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Simultaneous Analysis of Three Flame Retardants in Textile Products Prohibited in Japan by Liquid Chromatography/Tandem Mass Spectrometry

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Three flame retardants, tris(1-aziridinyl) phosphine oxide (APO), tris(2,3-dibromopropyl) phosphate (TDBPP), and bis(2,3-dibromopropyl) phosphate (BDBPP), are prohibited for use in textile products under the "Act on the Control of Household Products Containing Harmful Substances" in Japan. The currently used method for analyzing these substances by the Ministry of Health, Labor and Welfare in Japan (MHLWJ), is GC, which uses carcinogenic solvents and hazardous reagents during sample processing. Furthermore, due to the global shortage of helium gas used as carrier gas for GC, the development of an alternative method is necessary. In this study, we developed a method to simultaneously analyze the three flame retardants using LC–MS/MS. In addition, this method is an improvisation of ISO 17881-2, which involves extraction with acetone under acidic conditions and replacing it with acetonitrile. This prevents the acid decomposition of APO and the thermal decomposition of BDBPP. For accurate and sensitive quantification, deuterated compounds, APO-d_{12}, TDBPP-d_{15}, and BDBPP-d_{10}, were used as surrogate standards. The calibration curve displayed linearity within the 0.005–1.0 µg/mL range for APO and the 0.01–2.0 µg/mL range for TDBPP and BDBPP. The limits of detection for APO, TDBPP, and BDBPP were 0.19, 0.87, and 1.0 µg/g, respectively. These values were 2.2, 9.2, and 10 times more sensitive than the current detection limits of 0.4, 8, and 10 µg/g, respectively. The recovery of the three flame retardants in various textile products using the developed analytical method was 83.7–120.8%, which confirms their satisfactory analyses.

Key words flame retardants, LC-MS/MS, textile products, tris(1-aziridinyl) phosphine oxide, bis(2,3-dibromopropyl) phosphate, tris(2,3-dibromopropyl) phosphate

INTRODUCTION

In 1978, tris(1-aziridinyl) phosphine oxide (APO), an organophosphorus flame retardant, was reported to cause hematopoietic dysfunction in animal experiments.[1)] In addition, it was reported in animal experiments that tris(2,3-dibromopropyl) phosphate (TDBPP), another flame retardant, is carcinogenic and mutagenic.[2,3)] Therefore, these flame retardants are prohibited for use in textile products such as curtains, carpets, and sleepwear under the "Act on the Control of Household Products Containing Harmful Substances" in Japan.[4)] Furthermore, in 1981, bis(2,3-dibromopropyl) phosphate (BDBPP) was designated as a hazardous substance because it was confirmed to have the same toxicity as TDBPP.[5)] Consequently, its use in textile products was prohibited.[4,5)] Fig. 1 shows the chemical structure of these flame retardants.

The equipment for chemical analysis has made remarkable progress, advancing from packed column gas chromatography (GC) to capillary GC. Moreover, gas chromatography–mass spectrometry (GC-MS) has become popular as it enables highly selective and sensitive analysis. Official analytical methods by the Ministry of Health, Labor and Welfare in Japan (MHLWJ) employ packed column GC for analyzing APO, BDBPP, and TDBPP; however, these methods have not been revised for more than 40 years.[6)] Additionally, these methods use dichloromethane for analyzing APO and benzene for analyzing TDBPP and BDBPP, although these solvents are carcinogenic.[7,8)] In addition, the diazomethane solution used for the methyl derivatization of BDBPP is a carcinogenic and explosive reagent when prepared in the laboratory.[6)] Therefore, this poses a hazard to the health of the analyst. We previously reported GC-MS methods in which the solvent was changed to safer alternatives, such as acetone or ethyl acetate, and the methyl derivatization reagent was substituted with a commercially available, safer alternative.[9,10)] However, problems persist: TDBPP thermally decomposes at temperatures above 260–300°C,[5)] and BDBPP thermally decomposes at the GC-MS injection port.[9)] Furthermore, due to the global shortage of helium gas used as carrier gas for GC, an alternative method is

