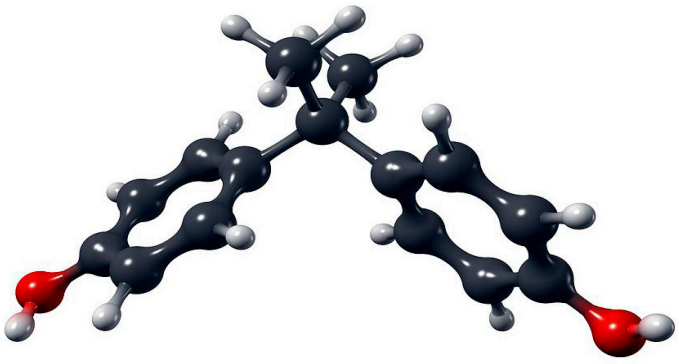
Determination of Bisphenol A in Beverages by QuEChERS and LC-MS/MS



UCT Part Numbers

RFV0050CT
50 mL centrifuge tubes

ECMSSC50CT-MP
Mylar Pouch contains 4000 mg $MgSO_4$ and 1000 mg NaCl

Summary:

Bisphenol A (BPA) is a chemical widely used in manufacturing polycarbonate and epoxy resin, materials that are commonly used in the production of various types of food and beverage containers, including the lining of metal cans. BPA is an endocrine disrupter that can mimic human hormones, recent studies have found that human exposure to BPA results in adverse health effects, such as altered hormone levels, reproductive effects, or increased incidence of diseases, including cancer. The U.S. EPA has set the current safety level of BPA exposure to 50 µg/kg/day. Meanwhile, the FDA has banned the use of BPA in baby bottles, sippy cups, and infant formula packaging.

This application presents a simple, fast, and effective method for the determination of the BPA levels in bottled or canned beverages. BPA in beverages was extracted into acetonitrile (MeCN) by the original QuEChERS procedure.

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Procedure

1. Sample Preparation

a) Pour the entire bottle or can of beverages into 500-mL beakers, stir for 1 hr at high speed to remove the dissolved gases.

2. QuEChERS Extraction

a) Transfer 10 mL of the degassed beverage sample into a 50-mL centrifuge tube (**RFV0050CT**).
b) Add 50 µL of 50 ppm BPA d16 solution as internal standard (IS) to all samples, and appropriate amounts of BPA solution to fortified samples.
c) Add 10 mL of MeCN. Cap and shake for 1 min at 1000 strokes/min using a Spex 2010 Geno-Grinder.
d) Add salts, 4 g $MgSO_4$ and 1 g NaCl from Mylar pouch (**ECMSSC50CTMP**), and vortex for 10 sec to break up salt agglomerates.
e) Shake 1 min at 1000 strokes/min using the Spex Geno-Grinder.
f) Centrifuge at 3830 rcf for 5 min.
g) Transfer 0.5 mL of the supernatant into a 2-mL autosampler vial, add 0.5 mL of reagent water, and vortex for 1 min.
h) The samples are ready for LC-MS/MS analysis

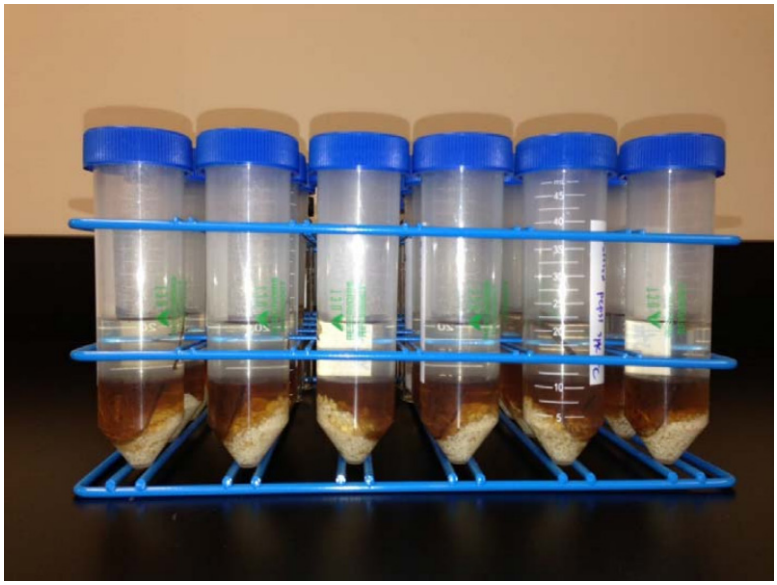

Figure 1. Coke samples extracted by QuEChERS, BPA was extracted into the clear colorless upper layer.

LC-MS/MS Method	
HPLC	Thermo Scientific Dionex UltiMate 3000® LC System
Column	Thermo Scientific, Accucore™ C18, 100 x 2.1 mm, 2.6 µm
Guard Column	Thermo Scientific, Accucore™ C18, 10 x 2.1 mm, 2.6 µm
Column Temperature	40°C
Column Flow Rate	0.400 mL/min
Auto-sampler Temperature	10°C
Injection Volume	20 µL
Gradient Program	**Mobile Phase A:** 0.1 % ammonia in water; **B:** methanol

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Gradient Program		
Time (min)	Mobile Phase A (%)	Mobile Phase B (%)
0	95	5
2	95	5
4	10	90
8	10	90
8.1	95	5
10	95	5

Divert mobile phase to waste from 0 - 3 and 8.5 - 10 min to prevent ion source contamination.

