

## MIGRATION OF BISPHENOL A FROM POLY-CARBONATE PRODUCTS

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Polycarbonate is mainly a condensed polymer of bisphenol A and carbonyl chloride or diphenyl carbonate. Since it is transparent, has excellent heat resistance and impact resistance, and can be used for high-temperature uses and for microwave ovens, it is used in items such as children's tableware, tableware for meat services, coffee makers and food containers.

Bisphenol A is a compound with high reactivity having two phenol groups, is a raw material of epoxy resin, polysulfone, etc., in addition to polycarbonate, and is also used as an antioxidant and a stabilizer. Dodds *et al.*<sup>1</sup> report that it has an estrogen-like action of toxicity; moreover, Krishnan *et al.*<sup>2</sup> report that it elutes from a flask made of polycarbonate and causes abnormal multiplication of breast cancer cells, and that its activity is about 1/5,000 that of estradiol. Additionally, it is reported<sup>3</sup> that it causes fetal toxicity in mice.

Concerning bisphenol A in tableware and food container packages, Sugita *et al.*<sup>4</sup> report that it is found in polycarbonate and the elution, and Brotons *et al.*<sup>5</sup> report its elution from an inner surface coating of canned goods. Also, Hanai *et al.* (published on September 26, 1997 by Mainichi Newspaper) report elution from a plastic nursing bottle on the market. However, the elution behavior of bisphenol A from polycarbonate products has seldom been clarified.

In the Food Hygiene Law, the standard for bisphenol A in polycarbonate is prescribed by the total value of phenol and *p-tert*-butylphenol being added as polymerization adjustors, and it is determined as 500 ppm or less in a material test and 25 ppm or less in an elution test on container packaging.

In the fall of 1997, the total of bisphenol A, phenol, and *p-tert*-butylphenol was detected as being more than 500 ppm in children's tableware made from a polycarbonate containing an antibacterial agent, and many products were recalled due to violation of the standard (published on October 4, 1997 by Nikkei Newspaper, etc.). Since some of these products had been on the market for some time, elution of bisphenol A from them has been a cause for concern.

Accordingly, using the above-mentioned recalled products and commercially available nursing bottles, the elution behavior of bisphenol A was reviewed under various kinds of conditions, and the elution of phenol and *p-tert*-butylphenol was also reviewed. Furthermore, material and elution tests were conducted on polycarbonate products such as commercially available children's tableware and nursing bottles. The results of these tests are reported in this paper.

### EXPERIMENTAL METHODS

#### 1. Test Materials

Chosen as test materials were 14 items in total (one of each of the following): children's tableware rice bowl, mug, soup cup, and dish from among the recalled merchandise, and three mugs, two rice bowls, four nursing bottles, and one measuring cup from among products still on the market.

Among the test materials, two new samples were used for each test condition. When unwashed samples were used, they were washed with water according to the Food Hygiene Law and tested. In a repeated elution test, after one elution test was finished, the samples were washed thoroughly with water and then subjected to the elution test. In a boiling treatment, water was boiled in a pot, and the samples were dipped into it. Then boiling was continued for 5 min.

Table 1 shows the sample number, use, color, existence of antibacterial properties, and safety for use in microwave ovens, product history, amount of solvent used in the elution test, and surface area of the samples in contact with the solvent.

## 2. Reagents

Bisphenol A (2,2-bis(4-hydroxyphenyl)propane) and *p-tert*-butylphenol: primary reagent, made by Tokyo Kasei Kogyo K.K.

Phenol and n-heptane: special reagent, made by Wako Pure Chemical Industries, Ltd. Acetonitrile, dichloromethane, and ethanol: for HPLC, made by Katayama Kagaku Kogyo K.K.

Acetic acid: for precision analysis, made by Wako Pure Chemical Industries, Ltd. Water: purified by MILLI-Q SP (made by Millipore Co.)

