

# Analysis of phthalates with IEC standard method using non-deactivated stainless steel sample cups

**[Background]** Thermal desorption (TD)-GC/MS is incorporated into the IEC standard method for the analysis of phthalates in plastics (IEC 62321-8). The sample cup specified in the method is made of deactivated (*via* the vapor deposition of a thin quartz film on the surface) stainless steel. Although the sample cups are expensive, they are reusable if residuals are removed using either heat or selected chemical treatments. An alternative is to use non-deactivated sample cups. These one-time-use cups are inexpensive and eliminate all concerns about sample cross contamination; however, there is a concern about catalytic pyrolysis on the metal surface. In this note, a side-by-side comparison of a PVC standard is described using deactivated and non-deactivated (cleaned) sample cups. Both reproducibility and recovery data clearly show that there is no effect on data quality when non-deactivated cups are used for the analysis of phthalates in plastic.

**[Experimental]** A typical sample cup is shown in Fig. 1. Four deactivated sample cups and four non-deactivated sample cups were used for repetitive analysis ( $n=4$ ). The sample was a PVC standard (obtained from AIST: NMIJ CRM 8152-a) containing eight different phthalates and an adipate, each at 900 ppm). The analytical system consisted of a Multi-Shot Pyrolyzer (EGA/PY-3030D) interfaced directly to the GC split/splitless injector. The sample was freeze-ground, placed in a sample cup, and analyzed using the method described in the IEC standard.

**[Results]** Representative TD chromatograms obtained using two different sample cups are shown in Fig. 2. The two chromatograms are virtually identical. The reproducibility ( $n=4$ ) of peak areas calculated using the areas of the extracted ion chromatograms of the characteristic ion of each phthalate and the relative recovery (based on values obtained using the deactivated sample cup) are summarized in Table 1. Reproducibility is comparable; RSD values are 4% or less. Also, the relative recoveries obtained from the peak area ratios between the two types of cups are 94 – 101% for all of the phthalates. Comparable results were obtained for both non-deactivated and deactivated sample cups; therefore, using non-deactivated sample cups within the guidelines specified in IEC 62321-8 will have no effect on the analytical determination of phthalates in plastics.

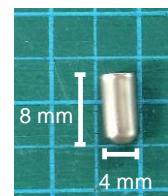


Fig. 1 Sample cup.

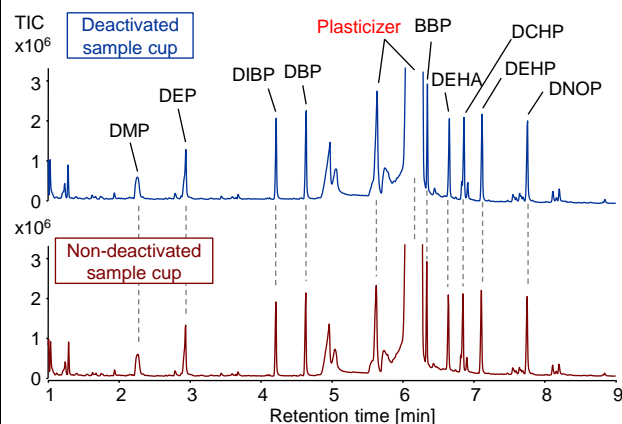


Fig. 2 TD chromatograms of phthalates in PVC obtained using two different types of sample cups.

Furnace temp.: 200 – 300 (20 °C/min) – 340 °C (5 °C/min)  
 Column: UA-PBDE (dimethylpolysiloxane,  $L=15$  m, i.d.=0.25 mm, df=0.05  $\mu$ m)  
 Column flow rate: 1 mL/min (He), Split ratio: 1/50  
 GC oven: 80 – 300 °C (20 °C/min), Sample amount: ca. 0.5 mg

Table 1 Reproducibility of peak area and relative recovery rate

Phthalate	$m/z$	Deactivated sample cup		Non-deactivated sample cup		Relative Recovery [%]
		Average area $\pm$ s [ $10^6$ ]	RSD [%]	Average area $\pm$ s [ $10^6$ ]	RSD [%]	
DMP	163	21.4 $\pm$ 0.2	0.72	21.0 $\pm$ 0.5	2.54	98.3
DEP	149	20.6 $\pm$ 0.3	1.29	20.8 $\pm$ 0.2	0.86	101
DIBP	149	28.1 $\pm$ 0.2	0.79	28.4 $\pm$ 0.3	1.05	101
DBP	149	36.7 $\pm$ 0.5	1.25	34.6 $\pm$ 0.3	0.80	94.3
BBP	149	14.0 $\pm$ 0.4	2.87	13.9 $\pm$ 0.1	0.97	98.9
DEHA	129	11.56 $\pm$ 0.1	0.97	11.6 $\pm$ 0.1	0.94	101
DCHP	149	22.9 $\pm$ 0.5	2.10	22.4 $\pm$ 0.8	3.63	97.7
DEHP	149	20.2 $\pm$ 0.3	1.62	20.4 $\pm$ 0.2	1.16	101
DNOP	149	33.5 $\pm$ 0.4	1.22	33.7 $\pm$ 0.9	2.67	101

- Recovery rate is normalized to the peak area obtained using deactivated sample cups being 100.
- Due to high volatility of DMP and DEP, the data may not be reliable.

**Keywords :** IEC 62321-8, RoHS regulation, Phthalate ester, PVC, Thermal desorption, Non-deactivated sample cup

**Products used :** Multi-functional Pyrolyzer, Auto-Shot Sampler, Vent-free GC/MS adapter, UA-PBDE, Eco-Cup LF, RoHS cup LN

**Applications :** General polymer analysis, Phthalates analysis

**Related technical notes :**

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