CASE STUDY

Quantitation of Bisphenol A (BPA) Leaching from Plastic Drinking Bottles

OBJECTIVE

The purpose of this work was to quantify the levels of Bisphenol A (BPA) in a polycarbonate water bottle through possible stages of degradation.

ANALYTICAL STRATEGY

The water bottle was tested in an as-received state, after three dishwashing cycles, and after heating in an oven at 130°C for 2 days. QTOF-LCMS was used to quantify the amount of BPA in the samples.

CONCLUSIONS

The as-received sample was found to contain 59 ppm of BPA. Subjecting the bottle to three dishwashing cycles increased this concentration to 78 ppm. Heating the water bottle at 130°C for 2 days resulted in a 10 fold increase to 586 ppm.

Read the following report to see the full analysis.

Final Report

Jordi Labs LLC
Case Study
Quantitation of Bisphenol A (BPA)
Leaching from Plastic Drinking Bottles

Date: 11/12/10

Prepared by:
Dr. Mark Jordi
President
Jordi Labs LLC

Report Number: J5217

November 12, 2010

Client Name
Company Name
Address

Dear Valued Client:

Please find enclosed the test results for your samples described as:

1. *Polycarbonate Water Bottle*

The following tests were performed:

1. Quadrupole Time-of-Flight LCMS (QTOF-LCMS)

Objective

The purpose of this work was to quantify the levels of Bisphenol A (BPA) in a polycarbonate water bottle. Quantification is the science of determining the quantity of a component in a sample. This is one of the many analytical services offered at Jordi Labs.

Summary of Results

Samples were collected from a reusable polycarbonate water bottle in the as-received state, after three dishwashing cycles, and after heating in an oven at 130°C for 2 days. The plastic material was cryoground and Bisphenol A (BPA) was extracted in methanol. QTOF-LCMS was then used to quantify the amount of BPA in the samples.

The amount of extracted BPA in each sample can be found in **Table 2**. The as-received sample was found to contain 59 ppm of BPA. Subjecting the bottle to three dishwashing cycles increased this concentration to 78 ppm. Heating the water bottle at 130°C for 2 days resulted in a 10 fold increase to 586 ppm. Analysis of a control showed 106% percent recovery of the spiked quantity indicating the method is suitable for the intended purpose.

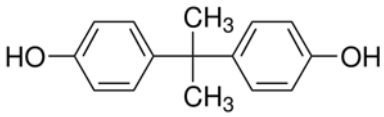
Individual Test Results

A summary of the individual test results is provided below. All accompanying data, including spectra, has been included in the data section of this report.

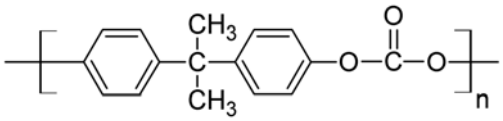
Background

Bisphenol A (BPA), along with phosgene, are the two components reacted to form polycarbonate, a widely used plastic for household items. Polycarbonate has been used for many years in the food and beverage packaging industry, most notably in the production of reusable water bottles. Recently, the scientific community has brought into question the safety of BPA exposure.[1] This has resulted in a need for a method for quantification of BPA levels in these reusable water bottles. Additionally, the effect of thermal treatment, such as dishwashing, can also be examined.

Definitions



Bisphenol A (BPA)



Polycarbonate (PC)

Sample Preparation

Polycarbonate samples were collected from the as-received bottle, after three dishwashing cycles, and after the bottle was heated in an oven at 130°C for 2 days. Each polycarbonate sample was cryoground to ensure sample homogeneity.

Soxhlet extraction with methanol was then carried out on 2g ± 1mg of the ground material for 72 hours. A fourth sample was prepared from the ground untreated polymer that was spiked with 5ml of a 0.1 mg/ml BPA/methanol solution during the soxhlet extraction. Each extracted sample was completely dried and the mass of the residual solids was measured. The residual soxhlet masses can be found in **Table 1**.

Table 1: Residual Mass of Extracted Components

Sample	Residual Mass (mg)	% Extractables
Untreated Bottle	4.2	0.210
Untreated (spiked)	6.1	0.305
Dishwasher 3x	4.9	0.245
Oven	7.6	0.380

Residual mass is from the extract of a 2g sample

QTOF-LCMS

The dried extraction samples were brought back up in 5ml of a methanol solution containing 0.1 mg/ml Palmitic Acid as an internal standard. Standard solutions of BPA in methanol (with the same concentration internal standard) were also prepared. Solution concentrations of 1, 0.5, 0.1, 0.02, 0.01, 0.001, and 0.0001 mg/ml were used to construct a calibration curve. The calibration curve, fit equation, and R^2 value can be found in **Figure 1**.

