

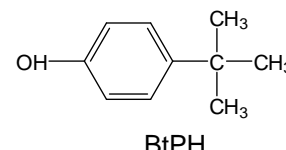
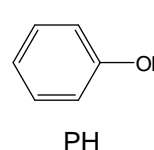
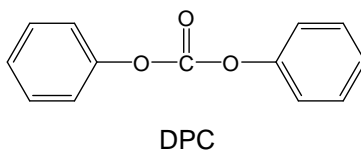
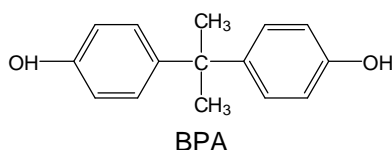


Analysis of Bisphenol A and Diphenyl Carbonate in Polycarbonate Container by HPLC

Polycarbonate (PC) is a transparent noncombustible thermoplastic and has excellent heat-resistance, acid-resistance, and impact-resistance properties. As a result, it is used in dishes and containers for microwave heating. The main raw materials for PC are bisphenol A (BPA), and diphenyl carbonate (DPC), and phenol (PH) and p-tert-butylphenol (BtPH) are used as polymerization 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. This time, based on the Food Sanitation Act “Standards and Criteria for Food and Food Additives, etc.,” the material test and elution test for PC containers were performed.



High-Performance Liquid Chromatograph Chromaster®



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 µg/mL each).
- ✓ The standard solutions are prepared by transferring 1, 2, 3, 4, and 5 mL of the standard stock solution to separate 20 mL volumetric flasks and adding purified water to make the volume to 20 mL (5, 10, 15, 20, and 25 µg/mL).

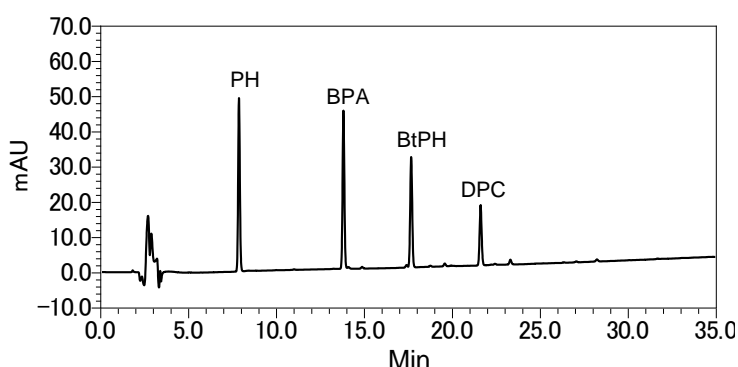


Figure 1 Chromatogram of Standard Solution (5 µg/mL)

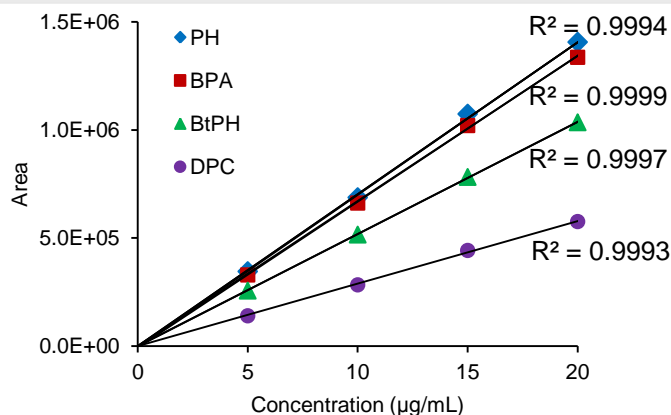


Figure 2 Calibration Curve

Table 1 Analytical Conditions

Column	LaChrom II C18 (5 µm) 4.6 mm I.D. X 250 mm
Mobile phase	(A) H ₂ O (B) CH ₃ CN (v/v) 30% B (0 min)→100% B (35-45 min) →30% B (45.1-60 min)
Flow rate	1.0 mL/min
Column temperature	40°C
Detection wavelength	UV 217 nm (DAD)
Injection vol.	20 µL

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

Component	PH	BPA	BtPH	DPC
Retention time (min)				
Mean	7.917	13.859	17.714	21.662
%RSD	0.051	0.023	0.018	0.014
Area				
Mean	342788	329071	256259	140201
%RSD	0.121	0.442	0.434	0.280

- ✓ The calibration curves of BPA, PH, BtPH, and DPC (5, 10, 15, 20, 25 µg/mL) showed a correlation coefficient (R²) of 0.9993 or higher, indicating a good linearity (Figure 2).
- ✓ Table 2 shows the reproducibility (n=6) for 5 µg/mL. A good reproducibility result was obtained for each component.



