Analysis of Bisphenol A and Diphenyl Carbonate in Polycarobonate Container by UHPLC

Polycarbonate (PC) is a transparent noncombustible thermoplastic and used in dishes and containers for microwave heating. The main raw materials for the PC are bisphenol A (BPA), and diphenyl carbonate (DPC) and phenol (PH) and p-tert-butylphenol (BtPH) are used as the polymerizaton modifiers. In the Food Sanitation Act, to prevent the health hazards caused by the elution of chemical substances from these raw materials to food and drink, the standards for the materials and eluates are specified and the HPLC method for BPA (incl. PH and BtPH) and DPC in PC containers are described. This time, the specified standard HPLC method (see AS/LC-052) was converted to a UHPLC method with ultra high-performance liquid chromatograph ChromasterUltra Rs DAD system. By using this method, the conventional analysis time (60 min) can be shortened to about 1/8 (7 min), and the mobile phase consumption can also be reduced to 1/10. It was also possible to obtain the analysis results with good reproducibility even by a short-time gradient analysis, and the results are introduced here.

Material Test (Analysis of Standard Solution)

- ✓**The standard stock solutions are prepared by weighing 10 mg each of BPA, PH, BtPH, and DPC, and adding methanol to make the volume to 100 mL (100 ug/mL each).**
- ✓**The standard solutions are prepared by transferring 1, 2, 3, 4, and 5 mL of the standard stock solutions into separate 20 mL volumetric flasks and adding purified water to make the volume to 20 mL (5, 10, 15, 20, and 25 µg/mL).**

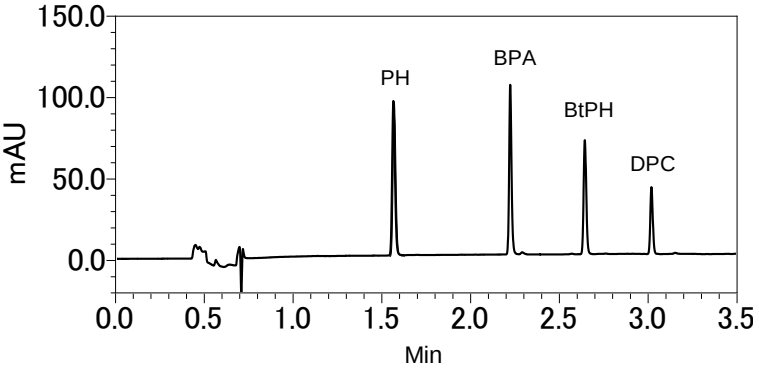

Figure 1 Chromatogram of Standard Solution (5 µg/mL)

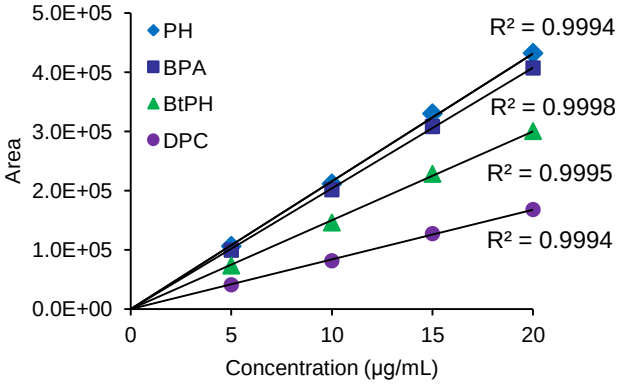

Figure 2 Calibration Curve

Table 1 Analytical Conditions

Column	LaChromUltra II C18 (1.9 µm) 3 mm I.D. X 100 mm
Mobile phase	(A) H_2O (B) CH_3CN (v/v) 30% B (0 min)→100% B (3.5-4.5min) →30% B (4.51-7 min)
Flow rate	0.8 mL/min
Column temperature	40°C
Detection wavelength	UV 217 nm (DAD)
Injection vol.	5 µL

Table 2 Reproducibility (n=6) for Standard Solution (5 µg/mL)