Standard solutions: Phenol, bisphenol A, and *p-tert*-butylphenol were respectively dissolved in methanol to give stock solutions of 1,000 ppm. These stock solutions were appropriately mixed and diluted with water, so that standard solutions for each concentration were prepared.

Filter: LCR 13-LM filters with a pore diameter of 0.5  $\mu\text{m}$  and a diameter of 13 mm, made by Millipore Co.

## 3. Apparatuses

High-speed liquid chromatograph: pump LC-10A, column oven CTD-10 Avp, ultraviolet-visible detector SPD-10 AVvp, system controller SCL-10A, data processor C-R7A plus, auto injector SIL-10 Axl, made by Shimadzu Corporation.

Isothermal chamber: ST-120 made by Tabai Espec Corporation.

Microwave oven: RE-E3, rated output 500 W, made by Sharp Corporation.

## 4. HPLC measurement conditions

**Table 1. List of Test Materials**

| Sample | Use            | Color | Antibacteria <sup>a</sup> | Microwave Oven <sup>b</sup> | History  | Solvent Vol. (mL) | No. (cm <sup>2</sup> ) |
|--------|----------------|-------|---------------------------|-----------------------------|----------|-------------------|------------------------|
| 1      | Ricebowl       | White | •                         | •                           | Recalled | 160               | 92                     |
| 2      | Mug            | White | •                         | •                           | Recalled | 160               | 140                    |
| 3      | Soup Cup       | White | •                         | •                           | Recalled | 160               | 124                    |
| 4      | Dish           | White | •                         | •                           | Recalled | 160               | 97                     |
| 5      | Mug            | White | •                         | •                           | Marketed | 160               | 127                    |
| 6      | Mug            | White |                           |                             | Marketed | 160               | 131                    |
| 7      | Ricebowl       | White |                           | •                           | Marketed | 160               | 92                     |
| 8      | Ricebowl       | White |                           | •                           | Marketed | 160               | 88                     |
| 9      | Mug            | Clear |                           |                             | Marketed | 160               | 132                    |
| 10     | Nursing Bottle | Clear |                           |                             | Marketed | 100               | 125                    |
| 11     | Nursing Bottle | Clear |                           |                             | Marketed | 200               | 184                    |
| 12     | Nursing Bottle | Clear |                           |                             | Marketed | 100               | 91                     |
| 13     | Nursing Bottle | Clear |                           |                             | Marketed | 200               | 293                    |
| 14     | Measuring Cup  | Clear |                           |                             | Marketed | 500               | 281                    |

<sup>a</sup> Indication of anti-bacteria

<sup>b</sup> Indication of microwave oven useable

Condition 1 (for the material test)

Column: TSKgel ODS-80Ts (inner diameter of 4.6 mm, a length of 250 mm, and particle diameter of 5 mm) made by Tosoh Corporation.

Column jacket: TSKguardget ODS-80Ts (inner diameter of 32 mm and length of 15 mm) made by Tosoh Corporation.

Column temperature: 40°C

Mobile phase: Acetonitrile-water (1:1) was used as an initial concentration, with undiluted acetonitrile used following a 20 min linear gradient.

Flux: 1.0 mL/min

Detecting wavelength: 217 nm

Amount injected: 10 µL

Condition 2 (for the elution test)

Mobile phase: acetonitrile-water (1:1)

Amount injected: 100 µL

The other conditions are the same as condition 1.

## 5. Measuring methods

### 5.1 Material test

A 1.0g sample was precisely weighed and dissolved in 20 mL dichloromethane. 100 mL acetonitrile was slowly dripped into the solution while stirring with a stirrer, so that a high-molecular compound was precipitated. The suspension was centrifuged at 3,000 rpm for 10 min, and the supernatant fluid was concentrated under reduced pressure (at 40°C or lower) to about 2 mL. The concentrate, and 8 mL acetonitrile used to rinse the reduced pressure vessel, were transferred to a volumetric flask and adjusted to 20 mL with water. An appropriate amount was filtered and analyzed by the HPLC (condition 1).