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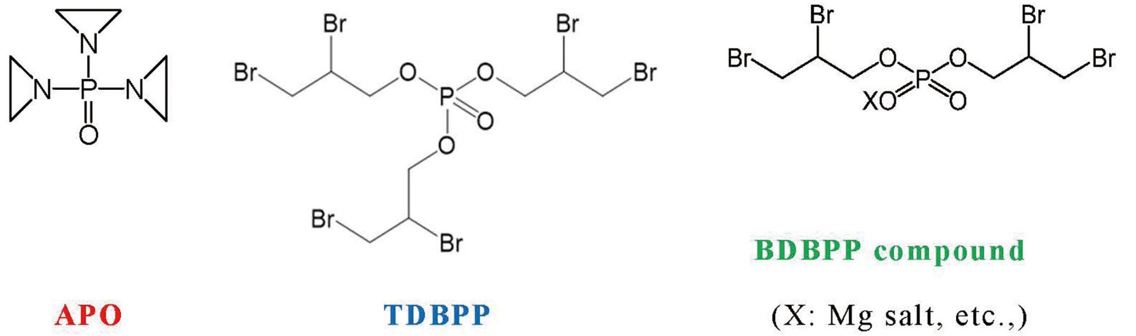

Fig. 1. Chemical Structures of the Analyzed Three Flame Retardants

necessary. Thus, a liquid chromatography-tandem mass spectrometry (LC-MS/MS) method that does not use helium and eliminates the risk of thermal decomposition is necessitated. ISO 17881-2[11] already includes a method using LC-MS/MS for analyzing TDBPP and APO in textile products; however, this method cannot be utilized for BDBPP. Therefore, in this study, to simultaneously analyze three flame retardants, APO, TDBPP, and BDBPP, we developed an LC-MS/MS method that prevents the thermal decomposition of BDBPP and suppresses the acid decomposition of APO.

MATERIALS AND METHODS

Samples Seven commercially available textile products labeled as flame retardant were purchased: three curtains, one blanket, one apron, one pair of pants, and one T-shirt. One commercially available infant underwear, made of 100% untreated white cotton cloth, was purchased. Two untreated white cloths, one made of 100% wool and the other made of 100% silk, manufactured by the Japanese Standards Association (Tokyo, Japan), were purchased. These samples were purchased in Japan between October 2019 and August 2022 (Table 1).

Chemical and Reagents APO (CAS 545-55-1), TDBPP (CAS 126-72-7), and BDBPP (CAS 5412-25-9) were purchased from Toronto Research Chemicals (Toronto, Canada). Their deuterated forms, APO-d$_{12}$, TDBPP-d$_{15}$, and BDBPP-d$_{10}$, were also purchased from Toronto Research Chemicals (Toronto, Canada) as surrogate standards. Pesticide residue and polychlorinated biphenyl test-grade acetone and hexane and LC-MS-grade acetonitrile and formic acid were purchased from FUJIFILM Wako Pure Chemical Corporation (Osaka, Japan). LC-grade ammonium acetate aqueous solution (1 mol/L) was purchased from Nacalai Tesque Inc. (Kyoto, Japan). Milli-Q water was produced using a Direct-Q 5UV instrument (Merck Millipore, Massachusetts, USA).

Instrument The LC-MS/MS device used was a Triple Quad 4500 (MS) equipped with an Exion LC AD (LC) from AB Sciex (Massachusetts, USA). The column and LC gradient conditions were in accordance with ISO 17881-2.[11] Additionally, a 5 mmol/L ammonium acetate solution was used as the aqueous mobile phase, and acetonitrile was used as the solvent mobile phase. APO and TDBPP were measured using the positive mode (ESI+), and BDBPP was measured using the negative mode (ESI-) of atmospheric pressure electrospray ionization.

