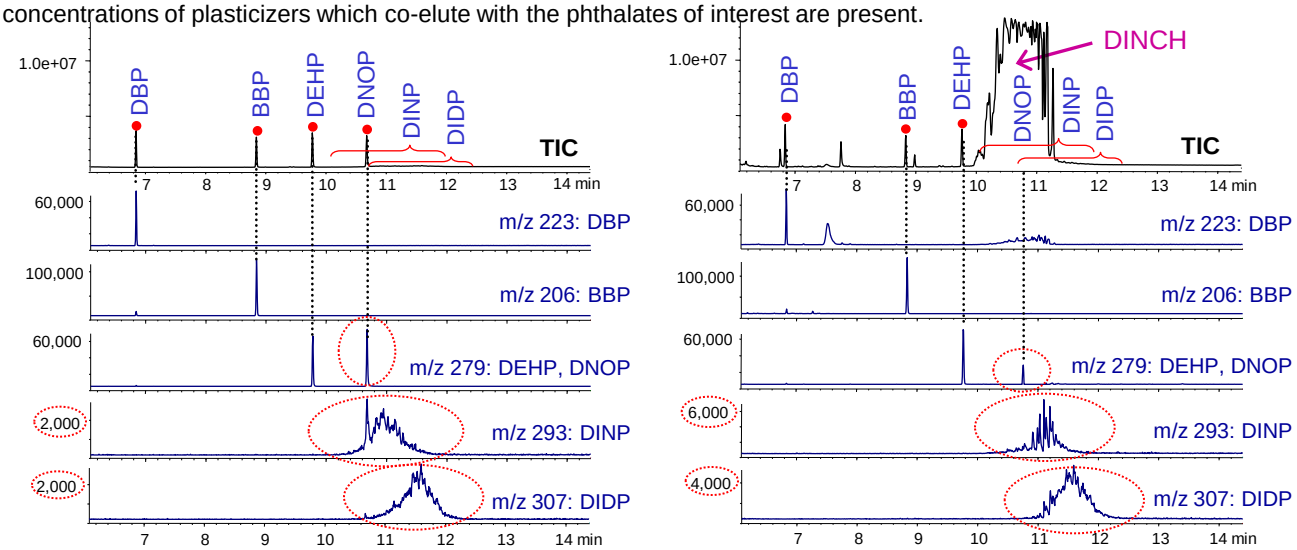
Determination of phthalates in PVC by thermal desorption-GC/MS

Part 2: Calibration using absolute calibration method and standard addition

[Background] Using the thermal desorption temperature zone described in PYA1-068E, two different calibration methods (absolute calibration and standard addition) were evaluated in order to determine which method most effectively reduces or eliminates the matrix effects caused by the presence of a large amount of plasticizers in the sample.

[Experimental] The thermal desorption (TD)-GC/MS system consisted of a Multi-shot pyrolyzer (EGA/PY-3030D) interfaced to the split/splitless injection port of a GC/MS. A thin film (0.2mg) of the PVC-DINCH sample was prepared as described in PYA1-068E. The thermal desorption temperature zone was 100-320ºC. A calibration standard (Ph-Mix) containing 0.1% of each phthalates was used. Identification was based on the retention time of the each phthalate's characteristic ion and the common m/z149 ion. Quantitation was based on the peak area of the characteristic ion.

[Results] No DINCH interferences were observed for DBP, BBP, and DEHP, and both methods gave similar results – Fig.1. However, DNOP, DINP, and DIDP co-elute with the DINCH peak envelop which potentially will degrade the accuracy of the phthalate concentration determination. Specifically, if there is no DINCH in the sample the retention time of DNOP shifts and the peak width at half-height is double what it is when the sample contains DINCH. In addition, the peak height is 60% lower when DINCH is present; consequently, when the absolute calibration method is used, the concentrations of DNOP, DINP, and DIDP are higher than the true value of 0.1% On the other hand, the concentration of the phthalates obtained using the standard addition method are very close to the true value of 0.1%. The results show that standard addition is the preferred method when quantitating phthalates in PVC using TD-GC/MS. Standard addition minimizes the interference when high concentrations of plasticizers which co-elute with the phthalates of interest are present.



(a) Ph-Mix (0.1% each)

(b) PVC-DINCH (Contains 0.1% each Ph)

Fig.1 TD chromatograms of samples and effects of DINCH interference to phthalates (TD: 100-320ºC/min, 5 min hold)

Table 1 Reproducibility of the phthalate peak area when using two calibration methods

Phthalate: 0.1% each		DBP	BBP	DEHP	DNOP	DINP	DIDP
%RSD (n=5)		0.79	0.85	0.69	1.59	1.59	0.98
Quantified value (%)	Absolute calibration	0.122	0.117	0.121	**0.029**	**0.126**	**0.193**
	Standard addition	0.115	0.093	0.096	**0.098**	**0.103**	**0.088**

Keywords : Restricted phthalate, plasticizer, absolute calibration, standard addition, thermal desorption, infant plastic toy

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

Applications : Restricted phthalates, plasticizer

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

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