



Modified QuEChERS Procedure for Analysis of Bisphenol A in Canned Food Products

UCT Products:

ECQUEU750CT-MP (each pouch contains 4000 mg MgSO₄, 1000 mg NaCl, 500 mg Na citrate dibasic sesquihydrate, 1000 mg Na citrate tribasic)

ECPSAC1856 (500 mg of PSA and 500 mg endcapped C18, 6 mL cartridge)

ECPSACB6 (400 mg PSA and 200 mg GCB, 6 mL cartridge)

SMTBSTFA-1-1 (MTBSTFA w/1% TBDMCS)

ECSS10K (Sodium sulfate, anhydrous, ACS Grade, Granular 60 Mesh)

July 29, 2011

Procedure

(Developed for tuna, baby food, pineapple and tea)

1) Sample Preparation

- a) Weigh 10 g homogenized sample into a 50 mL QuEChERS tube
- b) Spike with 25 ng/g internal standard: Bisphenol A d16 (BPA d16)
- c) Add 10 mL MeCN
- d) Add the contents of **ECQUEU750CT-MP**
- e) Shake vigorously for 2 min.
- f) Centrifuge at 3500 rpm for 3 min.

2) Clean-up (products without pigments)

- a) Attach **ECPSAC1856** to the vacuum manifold
- b) Add 3 g muffled Na₂SO₄ to the cartridge
- c) Rinse with 2 x 2 mL MeCN
- d) Insert test tubes into the manifold
- e) Load 5 mL of the supernatant to the cartridge
- f) Turn on vacuum to collect extract dropwise
- g) Transfer 1 mL of cleaned extract into a 5 mL test tube
- h) Concentrate to dryness by N₂ at 35 °C

3) Clean-up (products with pigments)

- a) Attach ECPSACB6 to the vacuum manifold
- b) Add 3 g muffled Na_2SO_4 to the cartridge
- c) Rinse with 2 x 2 mL MeCN
- d) Insert test tubes into the manifold
- e) Load 5 mL of the supernatant to the cartridge
- f) Turn on vacuum to collect extract dropwise
- g) Transfer 1 mL of cleaned extract into a 5 mL test tube
- h) Concentrate to dryness by N_2 at 35 °C

4) Derivatization

- a) Add 50 μL pyridine to the dried test tube and vortex
- b) Add 50 μL MTBSTFA/1%TBDMCS and cap the test tube
- c) Heat at 75° C for 30 min.
- d) Cool then concentrate to dryness by N_2 at 35 °C
- e) Reconstitute with 75 μL toluene and 25 μL surrogate: 2 ppm triphenyl phosphate (TPP)
- f) Vortex
- g) Transfer to auto-sampler vial with 100 μL insert
- h) Inject 1 μL to GC/MS

5) GC/MS Analysis

GC/MS

- Agilent 6890N GC coupled with 5975C MSD, equipped with 7683 auto sampler. Chemstation software for data acquisition and analysis. Equivalent instrumentation may be used

Injector

- 1 μL splitless injection at 250 °C, split vent of 30 mL/min at 1 min.

Liner

- 4 mm splitless gooseneck, 4mmID x 6.5mmOD x 78.5mm (UCT #: **GCLGN4MM**)

Glass wool for liner

- Restek® Deactivated Wool

GC capillary column

- Restek® Rxi-5sil MS 30m x 0.25mm x 0.25µm

Oven temperature program

- Initial oven temperature of 100 °C, hold for 1 min.
- Ramp at 20 °C/min. to 300 °C, hold for 1 min.
- Ramp at 40 °C/min. to 320 °C, hold for 2.5 min.

Total run time 15 min. Data acquisition begins at 9 min.

Carrier Gas

- He constant flow 1.2 mL/min.

MSD Conditions

- Aux temperature: 280 °C, MS Source: 230 °C, MS Quad: 150 °C

Simultaneous Scan/SIM:

Scan range: 50-500

SIM:

Group 1: 9.0 min.: 326.1, 325.1 (Triphenyl phosphate)

Group 2: 10.5 min.: 441.3, 456.3, 442.3 (derivatized: Bisphenol A-2TBDMS)

452.4, 470.4, 453.4 (derivatized: Bisphenol A d16-2TBDMS)

Dwell time: 100 ms for all ions

Matrix matched curves are generated with the adjustment of the concentrations in the blank and baby food sample