Table 1. Sample Information

Usage	Material	Color
Flame-retardant textile products		
Curtain 1	100% Polyester	Blue
Curtain 2	65% Polyester, 35% Cotton	Blue
Curtain 3	50% Polyester, 50% Acrylic	White
Blanket	100% Wool	Green
Apron	100% Cotton	Navy
Pants	100% Cotton	Beige
T-shirts	55% Acrylic, 45% Cotton	Navy
Textile products		
Infant underwear	100% Cotton	White
Wool cloth	100% Wool	Beige
Silk cloth	100% Silk	Off-White

The optimum conditions for selected reaction monitoring (SRM) were determined using the infusion method, in which each standard solution (0.1 µg/mL in acetonitrile) was directly introduced into the ion source. During LC, the 5 mmol/L ammonium acetate solution was used as the aqueous mobile phase, and acetonitrile was used as the solvent-based mobile phase. Retention times, collision energies, quantifying and qualifying ions of the chemicals studied are listed in Tables 2 and 3.

Preparation of Standard Solution APO and APO-d$_{12}$ standard stock solutions (200 and 100 µg/mL, respectively) were prepared by accurately weighing 2 mg of APO and 1 mg of APO-d$_{12}$ and adjusting the volume to 10 mL with methanol. Each standard stock solution was diluted with acetone to prepare a 10 µg/mL standard solution.[10] TDBPP and BDBPP standard stock solutions (200 µg/mL each) were prepared by accurately weighing 2 mg of each compound and adjusting the volume to 10 mL with acetone. TDBPP-d$_{15}$ and BDBPP-d$_{10}$ standard stock solutions (100 µg/mL each) were prepared by accurately weighing 1 mg of each compound and adjusting the volume to 10 mL with acetone. A 50 µg/mL mixed standard solution of TDBPP and BDBPP was prepared by taking 2.5 mL of each standard stock solution and diluting it to 10 mL with acetone. A 50 µg/mL mixed standard solution of TDBPP-d$_{15}$ and BDBPP-d$_{10}$ was prepared by mixing equal volumes of these standard stock solutions. The APO standard solution

Table 2. LC-MS/MS Experimental Parameters of the Three Flame Retardants: APO, TDBPP, and BDBPP[12-15]

Flame retardant	Retention time (min)	DP (V)	Quantification ion		Qualifier ion		Detection limit (µg/g)
			Q1 > Q3	CE (V)	Q1 > Q3	CE (V)	
APO	1.91	51	174.0 > 131.0	17	174.0 > 90.0	25	0.19
TDBPP	6.51	81	698.6 > 99.1	75	698.6 > 299.2	25	0.87
BDBPP	4.70	−35	497.0 > 80.9	−58	497.0 > 79.0	−56	1.0

DP: declustering potential, Q1: precursor ion, Q3: product ion, CE: collision energy

Table 3. LC-MS/MS Experimental Parameters of APO-d$_{12}$, TDBPP-d$_{15}$, and BDBPP-d$_{10}$

Flame retardant	Retention time (min)	DP (V)	Quantification ion	
			Q1 > Q3	CE (V)
APO-d$_{12}$	1.91	56	185.8 > 139.2	19
TDBPP-d$_{15}$	6.46	76	714.0 > 102.1	81
BDBPP-d$_{10}$	4.69	−45	506.5 > 80.8	−62

DP: declustering potential, Q1: precursor ion, Q3: product ion, CE: collision energy

(10 µg/mL) and the TDBPP and BDBPP mixed standard solution (50 µg/mL) were diluted stepwise with acetonitrile as appropriate, and calibration curves were created for each: 0.005–1.0 µg/mL for APO and 0.01–2.0 µg/mL for TDBPP and BDBPP. At the same time, APO-d$_{12}$ was added to each standard solution at 0.025 µg/mL, while TDBPP-d$_{15}$ and BDBPP-d$_{10}$ were each added at 0.1 µg/mL.