Component	PH	BPA	BtPH	DPC
Retention time (min)				
Mean	1.572	2.230	2.650	3.026
%RSD	0.085	0.068	0.065	0.080
Area				
Mean	105754	99214	73175	40769
%RSD	0.168	0.115	0.065	0.135

- ✓ **The system pressure (Max) under the analytical conditions shown in Table 1 and Table 5 is 66 MPa.**
- ✓ **The calibration curves of BPA, PH, BtPH, and DPC (5, 10, 15, 20, 25 µg/mL) showed correlation coefficients (R^2) of 0.9994 or higher, indicating good linearity (Figure 2).**
- ✓ **Table 2 shows the reproducibility (n=6) for 5 µg/mL. A good result was obtained for each component. Highly reliable analysis can be performed even by a short-time gradient analysis.**

Material Test (Analysis of Sample Solution)

- ✓ **The standard for the material test of PC containers specified in the Food Sanitation Acts is not more than 500 µg/g for the total of BPA, PH, and BtPH and not more than 500 µg/g for DPC.**
- ✓ **Unused PC containers were used as the samples. The samples were prepared in accordance with the procedure described under the material test of the Food Sanitation Act (Figure 3) so as to extract the sample solutions.**
- ✓ **The calculation formula for BPA (incl. PH and BtPH) and DPC in materials.**

$$\text{Content in material (µg/g)} = \frac{\text{Sample solution concentration (µg/mL) X 20 (mL)}}{\text{Sample weight (g)}}$$