### 5.2 Elution test

For each test, duplicate samples of each test material was filled with solvent and held at the test temperature for a set time. When the amount of solvent was reduced during testing, it was restored by adding solvent.

When water, 20% ethanol, and 4% acetic acid were used as solvents, the eluates obtained were filtered and measured by the HPLC (condition 2).

For n-heptane, 26 mL of the eluate obtained was transferred to a separatory funnel, extracted twice with 10 mL acetonitrile, and the volume adjusted to 25 mL by adding acetonitrile. Five mL of the eluate was concentrated under a nitrogen gas flow up to about 1 mL, and diluted to 5 mL with water. An appropriate quantity was filtered and measured by the HPLC condition 2).

The calculated amount eluted was obtained when the eluent was set to 2 mL per 1 cm<sup>2</sup>, as specified in the Food Hygiene Law:

$$\text{Calculated amount eluted} = \frac{\{\text{amount eluted (measured value)} \times \text{amount of eluent}\}}{(\text{surface area} \times 2)}$$

## RESULTS AND DISCUSSION

### 1. Review of the measuring methods

The material test was based on the previous report<sup>4</sup>; however, the initial concentration of the HPLC mobile phase was changed to acetonitrile-water (1:1) followed by a linear gradient for 20 min. The retention time was 5.1 min for phenol, 7.5 min for bisphenol A, and 10.5 min for *p-tert*-butylphenol. The detection limit was 20 ppb for each, and the quantification limit was 50 ppb per testing solution and 1 ppm per material.

In the elution tests with water, 20% ethanol, and 4% acetic acid, peaks which were ascribed to the solvents appeared at 6.4 min; however, this delayed peak did not overlap with

the three eluted compounds. Thus, the mobile phase of the HPLC was an isocratic gradient. The retention time was 5.3 min for phenol, 7.9 min for bisphenol A, and 13.3 min for *p-tert*-butylphenol. Even if the amount analyzed was 100  $\mu\text{L}$ , the baseline was stable, and no hindering peak was observed. Thus, quantification was possible down to 0.6 ppb per eluate (Figure 1).

According to a previous report<sup>4</sup>, when the *n*-heptane elution solution was replaced with 100% acetonitrile and the elutes measured by HPLC, the peak solvent floated. Consequently, the elution solution was concentrated and water added so that it was about 80% water. Thus, bisphenol A and *p-tert*-butylphenol could be measured by condition 2. However, for phenol, a hindering peak existed and it therefore could not be measured. If the HPLC is set to the gradient elution of condition 1, the hindering peak can be separated; however, if the amount being analyzed is increased to 100  $\mu\text{L}$ , the baseline is disturbed, so that the quantification limit value has to be raised. In this experiment, since the main purpose was high-sensitivity analysis of bisphenol A, the phenol in the *n*-heptane elution was measured but not quantified.

## **2. Comparison of the amount eluted under the various conditions of the Food Hygiene Law**

Among the polycarbonate products, four recalled products which violated the material standard and one nursing bottle currently on the market were subjected to the material test and the elution test under six test conditions prescribed by the Food Hygiene Law (Table 2 and Figure 2).

The amount of residual bisphenol A in the material of four recalled products was 379-599 ppm, and phenol and *p-tert*-butylphenol were 8-20 ppm and 81-154 ppm. The total amount of the three compounds was 510-779 ppm, and each of them exceeded the 500 ppm standard value.

In the comparison of the amount of bisphenol A eluted under various test conditions, *n*-heptane was the highest at 28.8-39.1 ppb. Water and 4% acetic acid at 95°C gave intermediate values at 19.0-26.3 ppb and 21.1 ppb, as did 20% ethanol at 7.4-28.9 ppb. Water and 4% acetic acid at 60°C were the lowest at 6.1-11.8 ppb and 10.4 ppb. The higher the fat solubility of the eluent or the elution temperature, the larger the amount eluted; however, the difference was only on the order of several fold. Also, even in the sample with a residual amount of about 600 ppm, the amount eluted was 40 ppb or less. The elution of phenol could not be measured in the *n*-heptane; however, it was as high as 1.1-7.5 ppb in 20% ethanol. Then, water at 95°C followed, and water at 60°C and 4% acetic acid at 60°C and 95°C were low. The reason why 4% acetic acid at 95°C was not as high as the water was unknown. As to why the amount of phenol eluted was large compared to the residual amount, it was presumed that, since the molecular weight was small, elution was easy.