Experimental Data

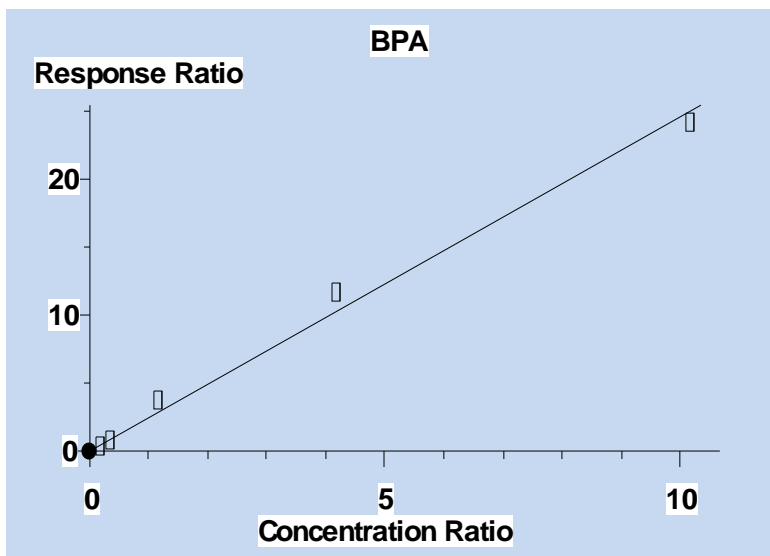
Calculations

C_{blank} and C_{sample} were calculated by equations 1 and 2:

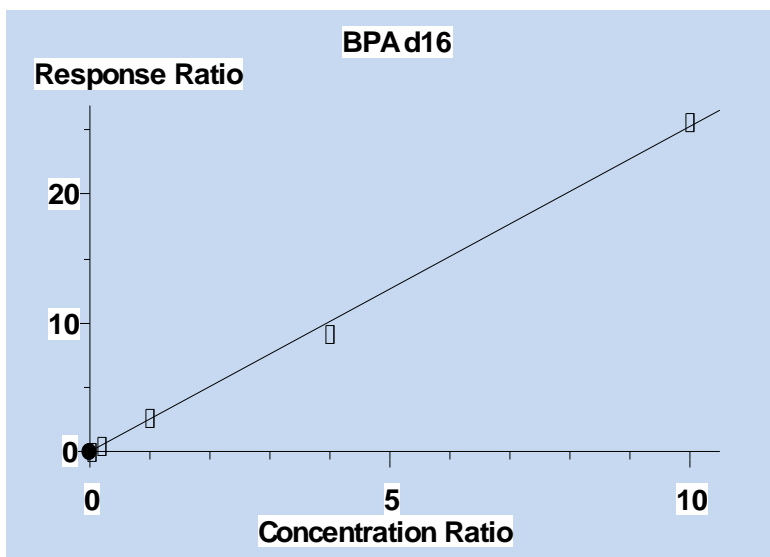
$$\text{Equation 1: } A_{\text{sample}}/A_{\text{blank}} = (C_{\text{sample}} + C_{\text{blank}}) * R_1\% / C_{\text{blank}} * R_1\%$$

$$\text{Equation 2: } A_{\text{spiked sample}}/A_{\text{sample}} = (C_{\text{sample}} + C_{\text{blank}} + C_{\text{spike}}) * R_2\% / (C_{\text{sample}} + C_{\text{blank}}) * R_2\%$$

Tuna: Tuna in pure olive oil. (3oz can)