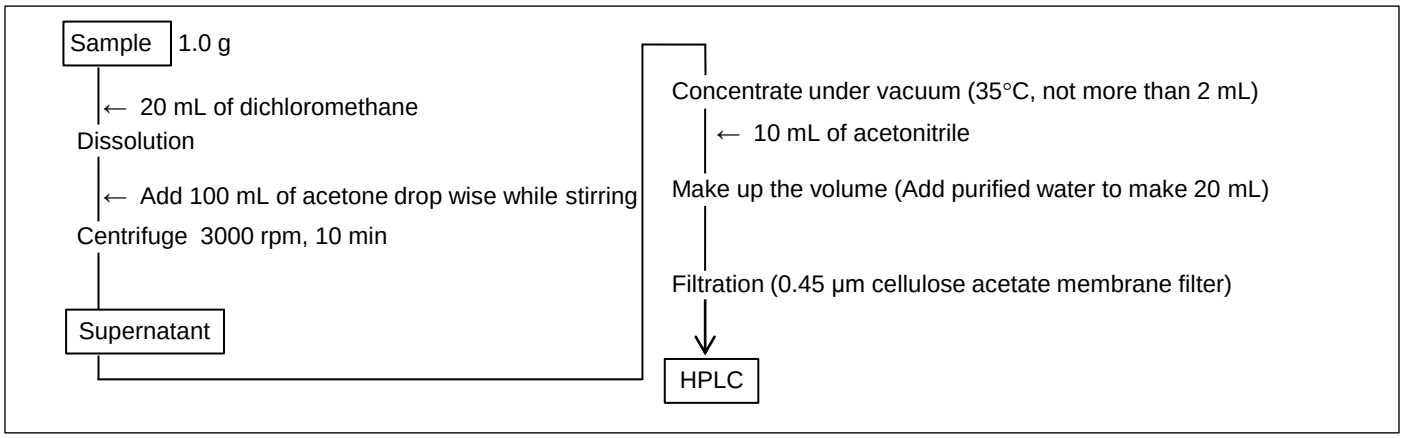

Figure 3 Preparation Method for Material Test

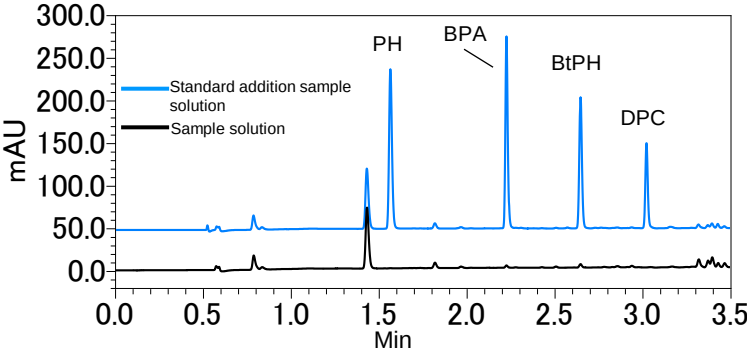

Figure 4 Chromatogram of Sample Solution

Table 3 Quantitative Analysis Results

		Sample solution		Standard addition sample solution (Addition concentration: 10 µg/mL)		
		Sample solution concentration (µg/mL)	Content (µg/g)	Sample solution concentration (µg/mL)	Content (µg/g)	Recovery rate (%)
Sample (1.151 g)	PH	n.d.	n.d.	9.885	171.764	98.9
	BPA	0.125	2.172	10.489	182.259	103.6
	BtPH	0.293	5.091	10.661	185.248	103.7
	DPC	n.d.	n.d.	10.494	182.346	104.9

- ✓ **The result obtained from the material test for PC containers showed that BPA (incl. PH and BtPH) was less than the specified standard and DPC was not detected (Figure 4, Table 3).**
- ✓ **When the recovery rates were determined by adding BPA, PH, BtPH and DPC at the concentration of 10 µg/mL each, the rates were found to be 98.9-104.9%, indicating good results (Figure 4, Table 3).**

Elution Test (Analysis of Standard Solution)

- ✓ **The standard solutions are prepared by transferring 2 mL each of the material test standard solutions (5, 10, 15, 20, and 25 µg/mL) to separate 20 mL volumetric flasks and making the volume to 20 mL by adding purified water (0.5, 1, 1.5, 2, 2.5 µg/mL).**
- ✓ **The target component for the elution test is BPA (incl. PH and BtPH). DPC is shown as a reference data.**

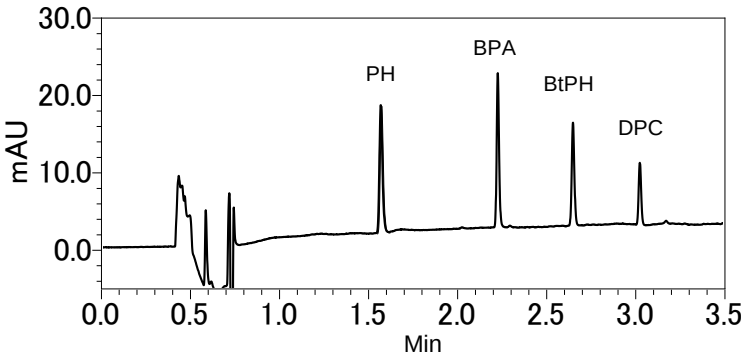

Figure 5 Chromatogram of Standard Solution (0.5 µg/mL)

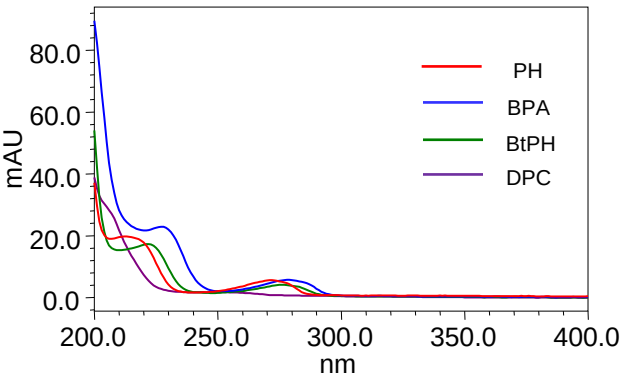

Figure 6 Absorption Spectrum of Standard Solution (0.5 µg/mL)