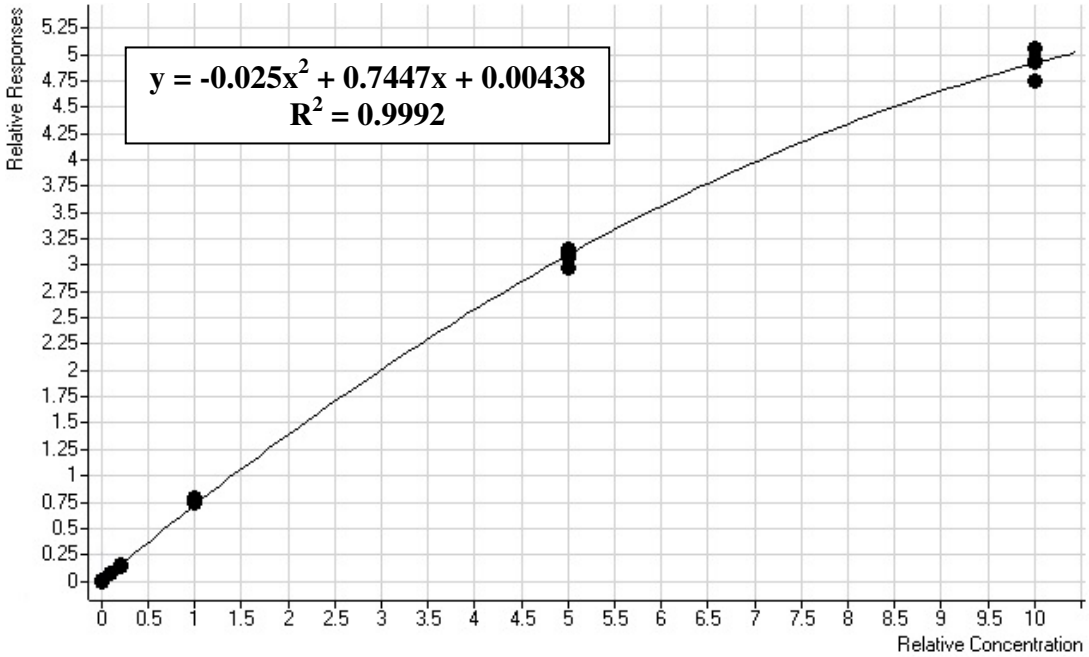


Figure 1: QTOF-LCMS calibration curve for Bisphenol A

Each sample was run four times to obtain an average value for the concentration of BPA in the extract solution. **Table 2** provides the individual and average concentrations of the extraction solutions as well as the total mass of BPA collected from the 2g of cryoground polycarbonate.

The oven-heated sample released the most BPA (factor of 10 increase) compared to the untreated sample (1.172 mg as compared to 0.12 mg). This is likely attributed to slow thermal degradation of the BPA at 130°C over the two days. The dishwasher sample released approximately 33% more BPA than the untreated sample.

It is possible to use the extracted mass of BPA and the starting mass of polycarbonate to calculate the BPA concentration in the solid. The BPA concentration in percent and ppm are shown in **Table 2**. The solid samples are approximately 59, 78, and 586 ppm for the untreated, dishwasher, and oven-treated samples, respectively.

As a quality control measure, a sample of the as-received material was spiked with .5 mg of BPA and analyzed along with the samples. The percent recovery for the spike value was determined to be 106% indicating that the method is suitable for the intended purpose.

Table 2: QTOF-LCMS Results

Sample	#	Concentration in Extract Solution (mg/ml)	Mass of BPA in Sample (2g)	% BPA	ppm BPA
Untreated Bottle	1	0.023	0.117	0.0059%	59
	2	0.024			
	3	0.021			
	4	0.025			
Untreated Bottle (spiked)	1	0.133	*0.647	*0.0324%	*324
	2	0.132			
	3	0.125			
	4	0.128			
Dishwasher	1	0.031	0.156	0.0078%	78
	2	0.029			
	3	0.033			
	4	0.032			
Oven	1	0.242	1.172	0.0586%	586
	2	0.259			
	3	0.223			
	4	0.213			

* This sample was spiked with .5mg of BPA as a quality control measure.

Analysis Conditions

This section of a Jordi report provides information on the methods used including instrument type, temperatures, solvents, sample preparation, etc. The specific conditions have been removed for this case study.

Closing Comments

Deformulation of an unknown material is intended to provide a best estimate of the chemical nature of the sample. All chemical structures are supported by the evidence presented but are subject to revision upon receipt of additional evidence. Additional factors such as material processing conditions may also affect final material properties.

Jordi Labs specializes in polymer testing and has 30 years experience doing complete polymer deformulations. We are one of the few labs in the country specialized in this type of testing. We will work closely with you to help explain your test results and solve your problem. We appreciate your business and are looking forward to speaking with you concerning these results.

Sincerely,

Mark Jordi

Mark Jordi, Ph. D.
President
Jordi Labs LLC

References

1. vom Saal FS, Akingbemi BT, Belcher SM, *et al* (2007). "Chapel Hill bisphenol A expert panel consensus statement: integration of mechanisms, effects in animals and potential to impact human health at current levels of exposure". *Reprod. Toxicol.* **24** (2): 131–8