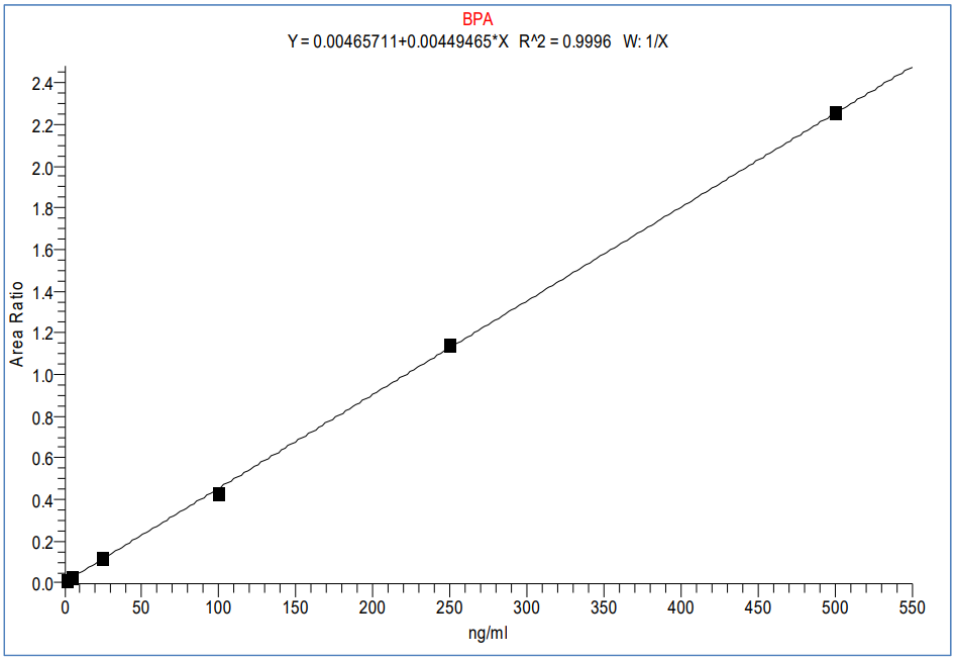
MS Parameters	
Polarity	ESI^-
Spray voltage V	4000 V
Vaporizer Temperature	350 °C
Ion transfer capillary temperature	300 °C
Sheath gas pressure	30 arbitrary units
Auxiliary gas pressure	55 arbitrary units
Q1 and Q3 peak width (FWHM)	0.4 and 0.7 Da
Collision gas and pressure	Ar at 1.5 mTorr
Scan type	SRM
Cycle time	0.5 sec
Acquisition method	EZ Method

SRM Transitions							
Compound	Rt (min)	Precursor ion	Product ion 1	CE 1	Product ion 2	CE 2	S-lens (V)
BPA d16	5.53	241.08	222.34	21	141.69	29	62
BPA	5.54	227.02	211.53	20	132.62	25	60

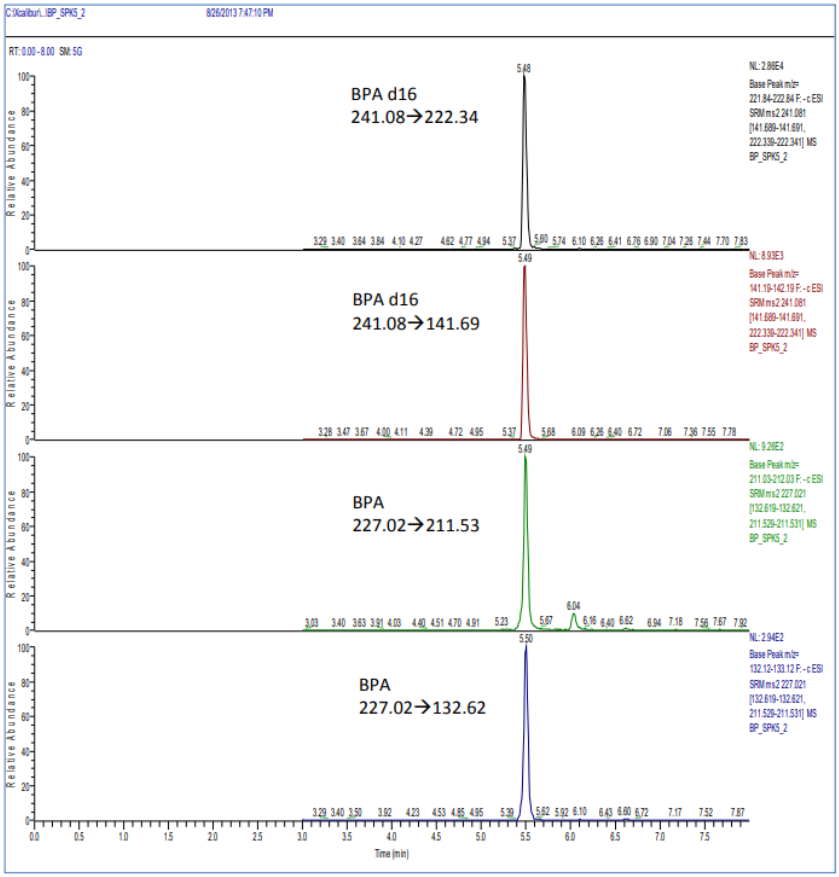
Results:

Recovery and RSD% Data from Spiked Coke Samples					
		Spiked at 5 ng/mL		Spiked at 100 ng/mL	
Samples	BPA Detected	Recovery %	RSD% (n=5)	Recovery %	RSD% (n=5)
bottled coke	< 1 ng/mL	94.9	8.5	96.9	6.2

Calibration Curve (Dynamic Linearity Range: 1 – 500 ng/mL)



Chromatogram of a Bottled Coke Sample Fortified with 5 ng/mL of BPA (IS: 250 ng/mL of BPA d16)



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