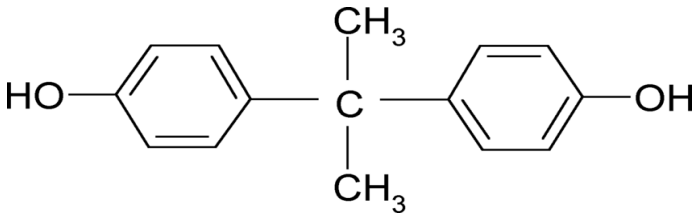
UCT Food Safety Application - Modified QuEChERS Procedure for the Analysis of Bisphenol A in Canned Food Products



UCT Part Numbers

ECQUEU750CT-MP
4000mg $MgSO_4$, 1000mg NaCl, 500mg Na citrate dibasic sesquihydrate, 1000mg Na citrate tribasic dihydrate

ECPSAC1856
500 mg of PSA and 500 mg endcapped C18, 6 ml cartridge

SMTBSTFA-1-1
MTBSTFA w/1% TBDMCS

ECPSACB6
400 mg PSA and 200 mg GCB, 6 ml cartridge

ECSS10K
Na_2SO_4, anhydrous, ACS Grade, Granular 60 Mesh

Abstract:

The concentration of bisphenol monomer in various pigmented and non-pigmented food products can be rapidly determined using a modified QuEChERS procedure. In this procedure, canned tuna, pineapple, peaches, and canned liquid tea are added directly to a centrifuge tube and extracted using a QuEChERS method. Extract cleanup is performed by solid-phase extraction (SPE). GC/MS instrumentation is used to quantify the results with excellent recovery and reproducibility.

Introduction:

Bisphenol A (BPA, CASRN 80-05-7) monomer is used to prepare polycarbonate and epoxy resins. These polymers are often found as coatings on the interior walls of canned food products and are the basis of many bottles used as beverage containers. Though BPA has been known to be an estrogen mimic since the 1930's, only recently have concerns grown as a result of trace quantities of BPA being discovered in foods. Consumers have become alarmed over that last few years because of the potential for BPA to alter normal endocrine function in humans and animals.

Unreacted monomers have been found to leach into foods and are then ingested when the food product is consumed.

A rapid and efficient method was needed to assess the quality of the food supply. This application demonstrates how BPA concentrations in a variety of canned and bottled food products can be accurately assessed using a modified QuEChERS approach.

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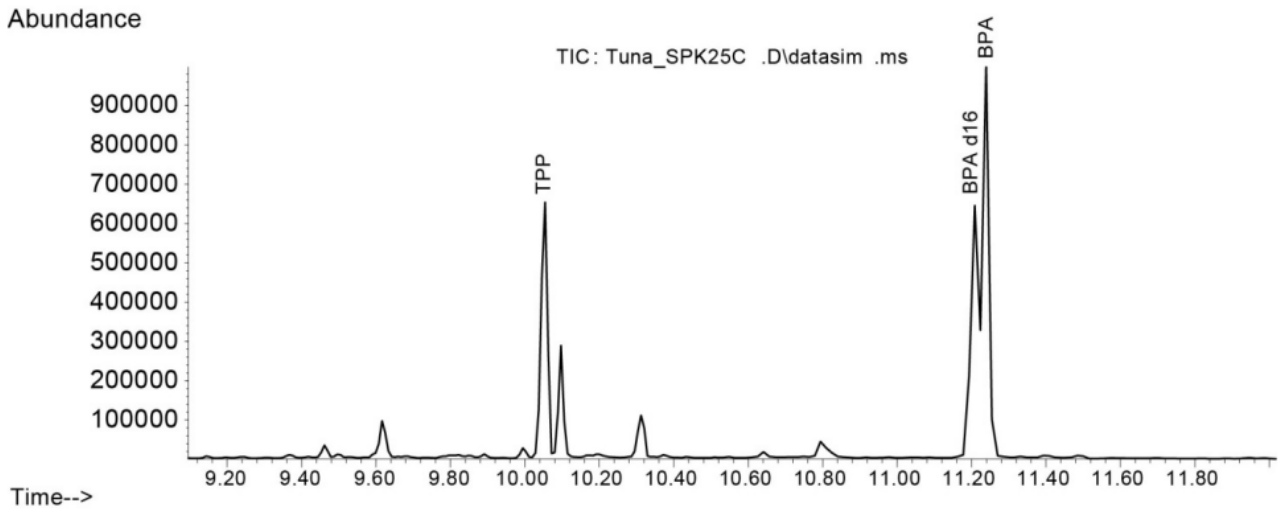
Food samples were purchased from a local grocery store and used directly in this analysis

