

## Determination of aqueous bisphenol A and tetrabromobisphenol A using molecular-complex-based liquid-liquid microextraction

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### ABSTRACT

A sample preparation method for the extraction of bisphenol A (BPA) and tetrabromobisphenol A (TBBPA), potential endocrine-disrupting chemicals common in manufacturing, from environmental water samples was established by means of molecular-complex-based liquid-liquid microextraction coupled to high-performance liquid chromatography with diode array and mass spectrometric detection. In this method, tributyl phosphate acts as an extractant of BPA and TBBPA via hydrogen bond interactions. Additionally, no dispersants are needed. Attenuated total reflection Fourier transform infrared spectroscopy provides robust evidence for the formation of hydrogen bonds between the extractant and phenols in the molecular complexes, which supports the experimental results. Under optimised conditions, calibration plots were linear in the range of 0.05–2  $\mu\text{g mL}^{-1}$ , and detection limits ranged from 0.16 to 0.23 ng  $\text{mL}^{-1}$ . The relative standard deviations of intra-day and inter-day repeatability were in the ranges of 3.5–4.9% and 5.1–6.8%, respectively, and recoveries ranged from 84.5 to 105.6%. The method we developed has been successfully employed in the measurement of BPA and TBBPA in four aqueous environmental samples.

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## 1. Introduction

Bisphenol A (BPA) is widely used in the production of *polycarbonate plastics* and *epoxy resins*, and tetrabromobisphenol A (TBBPA) is commonly used as a flame retardant. Due to mass production and utilisation, these compounds can easily migrate into the environment during production and application. According to recent research reports, BPA and TBBPA have been found in water [1,2], soil [3], and animals [4].

BPA and TBBPA are potential endocrine-disrupting chemicals [5,6]. Therefore, an effective monitoring system is required to prevent their harmful effects. At present, high-performance liquid chromatography with UV detection (HPLC-UV) [7], HPLC tandem mass spectrometry (HPLC-MS/MS) [8], and HPLC inductively coupled plasma MS (HPLC-ICP-MS) [9] are used for their analyses. Liquid-liquid, solid-phase, and solvent extraction have been developed as sample preparation methods to extract these compounds [10,11]. However, these extraction

procedures are often tedious and time-consuming and involve the consumption of large quantities of organic solvents.

Since 2006, dispersive liquid-liquid microextraction (DLLME) has attracted attention because of its ease of operation, low organic solvent consumption, and high efficiency and sensitivity [12–15]. In this method, a mixture of extraction and disperser solvents is rapidly injected into the sample to form a cloudy solution. After centrifugation, the enriched analytes in the extractant phase are collected and measured by conventional chromatography [16]. However, conventional DLLME has notable drawbacks including unsuitability for extraction of highly polar or hydrophilic analytes, reliance on highly toxic extraction solvents (e.g. halogenated or aromatic hydrocarbons), and millilitre volumes of disperser solvents [12,14,17]. Ionic liquids and extractants with densities less than water have been evaluated to replace highly toxic extraction solvents [16,18]. To reduce the use of dispersants, air-assistance [19], sonication [20], and magnetic stirring [21] have been used to assist in dispersing the extractant. However, development of new liquid-phase microextraction techniques for hydrophilic compounds remains a challenge.

In 2010, Feng's group introduced a molecular-complex-based DLLME (MC-DLLME) method [22] in which a molecular complex is formed between the extractant and analytes via hydrogen bond

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interactions. This technique has been applied to the separation and enrichment of trace analytes in different matrices, e.g. phthalate sums in edible oils [23], sodium benzoate in ibuprofen oral solutions [24], and traces of chromium in low-cost plastic toys [25]. Inspired by these applications, a procedure for the analysis of BPA and TBBPA in water samples by molecular-complex-based liquid-liquid microextraction (MC-LLME) has been established in this study. HPLC using both diode array (DAD) and MS detection were used and compared. Moreover, attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) was used to understand the interaction between the analyte and extractant in the resulting equilibrium complex.

