BUILDING YOUR ANALYTICAL TESTING FOUNDATION
CORNERSTONE ANALYTICAL LABORATORIES

Analysis of Phthalate Esters

Overview and Application of Phthalate Testing

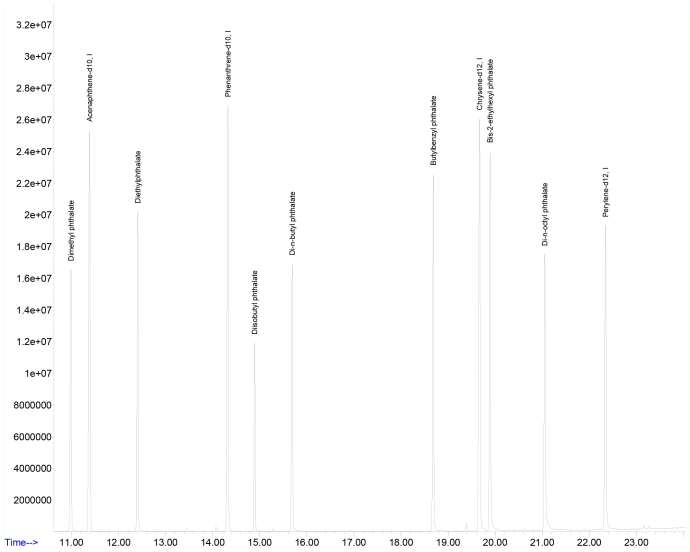

Figure 1. Typical Chromatogram of Phthalate Standard Mix

Introduction
Phthalates (phthalate esters) are plasticizers added to improve the properties of a plastic. Phthalates are categorized based on the number of carbons in their side chains. Low molecular weight phthalates have 3-6 carbon side chains. High molecular weight phthalates have 6 or more carbons in their sidechains.

In general, the higher weight phthalates are more durable in finished plastics and are believed to represent a reduced toxicological risk.

Sample Types
Phthalate analysis can be applied to many types of samples, but is generally applied to plastics and polymers used in consumer products.

Method
Our internal method is designed to handle a wide range of sample types. It is based on EPA Method 8061, yet with GC-MS in lieu of GC-ECD.

Sample Preparation
Since phthalates are not chemically (covalently) bound to the polymers in a plastic, phthalates may be extracted from the sample using a solvent along with agitation and heat. It is therefore important to finely divide the sample prior to analysis. In most cases, the product is cut, drilled or ground to provide small particles. These particles are then weighed and a known volume of a solvent (such as 1:1 hexanes: acetone) is used to extract the sample. The sample is added to a glass vial with solvent resistant cap and is sonicated, shaken and may be heated to affect maximum extraction of phthalates into the solvent.

Calibration
The GC-MS is calibrated using a mixture of common phthalate esters. If a phthalate ester does not appear in the mix that is of interest to the client, Cornerstone will prepare a separate calibration source from a standards grade source. The calibration curve contains a minimum of 4 standards.

Analysis
Sample analysis is conducted on a GC-MS that has been externally calibrated with a series of phthalate standards. The calibration standards are matrix-matched as close as practical to the samples. For the result to be considered valid, all quality control must be within the acceptance criteria.

Calculations
The phthalate concentration of the sample calculated based on the mass of sample taken for the analysis to give the results on the weight/weight basis, such as ppm or ppb. Results that are below the method limit of quantitation are reported as a less-than value (e.g. <10 ppm).

Reporting
Results are reported based on the mass of sample taken for the analysis (wt/wt basis). If a sample is not dissolved during the preparation step, the results are reported as “leachable."

Limit of Detection and Quantitation
The limit of detection and quantitation are a function of the mass of sample taken for the analysis and the instrument limits. The limit of quantitation of this analysis is easily dialed very low by taking a relatively large sample size. Contact us for specific LOD/LOQ we can reach in your sample.

Phthalate Method Development and Validation
Cornerstone can develop and validate an analytical method for phthalate analysis in your sample. Please view our page on method validation for more information on our process of developing and validating methods. Once validated, we can transfer it to your facility.

Scope

The following is a list of phthalates provided in our routine service. Other phthalates are available - please contact us:

- Benzyl butyl phthalate (CAS 85-68-7)
- Bis(2-n-butoxyethyl)phthalate (CAS 117-83-9)
- Bis(2-ethoxyethyl)phthalate (CAS 605-54-9)
- Bis(2-ethylhexyl)phthalate (CAS 117-81-7)
- Bis(2-methoxyethyl)phthalate (CAS 117-82-8)
- Bis(4-methyl-2-pentyl)phthalate (CAS 146-50-9)
- Di-n-butylphthalate (84-74-2)
- Diethylphthalate (CAS 84-66-2)
- Di-n-hexyl phthalate (CAS 84-75-3)
- Dimethylphthalate (CAS 131-11-3)
- Di-nonyl phthalate (CAS 84-76-4)
- Di-n-octyl phthalate (CAS 117-84-0)
- Dipentylphthalate (CAS 131-18-0)
- Phthalic acid dicyclohexyl ester (CAS 84-61-7)
- Phthalic acid diisobutyl ester (CAS 84-69-5)

References

1. EPA Method 8061A, Revision 1, *Phthalate Esters by Gas Chromatography with Electron Capture Detection (GC-ECD)*, US Environmental Protection Agency, December 1996.
2. AP-CHROM-101, *Analysis of Phthalates by GC-MS*, Version 1, Cornerstone Analytical Laboratories, December 2015.