Food Matrix	
Peaches, for about 6 months old, (glass jar with lined metal lid)	Green Tea with Ginseng and Honey (canned)
Pineapple, crushed in 100% pineapple juice (canned)	Tuna in pure olive oil



Experimental:

Extraction Procedure

Food samples were purchased from a local supermarket. 10g of sample was added directly from its container to a 50 mL centrifuge tube and then spiked with IS bisphenol A d16 (BPA d16) and target analyte bisphenol A (BPA). 10 mL of MeCN and the contents of packet **ECQUEU750CT-MP** were added to the centrifuge tube. The sample was shaken for 2 minutes then placed in a centrifuge at 3500 rpm for 3 minutes.

Clean-up Procedure

SPE cartridges **ECPSAC1856** (for nonpigmented samples) and SPE cartridges **ECPSACB6** (for pigmented samples) were attached to a vacuum manifold and 3 grams of muffled Na_2SO_4 was added to each cartridge. MeCN in two 2 mL portions was used to wash/condition each cartridge. 5 mL of supernatant from the extraction was added to a cartridge and drawn through in a dropwise fashion. One mL of the cleaned extract was concentrated to dryness at 35 °C. The dried extract was derivatized by adding 50 µL pyridine and vortexed. 50 µL MTBSTFA/1%TBDMCS was then added and heated to 75 °C for 30 min before drying again with N_2 at 35 °C. The extract was reconstituted with 75 µL toluene and 25 µL of 2 ppm triphenyl phosphate (TPP) surrogate. After transfer to a GC vial the sample was ready for analysis by GC/MS/SIM using matrix matched calibration.

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GC Method

An Agilent 6890N gas chromatograph coupled with 5975C MSD was equipped with 7683 auto sampler. A Restek low polarity Rxi-5sil MS 30m x 0.25mm x 0.25um capillary column was used along with Agilent Chemstation software for data acquisition and analysis.

GC/MS Parameters for Derivatized Bisphenol A

Instrument Parameters	Information
Injection	1 µL splitless injection at 250 °C, split vent of 30 mL/min at 1 min
Liner	4 mm splitless gooseneck, 4mmID*6.5mmOD*78.5mm (UCT#: **GCLGN4MM**)
Oven Temperature Program	Initial oven T 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 is 15 min
Carrier gas	He @ constant flow of 1.2 mL/min
MSD condition	Aux temperature: 280 °C, MS Source: 230 °C, MS Quad: 150 °C
Simultaneous Scan Range/SIM	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 A2TBDMS) 452.4, 470.4, 453.4 (derivatized: Bisphenol A d16-2TBDMS)
Dwell time	100 ms for all ions

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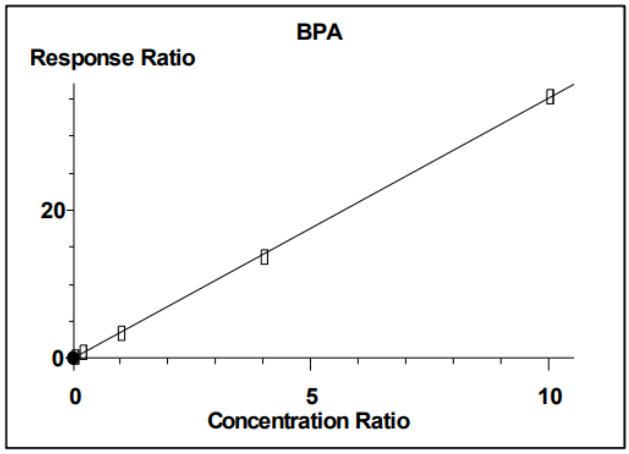
Calibration Curves for Peaches, Tea, Pineapple, and Tuna