Sample Processing The textile products were cut into small pieces (approximately 1×1 cm), and 0.2 g of the sample was weighed and collected in a glass tube. To this, 75 µL of the APO-d$_{12}$ surrogate standard solution and 60 µL of the TDBPP-d$_{15}$ and BDBPP-d$_{10}$ mixed surrogate standard solution (i.e., 0.75 µg of APO-d$_{12}$, 3.0 µg of TDBPP-d$_{15}$ and BDBPP-d$_{10}$) were added. Furthermore, 10 mL of acetone and 0.1 mL of 1 mol/L aqueous formic acid solution were added, and the glass tube was placed in an ultrasonic generator at 30°C, and ultrasonic wave extraction was performed for 30 min. Next, 1 mL of the sonicated solution was collected and concentrated to 0.1 mL under a nitrogen stream, and 0.1 mL of acetonitrile was added to it. This solution was again concentrated to 0.1 mL under a nitrogen stream, and the volume was adjusted to 3 mL with acetonitrile. The solution was filtered using a polytetrafluoroethylene (PTFE) filter (pore size 0.20 µm) and analyzed via LC-MS/MS.

However, if the acetone solution containing 1 mol/L aqueous formic acid solution became viscous, 2 mL of the sonicated solution was collected, and 1 mL of hexane was added dropwise under ultrasonic irradiation. This resulted in poor solubility, causing the polymer to precipitate, and 1 mL of the supernatant was collected. The solution was then concentrated to 0.1 mL under a nitrogen stream, and the volume was adjusted to 2 mL with acetonitrile. The dilution ratio was the same as the previous test solution. The solution was then filtered using a PTFE filter (pore size 0.20 µm) and analyzed by LC-MS/MS.

The spike recovery test was carried out by adding 150 µL of the APO standard solution and 60 µL of the TDBPP and BDBPP mixed standard solution (i.e., 1.5 µg of APO, 3.0 µg of each of TDBPP and BDBPP) to 0.2 g of the cut sample. Pretreatment was performed according to sample processing (n=3).

RESULTS AND DISCUSSION

LC-MS/MS Conditions Infusion analysis showed that the products ions of APO, TDBPP, and BDBPP were estimated based on previous studies[12-15] as $[M-C_2H_4N]^+$ m/z=131.0 and $[M-C_4H_8N_2+H]^+$ m/z=90.0 (APO), $[M-C_9H_{15}Br_6+4H]^+$ m/z= 99.1 and $[M-C_6H_{10}Br_4+3H]^+$ m/z=299.2 (TDBPP), and $[M-C_6H_{10}Br_3PO_4X]^-$ m/z=80.9 and 79.0 (BDBPP). These ions were used as quantification and qualification ions (Table 2 and 3).

As a result, the SRM chromatogram (Fig. 2) showed sufficient separation, and the calibration curves with surrogate correction showed linearity in the ranges of 0.005–1.0 µg/mL for APO and 0.01–2.0 µg/mL for TDBPP and BDBPP. The deviation was found to be within ±20%, using a calibration curve weighted by the reciprocal of the concentration. The calibration curve was completely satisfactory. The limits of detection calculated from the lower limit of the calibration curve[16] were 0.19 µg/g for APO, 0.87 µg/g for TDBPP, and 1.0 µg/g for BDBPP. Compared to the detection limits obtained using previously established methods—0.4 µg/g for APO,[17] 8 µg/g for TDBPP,[18] and 10 µg/g for BDBPP[19]—these values were 2.2, 9.2, and 10 times more sensitive, respectively. Moreover, in the absolute calibration curve method, a satisfactory calibration curve was obtained for all three compounds within the same concentration range as before, using a calibration curve weighted by the square of the reciprocal of the concentration.

Extraction According to ISO 17881-2,[11] TDBPP and APO are extracted from textiles with acetone, and TDBPP can also be extracted using methanol reflux extraction[6] or under acidic conditions.[9] In contrast, BDBPP can only be extracted under acidic conditions.[6] However, it has been reported that APO rapidly decomposes at pH 4.[1] Then, we investigated pretreatment methods to quickly remove formic acid to reduce the acid decomposition of APO. Therefore, as shown in sample processing, the extracted solution was concentrated under a nitrogen stream to remove volatile formic acid and then added to acetonitrile, which is suitable for LC-MS/MS analysis. Therefore, to improve the extraction efficiency of BDBPP, acetone containing formic acid was used as an extraction solvent. This reduced the time during which APO was exposed to acidic conditions.