The amount of *p-tert*-butylphenol eluted was 1.4-4.3 ppb, the difference in values between the test conditions was small, and the amount eluted was low in comparison with the residual amount.

**Table 2. Comparison of Test Condition on Migration of PH, BPA and PTBP From Recalled Samples and a Market Sample (No. 10)**

| No. | Chemical Residue | Migrant (ppb) |                |             |             |             |                 |              |
|-----|------------------|---------------|----------------|-------------|-------------|-------------|-----------------|--------------|
|     |                  | Water 60° C   | 20% EtOH 60° C | 4% AA 60° C | Water 95° C | 4% AA 95° C | n-Heptane 25° C |              |
| 1   | PH               | 12            | 0.7 (0.6)      | 3.1 (2.5)   | ---         | 1.8 (1.3)   | ---             | ---          |
|     | BPA              | 379           | 5.1 (4.2)      | 7.4 (6.1)   | ---         | 19.0 (16.5) | ---             | 28.8 (23.6)  |
|     | PTBP             | 119           | 1.8 (1.3)      | 2.8 (2.3)   | ---         | 2.4 (2.0)   | ---             | 2.9 (2.4)    |
|     | Total            | 510           | 7.4 (6.1)      | 13.3 (10.9) | ---         | 23.0 (18.8) | ---             | 31.7 (25.8)* |
| 2   | PH               | 20            | 0.8 (0.4)      | 7.5 (4.0)   | 0.8 (0.4)   | 7.8 (4.2)   | 0.6(0.3)        | ---          |
|     | BPA              | 599           | 11.8 (6.3)     | 28.9 (15.5) | 10.4 (5.6)  | 26.3 (15.9) | 21.1 (11.9)     | 37.2 (19.9)  |
|     | PTBP             | 154           | 2.2 (1.2)      | 2.8 (1.5)   | 2.3 (1.2)   | 2.8 (1.5)   | 2.2 (1.2)       | 4.3 (2.9)    |
|     | Total            | 773           | 14.8 (7.9)     | 39.2 (21.0) | 13.4 (7.2)  | 36.5 (21.6) | 23.9 (12.8)     | 41.5 (22.2)* |
| 3   | PH               | 11            | 0.9 (0.5)      | 5.4 (3.9)   | ---         | 1.6 (1.0)   | ---             | ---          |
|     | BPA              | 596           | 7.4 (4.5)      | 14.9 (9.0)  | ---         | 21.8 (12.9) | ---             | 32.6 (19.7)  |
|     | PTBP             | 140           | 1.6 (1.0)      | 2.2 (1.3)   | ---         | 1.9 (1.1)   | ---             | 1.4 (0.8)    |
|     | Total            | 747           | 8.9 (6.0)      | 23.5 (14.2) | ---         | 24.5 (18.0) | ---             | 34.0 (20.6)* |
| 4   | PH               | 8             | ND (ND)        | 3.9 (3.0)   | ---         | 1.9 (1.5)   | ---             | ---          |
|     | BPA              | 431           | 9.8 (7.6)      | 23.2 (18.0) | ---         | 24.6 (19.0) | ---             | 29.1 (30.2)  |
|     | PTBP             | 81            | 1.7 (1.3)      | 1.9 (1.5)   | ---         | 2.6 (2.0)   | ---             | 2.6 (2.0)    |
|     | Total            | 520           | 11.5 (8.9)     | 29.0 (22.5) | ---         | 29.1 (22.5) | ---             | 41.7 (31.2)* |
| 10  | PH               | ND            | ND (ND)        | ND (ND)     | ND (ND)     | ND (ND)     | ND (ND)         | ---          |
|     | BPA              | 20            | ND (ND)        | ND (ND)     | ND (ND)     | 0.5 (0.2)   | ND (ND)         | ND (ND)      |
|     | PTBP             | 4             | ND (ND)        | ND (ND)     | ND (ND)     | ND (ND)     | ND (ND)         | ND (ND)      |
|     | Total            | 24            | ND (ND)        | ND (ND)     | ND (ND)     | 0.5 (0.2)   | ND (ND)         | ND (ND)      |