BPA: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9933$

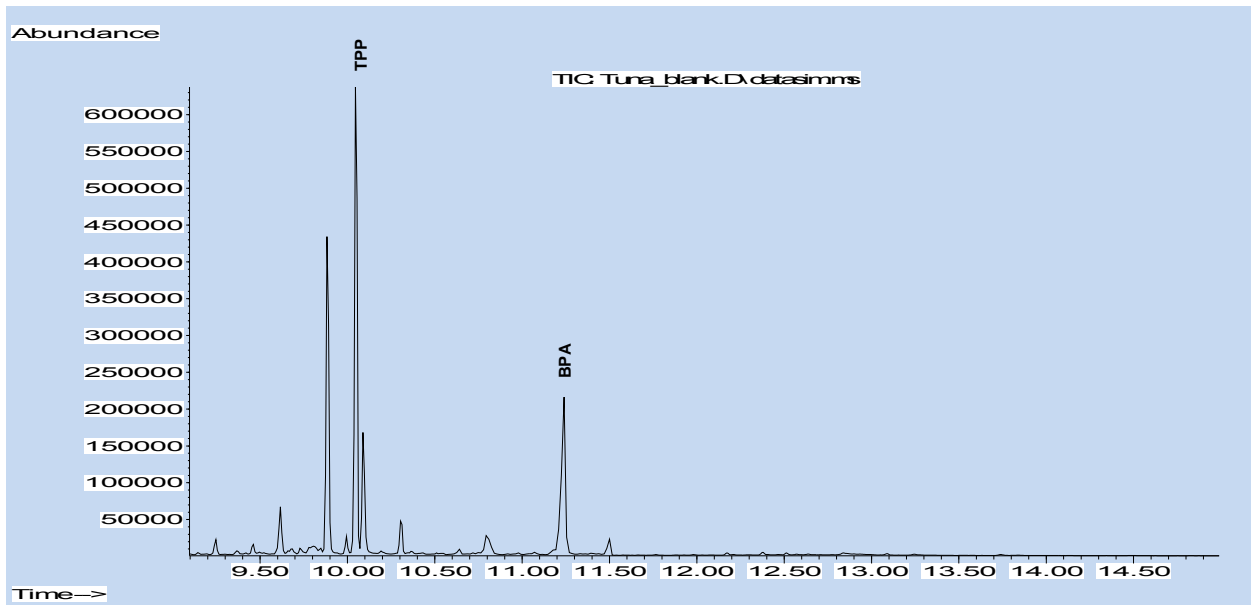


BPA d16: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9983$

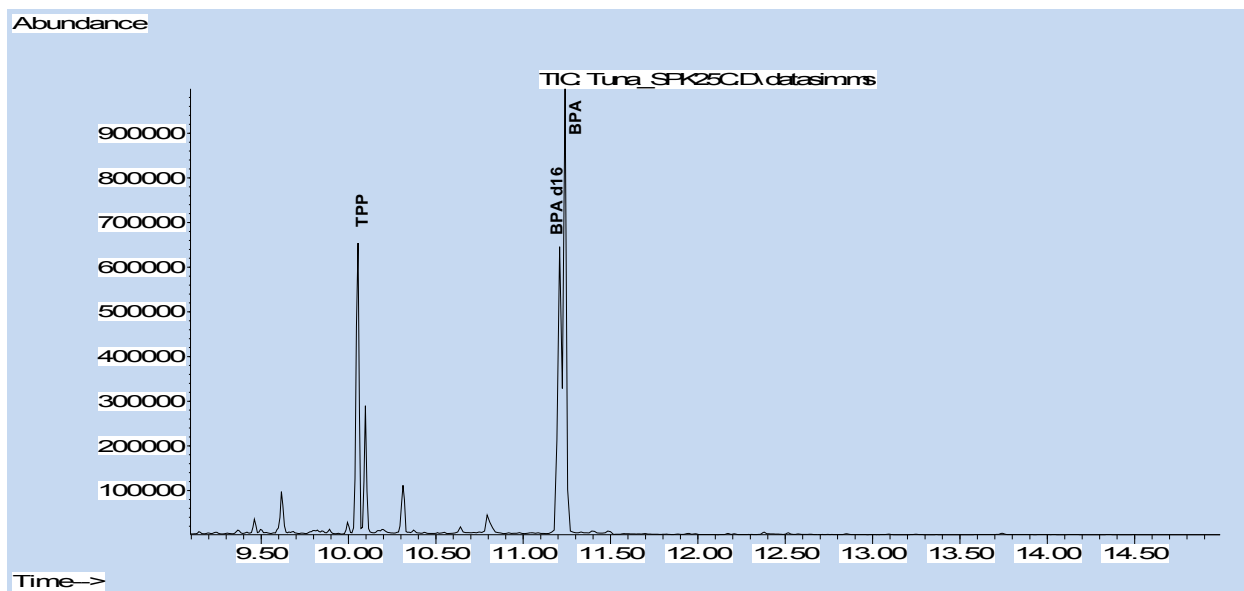
Bisphenol A in Canned Tuna

	BPA in blank	BPA in tuna	Tuna fortified with 25 ng/g BPA	Tuna fortified with 50 ng/g BPA
Analyte	Conc. (ng/g)	Conc. (ng/g)	Recovery% \pm RSD%	Recovery% \pm RSD%
BPA d16	0	0	74.0 \pm 6.4	76.5 \pm 5.9
BPA	0.56	6.64	86.6 \pm 7.5	100 \pm 8.0

