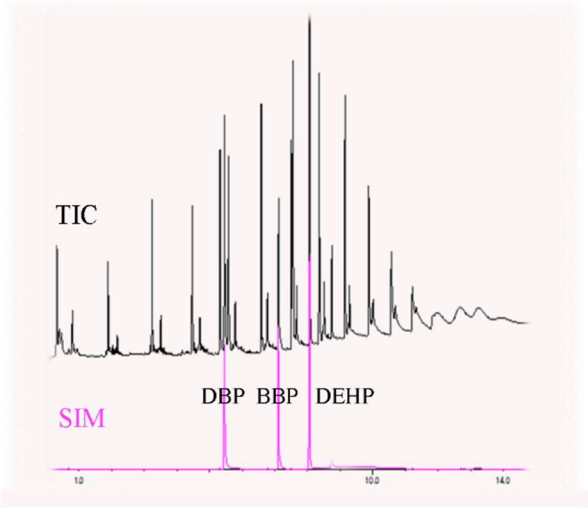


Fig. 3 PP

Fig. 3　All the phthalic esters were clearly detected as well as many aliphatic compounds that are decomposition products of PP.

CV values were 2.7% for DBP,4.8% for DBP and 2.2% for DEHP. (n=3)

Conclusion and Discussion

Using Thermal Desorption-GC/MS method, we heated the sample only up to 280°C for 15 minutes during which polymers are decomposed only partially and were able to obtain quantitative analysis results of the phthalic esters with high reproducibility.

The required time for one sample in this method was 30 minutes in total (15minutes each for thermal desorption and GC/MS analysis).

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