

Screening for Ten Phthalates in Four Food Standard Reference Materials (SRMs) by Gas Chromatography/Tandem Mass Spectrometry (GC/MS/MS).

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Why are Phthalates a Concern in Food?

Phthalates (diesters of 1,2-benzenedicarboxylic acid) are added to polymer formulations like polyvinyl chloride to increase flexibility. In such applications, phthalates are suspended in the polymer matrix and over time can leach from the material, possibly into food. There is concern that children's exposure to five phthalates, including diisononyl phthalate (DINP), di-n-pentyl phthalate (DPENP), di-n-hexyl phthalate (DHEXP), dicyclohexyl phthalate (DCHP), and diisobutyl phthalate (DIBP), can cause male reproductive development issues.



Screening for phthalates in food reference materials could estimate the levels of targeted phthalates in different food types. For this work, two infant/adult formula materials [SRMs 1849a (milk-based) and 1869 (milk/whey/soy-based)] and two Typical Diets (SRM 1548a and candidate SRM 1548b) were screened for 10 phthalates.

How are Phthalates Measured in Food?

Extraction: 0.5 g – 0.6 g weighed into glass centrifuge tubes, added 2 mL acetonitrile, shake for 1 min, centrifuge 5 min at 100 % power, filtered through 0.45 μm PTFE filters into amber glass vials. Blank processed through

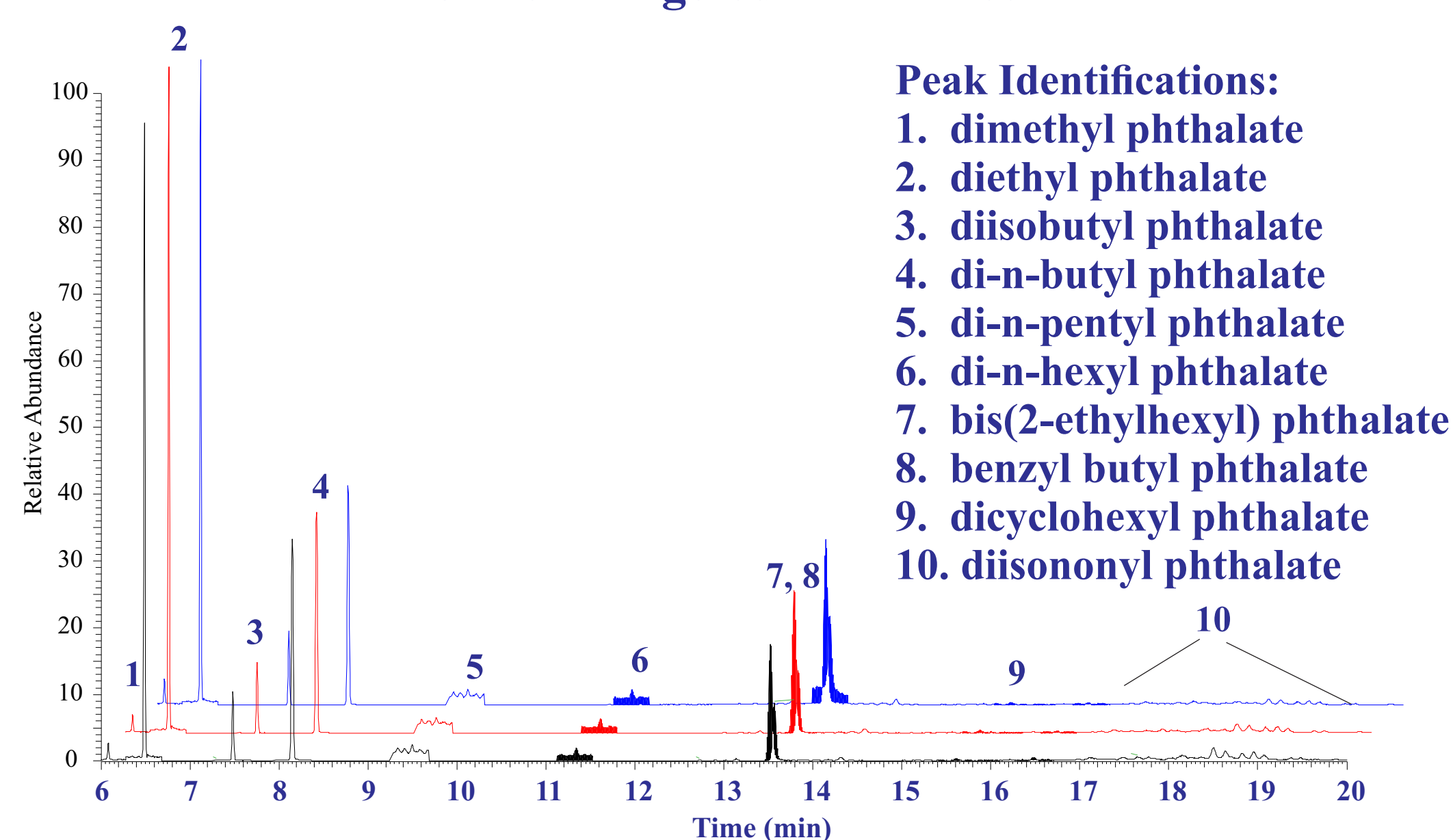
Multiple Reaction Monitoring (EI, see Ref. 1)