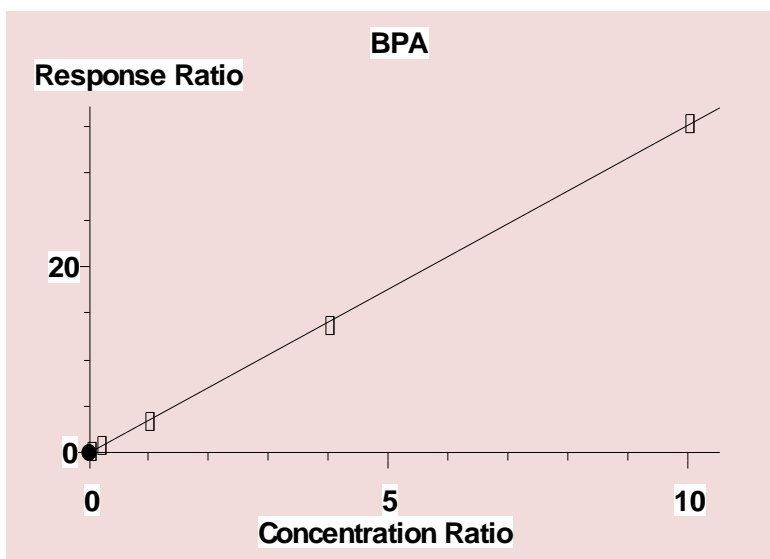
Chromatogram of Canned Tuna



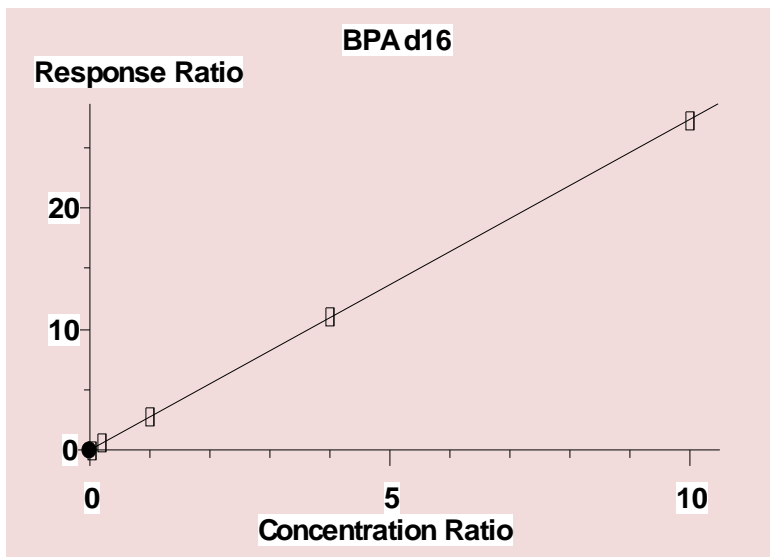
Chromatogram of canned tuna sample fortified with 25 ng/g BPA d16 and BPA