## 2. Experimental

### 2.1. Chemicals and reagents

BPA (99.8% pure), TBBPA (99.5% pure), analytical-grade tri-n-butyl phosphate (TBP), diethyl carbonate (DEC), 1-octanol, and sodium chloride were obtained from Aladdin Chemistry Co. Ltd. (Shanghai, China). HPLC-grade methanol and ethanol were purchased from Ke miou Chemical Reagent Co. Ltd. (Tianjin, China). The structures of BPA, TBBPA, TBP, DEC, and 1-octanol are illustrated in Fig. 1.

A stock solution of  $1 \text{ mg mL}^{-1}$  BPA and TBBPA was prepared in methanol. Mixed working standard solutions ( $5 \text{ }\mu\text{g mL}^{-1}$ ) were freshly prepared by suitable dilution of the stock solution with methanol.

### 2.2. Apparatus

Chromatographic analysis was performed on a LC-20A (Shimadzu, Japan) equipped with a quaternary LC-20AD pump, SIL-20AC autosampler, and SPD-M20A DAD. Separations were performed on a VP-ODS C<sub>18</sub> column (250 mm  $\times$  2.0 mm, 2.2  $\mu\text{m}$ , Shimadzu). The mobile phase was a mixture of methanol and water (75:25) at a flow rate of 0.2 mL min<sup>-1</sup>. UV detection was carried out at a wavelength of 270 nm.

MS detection was performed with a quadrupole MS equipped with an electrospray ion (ESI) source operated in negative ionisation mode. The ions *m/z* 227 and 541 in selected-ion monitoring (SIM) mode were used for BPA and TBBPA, respectively. The ESI parameters were as follows: drying temperature 350 °C, ESI temperature 300 °C, drying gas flow 10 L min<sup>-1</sup>, and nebuliser gas flow 1.5 L min<sup>-1</sup>. The mobile phase and separation column were the same as for the chromatographic analysis, and the injection volume was 5  $\mu\text{L}$ .

ATR-FTIR spectra were recorded on a Thermo Nicolet 7000 spectrometer equipped with a DTGS detector and ZnSe crystal ATR accessory. The spectra were measured from 4000 to 600 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup>.

### 2.3. Extraction procedures

The sample solution (3.5 mL) was placed in a disposable polyethylene pipette. Then, 100  $\mu\text{L}$  of TBP was injected rapidly into the aqueous solution. The pipette was placed in a 10-mL centrifuge tube and shaken for 3 min. After centrifugation for 3 min at 5000 rpm, the organic phase ( $90 \pm 2 \mu\text{L}$ ) floating in the narrow neck of the pipette was withdrawn by microsyringe. The extraction phase was dissolved in 50  $\mu\text{L}$  of HPLC-grade methanol, after which 20  $\mu\text{L}$  was injected into the chromatographic system.

## 3. Results and discussion

### 3.1. Method optimisation

#### 3.1.1. Type of extractant

It is well known that phenols form molecular complexes via hydrogen bonding [22,26]. TBP, DEC, and 1-octanol were tested as extractants in the optimisation experiment. The results (Fig. 3) revealed that TBP produces the highest extraction recoveries (ERs%). ATR-FTIR analysis was used to further understand the extraction mechanism. The spectrum of pure BPA contained a peak at 3340 cm<sup>-1</sup> corresponding to O—H stretching (Fig. 4). The O—H stretching peak of the TBP–BPA complex is red-shifted to 3261 cm<sup>-1</sup>. Compared with pure TBP, the positions of some characteristic peaks of TBP (asymmetric  $-\text{CH}_3$  stretch, 2961 cm<sup>-1</sup>; symmetrical C—H stretch, 2874 cm<sup>-1</sup>; asymmetric P—O—C stretch, 1022 cm<sup>-1</sup>) remained unchanged after BPA loading. However, the characteristic P=O peak of TBP red-shifted from 1281 to 1270 cm<sup>-1</sup> after BPA loading. This indicates that intermolecular P=O···HO hydrogen bonding occurs between TBP and BPA [27–29]. The hydrogen bond equilibrium reaction between TBP and BPA is illustrated in Fig. 2. A corresponding reaction occurs between TBP and TBBPA in the TBP–TBBPA complex. The spectra of DEC, 1-octanol, DEC–BPA, and 1-octanol–BPA are displayed in Fig. S1. The characteristic C=O peak of DEC and the O—H peak of 1-octanol barely shift after BPA loading. This indicates that the ability of TBP to form hydrogen bonds is stronger than that of DEC and 1-octanol. Hence, TBP was selected as the extractant for subsequent experiments.

