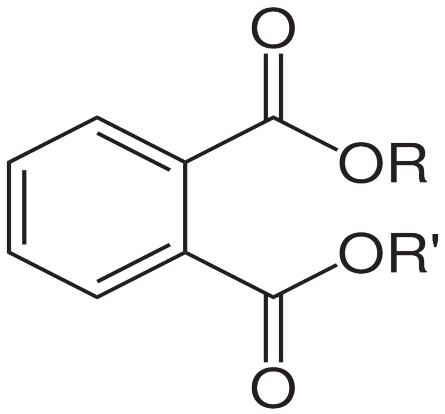
Determination of Phthalates Leached From Toys into Artificial Saliva



UCT Part Numbers

ECPAHFR50CT
50 mL polypropylene centrifuge tubes

ECQUUS2-MP
Mylar pouch with 4000 mg $MgSO_4$ and 2000 mg NaCl

Introduction:

Phthalates are plasticizers added to polymers to make them softer and more flexible. They are commonly used in the manufacturing of toys. Since phthalates are not permanently bonded in polymers, they may be released through touching, licking, or chewing. This new method artificially reproduces the licking and chewing actions of children by shaking cut toys in artificial saliva. Following extraction, phthalate levels are determined by GC/MS.

Preparation of Artificial Saliva (AS)1

AS was prepared by mixing 0.18 g xanthan gum, 1.2 g potassium chloride, 0.85 g sodium chloride, 0.05 g magnesium chloride, 0.13 g calcium chloride, and 0.13 g di-potassium hydrogen orthophosphate with 1 L of reagent water (1) and stirred for 4 hours.

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Procedure:

a) Cut plastic toys into small pieces
b) Weigh 1 to 2 g of toy samples to a 50 mL centrifuge tube **ECPAHFR50CT**
c) Add 10 mL of AS and two stir bars to the 50 mL tube
d) Shake for 1 hour using a horizontal shaker
e) Transfer the AS to a new 50 mL tube, add 10 mL of ethyl acetate and shake for 1 min
f) Add salts from pouch ECQUUS2-MP and shake vigorously for 1 min
g) Centrifuge at 5000 rpm for 5 min
h) Transfer 1 mL of the supernatant into a 2 mL auto-sampler vial
i) Add 10 µL of 50 ppm triphenyl phosphate (TPP) as internal standard
j) The sample is ready for GC/MS analysis

Instrument Conditions	
GC/MS	Agilent 6890N GC coupled with 5975C MSD, equipped with 7683 auto sampler
Injector	1µL splitless injection at 250 °C, 30 mL/min split vent at 1 min
Liner	4 mm splitless gooseneck, 4mmID*6.5mmOD*78.5mm (UCT cat#: **GCLGN4MM**)
GC capillary column	Restek Rxi-5sil MS 30m*0.25mm*0.25µm integrated with 10m guard column
Oven temperature program	Initial temperature of 70 °C, hold for 1 min; ramp at 20 °C/min to 315 °C, hold for 4.75 min. Acquire data from 6 to 14 min.
Carrier gas	He at a constant flow of 1.2 mL/min.
MSD	Transfer line: 280 °C; MS Source (EI): 250 °C; MS Quad: 150 °C
Simultaneous Scan/SIM	Scan range: 55-350 amu

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SIM Table								
Compound	**Abbreviation**	**Rt (min)**	**Group #**	**Start (min)**	**Dwell time (ms)**	**Quantify ion**	**Qualifier ion 1**	**Qualifier ion 2**
Dimethyl phthalate	DMP	6.989	1	6	25	163	194	133
Diethyl phthalate	DEP	7.858	2	7.5	25	149	177	105
Dibutyl phthalate	DBP	9.865	3	9	25	149	223	150
Benzyl butyl phthalate	BBP	11.716	4	11	25	149	91	206
Triphenyl phosphate	TPP	11.964			25	326	325	
Bis(2-ethylhexyl) phthalate	DEHP	12.432	5	12.3	25	149	167	279
Di-n-octyl phthalate	DOP	13.138			25	149	279	150

Calibration Curves:

Matrix matched calibration curves are constructed by analyzing matrix matched standards (0-1000 ng/mL). The responses were linear with correlation coefficient higher than 0.9973. The limit of quantification of this method is 25 ng/mL.