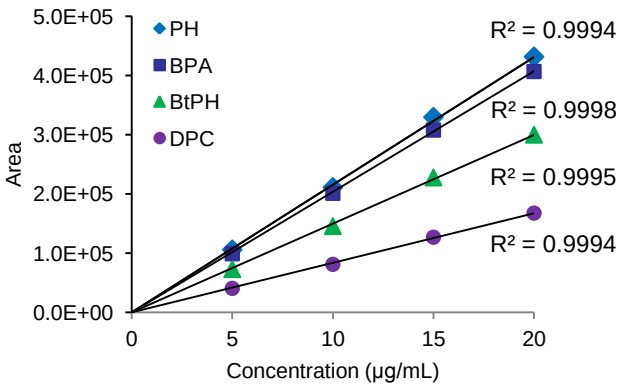

Figure 7 Calibration Curve

Table 4 Reproducibility (n=6) of Standard Solution

Component	PH	BPA	BtPH	DPC
Retention time (min)				
Mean	1.574	2.231	2.653	3.028
%RSD	0.040	0.028	0.050	0.044
Area				
Mean	19895	18926	13944	7856
%RSD	0.310	0.327	0.417	0.350

Table 5 Analytical Conditions

Column	LaChromUltra II C18 (1.9 µm) 3 mm I.D. X 100 mm
Mobile phase	(A) H_2O (B) CH_3CN (v/v) 30% B (0 min)→100% B (3.5-4.5min) →30% B (4.51-7 min)
Flow rate	0.8 mL/min
Column temperature	40°C
Detection wavelength	UV 217 nm (DAD)
Injection vol.	10 µL

- ✓ **The calibration curves for those components (0.5, 1, 1.5, 2, 2.5 µg/mL) showed correlation coefficients (R^2) of 0.9994 or higher, indicating good linearity (Figure 7)**
- ✓ **Table 4 shows the reproducibility (n=6) for 0.5 µg/mL. A good result was obtained for each of the components.**

Elution Test (Analysis of Sample Solution)

- ✓ **The standard for the elution test of PC containers specified in the Food Sanitation Acts is not more than 2.5 µg/g for the total of BPA, PH, and BtPH.**
- ✓ **The elution test was performed with reference to "Implements or containers/packages that contact with food fats and oils or fat-rich foods." Unused PC containers were used as the samples. For the sample preparation, the procedures of the solvent switch from heptane to acetonitrile and concentration[1)] were added to the procedure conforming to the elution test described in the Food Sanitation Act (Figure 8).**

1) 2010 Method of Analysis in Health Science, Pharmaceutical Society of Japan, p. 622