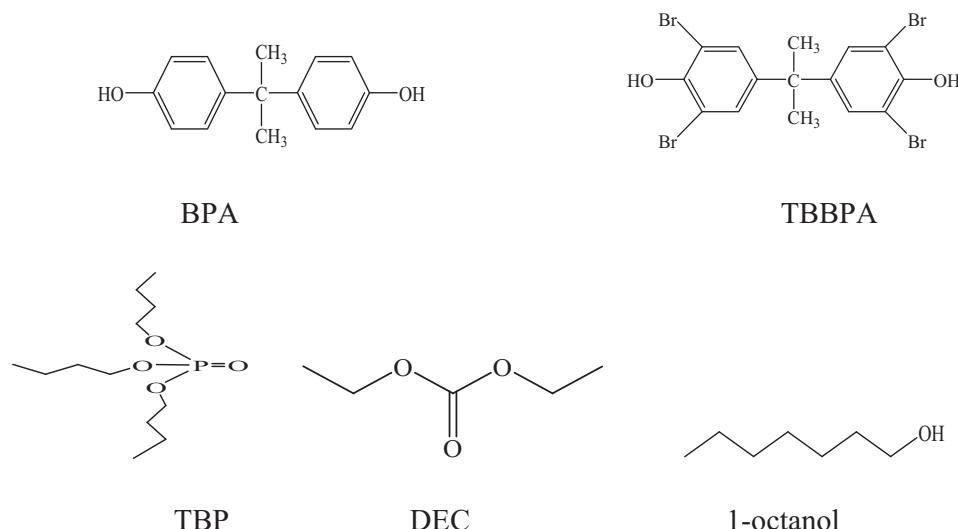
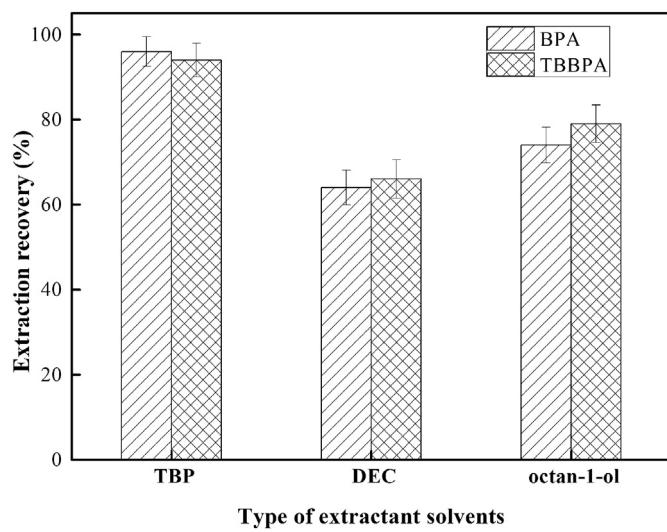
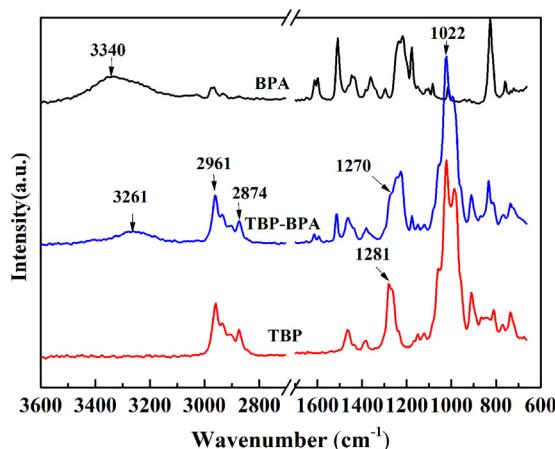


Fig. 1. Structures of organic compounds used in this study.