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 the BPA (incl. PH and BtPH) and DPC contents in materials.

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

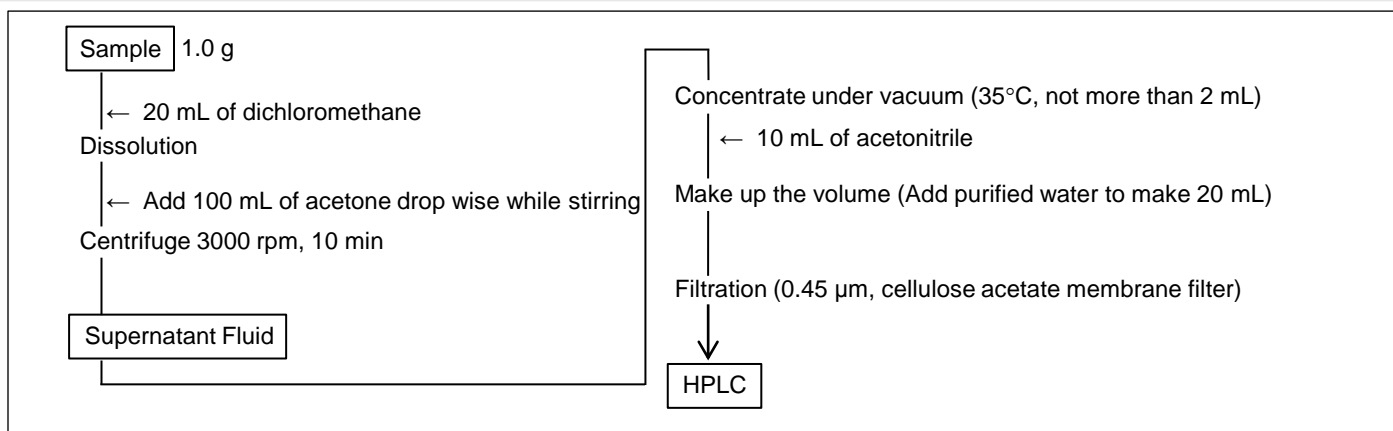


Figure 3 Preparation Method for Material Test

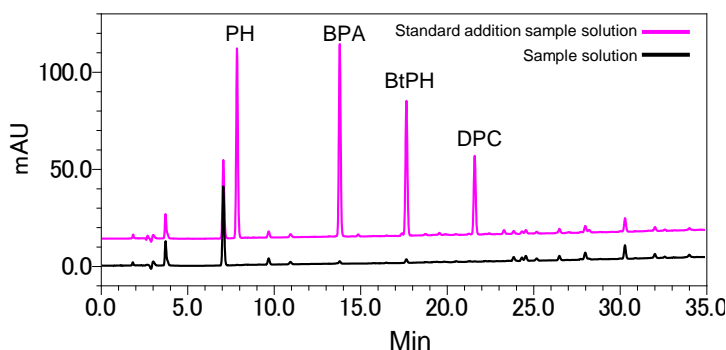


Figure 4 Chromatogram of Sample Solution

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.	9.958	173.032	99.6
	BPA	0.167	2.902	10.521	182.820	103.5
	BtPH	0.285	4.952	10.638	184.840	103.5
	DPC	n.d.	n.d.	10.375	180.275	103.8

- ✓ 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 99.6-103.8%, 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 $\mu\text{g/mL}$) 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 $\mu\text{g/mL}$).
- ✓ The target component for the elution test is BPA (incl. PH and BtPH). DPC is shown as a reference data.