Measurements A spike recovery test was conducted as per sample processing. Curtain 3 was a flame-retardant product consisting of 50% polyester and 50% acrylic. Because the acetone extract became viscous, it was pretreated using method 5 for viscous solutions. Although the extracted solution for

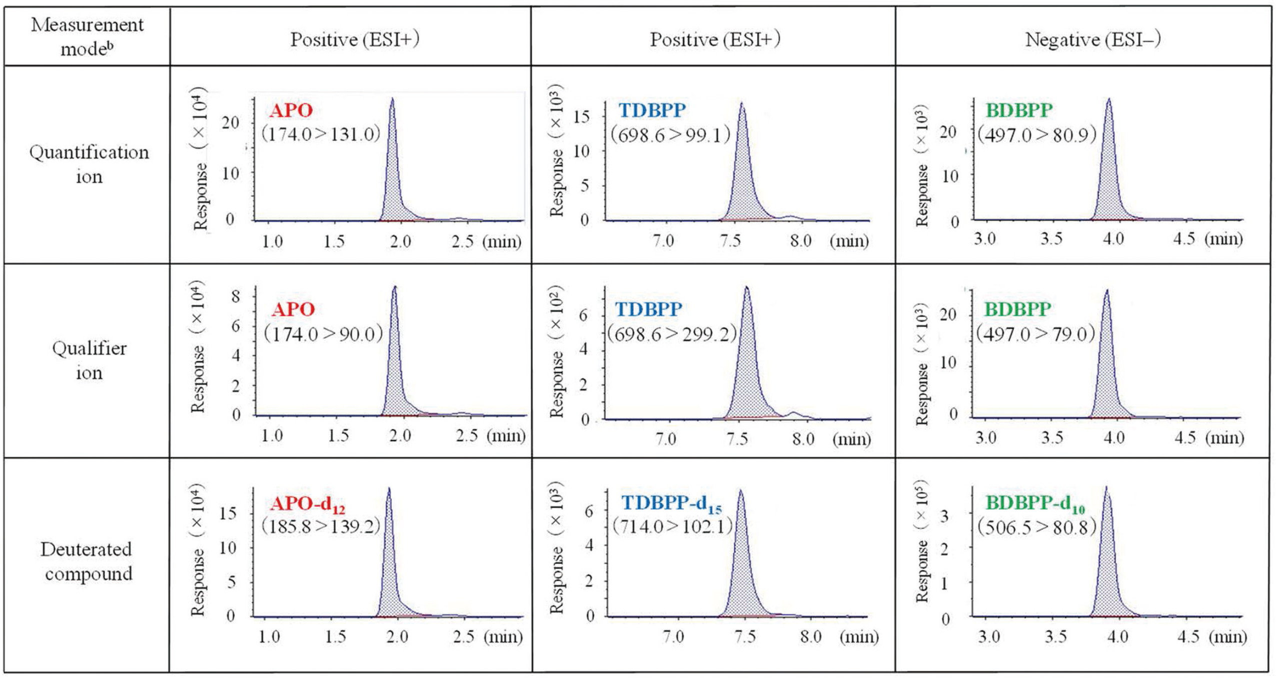

Fig. 2. SRM Chromatograms of APO, TDBPP, and BDBPP and Their Deuterated Compounds ^{a}

^{a} The concentrations of APO, TDBPP, and BDBPP are 0.05, 0.1, and 0.1 µg/mL, respectively. The concentrations of deuterated compounds APO-d_{12}, TDBPP-d_{15}, and BDBPP-d_{10} as surrogate standards are 0.025, 0.1, and 0.1 µg/mL, respectively. The numbers in parentheses represent Q1 > Q3. ^{b} Atmospheric pressure electrospray ionization measurement mode of LC-MS/MS.