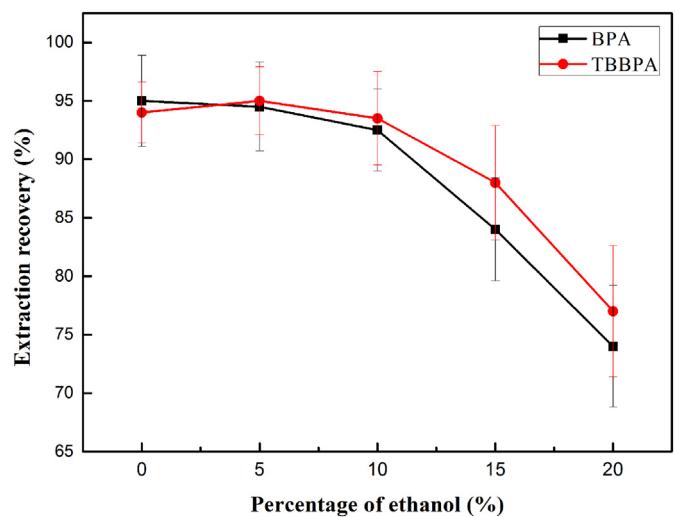
**Fig. 2.** Effect of extractant type on the extraction rates of BPA and TBBPA ( $n = 3$ ). Conditions: 3.5 mL spiked ( $0.5 \mu\text{g mL}^{-1}$  of each analyte) water solution, 100  $\mu\text{L}$  extractant, no disperser or salt, 3 min extraction time, and centrifugation at 5000 rpm for 3 min.



**Fig. 3.** ATR-FTIR analysis of BPA, TBP, and TBP-BPA complexation.

### 3.1.2. Disperser solvent

Methanol, ethanol, and acetonitrile are used as dispersants in classical DLLME, because of their miscibility with most extractants. In the present work, 100  $\mu\text{L}$  of TBP mixed with different percentages of ethanol was used to investigate the influence of the disperser solvent on the ERs% of the analytes. As illustrated in Fig. 5, there was no noticeable change in ER% when the percentage of ethanol increased from 0 to 10%. Upon further increasing the ethanol content, the ERs% decreased gradually. This phenomenon is inconsistent with conventional DLLME. Obviously, adding water-



**Fig. 5.** Effect of disperser ratio on the extraction rates of BPA and TBBPA ( $n = 3$ ).

miscible solvents could increase the dissolution of analytes in an aqueous solution. Addition of excess ethanol also may affect the formation of hydrogen bonds between TBP and the analytes [23]. ATR-FTIR was used to investigate how ethanol affects the extraction performance of TBP. The peak at  $3327 \text{ cm}^{-1}$  in the spectrum of pure ethanol (Fig. 6) is assigned to the O—H stretching vibration. This peak shifts to higher wavenumber ( $3437 \text{ cm}^{-1}$ ) in TBP-ethanol. The characteristic P=O peak of TBP after ethanol loading red-shifted from  $1281 \text{ cm}^{-1}$  to  $1260 \text{ cm}^{-1}$  compared with that of pure TBP. This indicates that ethanol also can form hydrogen bonds with TBP and compete with BPA and TBBPA [27–29]. Therefore, no extra dispersants were needed.