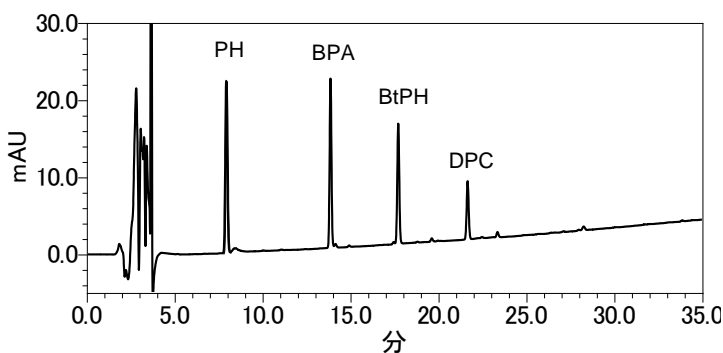


Figure 5 Chromatogram of Standard Solution (0.5 $\mu\text{g/mL}$)

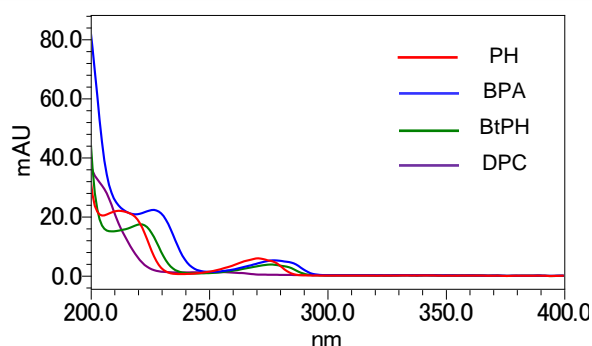


Figure 6 Absorption Spectrum of Standard Solution (0.5 $\mu\text{g/mL}$)

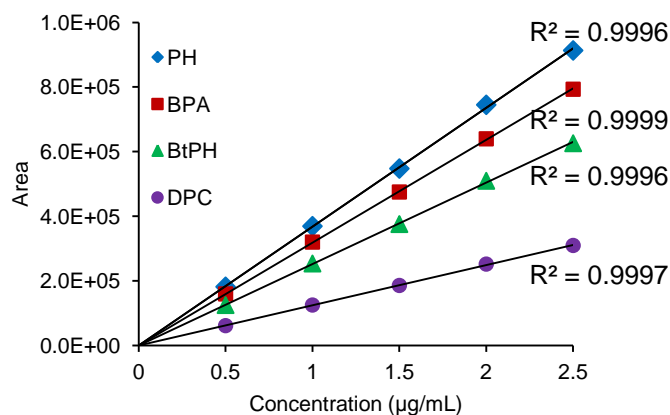


Figure 7 Calibration Curve

Table 4 Reproducibility (n=6) for Standard Solution (0.5 $\mu\text{g/mL}$)

Component	PH	BPA	BtPH	DPC
Retention time (min)				
Mean	7.952	13.886	17.736	21.682
%RSD	0.026	0.015	0.009	0.009
Area				
Mean	183755	161721	126865	62220
%RSD	0.342	0.421	0.379	0.255

Table 5 Analytical Conditions

Column	LaChrom II C18 (5 μm) 4.6 mm I.D. X 250 mm
Mobile phase	(A) H_2O (B) CH_3CN (v/v) 30% B (0 min) \rightarrow 100% B (35-45 min) \rightarrow 30% B (45.1-60 min)
Flow rate	1.0 mL/min
Column temperature	40°C
Detection wavelength	UV 217 nm (DAD)
Injection vol.	100 μL (200 μL sample loop was used)

- ✓ Figure 6 shows the absorption spectrum of the standard solution (0.5 $\mu\text{g/mL}$). By using a diode array detector, the absorption spectrum of each component can be confirmed.
- ✓ The calibration curves for those components (0.5, 1, 1.5, 2, 2.5 $\mu\text{g/mL}$) showed correlation coefficients (R^2) of 0.9996 or higher, indicating a good linearity (Figure 7)
- ✓ Table 4 shows the reproducibility (n=6) of 0.5 $\mu\text{g/mL}$. A good result was obtained for each component.