Baby Food: Peaches, Stage 2



BPA: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9998$

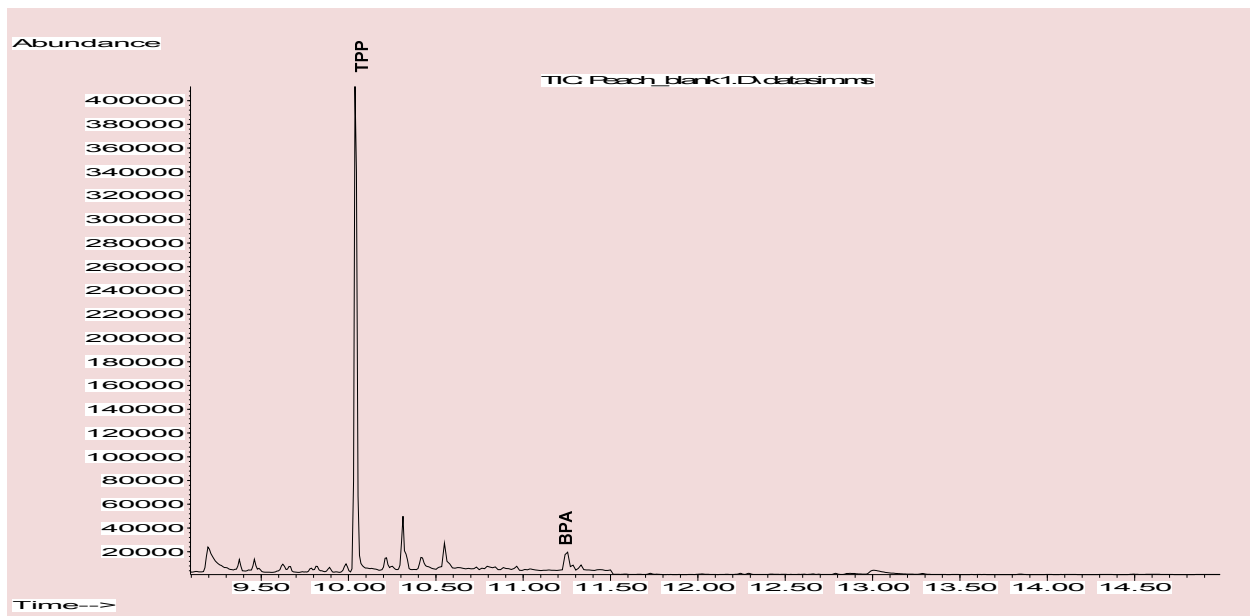


BPA d16: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9999$

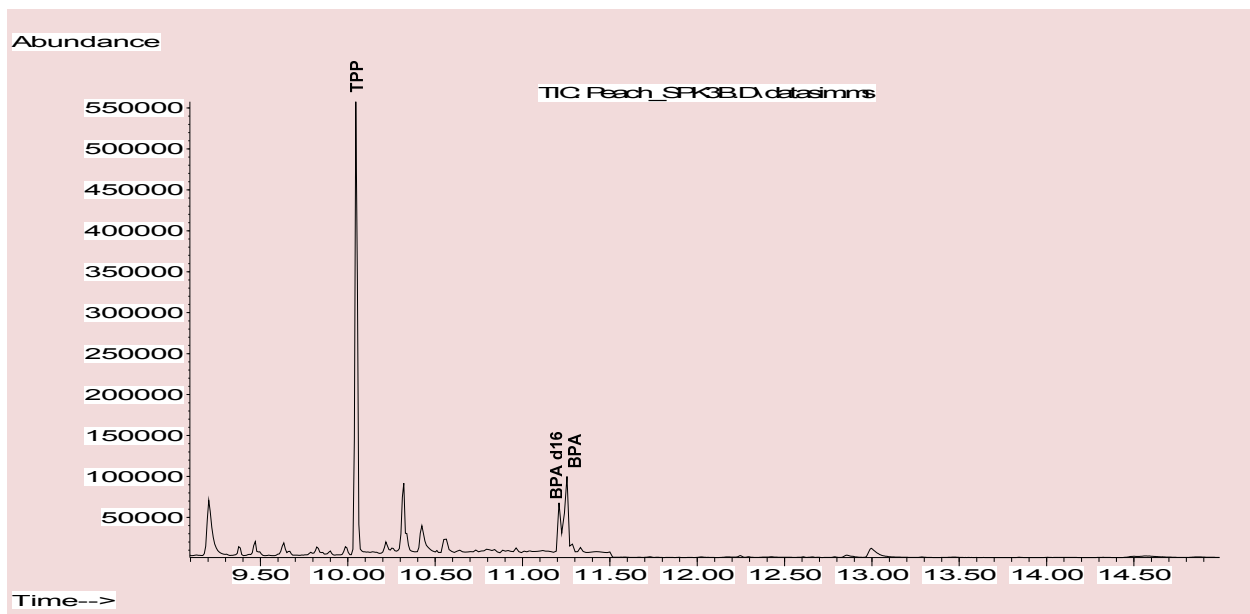
Bisphenol A in Baby Food

	BPA in blank (ng/g)	BPA in baby food (ng/g)	Baby food fortified with 3 ng/g BPA	Baby food fortified with 10 ng/g BPA
Analyte	Conc. (ng/mL)	Conc. (ng/g)	Recovery% \pm RSD%	Recovery% \pm RSD%
BPA d16	0	0	98.0 \pm 4.7	98.8 \pm 7.7
BPA	0.33	< 1 ng/g	99.2 \pm 2.0	95.3 \pm 11

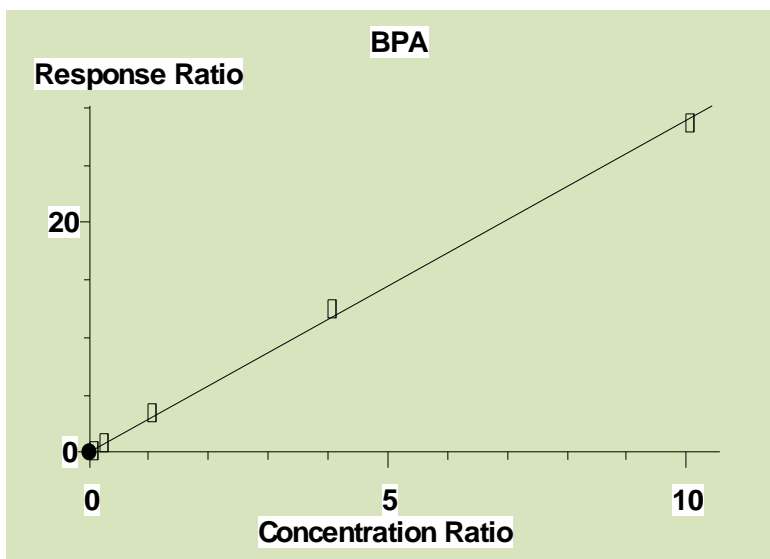
Chromatogram of baby food



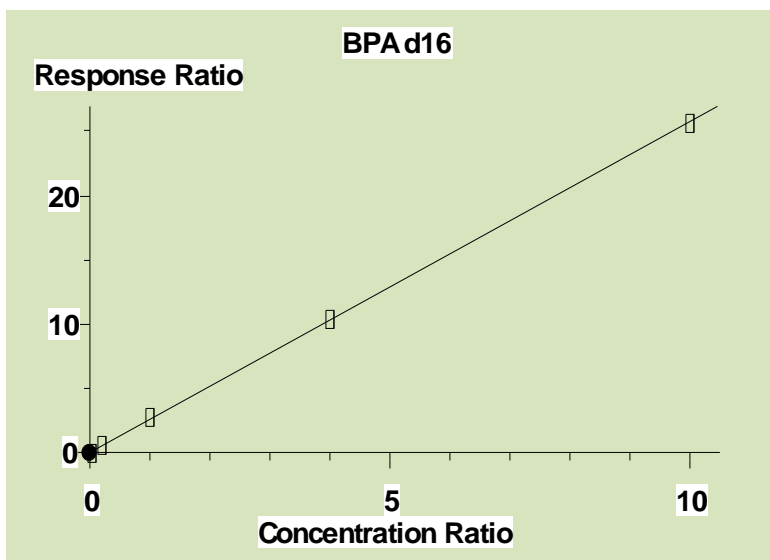
Chromatogram of baby food sample fortified with 3 ng/g BPA d16 and BPA