### 3.1.3. Extractant volume

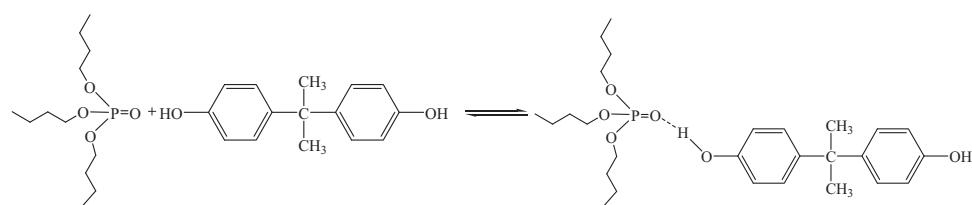
The influence of TBP volume was investigated within the range of 40–120  $\mu\text{L}$  at intervals of 20  $\mu\text{L}$ . As shown in Fig. 7, the ERs% rapidly increased upon increasing the volume of TBP from 40 to 100  $\mu\text{L}$ . Above 100  $\mu\text{L}$ , there was no obvious change in ER%. Thus, 100  $\mu\text{L}$  of TBP was used for further studies.

### 3.1.4. Salt addition

The influence of salt addition also was investigated by adding NaCl (0–20%, m/v) to the aqueous solution. No remarkable influence on ER% occurred with the addition of NaCl. Thus, salt was not added in further experiments.

### 3.1.5. Extraction and centrifugation time

Extraction times of 1 to 7 min were investigated. The corresponding results are shown in Fig. 8. The ERs% increases significantly as time increases from 1 to 3 min, but the ERs% reached a stable value above 3 min. Thus, 3 min was selected as the optimum extraction time. The centrifugation time in the range of 2–10 min at 5000 rpm was evaluated with 3 min being selected as optimum.



**Fig. 4.** Representative equilibrium equation of hydrogen bonding interaction between TBP and BPA.

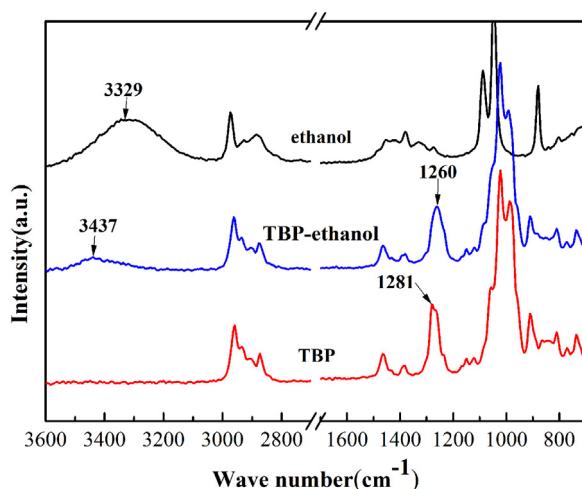


Fig. 6. ATR-FTIR analysis of ethanol, TBP, and TBP-ethanol complexation.

### 3.2. Method validation

The linear range, intra- and inter-day precisions, limits of detection (LODs), limits of quantification (LOQs), enrichment factors, and recovery rates were obtained under the optimum conditions. The range of linearity was  $0.05\text{--}2\text{ }\mu\text{g mL}^{-1}$  with correlation coefficients ( $r$ ) between 0.998 and 0.999. The LODs ( $S/N = 3$ ) were between 0.16 and 0.23  $\text{ng mL}^{-1}$ , and the LOQs ( $S/N = 10$ ) were between 0.53 and 0.76  $\text{ng mL}^{-1}$ . Intra- and inter-day precisions at a concentration of  $0.2\text{ }\mu\text{g mL}^{-1}$ , expressed as RSDs of peak areas, varied from 3.5 to 6.8%. Enrichment factors of 23.6 and 22.8 were achieved for BPA and TBBPA, respectively. As shown in Table 1, the recoveries ranged from 84.5 to 105.6% with RSDs  $<6.5\%$ . These results indicate that the developed MC-LLME method is reliable and feasible for the extraction of BPA and TBBPA from aqueous samples.