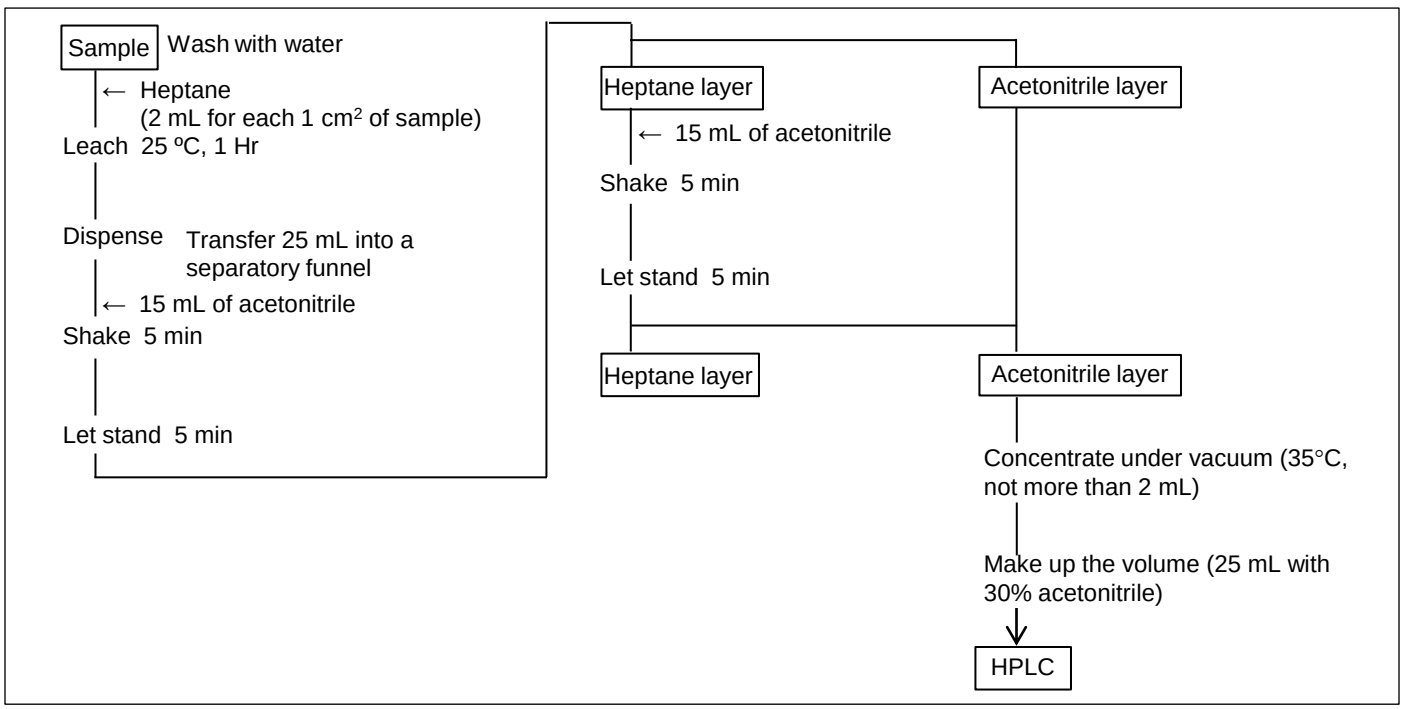

Figure 8 Preparation Method for Elution Test

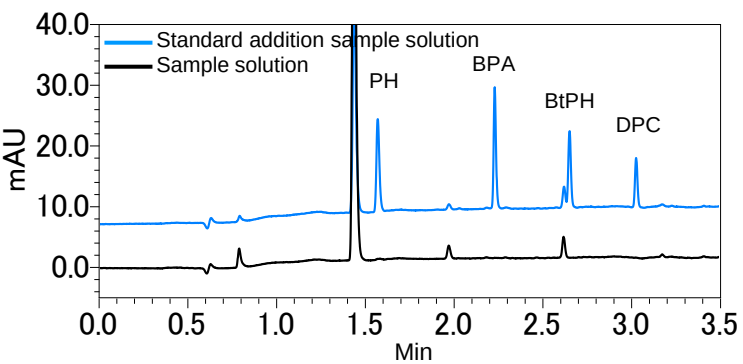

Figure 9 Chromatogram of Sample Solution

Table 6 Quantitative Analysis Results

		Sample solution	Standard addition sample solution (Addition concentration: 0.5 µg/mL)	
		Sample solution concentration (µg/mL)	Sample solution concentration (µg/mL)	Recovery rate (%)
Sample	PH	n.d.	0.488	97.7
	BPA	n.d.	0.503	100.6
	BtPH	n.d.	0.488	97.6
	DPC	n.d.	0.493	98.5

- ✓ **The result of the elution test for the PC containers showed that BPH (incl. PH and BtPH) was not detected (Figure 9, Table 6).**
- ✓ **When the recovery rates were determined by adding BPA, PH, and BtPH at the concentration of 0.5 µg/mL each, good results were obtained with the rates of 97.6–100.6% (Figure 9, Table 6).**

< Main System Configuration > ChromasterUltra Rs DAD System
6170 Binary Pump, 6270 Autosampler, 6310 Column Oven, 6430 Diode Array Detector

NOTE: These data are an example of measurement; the individual values cannot be guaranteed.

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