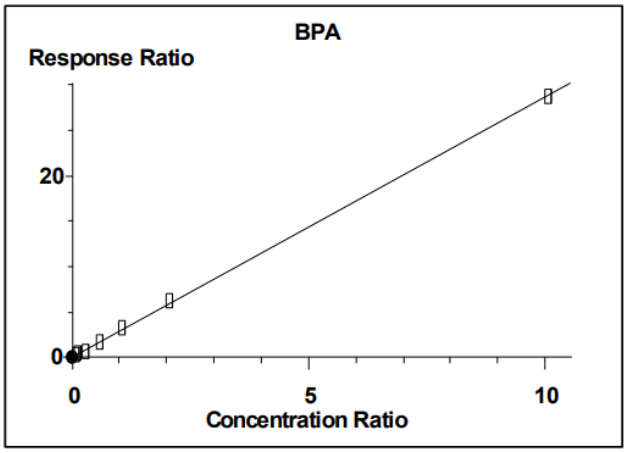
Figure 1

Peaches, Baby Food glass



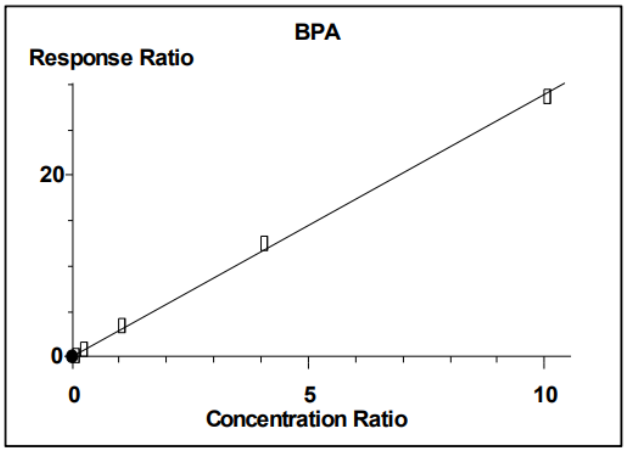
BPA: Linear dynamic range: 1-500 ng/mL in 1 mL extract; R^2=0.9998

Tea, liquid canned



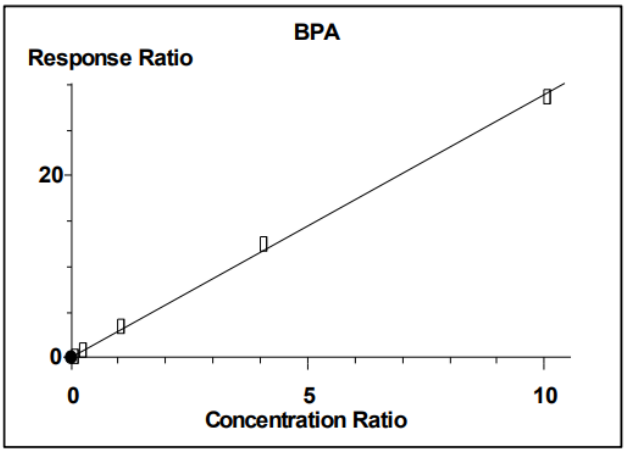
BPA: Linear dynamic range: 1-500 ng/mL in 1 mL extract; R^2=0.9998

Pineapple, canned



BPA: Linear dynamic range: 1-500 ng/mL in 1 mL extract; R^2=0.9989

Tuna, canned



BPA: Linear dynamic range: 1-500 ng/mL in 1 mL extract R^2=0.9989

Table 1

Analyte	BPA blank Conc. ng/mL	BPA found Conc. ng/g	Spike 3 ng/g BPA Recovery % + RSD % (n=3)	Spike 10 ng/g BPA Recovery % + RSD % (n=3)
Peaches, Baby Food Glass				
BPA d16	0	0	98.0 + 4.7	98.8 + 7.7
BPA	0.33	0.33	99.2 + 2.0	95.3 + 11
Pineapple, Canned				
BPA d16	0	0	112 + 2.3	93.4 + 6.1
BPA	0.33	1.65	112 + 5.7	96.1 + 5.7
Tea-Liquid Canned				
BPA d16	0	0	120 + 5.9	107 + 2.7
BPA	0.46	2.28	104 + 8.2	90.0 + 5.8
Tuna, Canned				
BPA d16	0	0	74.0 + 6.4	76.5 + 5.9
BPA	0.56	6.64	86.6 + 7.5	100 + 8.0

Results and Conclusion

Matrix calibration curves were prepared from 1-500 ng/mL. All components showed a correlation coefficient of 0.9989 R^2 or better. BPA was recovered with excellent yields in all food and beverage products within a range of 86.6 to 112%.

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References:

[1] Xiaoyan Wang, Don Shelly and Craig A Perman UCT, LLC, Bristol, PA 19007

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