### 3.3. Comparison with other procedures

A comparison of the MC-LLME-HPLC process with other methods reported in the literature for the analysis of TBBPA and its derivatives is given in Table 2. The comparison illustrates several advantages of the proposed method. First, the amounts and toxicity of the organic solvents consumed in our method are much less. Second, the total analysis time is shorter, which increases sample throughput and improves precision. Additionally, compared with the sensitivity of MS detection, our

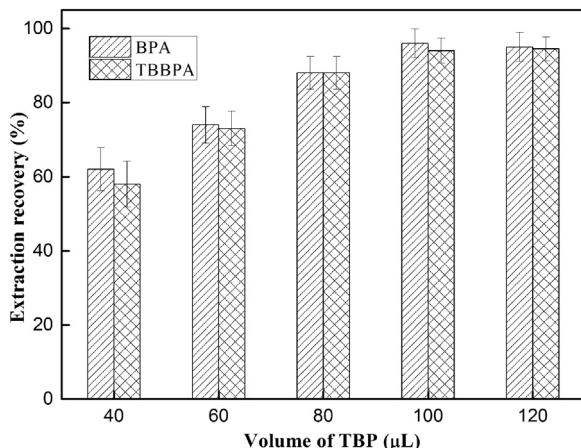


Fig. 7. Effect of extractant volume on the extraction rates of BPA and TBBPA ( $n = 3$ ).

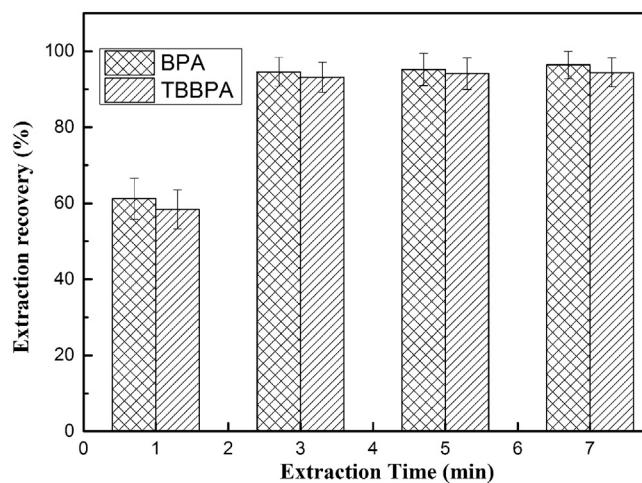


Fig. 8. Effect of extractant time on the extraction rates of BPA and TBBPA ( $n = 3$ ).

procedure offers the same low LODs because of the high enrichment effect of MC-LLME.

### 3.4. Real water sample analysis

The proposed MC-LLME-HPLC technique was employed in the analysis of four aqueous environmental samples. The results are listed in Table 3, and typical chromatograms are shown in Fig. 9. Concentrations of  $0.07\text{ }\mu\text{g mL}^{-1}$  BPA in the Fen River and  $0.12\text{--}0.18\text{ }\mu\text{g mL}^{-1}$  BPA and  $0.09\text{--}0.13\text{ }\mu\text{g mL}^{-1}$  TBBPA in wastewater from a local chemical plant were detected. To investigate the accuracy and reliability of the HPLC-DAD method, the chemical plant wastewater was also analysed qualitatively and quantitatively by HPLC-ESI-MS. Representative chromatograms are shown in Fig. S2. The MS results were consistent with those from DAD. The main advantage of MS detection is its higher sensitivity. However, due to their high price, MS detectors are not always available. The results demonstrate that DAD is also useful and reliable for measuring BPA and TBBPA in various water samples.

## 4. Conclusions

In this study, an MC-LLME approach followed by HPLC coupled to DAD and MS detection was successfully established for measuring BPA and TBBPA in aqueous samples. A significant advantage of the developed method is that TBP is used as the LLME extractant to extract BPA and TBBPA via hydrogen bonding interactions. ATR-FTIR analysis was used to help understand the interaction between the analytes and extractant in the equilibrium complex. Another significant innovation is that the proposed approach does not require a disperser solvent. This method may be useful for the analysis of various polar or hydrophilic organic compounds.

Table 1  
Recovery rates of BPA and TBBPA.

