

# GC/MS Analysis of Phthalate and Adipate Esters in Drinking Water

## Using New Restek Reference Mixes and a Low-Bleed Column

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- New calibration and quality control check mixes save preparation time and effort.
- Stable baseline with Rtx®-5Sil MS column—no interference with sensitive detection.
- Rapid analysis, excellent resolution.

Phthalate esters are of considerable interest because their extensive use in consumer products, mainly as plasticizers, leads to widespread human exposure and potential for environmental contamination. In the United States, the Environmental Protection Agency (EPA) established strict drinking water standards for two of these semivolatile compounds, bis(2-ethylhexyl)phthalate and bis(2-ethylhexyl)adipate, as potential carcinogenic agents. Because even trace amounts of these esters can have a harmful effect on drinking water quality, the goal is to extract the compounds efficiently and identify them accurately. EPA Method 506 offers a procedure for extracting, identifying, and quantifying seven phthalate and adipate esters in drinking water, using liquid/liquid extraction (methylene chloride / hexane) or liquid/solid extraction (octadecyl (C18) disk, e.g., Restek cat.# 24004), extract concentration to 1mL, and analysis by gas chromatography/mass spectrometry.

We have developed two new reference materials for analyses of the phthalate and adipate esters targeted by Method 506. We prepare 506 Calibration Mix in isooctane at 1000µg/mL, per method recommendation, and 506 Laboratory Performance Check Mix in purge-and-trap grade methanol at x10<sup>5</sup> the method detection limit (MDL) for each analyte.

### Rtx®-5Sil MS Column (fused silica)

(Selectivity equivalent to Crossbond® 5% diphenyl / 95% dimethyl polysiloxane) (temp. limits -60°C to 330°C)

30-Meter, 0.25mm ID, 0.25µm df  
cat.# 12723



### Resprep™-C18 & Resprep™-C8 SPE Disks

- 47mm glass fiber disks embedded with C18 or C8 bonded silica.
- Extract semivolatile organic compounds.
- Deep-pore design reduces clogging and allows faster flow rates.
- Meet requirements for US EPA Methods 525.1, 506, 550.1, 549.1.
- Lower cost than Teflon® disks.

Description	qty.	cat.#
Resprep™-C18		
47mm SPE Disks	20-pk.	24004
Resprep™-C8		
47mm SPE Disks	24-pk.	24048

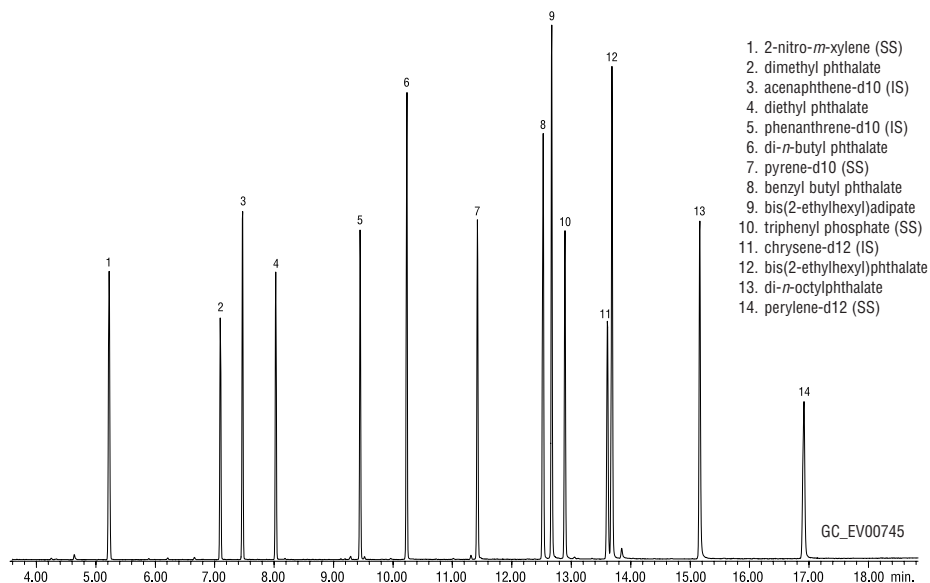
In our quest for superior chromatography and improved detection limits for this and other analyses, we have developed a series of low bleed polymeric stationary phases, using Crossbond® bonding technology. By providing stable baselines at higher temperatures, these phases allow higher signal-to-noise ratios, and thus greater sensitivity.

EPA Method 506 suggests low levels of phthalate and adipate esters be evaluated using a

photoionization detector. The method allows other approaches for detection, however, if equivalent performance can be demonstrated. Figure 1 shows a GC/MS analysis of the phthalates and adipates, using an Rtx®-5Sil MS column. Column bleed is low, even at the 310°C oven temperature needed to elute the phthalate esters with the highest boiling points. At this temperature, column bleed from an unstable column could have a significant effect on detection limits. The 80°C starting temperature and 18°C/min. temperature program ensure a fast analysis, without inhibiting resolution. US EPA 525.2 internal standards and surrogates were used since the method does not list specific monitoring compounds.

In analyses for phthalate and adipate esters, a low-bleed Rtx®-5Sil MS column can extend detection limits and help ensure you of reliable data from your samples.

**Figure 1** Rapid analysis of phthalates, with excellent resolution, using an Rtx®-5Sil MS column.



### 506 Laboratory Performance Check Mix

benzyl butyl phthalate	250µg/mL	di- <i>n</i> -octyl phthalate	650
bis(2-ethylhexyl)adipate	1200	diethyl phthalate	100
bis(2-ethylhexyl)phthalate	250	dimethyl phthalate	100
di- <i>n</i> -butyl phthalate	100		

Each	5-pk.	10-pk.
<b>In P&amp;T methanol, 1mL/ampul</b>		
31844	31844-510	—
w/data pack		
31844-500	31844-520	31944

### 506 Calibration Mix

benzyl butyl phthalate	di- <i>n</i> -octyl phthalate
bis(2-ethylhexyl)adipate	diethyl phthalate
bis(2-ethylhexyl)phthalate	dimethyl phthalate
di- <i>n</i> -butyl phthalate	

Each	5-pk.	10-pk.
<b>1,000µg/mL each in isooctane, 1mL/ampul</b>		
31845	31845-510	—
w/data pack		
31845-500	31845-520	31945

Column: Rtx®-5Sil MS, 30m, 0.25mm ID, 0.25µm (cat.# 12723)  
 Sample: 506 Calibration Mix, 1000µg/mL each analyte (cat.# 31845)  
 Method 525.2 Internal Standard Mix (cat.# 31825)  
 Method 525.2 Surrogate Standard Mix (cat.# 31826)  
 Inj.: 1.0µL, 20ppm each analyte using a 4mm splitless single gooseneck inlet liner (cat.# 20799) splitless hold time 0.40 min., 0.45 min. pressure pulse @ 50psi  
 GC: Agilent 6890  
 Inj. temp.: 270°C  
 Carrier gas: helium, constant flow  
 Flow rate: 1.0mL/min.  
 Oven temp.: 80°C (hold 0.5 min.) to 260°C @ 18°C/min., to 310°C @ 6°C/min. (hold 1 min.)  
 Det.: Agilent 5973 GC/MS  
 Transfer line temp.: 280°C  
 Scan range: 35–550 amu  
 Solvent delay: 3 min.  
 Tune: DFTPP