Pineapple: crushed pineapple in 100% pineapple juice (canned), pH=3



BPA: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9989$

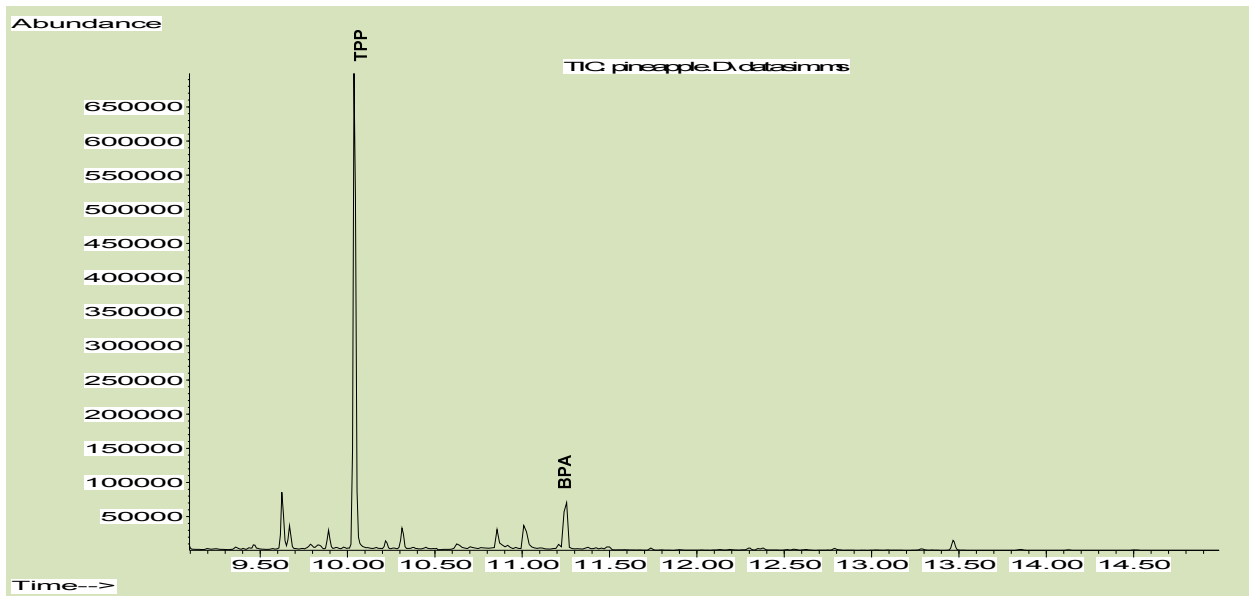


BPA d16: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9999$

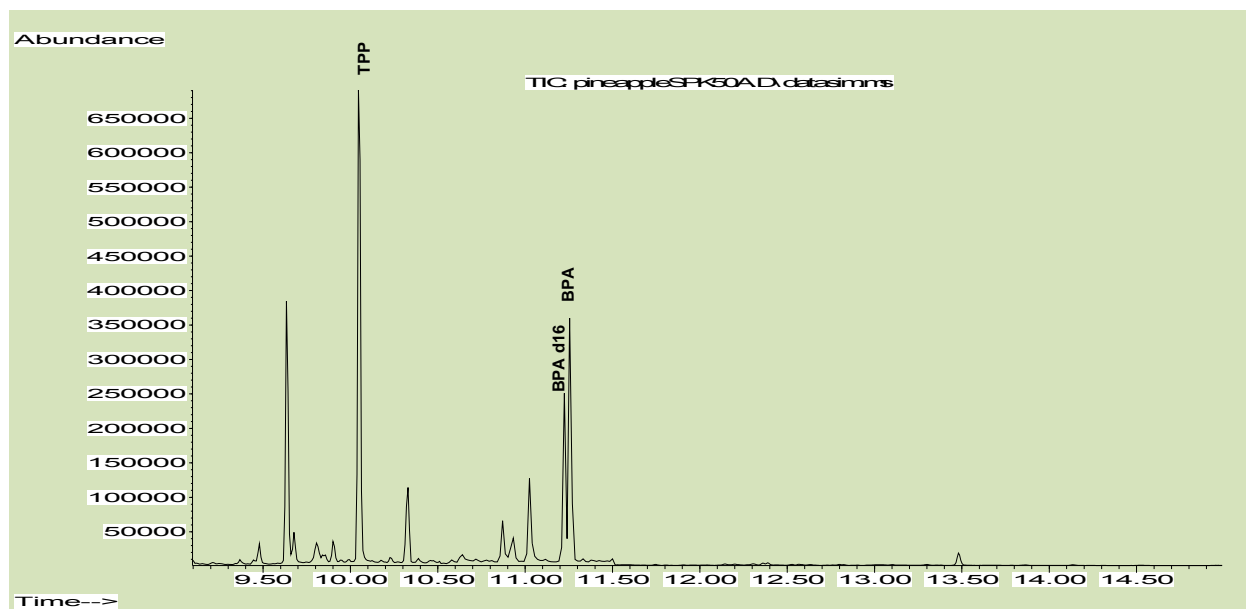
Bisphenol A in Canned Pineapple

	BPA in blank	BPA in pineapple	Pineapple fortified with 5 ng/g BPA	Pineapple fortified with 25 ng/g BPA