Samples	Spiked ( $\mu\text{g}$ )	Analytes	
		Average recovery (% $\pm$ RSD%, $n=3$ )	Average recovery (% $\pm$ RSD%, $n=3$ )
Tap water	0.2	$89.3 \pm 5.7$	$90.2 \pm 5.7$
	0.5	$100.2 \pm 3.1$	$103.2 \pm 6.1$
	1.0	$98.4 \pm 3.9$	$94.8 \pm 4.6$
	0.2	$85.2 \pm 5.4$	$84.5 \pm 6.5$
Chemical plant water 1	0.5	$93.2 \pm 3.8$	$90.6 \pm 3.8$
	1.0	$95.3 \pm 5.2$	$105.6 \pm 5.2$

**Table 2**

Comparison of the current work with previous procedure for the analysis of TBBPA and its derivatives.

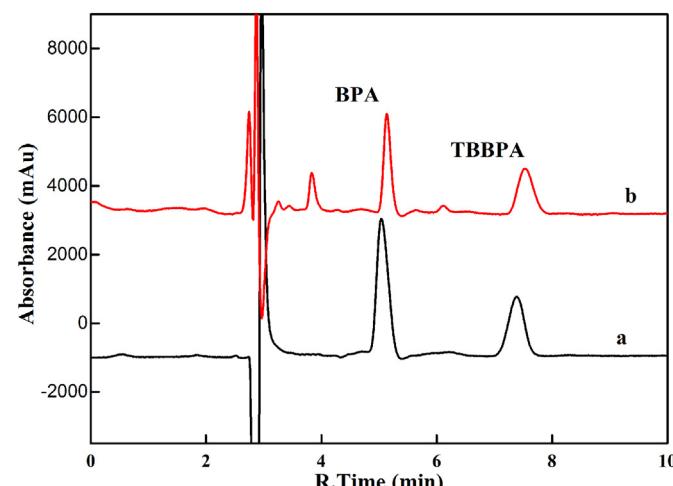
Methods	Pre-treatment	Extractant	Extraction time (min)	LOD	Reference
LC-ESI-MS/MS	SPE filtration	2 mL dichloromethane + 3 mL methanol	<20 min	300 ng g <sup>-1</sup>	[8]
HPLC-ICP-MS	Liquid-liquid extraction	100 mL DCM	<30 min	0.12–0.19 ng mL <sup>-1</sup>	[9]
HPLC-VWD	US-DLLME	0.6 mL THF + 25 μL chloroform	2	0.13–0.63 ng mL <sup>-1</sup>	[17]
HPLC-DAD	MC-LLME	100 μL TBP	3	0.16–0.23 ng mL <sup>-1</sup>	This work

**Table 3**

Mean contents of BPA and TBBPA in environmental aqueous samples.

Samples	Contents (μg mL <sup>-1</sup> ± RSD, n = 3)	
	BPA	TBBPA
Tap water	nd	nd
Fen river water	0.07 ± 6.2	nd
Chemical plant water 1	0.12 ± 4.9	0.09 ± 5.8
Chemical plant water 2	0.18 ± 3.5	0.13 ± 5.6

nd not detected.

**Fig. 9.** Chromatograms of blank sample spiked with 0.2 μg mL<sup>-1</sup> each analyte (a) and chemical plant wastewater (b) obtained using MC-LLME combined with HPLC-DAD under optimum conditions.**CRediT authorship contribution statement**

**Qilong Xie:**Conceptualization, Methodology, Investigation.  
**Jiangping Cao:**Methodology, Writing – original draft.  
**Dekui Sun:**Validation, Data curation, Formal analysis.  
**Huaiqian Lu:**Investigation, Writing – review & editing, Visualization.  
**Ming Xia:**Investigation, Writing – review & editing, Visualization.  
**Bo Hou:**Project administration.  
**Debao Li:**Supervision.  
**Litao Jia:**Funding acquisition.

**Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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**Appendix A. Supplementary data**

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.molliq.2020.112501>.

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