Target Phthalate	Mass (m/z)	Product Mass (m/z)	Collision Energy
dimethylphthalate	194	163	10
diethylphthalate	177	149	10
diisobutyl- and di-n-butyl phthalate	223	149	10
di-n-pentyl phthalate	237	149	10
di-n-hexyl phthalate	251	149	10
bis(2-ethylhexyl) phthalate	279	149	10
benzyl butyl phthalate	206	149	10
dicyclohexyl phthalate	249	149	10
diisononyl phthalate	293	149	10

GC/MS/MS: 60 m x 0.25 mm, ZB-50,
 2 mL/min He flow, 1 μL splitless injection
 at 285 $^{\circ}\text{C}$, oven 82 $^{\circ}\text{C}$ (1 min) • 50 $^{\circ}\text{C}/\text{min}$
 • 250 $^{\circ}\text{C}$ • 2 $^{\circ}\text{C}/\text{min}$ • 275 $^{\circ}\text{C}$ • 10 $^{\circ}\text{C}/\text{min}$
 • 300 $^{\circ}\text{C}$ (8 min).

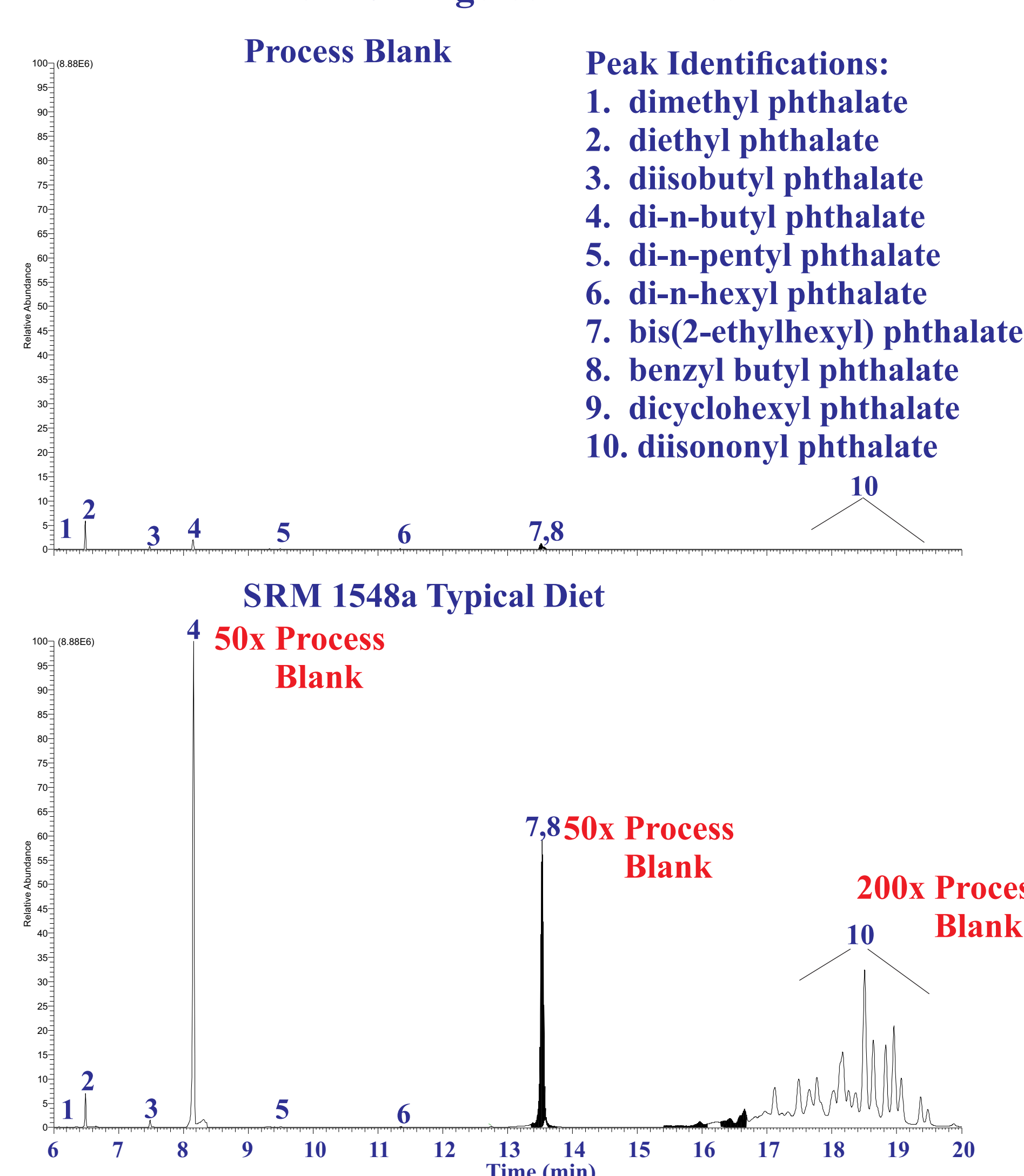
Which Phthalates were detected?