flame-retardant T-shirts (55% acrylic, 45% cotton) contained acrylic, it did not become viscous; hence, a general pretreatment was applied. As shown in Fig. 3, the results of the additive recovery test with surrogate correction were 84–113% for APO, 90–121% for TDBPP, and 105–118% for BDBPP. The relative standard deviation (RSD) was 2.1–11% for APO, 0.48–9.8% for TDBPP, and 1.1–7.5% for BDBPP, which were generally satisfactory. The surrogate recovery rate exceeded the surrogate recovery rate standard of 50%,[16] except for Curtain 3 for APO-d_{12} and Curtains 2, Curtain 3, and Wool cloth for BDBPP-d_{10}. This result showed that quantification by surrogate correction can be applied to various samples.

The recoveries determined by the absolute calibration curve method showed the same trend as the surrogate recoveries (Fig. 3). TDBPP showed recovery rates of 62–114% in 10 samples, and the RSD was 1.4–4.7%. Eight samples showed satisfactory recovery rates, except Blanket (62%) and Apron (69%), which showed recovery rates of less than 70% and were generally quantifiable. APO displayed a recovery rate of 36–78% (RSD 2.2–13%). Infant underwear (100% cotton) showed a recovery rate exceeding 70%, but other items showed lower recovery rates. The recovery rates of BDBPP were in the range of 36–90% (RSD 3.6–31%), with recoveries of over 70% in four samples: Curtain 1 (90%), Apron (73%), Pants (71%), and Silk cloth (75%). However, six samples displayed lower recovery rates. It has been reported that these flame-retardant finishing agents must be present in textile products at 5% or more to exhibit flame-retardant effects.[19-21] Therefore, based on the lower detection limits mentioned above, we concluded that these flame retardants can be detected even at a low recovery rate. Hence, the absolute calibration curve method can be used as a screening method.

In a previous study[10] using GC-MS for Apron, a 100% cotton flame-retardant textile product, the recovery rate of the APO surrogate was as low as 15% owing to interference. In contrast, the recovery rate of the APO surrogate obtained using LC-MS/MS was 73% (RSD 9.3%), which is approximately five times that obtained by GC-MS. In a previous method using GC-MS,[10] the solvent was replaced with acetone after methanol reflux extraction, and the sample was purified using a florisil cartridge column. The LC-MS/MS method differs significantly from the GC-MS method, in that it involves extracting the sample with acetone containing a 1 mol/L aqueous formic acid solution, thoroughly volatilizing the formic acid under a nitrogen stream, and replacing the solvent with acetonitrile. In particular, it differs in that methanol and acetone are used in the initial extraction process. When methanol is used in GC-MS, it extracts several substances that interfere with the analysis. In addition, the dilution factor of the test solution was 20 times for GC-MS and 150 times for LC-MS/MS. Therefore, we assumed that the interfering substances contained in Apron were diluted during LC-MS/MS, eliminating factors that would affect the analysis.

As described above, satisfactory results were obtained for determining APO, TDBPP, and BDBPP via surrogate correction using LC-MS/MS, regardless of whether flame-retardant-processed textile products or untreated textile products were used. Further, the simultaneous analysis of APO, TDBPP, and BDBPP, which are flame retardants subject to household product regulations, can be performed by LC-MS/MS.

Conclusion We developed an LC-MS/MS method for simultaneously analyzing three flame retardants (APO,