Testing time: n-heptane for 60 minutes, others for 30 minutes.  
 PH: phenol, BPA: bisphenol A, PTBP: *p-tert*-butylphenol, AA: acetic acid  
 Residue: ND < 1 ppm, Migrant < 0.5 ppb  
 --- : Not tested, \* Did not contain phenol, ( ): Calculated migration using 2 mL/cm<sup>2</sup> of solvent

The total eluted amount of these three compounds was 7.4-41.7 ppb. When the elution amount was calculated to 2 mL of solvent per cm<sup>2</sup> according to the Food Hygiene Law, total elution was 6.0-32.2 ppb, 1/800 or less of the standard value of 25 ppm for container packing. Thus, it was shown that even when the residual amount in the material exceeded the standard, the amount eluted was very low.

On the other hand, in sample 10, the marketed nursing bottle, the residual amount in the material was 20 ppm for bisphenol A, 4 ppm for *p-tert*-butylphenol, and an undetectable amount for phenol. The amount of bisphenol A eluted was detected as a very infinitesimal amount of 0.5 ppb for water at 95°C for 30 min, however it was not detected at all under the other conditions. Also, no phenol or *p-tert*-butylphenol were detected under any conditions.

### 3. Repeated Elution

For samples 2 and 10, the samples used in the elution test with water at 95°C for 30 min were repeatedly subjected to elution under the same conditions, and the change in the amount eluted during 5 cycles was investigated (Table 3).

In sample 2, the elution of bisphenol A was reduced from about 1/8 to 1/20: 26.9 ppb in the first cycle, 3.4 ppb in the second cycle, 2.4 ppb in the third cycle, 1.7 ppb in the fourth cycle, and 1.5 ppb in the fifth cycle. Also, phenol and *p-tert*-butylphenol were reduced. In sample 10, an infinitesimal amount of bisphenol A was detected in the initial cycle; however it was not detected at all after the second cycle.

As mentioned above, in the elution of bisphenol A, etc., from polycarbonate products, since the amount eluted was largely decreased after the second cycle, it was indicated that the amount being eluted in daily use was either a very infinitesimal amount or none at all.

**Table 3. Repeated Migration of PH, BPA and PTBP from a Recalled (No. 2) And a Market Sample (No. 10)**

| No. | Chemical | Migrant (ppb)   |                 |                 |                 |                 |
|-----|----------|-----------------|-----------------|-----------------|-----------------|-----------------|
|     |          | 1 <sup>st</sup> | 2 <sup>nd</sup> | 3 <sup>rd</sup> | 4 <sup>th</sup> | 5 <sup>th</sup> |
| 2   | PH       | 7.8 (4.2)       | 4.5 (2.4)       | 3.4 (1.8)       | 1.3 (0.7)       | 1.3 (0.7)       |
|     | BPA      | 26.3 (15.9)     | 3.4 (1.8)       | 2.4 (1.3)       | 1.7 (0.9)       | 1.5 (0.8)       |
|     | PTBP     | 2.8 (1.5)       | 1.5 (0.8)       | 0.9 (0.6)       | 0.9 (0.5)       | 0.6 (0.3)       |
|     | Total    | 36.9 (21.6)     | 9.4 (5.0)       | 6.7 (3.6)       | 3.9 (2.1)       | 3.4 (1.8)       |
| 10  | PH       | ND (ND)         | ND (ND)         | ND (ND)         | ND (ND)         | ND (ND)         |
|     | BPA      | 0.5 (0.2)       | ND (ND)         | ND (ND)         | ND (ND)         | ND (ND)         |
|     | PTBP     | ND (ND)         | ND (ND)         | ND (ND)         | ND (ND)         | ND (ND)         |
|     | Total    | 0.5 (0.2)       | ND (ND)         | ND (ND)         | ND (ND)         | ND (ND)         |

Test Conditions: Water at 95°C for 30 minutes and repeated.