Analyte	Conc. (ng/g)	Conc. (ng/g)	Recovery% \pm RSD%	Recovery% \pm RSD%
BPA d16	0	0	112 \pm 2.3	93.4 \pm 6.1
BPA	0.33	1.65	112 \pm 5.7	96.1 \pm 5.7

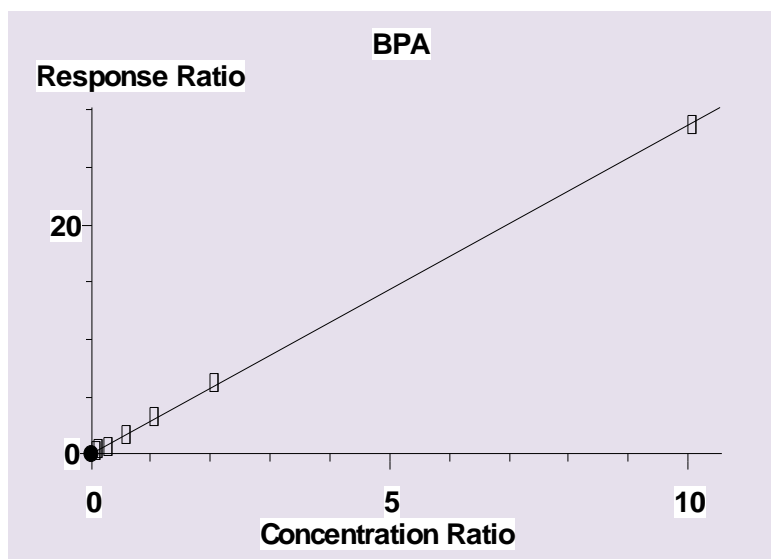
Chromatogram of canned pineapple



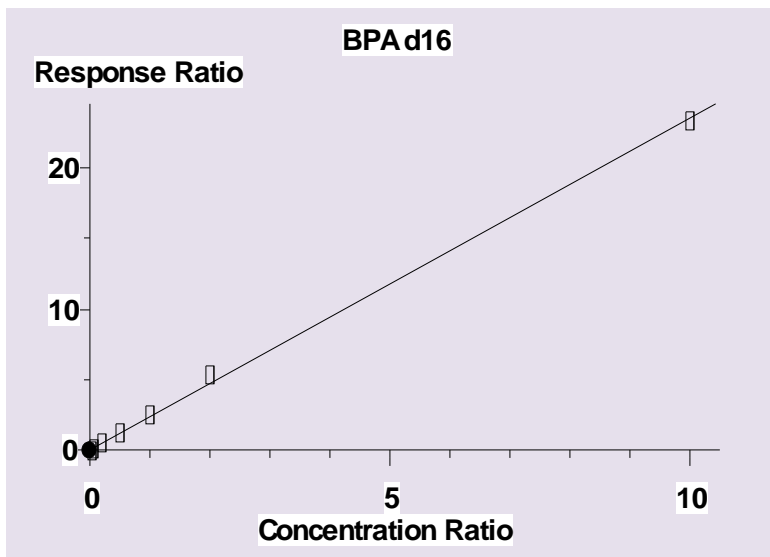
Chromatogram of canned pineapple sample fortified with 5 ng/g BPA d16 and BPA