Three GC/MS/MS Runs of Process Blank for Ten Targeted Phthalates

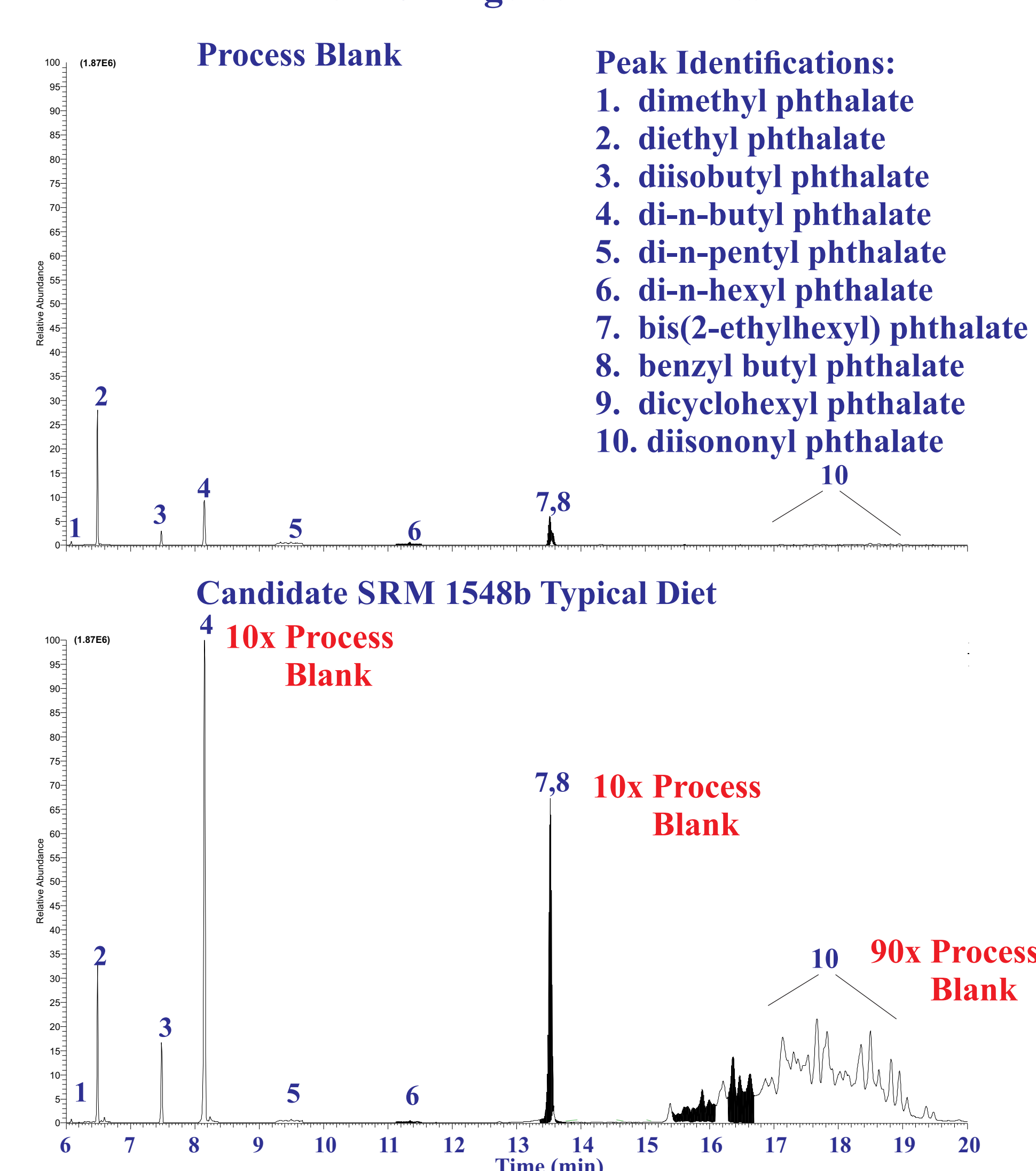


Results: As indication of the ubiquity of phthalates, 8 of the 10 targeted were detected in the process blank. None of the targeted phthalates were observed above process blank levels in SRM 1849a Infant/Adult Formula (milk-based). SRM 1548a Typical Diet was the most heavily contaminated of the four materials screened with levels of three phthalates 50 - 200x the process blank. Candidate SRM 1548b Typical Diet was the second most contaminated material yielding 10 - 90x the levels of the process blank for the same three phthalates.

GC/MS/MS of Process Blank and SRM 1548a Typical Diet for 10 Targeted Phthalates



GC/MS/MS of Process Blank and SRM 1548a Typical Diet for 10 Targeted Phthalates



Future Work: Optimize extraction method and quantify by GC/MS/MS using stable isotope labeled phthalates.

Reference

1. Food Chemistry 196, 211-219. 2016.

DISCLAIMER

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