PH: Phenol, BPA: bisphenol A, PTBP: *p-tert*-butylphenol

ND: < 0.5 ppb

( ): Calculated migration using 2 mL/cm<sup>2</sup> of solvent.

### 4. Elution by Microwave Oven Heating

Water was added to samples 2 and 10 and heated for 10 min in the microwave oven, and the amount eluted was investigated. Then, the samples were washed and reheated, and the amount eluted during the second cycle was investigated (Table 4).

The amount of bisphenol A eluted was 85.6 ppb during the first cycle and 4.7 ppb during the second cycle in sample 2. In sample 10, the amount was 0.7 ppb and at the detection limit or below. It was presumed that the reason why relatively high elution was shown during the first cycle was due to the harsh conditions, that is, the sample reached a boiling state after about 5 min and the boiling continued for the remaining 5 min. Also, during the second cycle, since the amount eluted was greatly decreased and was about the same as that during the second cycle of

the repeated elution at 95°C for 30 min, it was shown that decomposition is seldom observed. Consequently, it was demonstrated that even when the microwave oven was used, the amount eluted was infinitesimal.

## 5. Elution by pouring boiling water

The above mentioned Hanai *et al.*, reported that when boiling water was poured into the nursing bottle and held over night, 3.1-5.6 ppb bisphenol A was eluted. Accordingly, boiling water was poured into bottles of sample 10 and held at room temperature, and the amount eluted after 30 min and 24 h was investigated. Under those conditions, no elution was observed after 30 min; however 0.5 ppb bisphenol A was eluted after 24 h (Table 5). The amount eluted for 24 h at room temperature and at 95°C for 30 min after pouring boiling water is considered to be about the same.

**Table 4. Repeated Migration of PH, BPA and PTBP from Recalled (No. 2) and a Market Sample (No. 10) by Microwave Oven Heating**

| No. | Chemical | Migrant (ppb)   |                 |
|-----|----------|-----------------|-----------------|
|     |          | 1 <sup>st</sup> | 2 <sup>nd</sup> |
| 2   | PH       | 4.5 (2.4)       | 2.0 (1.1)       |
|     | BPA      | 85.6 (46.0)     | 4.7 (2.5)       |
|     | PTBP     | 2.9 (1.5)       | 1.3 (0.7)       |
|     | Total    | 93.2 (49.9)     | 8.0 (4.4)       |
| 10  | PH       | ND (ND)         | ND (ND)         |
|     | BPA      | 0.7 (0.3)       | ND (ND)         |
|     | PTBP     | ND (ND)         | ND (ND)         |
|     | Total    | 0.7 (0.3)       | ND (ND)         |

Test Conditions: Heating by microwave oven (500 W) with water for 10 minutes and repeated.  
 PH: phenol, BPA: bisphenol A,  
 PTBP: p-*tert*-butylphenol ND: < 0.5 ppb  
 ( ): Calculated migration using 2 mL/cm<sup>2</sup> of solvent.

## 6. Influence of washing, etc.

The influence of washing and boiling treatments on the samples before the elution test was investigated (Table 6). When no washing was applied, bisphenol A was 38.1 ppb in sample 2 and 3.9 ppb in sample 10, which were 1.5 times and 6.5 times those of the washed samples. Also, when the bottles were not washed, high measured values for phenol and p-*tert*-butylphenol were shown. On the other hand, after boiling for 5 min, the elution of bisphenol A was also largely decreased to the detection limit in sample 2, and no elution was detected in sample 10. Consequently, it was shown that the amount eluted was greatly decreased by washing and was greatly decreased in particular by boiling and disinfecting, which are usually carried out on nursing bottles and tableware for children.