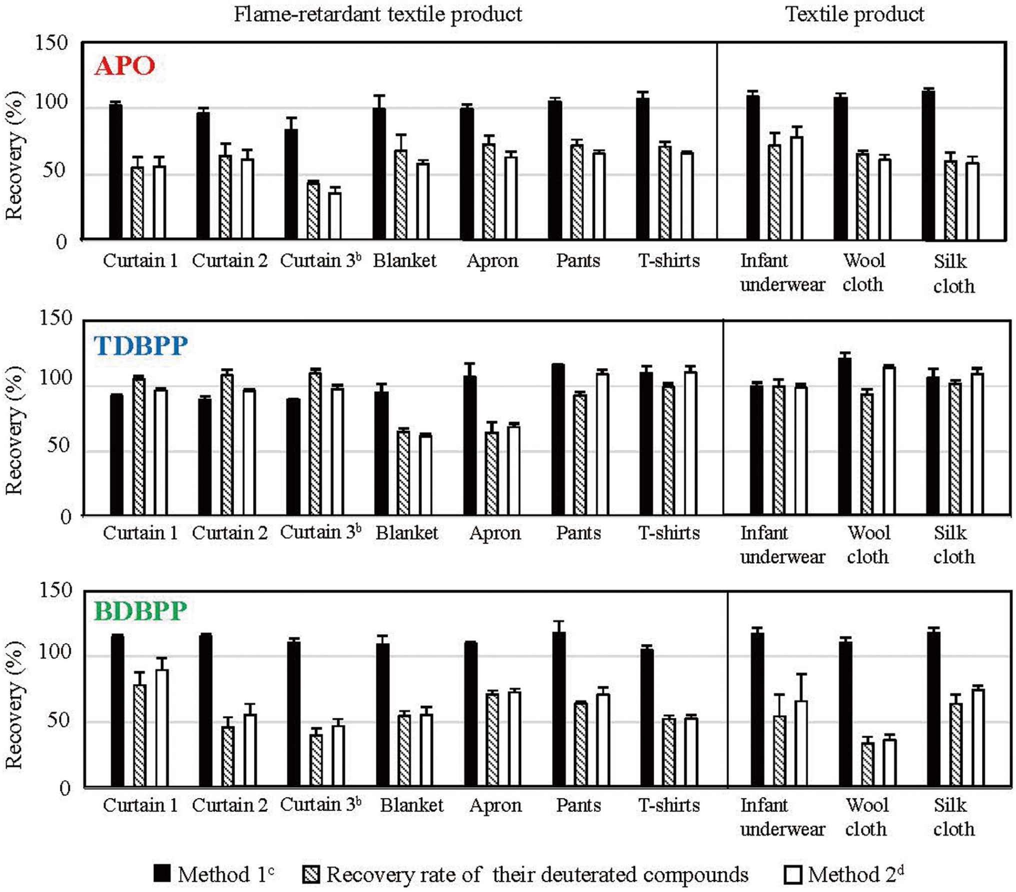

Fig. 3. Recovery rate of APO, TDBPP, BDBPP, and Their Deuterated Compounds in the Textile Products[a]

[a] Added amounts of APO, TDBPP, and BDBPP were 1.5, 3.0, and 3.0 µg, respectively. Added amounts of their deuterated compounds, APO-d$_{12}$, TDBPP-d$_{15}$, and BDBPP-d$_{10}$ were 0.75, 3.0, and 3.0 µg, respectively. [b] After polymer precipitation with hexane, the solution was pretreated and quantified. [c] Method 1: Quantified using the deuterated compounds as surrogate standard (n=3). [d] Method 2: Quantified using their external standards (n=3).

TDBPP, and BDBPP) using surrogate substances (APO-d$_{12}$, TDBPP-d$_{15,}$ and BDBPP-d$_{10}$) and obtained the following results:

1. BDBPP and BDBPP-d$_{10}$, both of which thermally decompose during GC-MS, were detected as a single peak in LC-MS/MS because they did not thermally decompose at the injection port.
2. The detection limits obtained using this analytical method with surrogate correction were compared with those from established methods: APO,[17)] TDBPP,[18)] and BDBPP.[19)] The limits of detection for APO, TDBPP, and BDBPP were 2.2, 9.2, and 10 times more sensitive than the current detection limits of 0.4, 8, and 10 µg/g, respectively.
3. We confirmed that the simultaneous analysis of APO, TDBPP, and BDBPP, which are flame retardants subject to household product regulations, can be performed via LC-MS/MS using surrogate correction. Screening analysis using the absolute calibration curve was also considered useful.

Therefore, the simultaneous analysis method using LC-MS/MS for flame retardants APO, TDBPP, and BDBPP enables a more sensitive analysis compared to GC-MS.

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Conflict of interest The authors declare no conflict of interest.

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