Accuracy and Precision Data (n=4)*		
Analyte	**Fortified 200 ng/mL**	**Fortified 500 ng/mL**
	Recovery ± RSD %	**Recovery ± RSD %**
Dimethyl phthalate	90.2 ± 2.6	94.6 ± 2.1
Diethyl Phthalate	91.1 ± 1.4	95.6 ± 2.0
Dibutyl phthalate	90.6 ± 3.8	97.0 ± 2.2
Benzyl butyl phthalate	85.5 ± 1.2	92.1 ± 2.5
Bis(2-ethylhexyl) phthalate	93.2 ± 2.2	92.7 ± 2.6
Di-n-octyl phthalate	88.8 ± 3.1	92.8 ± 1.3

*The control sample did not contain phthalates

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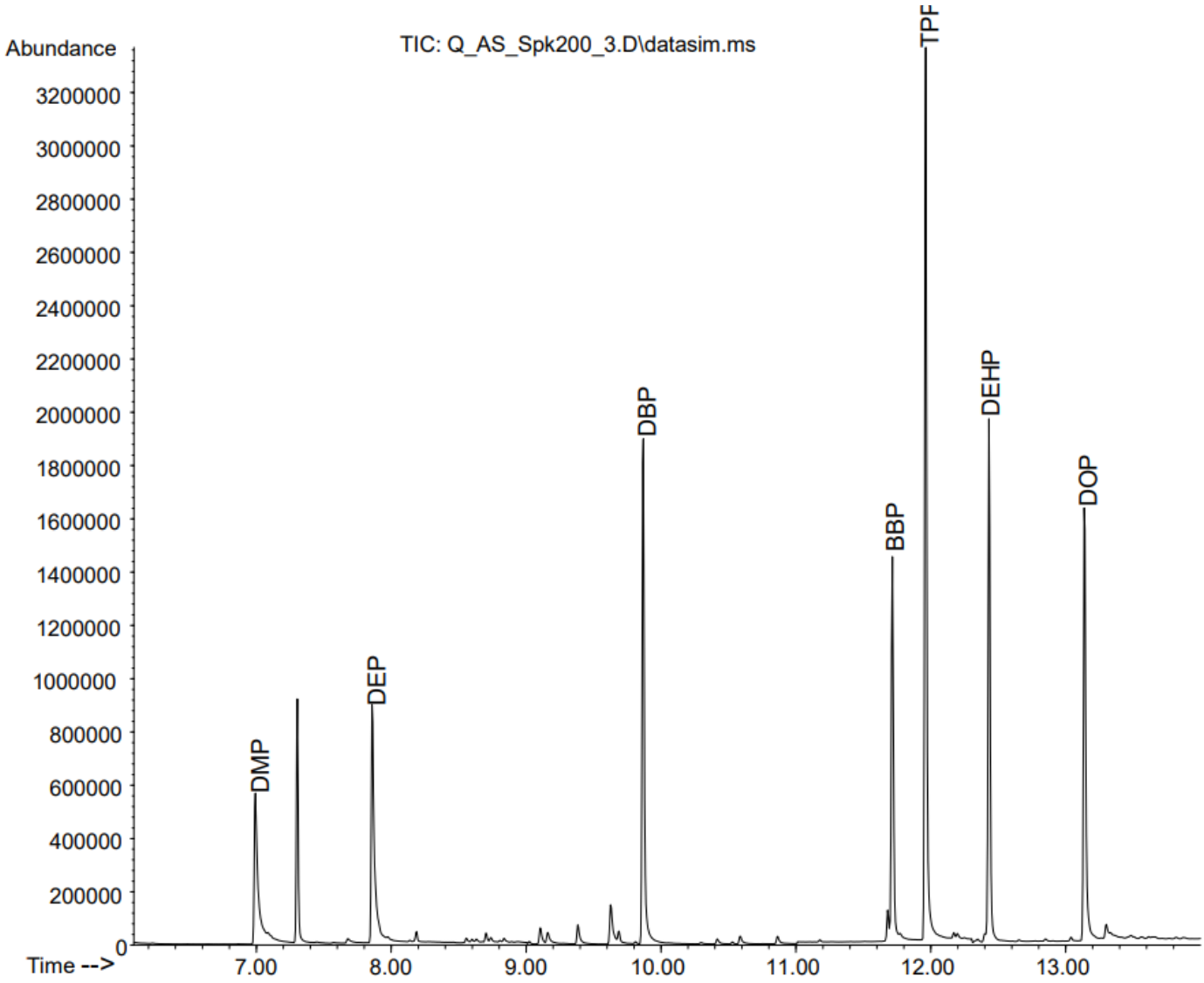
Chromatogram of AS fortified with 200 ng/mL phthalates



Results:

Diethyl phthalate was found leaching from toy samples into artificial saliva at a concentration of 285 ng/g (RSD=5.9%, n=3).

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References:

[1] A. Preetha and R. Banerjee, Comparison of Artificial Saliva Substitutes, *Trends Biomater. Artif. Organs* **18(2),**178-186 (2005).

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