**Table 5. Migration of PH, BPA and PTBP from a Market Sample Filled with Boiling Water and Held at Room Temperature**

| No. | Chemical | Migrant (ppb) |           |
|-----|----------|---------------|-----------|
|     |          | 30 min        | 24 hr     |
| 10  | PH       | ND (ND)       | ND (ND)   |
|     | BPA      | ND (ND)       | 0.5 (0.2) |
|     | PTBP     | ND (ND)       | ND (ND)   |
|     | Total    | ND (ND)       | 0.6 (0.2) |

Test conditions: Filled with boiling water and held for 30 minutes or 24 hours at room temperature.  
 PH: phenol, BPA: bisphenol A,  
 PTBP: *p-tert*-butylphenol ND: 0.5 ppb  
 ( ): Calculated migration using 2 mL/cm<sup>2</sup> of solvent.

**Table 6. Effect of Treatment Before Test on Migration of PH, BPA and PTBP from a Recalled (No. 2) and a Market Sample (No. 10)**

| No. | Chemical | Migrant (ppb) |             |           |
|-----|----------|---------------|-------------|-----------|
|     |          | No Washing    | Washing     | Boiling   |
| 2   | PH       | 19.5 (10.4)   | 7.8 (4.2)   | 0.6 (0.4) |
|     | BPA      | 39.1 (20.9)   | 26.3 (15.9) | 0.5 (0.3) |
|     | PTBP     | 20.0 (1.1)    | 2.8 (1.5)   | ND (ND)   |
|     | Total    | 60.6 (32.5)   | 36.9 (21.5) | 1.3 (0.7) |
| 10  | PH       | 0.7 (0.4)     | ND (ND)     | ND (ND)   |
|     | BPA      | 3.9 (2.1)     | 0.6 (0.3)   | ND (ND)   |
|     | PTBP     | 1.2 (0.8)     | ND (ND)     | ND (ND)   |
|     | Total    | 5.8 (3.3)     | 0.6 (0.2)   | ND (ND)   |

Test Conditions: Water at 96°C for 30 minutes.  
 PH: phenol, BPA: bisphenol A,  
 PTBP: *p-tert*-butylphenol ND: 0.5 ppb  
 ( ): Calculated migration using 2 mL/cm<sup>2</sup> of solvent

## 7. Investigation of products on the market

For nine polycarbonate products on the market, the residual amount of phenol, bisphenol A, and *p-tert*-butylphenol was measured, and the amount eluted in 20% ethanol at 60°C for 30 min and in water at 95°C for 30 min (Table 7).

The residual amount was ND-11 ppm in phenol, 5-80 ppm in bisphenol A, and ND-43 ppm in *p-tert*-butylphenol, and the total amount was 9-126 ppm. The white products, samples

5-8, showed a residual amount slightly higher than that of the transparent products (9-14) such as nursing bottles. Also, sample 5 showed an antibacterial characteristic; however, there was no problem in the residual amount. For bisphenol A and p-*tert*-butylphenol, residual levels were almost the same, or bisphenol A