Tea Sample: Green Tea with Ginseng and Honey (canned), pH=3.5



BPA: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9998$

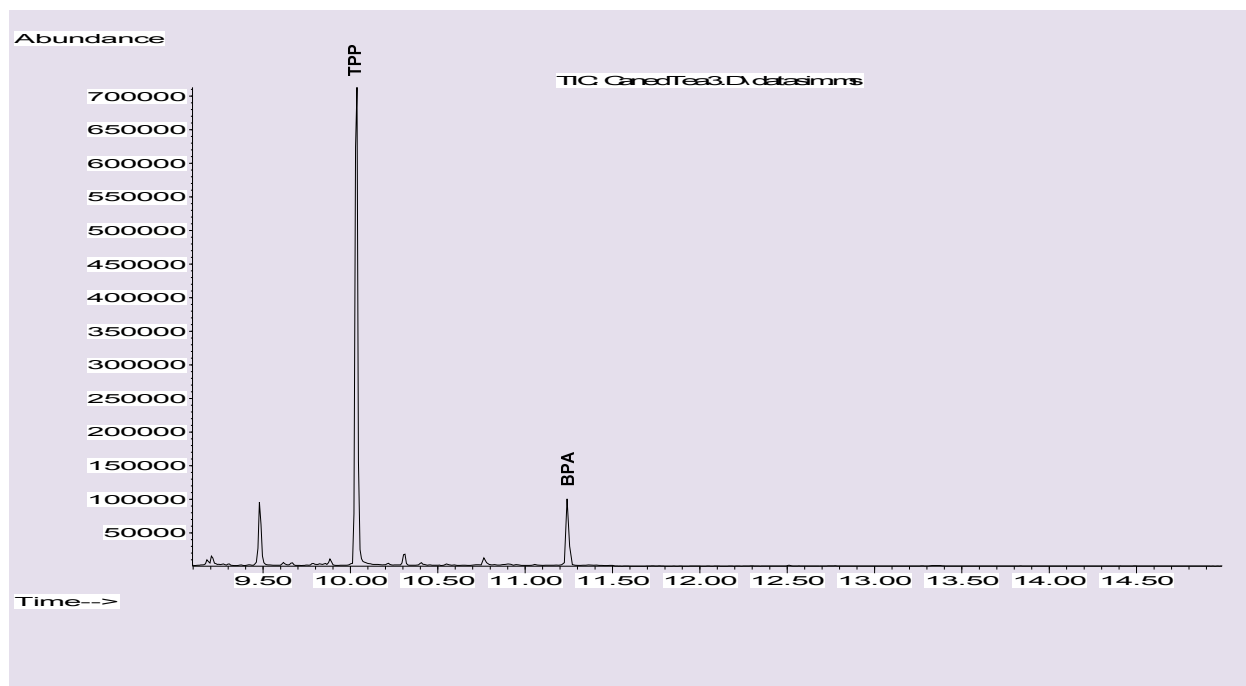


BPA d16: Linear dynamic range: 1-500 ng/mL (conc. per 1 mL extract) $R^2=0.9993$

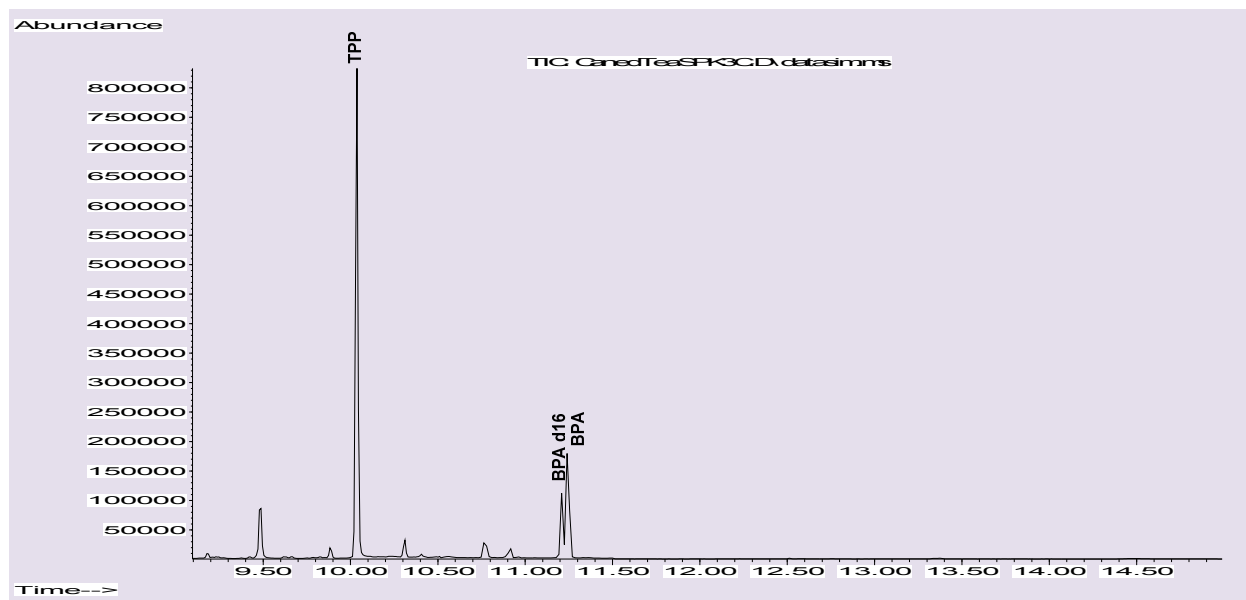
Bisphenol A in Canned Tea

	BPA in blank (ng/g)	BPA in tea (ng/g)	Tea fortified with	Tea fortified with
Analyte	Conc. (ng/mL)	Conc.	Recovery% \pm RSD%	Recovery% \pm RSD%
BPA d16	0	0	120 \pm 5.9	107 \pm 2.7
BPA	0.46	2.28	104 \pm 8.2	90.0 \pm 5.8

Chromatogram of canned tea sample



Chromatogram of canned tea sample fortified with 3 ng/mL BPA d16 and BPA



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