A Practical Applications Guide for Analytical Pyrolysis - GC/MS

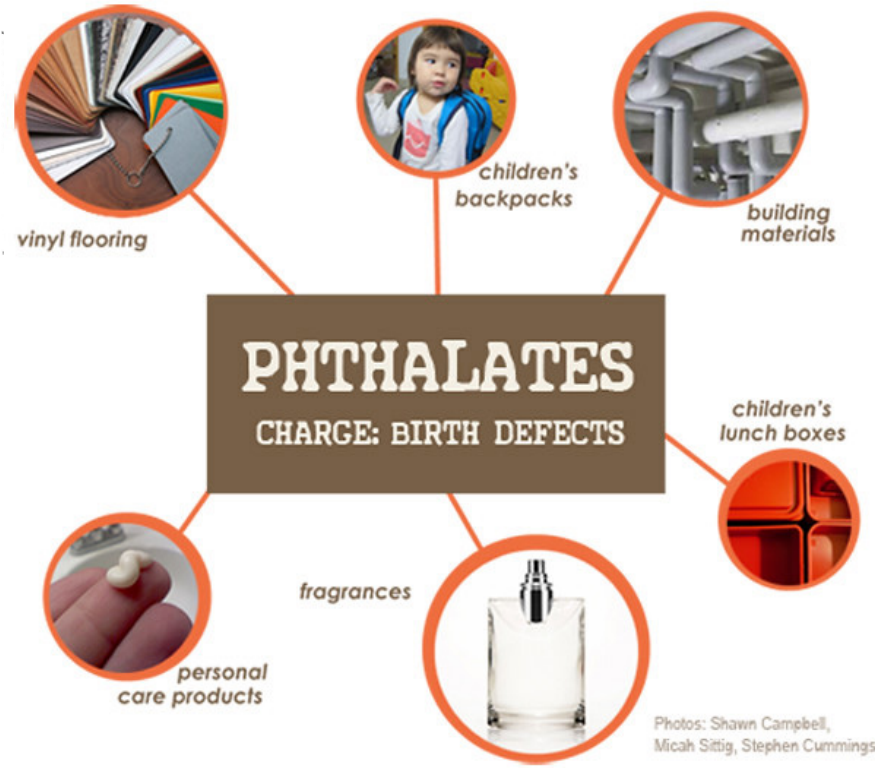
Phthalate

CDS Analytical
www.cdsanalytical.com

RoHS and IEC 62321-8

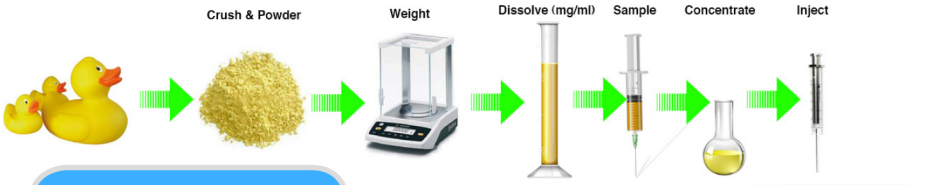
Restriction of Hazardous Substances, or RoHS, began in the European Union and restricts the use of specific hazardous materials found in electrical and electronic products. Businesses which sell these products may be subjected to RoHS requirements if they use restricted substances. The RoHS 3 with a deadline of July 22, 2019 requires the disclosure of the following 4 phthalates: Bis(2-Ethylhexyl) phthalate (DEHP), Benzyl butyl phthalate (BBP), di-n-butyl phthalate (DBP), Diisobutyl phthalate (DIBP). These phthalates are typically used as insulation plasticizers and are on the European Chemicals Agency REACH list of SVHC (Substances of Very High Concern).

In addition, the International Electrotechnical Commission (IEC) published a standard method for determining phthalates in electronic equipment. IEC 62321-8 defines approaches to determine previously mentioned DEHP, DIBP, BBP, DBP, as well as additional phthalates, di-n-octyl phthalate (DNOP), di-isononyl phthalate (DINP) and di-iso-decyl phthalate (DIDP) in electronics, by GC-MS and TD-GC-MS. The TD-GC-MS method involves two separate thermal desorption heating ramps for one GC run. This can be performed using a CDS 6000 Series Pyroprobe Autosampler.

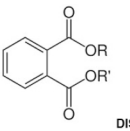


Thermal Extraction of Phthalates by CDS Pyroprobe

The traditional way of phthalates quantification involves with extensive wet chemistry in the sample preparation step, whereas the IEC 62321-8 method adopts the thermal extraction method by using a pyrolyzer to by pass the sample dissolution and concentration steps.



Temperature Ramp 1:
Pyroprobe :
Initial: 200°C
Ramp: 20°C/minute
Final: 300°C

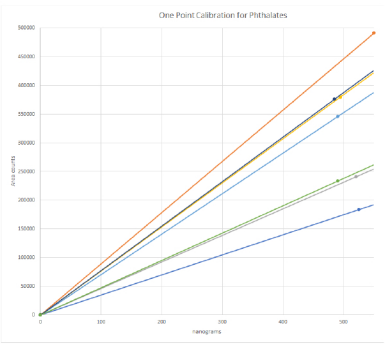


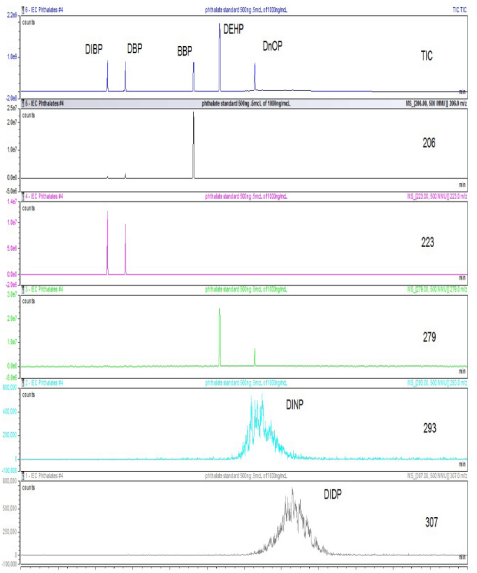
DISC Tube

sample 200 μg

Temperature Ramp 2:
Pyroprobe :
Initial: 300°C
Ramp: 5°C/minute
Final: 340°C hold 1 min

One Point Calibration for Phthalates





500ng Phthalate Standard TIC and Extracted Ion Chromatograms.

Results and Conclusions

TIC and extracted ion chromatograms match the chromatograms in Annex C.2 of the International Standard. Eight replicates of the standard presented area RSDs for most of the phthalates around or under 3%, which is 3 times better than other competitors' product. The MDL is averaged at 17 ppm, which is 5 times better than the method requirement.

Phthalate	Quant Ion	RSD	MDL(ppm)
DIBP	223	3.2 %	21.7
DBP	223	2.3 %	21.0
BBP	206	4.3 %	21.0
DEHP	279	2.9 %	14.7
DNOP	279	3.2 %	9.4
DINP	293	3.0 %	17.9
DIDP	307	3.2 %	13.6

RSDs and MDL of 7 regulated phthalates

The latest version of the Pyroprobe from CDS Analytical ensures repeatable, reliable results for thermal desorption of phthalates in accordance with standard regulations and methods, like RoHS and IEC 6321-8 for determination of phthalates in electromechanical products.

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