**Table 7. Migration of PH, BPA and PTBP from Market Samples**

| No. | Chemical | Residue (ppm) | Migrant (ppb) |            |
|-----|----------|---------------|---------------|------------|
|     |          |               | 20% E+OH 60°C | Water 95°C |
| 5   | PH       | 1             | ND (ND)       | 1.0 (0.6)  |
|     | BPA      | 43            | ND (ND)       | ND (ND)    |
|     | PTBP     | 43            | ND (ND)       | ND (ND)    |
|     | Total    | 87            | ND (ND)       | 1.0 (0.6)  |
| 6   | PH       | 5             | ND (ND)       | 1.0 (0.6)  |
|     | BPA      | 49            | ND (ND)       | ND (ND)    |
|     | PTBP     | 38            | ND (ND)       | ND (ND)    |
|     | Total    | 92            | ND (ND)       | 1.0 (0.6)  |
| 7   | PH       | 2             | ND (ND)       | ND (ND)    |
|     | BPA      | 47            | 1.7 (1.4)     | 3.2 (2.6)  |
|     | PTBP     | 24            | ND (ND)       | ND (ND)    |
|     | Total    | 73            | 1.7 (1.4)     | 3.2 (2.6)  |
| 8   | PH       | 11            | ND (ND)       | 0.7 (0.6)  |
|     | BPA      | 80            | 2.9 (2.5)     | 4.5 (9.8)  |
|     | PTBP     | 35            | ND (ND)       | ND (ND)    |
|     | Total    | 125           | 2.9 (2.5)     | 5.3 (4.5)  |
| 9   | PH       | ND            | ND (ND)       | 0.8 (0.5)  |
|     | BPA      | 5             | ND (ND)       | ND (ND)    |
|     | PTBP     | 4             | ND (ND)       | ND (ND)    |
|     | Total    | 9             | ND (ND)       | 0.8 (0.5)  |
| 11  | PH       | ND            | ND (ND)       | ND (ND)    |
|     | BPA      | 20            | ND (ND)       | ND (ND)    |
|     | PTBP     | 19            | ND (ND)       | ND (ND)    |
|     | Total    | 39            | ND (ND)       | ND (ND)    |
| 12  | PH       | 3             | 0.8 (0.4)     | 0.8 (0.4)  |
|     | BPA      | 18            | ND (ND)       | ND (ND)    |
|     | PTBP     | ND            | ND (ND)       | ND (ND)    |
|     | Total    | 21            | 0.8 (0.4)     | 0.8 (0.4)  |
| 13  | PH       | ND            | ND (ND)       | ND (ND)    |
|     | BPA      | ND            | ND (ND)       | ND (ND)    |
|     | PTBP     | ND            | ND (ND)       | ND (ND)    |
|     | Total    | 37            | ND (ND)       | ND (ND)    |
| 14  | PH       | ND            | ND (ND)       | ND (ND)    |
|     | BPA      | 12            | ND (ND)       | ND (ND)    |
|     | PTBP     | 2             | ND (ND)       | ND (ND)    |
|     | Total    | 14            | ND (ND)       | ND (ND)    |

was considerably higher, or *p-tert*-butylphenol was not detected. These differences were considered to be caused by conditions during manufacturing and processing of raw material pellets.

Elution of bisphenol A was observed in samples 7 and 8, where it was 1.7 and 2.9 ppb in the 20% ethanol and 3.2 and 4.6 ppb in water at 95°C. No elution was seen in the other products. Also, 1 ppb or less of phenol was detected from the fifth product; however elution of *p-tert*-butylphenol was not seen.

Based on these results, bisphenol A, etc., largely remained in the products currently on the market, since the residual amounts were low. Elution was observed in small amounts only in some of the products, and in many products no elution was seen.

## CONCLUSION

When elution tests were carried out using four products which violated the material standards, having a residual amount of bisphenol A, etc., of more than 500 ppm, the amount of bisphenol A eluted was 40 ppb or less and the total amount of residual phenols was merely 50 ppb or less. Among ten marketed products, elution of small amounts of bisphenol A (less than 5ppb) were observed in three products and no elution observed in the other products. Also, the amount eluted was considerably lowered by repeated elution, and no increase of bisphenol A due to the decomposition of polycarbonate was seen in a microwave oven. Slightly higher elution was seen in unwashed products; however, elution was greatly decreased by washing or boiling.

Consequently, it is believed that only the bisphenol A existing on the surface or near the surface is eluted from polycarbonate products. For this reason, even in the recalled products containing bisphenol A, etc., in excess of material standards, the amount being eluted is low. Also, it was confirmed that, for everyday use of nursing bottles, children's tableware, etc., currently on the market, the amount eluted was infinitesimal or seldom observed.

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