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 concentration¹⁾ 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

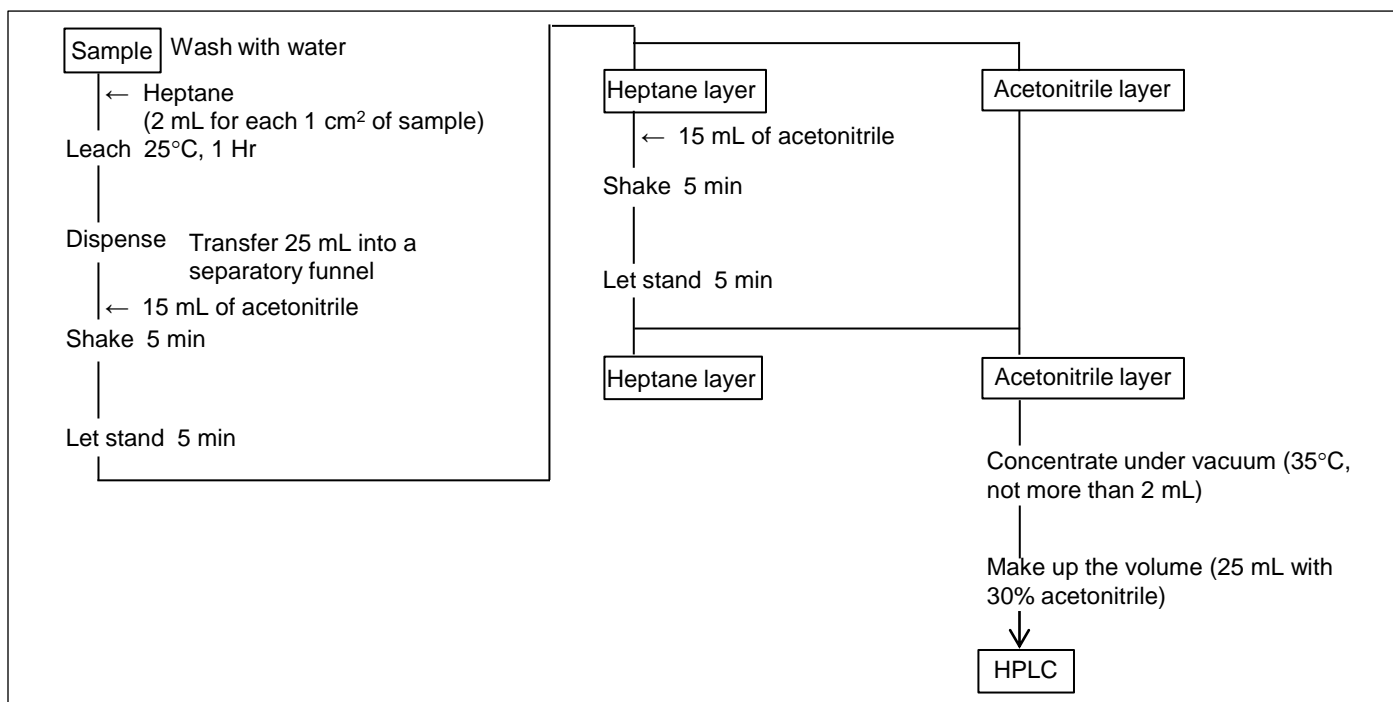


Figure 8 Preparation Method for Elution Test

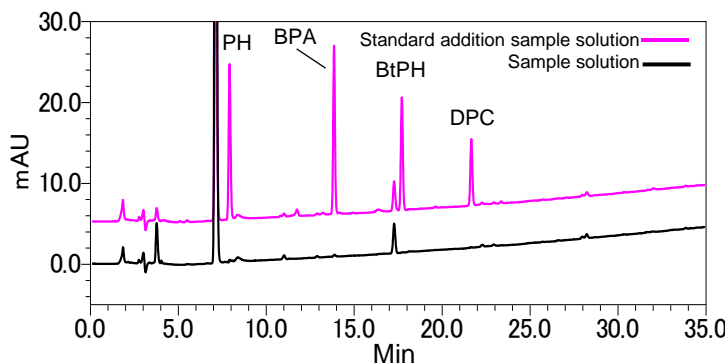


Figure 9 Chromatogram of Sample Solution

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.	0.477	95.5
	BPA	n.d.	0.500	100.0
	BtPH	n.d.	0.484	96.8
	DPC	n.d.	0.505	101.0

- ✓ The result of the elution test for the PC containers indicated 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 95.5-100% (Figure 9, Table 6).

<Main System Configuration>

Chromaster 5160 Pump, 5260 Autosampler, 5310 Column Oven, 5430 Diode Array Detector (DAD)

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